UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
NEW ENGLAND REGION
FIVE POST OFFICE SQUARE, SUITE 100, BOSTON, MA 02109

October 8, 2020

Bruce Thompson
de maximis, inc.
200 Day Hill Road, Suite 200
Windsor, CT 06095

Re: Approval of de maximis inc. report titled *Remedial Design Work Plan – Appendix J Quality Assurance Project Plan (Rev. 3)* (the "QAPP"), dated October 2020.

Nuclear Metals, Inc. Superfund Site

Dear Mr. Thompson:

EPA, in consultation with the Massachusetts Department of Environmental Protection, has completed its review of the QAPP, dated October 2020. The QAPP was revised in response to EPA comments dated July 2, 2020 and August 31, 2020. The QAPP is subject to the terms and conditions specified in the Consent Decree (CD) for Remedial Design / Remedial Action (RD/RA) for the Nuclear Metals, Inc. Site, which has an effective Date of December 6, 2019.

EPA has reviewed the revisions to the QAPP and finds that they are acceptable. Therefore, EPA approves the QAPP.

If there is any conflict between the Performance Standards as stated in the Work Plan and the Performance Standards as stated in the CD and statement of work (SOW), the CD and SOW shall control.

Please do not hesitate to contact me at (617) 918-1339 or at smith.christopher@epa.gov should you have any questions in this regard.

Sincerely,

Christopher Smith
Project Manager

Final Remedial Design Work Plan
Appendix J

Nuclear Metals, Inc. Superfund Site

Remedial Design Work Plan Appendix J

Quality Assurance Project Plan (Rev. 3)

Nuclear Metals, Inc. (NMI) Superfund Site
Concord, Massachusetts

October 2020

Remedial Design Work Plan
Appendix J

Quality Assurance Project Plan (Rev. 3)

Nuclear Metals, Inc (NMI)
Superfund Site
Concord, Massachusetts

Prepared by:



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Date:
October 2020

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J-2 Chain of Custodies

1. Introduction

On October 17, 2019 the United States Environmental Protection Agency (USEPA) lodged a Consent Decree (CD) with the United States District Court for the District of Massachusetts Eastern Division in connection with Civil Action No. 19-12097-RGS. The CD was entered by the Court on December 6, 2019. The CD and its accompanying Statement of Work (SOW) describe the Remedial Design/Remedial Action (RD/RA) work to be performed for the Nuclear Metals, Inc. (NMI) Superfund Site in Concord, Massachusetts. The RD/RA work will be undertaken by the Settling Defendants (SDs) to the CD, with funding contributions from the Settling Federal Agencies (SFAs).

This Q*uality Assurance Project Plan* (QAPP) has been developed as a component of the *Remedial Design Work Plan* (RDWP) to address the requirement of Section 6.7 of the SOW, and it addresses the content specified in 6.7(d) to the SOW, including:

- This QAPP augments the FSP and addresses sample analysis and data handling regarding the Work.
- This QAPP includes a detailed explanation of SDs' quality assurance, quality control and chain of custody procedures for all treatability, design, compliance, and monitoring.
- Developed in accordance with, QA/R-5, EPA/240/B-01/003 (Mar. 2001, reissued May 2006); Guidance for Quality Assurance Project Plans., QA/G-5, EPA/240/R 02/009 (Dec. 2002);and Uniform Federal Policy for Quality Assurance Project Plans, Parts 1-3, EPA/505/B-04/900A though 900C (Mar. 2005).

The purpose of the QAPP is to support the sampling and analytical methods and procedures that will be used during the RD phases of the project, including pre-design investigations and SOW-specified groundwater monitoring events.

As described on USEPA's Quality System Website (http://www.epa.gov/QUALITY/qapps.html), a QAPP is designed to document the planning, implementation and assessment procedures for a particular project, as well as any specific quality assurance and quality control activities. It is designed to integrate all the technical and quality aspects of the project to provide a "blueprint" for obtaining the type and quality of environmental data and information needed for a specific decision or use.

In March 2005, the Intergovernmental Data Quality Task Force developed a policy and guidance document to fulfill the project-specific requirements of Part B of American National Standards Institute (ANSI)/American Society for Quality Control (ASCQ) E4, to ensure that Federal departments and agencies will produce consistent QAPPs that reflect a systematic planning approach to collection and use of environmental data. That guidance document—the *Uniform Federal Policy for Quality Assurance Project Plans* (UFP-QAPP) Manual V1, March 2005 (USEPA 2005b)—was followed in the development of this QAPP. Additionally, the provisions of this QAPP were reviewed against the Minimum Quality Assurance/Quality Control (QA/QC) Activities identified in the UFP-QAPP (USEPA 2005b).

As per the UFP-QAPP Manual V1, March 2005, the UFP approach was developed to be compliant with USEPA QA/R-5 and QA/G-5. Other existing QAPP guidance – including selected Region 1 (New England) QAPP guidance – was used as a point of reference for creation of the UFP-QAPP Manual. Therefore, use of the UFP-QAPP manual meets the substantive SOW requirements associated with the development of the QAPP for this site, as specified in Section 6.7 of the SOW.

As appropriate, pertinent state guidance available in the Massachusetts Department Environmental Protection (MASSDEP) have been adopted and incorporated into this QAPP. Given the USEPA lead on this project, the fact that the need for and scope of the QAPP were prescribed in the SOW, the historical dataset available for this site, and other site-specific factors, deviations from MASSDEP guidance potentially exist.

Periodic updates to the QAPP are prepared as necessitated by the project status. A QAPP revision history is provided as Table J-1.

1.1 Project Setting

The NMI Site includes the property located at 2229 Main Street in Concord, MA and downgradient properties where groundwater contamination has come to be located (see Figure J- 1). The areal extent of contaminants in groundwater extend beyond the Assabet River and include properties west of the river in the Town of Acton, MA. The NMI property is bordered by residential and woodland areas to the east and south, light commercial and

industrial areas to the west, and Main Street (Route 62) and the Assabet River to the north.

Additional information regarding the site location and setting is provided in the RDWP.

1.2 Applicability of the QAPP

This QAPP is applicable to all sampling activities performed at the Site. Consistent with Section 6.7(d) to the SOW, it will be updated as appropriate to address unique components of planned RD activities.

1.3 Remedial Activities

The overall purpose of RD/RA activities is to design and implement the remedial approach for the Site. The Scope of the Remedy includes the following actions described in Section L of the ROD, including:

- Excavation and off-site disposal of approximately 82,500 cubic yards of contaminated sediments, underground drain lines and debris, and non-Holding Basin soils (contaminated with DU, PCBs, and other contaminants of concern found in ROD Tables L-2 through L-4) in various areas of the Site (See Figure 3 of the RDWP);

- *In-situ* stabilization of DU contaminated soils in the Holding Basin via injection of a stabilization agent such as apatite (e.g., Apatite II™) or other comparable stabilization agent to prevent leaching of contaminants to groundwater, and *in-situ* treatment of DU in overburden groundwater and natural uranium in bedrock groundwater (See Figure 4 of the RDWP);

- Containment of HB stabilized soils with a low-permeability vertical wall and a low-permeability horizontal sub-grade cover to isolate the stabilized soils and further limit mobility of contaminants by removing the flow of groundwater (See Figure 5 of the RDWP;

- Extraction and *ex-situ* treatment of volatile organic compounds (VOCs), 1,4-dioxane and other contaminants found in ROD Table

L-1 in overburden and bedrock aquifers (See Figure 6a of the RDWP);

- Long-term monitoring to monitor effectiveness of *in*- and *ex-situ* treatment; and

- Institutional Controls to: 1) prevent unacceptable exposures to, and to prevent disturbance of, the HB area; 2) prohibit use of contaminated groundwater until cleanup levels are met; and 3) require installation of vapor mitigation systems should future structures be built above the VOC plume before groundwater cleanup levels are met, unless an evaluation of vapor intrusion risks is performed to show such systems are not required.

2. Document Overview

The UFP-QAPP guidance (USEPA 2005b) includes a series of 37 worksheets that can be used to present the critical information required in a QAPP. Since the worksheets were designed to "ensure consistent content and presentation of information in a project-specific QAPP... and streamline the review of QAPPs by regulators and others" (USEPA 2005b), this QAPP has been developed using these worksheets, which provide information associated with four key topics:

- Project Management and Objectives

- Measurement and Data Acquisition

- Assessment and Oversight

- Data Review

A summary of the required QAPP elements and corresponding QAPP sections and general scope/content of the 37 worksheets can be found in Worksheet 2 within this QAPP.

Two appendices are also included with this QAPP. The first (Appendix J-1) is available on Project Portal and includes the following:

- Laboratory Quality Assurance Plans and Standard Operating Procedures (SOPs)

- Laboratory certifications

Appendix J-2 contains a copy of an example chain of custody(s) form.

When elements required by the UFP-QAPP are present in other documents (e.g., SOPs), careful cross-referencing of these other documents can be used in lieu of repeating information (USEPA 2005b). Following this guidance, this QAPP provides certain relevant information by reference to other supporting documents, including the RDWP, RD POP, and supporting plans attached thereto. All of these planning documents are companion documents to this QAPP.

3. References

The following references are cited within and/or considered in the development of this QAPP.

USEPA. 2001. *EPA Requirements for QA Project Plans for Environmental Operations*. EPA-QA/R-5. Office of Environmental Information. March 2001.

USEPA. 2002. *Guidance for QA Project Plans*. EPA-QA/G-5. Office of Environmental Information. December 2002.

USEPA. 2004. Office of Environmental Measurement and Evaluation, Region I, EPA-New England Part III Pesticides/PCBs Data Validation Functional Guidelines, (February, 2004).

USEPA. 2005a. Record of Decision Summary, Solvents Recovery Service of New England, Inc. (SRSNE) Site, Southington, Connecticut. September 2005.

USEPA. 2005b. *Uniform Federal Policy for Quality Assurance Project Plans* (UFP-QAPP) Manual V1. March 2005.

American Association for Laboratory Accreditation, A2LA Guide for the Estimation of Measurement Uncertainty in Testing, December 2014.

USEPA.2018. EPA New England Quality Management Plan, EPA NE QMP. May 2016,

USEPA. 2006. Guidance on Systematic Planning using the Data Quality Objectives Process. EPA-QA/G-4. Office of Environmental Information. February 2006.

USEPA. 2008. Office of Environmental measurement and Evaluation, Region I, EPA-New England Part IV Inorganic Data Validation Functional Guidelines, (November, 2008).

USEPA. 2018. REGION 1 - EPA NEW ENGLAND ENVIRONMENTAL DATA REVIEW SUPPLEMENT For Region 1 Data Review Elements and Superfund Specific Guidance/Procedures. EQADR–Supplement1. June 2018.

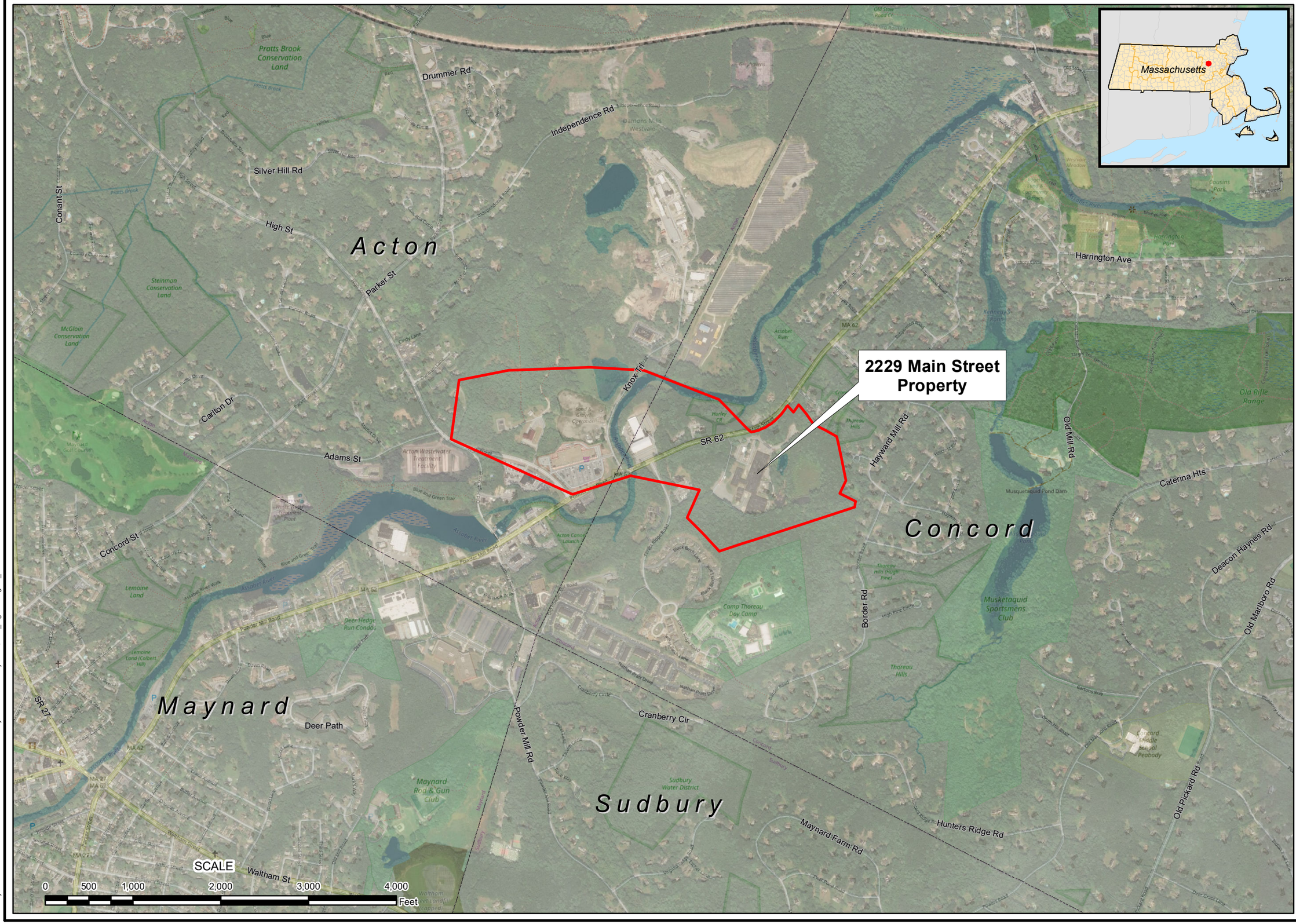
Tables

Table J-1.
QAPP Revision History
NMI, Inc. Superfund Site, Concord, MA

Revision #	Date	Description of Revision
1	February 2020	Initial draft version of QAPP
2	August 2020	Draft with revisions based on RTC received 7/2/31
3	October 2020	Draft with revisions based on RTC Received 8/31/20

Notes:
QAPP = Quality Assurance Project Plan

Figures



J-1

Site Location Map

Nuclear Metals, Inc. Site
Attachment J QAPP

Concord, Massachusetts

Description:

2229 Main Street Property

Map Legend:

Site Boundary

Spatial Projection:

Coordinate System:
MA State Plane Mainland
FIPS Zone: 2001
Units: US Survey Feet
Datum: NAD83

Plot Info:

File: FigJ-1_SiteLoc.mxd
Project No.: 3252
Plot Date: 3/3/2020
Arc Operator: LS
Reviewed by: HG

ddms
www.ddmsinc.com

de maximis, inc.

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J-2

Study Area

Nuclear Metals, Inc. Site
Attachment J QAPP

Concord, Massachusetts

Description:

Well sample locations.

Map Legend:

Well Formation

- Undefined
- Shallow Overburden (SOB)
- Middle Overburden (MOB)
- Till
- Weathered Bedrock (WBR)
- Bedrock (BR)
- Site Boundary
- Former Building Concrete Foundations
- Remediation Zone (Holding Basin)

Spatial Projection:

Coordinate System:
MA State Plane Mainland
FIPS Zone: 2001
Units: US Survey Feet
Datum: NAD83

Plot Info:

File: FigJ-2_Study_Area.mxd
Project No.: 3252
Plot Date: 3/3/2020
Arc Operator: LS
Reviewed by: HG

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SCALE
0 100 200 400 600 800 1,000 Feet

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Worksheets

QAPP Worksheet #1 Title and Approval Page

Site Name/Project Name:	**Nuclear Metals, Inc. (NMI) – Remedial Design / Remedial Action**
Site Location:	Concord, MA
Document Title:	*Quality Assurance Project Plan*
Lead Organization:	USEPA Region 1
Preparer's Name and Organizational Affiliation:	Jessie McCusker, ***de maximis, inc.***
Preparer's Contact Information:	Jessie@demaximis.com
Preparation Date:	February 2020 (Rev 1), August 2020 (Rev 2), October 2020
Project Coordinator:	
	Signature
	Bruce Thompson, ***de maximis, inc.***
Investigative Organization's Project Manager:	
	Signature
	Bruce Thompson, ***de maximis, inc.***
Investigative Organization's Project QA Officer:	
	Signature
	Jessie McCusker, ***de maximis, inc.***
Investigative Organization's Project Manager:	
	Signature
	Haley & Aldrich, Inc.

QAPP Worksheet #1 Title and Approval Page

Investigative Organization's Project Manager:	
	Signature
	Geosyntec Consultants
Investigative Organization's Project Manager:	
	Signature
	O&M, Inc.
Lead Organization's Program Manager:	
	Signature
	Chris Smith, USEPA Region 1
Lead Organization's Quality Assurance Manager:	
	Signature
	Nora Conlon, USEPA Region 1

QAPP Worksheet #2 QAPP Identifying Information

Site Name/Project Name:	Nuclear Metals, Inc. (NMI) Superfund Site – Remedial Design / Remedial Action (RD/RA)
Site Location:	2229 Main Street, Concord, MA
Site Number/Code:	MAD062166335
Operable Unit:	NA
Contractor Name:	NA
Contractor Number:	NA
Contract Title:	NA
Work Assignment Number:	NA
Identify guidance used to prepare QAPP:	EPA Requirements for Quality Assurance Project Plans, QA/R-5, EPA/240/B-01/003 (Mar. 2001, reissued May 2006); Guidance for Quality Assurance Project Plans., QA/G-5, EPA/240/R 02/009 (Dec. 2002); and Uniform Federal Policy for Quality Assurance Project Plans, Parts 1-3, EPA/505/B-04/900A though 900C (Mar. 2005)
Identify regulatory program:	Comprehensive Environmental Response, Compensation and Liability Act (CERCLA)
Identify approval entity:	USEPA Region 1 (with reasonable opportunity for review and comment by the MassDEP)
Indicate whether the QAPP is a generic or a project-specific QAPP?	This NMI QAPP is a project-specific QAPP to serve the needs of the Remedial Design and monitoring activities at the Site, as described in the Remedial Design Work Plan
List dates of scoping sessions that were held:	(See Worksheet 9).

QAPP Worksheet #2 QAPP Identifying Information

List dates and titles of QAPP documents written for previous site work, if applicable:	
Quality Assurance Project Plan	MACTEC, Quality Assurance Project Plan, August 1996. Geosyntec, QAPP Addendum for Groundwater NTCRA, September 2017. These documents were approved by USEPA Region 1.
List organizational partners (stakeholders) and connection with lead organization:	The Settling Defendants to a December 2019 Consent Decree and Statement of Work for Remedial Design/Remedial Action.
List data users:	USEPA Region 1 and their oversight contractor, MassDEP, NMI Settling Defendants, their General Contractor (***de maximis, inc***.) and sub-contractors.
Lead Organization's Program Manager:	Chris Smith, USEPA Region 1

QAPP Worksheet #2 QAPP Identifying Information

Required QAPP Element(s) and Corresponding QAPP Section(s) (per UFP QAPP 2005)	Required Information	Crosswalk to Related Information and Documents
Project Management and Objectives		
2.1 Title and Approval Page	- Title and Approval Page	Worksheet #1 Title and Approval Page
2.2 Document Format and Table of Contents 2.2.1 Document Control Format 2.2.2 Document Control Numbering System 2.2.3 Table of Contents 2.2.4 QAPP Identifying Information	- Table of Contents - QAPP Identifying Information	The Table of Contents is provided following the QAPP cover page. Worksheet #2 QAPP Identifying Information
2.3 Distribution List and Project Personnel Sign-Off Sheet 2.3.1 Distribution List 2.3.2 Project Personnel Sign-Off Sheet	- Distribution List - Project Personnel Sign-Off Sheet	Worksheet #3 Distribution List and Worksheet #4 Project Personnel Sign-Off
2.4 Project Organization 2.4.1 Project Organizational Chart 2.4.2 Communication Pathways 2.4.3 Personnel Responsibilities and Qualifications 2.4.4 Special Training Requirements and Certification	- Project Organizational Chart - Communication Pathways - Personnel Responsibilities and Qualifications Table - Special Personnel Training Requirements Table	Worksheet #5 Project Organization Chart, Worksheet #6 Communication Pathways, Worksheet #7 Personnel Responsibilities and Qualifications and Worksheet #8 Special Personnel Training Requirements
2.5 Project Planning/Problem Definition 2.5.1 Project Planning (Scoping) 2.5.2 Problem Definition, Site History and Background	- Project Planning Session Documentation (including Data Needs tables) - Project Scoping Session Participants Sheet - Problem Definition, Site History and Background - Site Maps (historical and present)	Worksheet #8 Special Personnel Training Requirements, Worksheet #9 Project Team Planning Sessions Participants Sheet and Worksheet #10 Problem Definition for Project DQOs Site history, maps and more detail concerning the project DQOs can be found in the *Remedial Design Work Plan, February 2020*

QAPP Worksheet #2 QAPP Identifying Information

Required QAPP Element(s) and Corresponding QAPP Section(s) (per UFP QAPP 2005)	Required Information	Crosswalk to Related Information and Documents
2.6 Project Quality Objectives and Measurement Performance Criteria 2.6.1 Development of Project Quality Objectives Using the Systematic Planning Process 2.6.2 Measurement Performance Criteria	- Site-Specific Project Quality Objectives (PQOs) - Measurement Performance Criteria Table	Worksheet #11 Project Quality Objectives/Systematic Planning Process Statements and Worksheet #12-1 though #12-13 Measurement Performance Criteria for project analytes Details concerning the project objectives can be found in the *Remedial Design Work Plan, February 2020*
2.7 Secondary Data Evaluation	- Sources of Secondary Data and Information - Secondary Data Criteria and Limitations Table	Worksheet #13 Secondary Data Criteria and Limitations
2.8 Project Overview and Schedule 2.8.1 Project Overview 2.8.2 Project Schedule	- Summary of Project Tasks - Reference Limits and Evaluation Table - Project Schedule/Timeline Table	Worksheet #14 Summary of Project Tasks, Worksheet #15-1 through #15-3 Reference Limits and Evaluation for specific monitoring activities and Worksheet #16 Project Schedule/Timeline More details concerning the project schedule can be found in the *Remedial Design Work Plan, February 2020*

QAPP Worksheet #2 QAPP Identifying Information

Required QAPP Element(s) and Corresponding QAPP Section(s) (per UFP QAPP 2005)	Required Information	Crosswalk to Related Information and Documents
Measurement/Data Acquisition		
3.1 Sampling Tasks 3.1.1 Sampling Process Design and Rationale 3.1.2 Sampling Procedures and Requirements 3.1.2.1 Sampling Collection Procedures 3.1.2.2 Sample Containers, Volume and Preservation 3.1.2.3 Equipment/Sample Containers Cleaning and Decontamination Procedures 3.1.2.4 Field Equipment Calibration, Maintenance, Testing and Inspection Procedures 3.1.2.5 Supply Inspection and Acceptance Procedures 3.1.2.6 Field Documentation Procedures	- Sampling Design and Rationale - Sample Location Map - Sampling Locations and Methods/SOP Requirements Table - Analytical Methods/SOP Requirements Table - Field Quality Control Sample Summary Table - Sampling SOPs - Project Sampling SOP References Table - Field Equipment Calibration, Maintenance, Testing and Inspection Table	Worksheet #17 Sampling Design and Rationale, Worksheet #18Sampling Locations and Methods/SOP Requirements for the project, Worksheet #19 Analytical SOP Requirements (Sample Containers Preservation and Holding Times), Worksheet #20 Sample Quantities and Control Frequencies, Worksheet #21 Field Sampling SOP References and Worksheet #22 Field Equipment Calibration, Maintenance, Testing and Inspection More details concerning the sampling design and rationale can be found in the *Remedial Design Work Plan, February 2020* The analytical SOPs can be found in Appendix C-1 of this QAPP

QAPP Worksheet #2 QAPP Identifying Information

Required QAPP Element(s) and Corresponding QAPP Section(s) (per UFP QAPP 2005)	Required Information	Crosswalk to Related Information and Documents
3.2 Analytical Tasks 3.2.1 Analytical SOPs 3.2.2 Analytical Instrument Calibration Procedures 3.2.3 Analytical Instrument and Equipment Maintenance, Testing and Inspection Procedures 3.2.4 Analytical Supply Inspection and Acceptance Procedures	- Analytical SOPs - Analytical SOP References Table - Analytical Instrument Calibration Table - Analytical Instrument and Equipment Maintenance, Testing and Inspection Table	Worksheet #23 Analytical SOP References, Worksheet #24 Analytical Instrument Calibration and Worksheet #25 Analytical Instrument and Equipment Maintenance, Testing and Inspection The analytical SOPs can be found in Appendix C-1 of this QAPP
3.3 Sample Collection Documentation, Handling, Tracking and Custody Procedures 3.3.1 Sample Collection Documentation 3.3.2 Sample Handling and Tracking System 3.3.3 Sample Custody	- Sample Collection Documentation Handling, Tracking and Custody SOPs - Sample Container Identification - Sample Handling Flow Diagram - Example Chain-of-Custody (COC) Form and Seal	Worksheet #27 Sample Custody Requirements More details concerning the field sampling procedures can be found in the *Field Sampling Plan* (FSP) Attachment B to the RD POP. February 2020 An example of the COC form can be found in SOP # NMI-001
3.4 Quality Control Samples 3.4.1 Sampling Quality Control Samples 3.4.2 Analytical Quality Control Samples	- QC Samples Table - Screening/Confirmatory Analysis Decision Tree	Worksheet #28-1 through #28-16 present QC sample information for project analytes
3.5 Data Management Tasks 3.5.1 Project Documentation and Records 3.5.2 Data Package Deliverables 3.5.3 Data Reporting Formats 3.5.4 Data Handling and Management 3.5.5 Data Tracking and Control	- Project Documents and Records Table - Analytical Services Table - Data Management SOPs	Worksheet #29 Project Documents and Records and Worksheet #30 Analytical Services Data Management is described in the *Remedial Design Work Plan, February 2020*

QAPP Worksheet #2 QAPP Identifying Information

Required QAPP Element(s) and Corresponding QAPP Section(s) (per UFP QAPP 2005)	Required Information	Crosswalk to Related Information and Documents
Assessment/Oversight		
4.1 Assessments and Response Actions 4.1.1 Planned Assessments 4.1.2 Assessment Findings and Corrective Action Responses	- Assessments and Response Actions - Planned Project Assessments Table - Audit Checklists - Assessment Findings and Corrective Action Responses Table	Worksheet #31 Planned Project Assessments and Worksheet #32 Assessment Findings and Corrective Action Responses Laboratory Certifications can be found in Appendix C-1
4.2 QA Management Reports	- QA Management Reports Table	Worksheet #33 QA Management Reports
4.3 Final Project Report		

QAPP Worksheet #2 QAPP Identifying Information

Required QAPP Element(s) and Corresponding QAPP Section(s) (per UFP QAPP 2005)	Required Information	Crosswalk to Related Information and Documents
Data Review		
5.1 Overview		
5.2 Data Review Steps 5.2.1 Step I: Verification 5.2.2 Step II: Validation 5.2.2.1 Step IIa Validation Activities 5.2.2.2 Step IIb Validation Activities 5.2.3 Step III: Usability Assessment 5.2.3.1 Data Limitations and Actions from Usability Assessment 5.2.3.2 Activities	- Verification (Step I) Process Table - Validation (Steps IIa and IIb) Process Table - Validation (Steps IIa and IIb) Summary Table - Usability Assessment	Worksheet #34 Verification (Step I) Process, Worksheet #35 Validation (Steps IIa and IIb) Process, Worksheet #36 Validation (Steps IIa and IIb) Summary and Worksheet #37 Usability Assessment
5.3 Streamlining Data Review 5.3.1 Data Review Steps to Be Streamlined 5.3.2 Criteria for Streamlining Data Review 5.3.3 Amounts and Types of Data Appropriate for Streamlining	None	NA

QAPP Worksheet #3 Distribution List

QAPP Recipients	Title	Organization	Telephone Number	E-mail Address
Chris Smith	Remedial Project Manager	USEPA	617.918.1339	Smith.christopher@epa.gov
Nora Conlon	QAPP Reviewer	USEPA	617.918.8335	conlon.nora@epa.gov
Garry Waldeck	Project Manager	MassDEP	617.348.4017	Garry.waldeck@mass.gov
Bruce Thompson	Project Coordinator	***de maximis, inc.***	860.298.0541	brucet@demaximis.com
Jessie McCusker	Alternate Project Coordinator / QA Coordinator / Task Manager – Sitewide Soils and Sediments and Holding Basin Remedies	***de maximis, inc.***	860.817.7544	jessie@demaximis.com
Amy Hoffmann	Task Manager – ISS, Field Team Coordinator – Groundwater Sampling	***de maximis, inc***	978.793.7163	ahoffmann@demaximis.com
Nathan Hunt	Task Manager – Ex-situ Groundwater Treatment and 1,4-Dioxane and VOCs in Bedrock	***de maximis, inc***	774.258.1015	nhunt@demaximis.com
Polly Newbold	QA Manager/Data Validator	ddms, Inc.	908.619.9132	pnewbold@ddmsinc.com
Heidi Gaedy, PMP	Database Manager	ddms, Inc	651.842.4236	hgaedy@ddmsinc.com
Bryan Heald	Data Management	Geosyntec	603.782.4073	bheald@geosyntec.com

QAPP Worksheet #3 Distribution List

QAPP Recipients	Title	Organization	Telephone Number	E-mail Address
Kelly Chatterton	Data Management	Haley & Aldrich, Inc.	617.886.7355	kchatterton@haleyaldrich.com
Mark Kelley, PE	Project Manager – Sitewide Soils and Sediment Remedy	Haley & Aldrich, Inc.	617.886.7338	mkelley@haleyaldrich.com
Kenneth Alepidis	Field Operations Lead– Sitewide Soils and Sediment Remedy/Holding Basin Containment	Haley & Aldrich, Inc.	617.886.7366	kalepidis@haleyaldrich.com
Elizabeth Christmas, EIT	Design and Reporting – Sitewide Soils and Sediment Remedy	Haley & Aldrich, Inc.	617.886.7581	echristmas@haleyaldrich.com
David Adilman, PG	Project Manager – ISS / 1,4-Dioxane and VOCs in Bedrock Groundwater	Geosyntec Consultants	978.206.5769	dadilman@geosyntec.com
Dariusz Chlebica, PG	Field Team Coordinator - ISS / 1,4-Dioxane and VOCs in Bedrock Groundwater	Geosyntec Consultants	978.206.5781	dchlebica@geosyntec.com
Chapman Ross, PE	Design Engineer - ISS / 1,4-Dioxane and VOCs in Bedrock Groundwater	Geosyntec Consultants	978.206.5779	cross@geosyntec.com
Tim Crowl, PE	Project Manager – Holding Basin Containment	Haley & Aldrich, Inc.	860.290.3121	tcrowl@haleyaldrich.com
Wystan Carswell, PE, PhD	Design and Reporting – Holding Basin Containment	Haley & Aldrich, Inc.	617.886.7357	wcarswell@haleyaldrich.com
Nicholas Carabillo	Project Manager / Field Team Coordinator – Ex-situ Groundwater Treatment	O&M, Inc.	860.819.0335	ncarabillo@oandm-inc.com

QAPP Worksheet #3 Distribution List

QAPP Recipients	Title	Organization	Telephone Number	E-mail Address
Dave Sanford	Laboratory Project Manager	Alpha Analytical Westborough	508.439.5157	dsanford@alphalab.com
Jim Todaro	Laboratory QA Manager	Alpha Analytical Westborough	508.439.5101	jtodaro@alphalab.com
Pete Henriksen	Laboratory Project Manager	Alpha Analytical Mansfield	508.844.4113	phenriksen@alphalab.com
Edie Kent	Laboratory Project Manager	GEL	843.769.7385	emk@gel.com
Robert Pullano	Laboratory QA Manager	GEL	843.556.8171	rlp@gel.com

Note: Copies of the QAPP will be distributed to the individuals above. The copies will consist of the following documents: QAPP, and any subsequent QAPP revisions and addenda.

QAPP Worksheet #4-1 Project Personnel Sign-Off (*de maximis*)

Organization/Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Bruce Thompson (***de maximis)***	Project Coordinator	860.298.0541		
Jessie McCusker (***de maximis)***	Alternate Project Coordinator / Project Manager – Sitewide Soils and Sediments and Holding Basin Remedies	860.817.7544		
Amy Hoffmann (***de maximis)***	Project Manager – ISS / Field Team Coordinator – Groundwater Sampling	978.793.7163		
Nathan Hunt (***de maximis)***	Project Manager – ISS / Field Team Coordinator – Groundwater Sampling	774.258.1015		
Polly Newbold (ddms)	QA Manager /Data Validator	908.619.9137		
Jeri Rossi (ddms)	QA Coordinator/Data Validator	908.370.3431		
Heidi Gaedy,PMP,GISP (ddms)	Database Manager	651.842.4236		
Nicholas Carabillo (O&M)	Project Manager / Field Team Coordinator – Ex-situ Groundwater Treatment	860.819.0335		

QAPP Worksheet #4-1 Project Personnel Sign-Off (*de maximis*)

Organization/Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Dave Fuerst (O&M)	Project Manager	865.804.6314		

Note: The project personnel sign-off table above documents key project personnel who have read the applicable sections of the QAPP and will perform the tasks as described in the QAPP.

QAPP Worksheet #4-2 Project Personnel Sign-Off (Haley & Aldrich)

Organization/Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Damien Seibert	Structural Analysis-Holding Basin Containment	617.886.7399		
Travis Hinman	Field Operations Lead--Holding Basin Containment	925.929.4406		
Wystan Carswell, PE, PhD	Design and Reporting--Holding Basin Containment	617.886.7357		
Kimberly Scalise	Reporting--Holding Basin Containment	617.886.7416		
Kelly Chatterton	Data Management	617.886.7355		
Mark Kelley, PE	Project Manager – Sitewide Soils and Sediment Remedy, cover design HB Containment	617.886.7338		
Peter Zawadzkas	PM-FSP, HASP	617.886.7335		
Denis Conley	QAPP	585.321.4245		

QAPP Worksheet #4-2 Project Personnel Sign-Off (Haley & Aldrich)

Organization/Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Nadia Glucksberg	TSCA/Radiological Regulatory, ARARs support	207.482.4623		
Jay Peters	Cleanup Goal Compliance / Risk Assessment	603.391.3312		
Kenneth Alepidis	Field Operations Lead-Sitewide Soils and Sediment Remedy	617.886.7366		
Elizabeth Christmas, EIT	Design and Reporting-Sitewide Soils and Sediment Remedy	617.886.7581		

Note: The project personnel sign-off table above documents key project personnel who have read the applicable sections of the QAPP and will perform the tasks as described in the QAPP.

QAPP Worksheet #4-3 Project Personnel Sign-Off (Geosyntec Consultants)

Organization/Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Carl Elder, PhD., PE	Project Manager- ISS / 1,4-Dioxane and VOCs in Bedrock Groundwater	978.206.5768		
Jeff Gillow, PhD	Geochemistry and Uranium Sequestration - ISS	720.286.2249		
Chris Robb, PE	In-Situ Soil Mixing - ISS	262.834.0232		
Peter Zeeb, PhD, PG, BCEE	Geochemistry - ISS	978.263.9588		
Doug Larson, PhD, PE	Remediation - ISS	978.206.5774		
Juan Pestana, ScD, PE	Seismic / Stability Analysis (peer review)-Holding Basin Containment	978.206.5753		
Bryan Heald	Data Management	603-782-4073		
David Adilman, PG	Project Manager – ISS / 1,4-Dioxane and VOCs in	978.206.5769		

QAPP Worksheet #4-3 Project Personnel Sign-Off (Geosyntec Consultants)

Organization/Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
	Bedrock Groundwater			
Dariusz Chlebica, PG	Field Team Coordinator - ISS / 1,4-Dioxane and VOCs in Bedrock Groundwater, QA Manager,FSP	978.206.5781		

Note: The project personnel sign-off table above documents key project personnel who have read the applicable sections of the QAPP and will perform the tasks as described in the QAPP.

QAPP Worksheet #4-4 Project Personnel Sign-Off (Alpha)

Organization/Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Pete Henriksen	Laboratory Project Manager	508.844.4113		
Dave Sanford	Laboratory Project Manager	508.439.5157		
Jim Todaro	Laboratory QA Manager	508.439.5101		

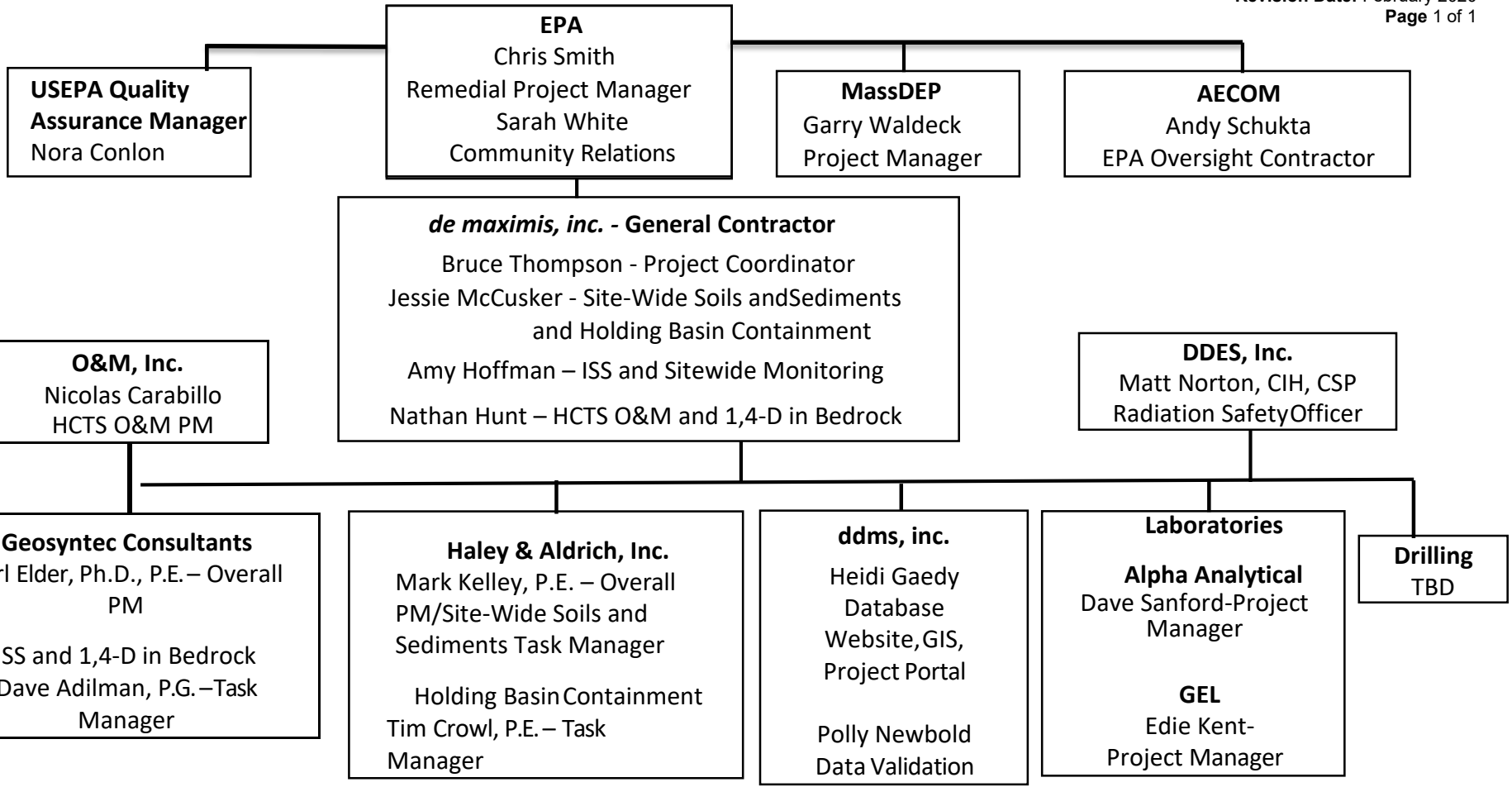
Note: The project personnel sign-off table above documents key project personnel who have read the applicable sections of the QAPP and will perform the tasks as described in the QAPP.

QAPP Worksheet #4-5 Project Personnel Sign-Off (GEL)

Organization/Project Personnel	Title	Telephone Number	Signature	Date QAPP Read
Edie Kent	Laboratory Project Manager	843.769.7385		
Robert Pullano	Laboratory QA Manager	843.556.8171		

Note: The project personnel sign-off table above documents key project personnel who have read the applicable sections of the QAPP and will perform the tasks as described in the QAPP.

QAPP Worksheet #5 NMI Project Organization Chart



QAPP Worksheet #6 Communication Pathways

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (e.g., Timing, Pathways)
Point of Contact with USEPA Remedial Project Manager (RPM)	Lead Organization Project Manager	Chris Smith	617.918.1339	Planning documents will be provided to Chris Smith, USEPA by Bruce Thompson, de maximis.
Manage all Project Phases	Project Coordinator	Bruce Thompson	860.298.0541	Bruce Thompson will be the liaison to the SRSNE Site Group and all agencies.
Coordinate Field Program	Alternate Project Coordinator/Project Manager/Field Team Manager/Coordinator	Jessie McCusker/Nathan Hunt/Amy Hoffmann/Kenneth Alepidis/Dariusz Chelbica	860.817.7544/774.258.1015/ 978.793.7163/617.886.7366/ 978.206.5781	To be notified of field-related questions or problems by phone, e-mail, or fax by COB the next business day.
QAPP changes in the field	Project Manager/Field Team Manager/Coordinator	Jessie McCusker/Nathan Hunt/Amy Hoffmann/Kenneth Alepidis/Dariusz Chelbica	860.817.7544/774.258.1015/ 978.793.7163/617.886.7366/ 978.206.5781	To be notified of any changes to QAPP made in the field and the reasons within 2 business days. Will notify the Project Manager and the Project Manager and Project Manager/Coordinator of any such changes immediately.
Daily Field Progress Reports	Field Team Manager/Coordinator	Kenneth Alepidis/Dariusz Chelbica	860.817.7544/774.258.1015/ 978.793.7163/617.886.7366/ 978.206.5781	To be provided daily field progress reports. Will provide complete sets of daily field progress reports, sampling logs, chains-of-custody forms, and other information to the Project Manager and Field Program Manager/Coordinator.

QAPP Worksheet #6 Communication Pathways

Communication Drivers	Responsible Entity	Name	Phone Number	Procedure (e.g., Timing, Pathways)
Reporting Lab Data Quality Issues	Laboratory Project Manager	Dave Sanford/Pete Henriksen/Edie Kent	508.439.5157/508.884.4113/ 843.556.8171	All QA/QC issues with project field samples will be reported by Ruth Welsh to Polly Newbold within 1 business day.
Field and Analytical Corrective Actions	QA Manager	Jim Todaro/Robert Pullano/ Polly Newbold	508.439.5101/843.556.8171/ 908.619.9132	The need for corrective action for field and analytical issues will be determined by Dennis Capria/Polly Newbold in conjunction with the Project Coordinator, the Field Program Coordinator or the Laboratory QA Manager, as appropriate.
Release of Analytical Data	Data QA Manager	Polly Newbold	908.619.9132	No final analytical data can be released until validation is completed and Polly Newbold has approved the release. Unvalidated data may be released at any time
QAPP Amendments	QA Coordinator	Jeri Rosi	908.370.3431	Any major changes to the QAPP must be approved by Jeri Rosi and the Alternate Project Coordinator (Jessie McCusker) and USEPA before the changes can be implemented.

QAPP Worksheet #7 Personnel Responsibilities and Qualifications

Name	Title	Organizational Affiliation	Education and Experience Qualifications
Chris Smith	Remedial Project Manager / QAPP Reviewer	USEPA	Designated as the USEPA's Remedial Project Manager
Nora Conlon	QAPP Reviewer	USEPA	Designated as the USEPA's Project QA Manager
Bruce Thompson	Project Coordinator	***de maximis, inc.***	B.S., Oceanography, 29 years of experience
Jessie McCusker	Alternate Project Coordinator / Project Manager – Sitewide Soils and Sediments and Holding Basin Remedies	***de maximis, inc.***	B.S., Facilities / Environmental Engineering, 19 years of experience
Amy Hoffmann	Project Manager – ISS, Field Team Coordinator – Groundwater Sampling	***de maximis, inc.***	B.S., Geology, 9 years of experience
Nathan Hunt	Project Manager – Ex-situ Groundwater Treatment and 1,4-Dioxane and VOCs in Bedrock	***de maximis, inc***	B.S., Environmental Science, 9 years of experience
Polly Newbold	QA Manager /Data Validator	ddms, Inc.	B.S., in Textile Science, 36 years of experience
Jeri Rosi	QA Coordinator /Data Validator	ddms, Inc	B.S., in Environmental Science, 31 years of experience
Heidi Gaedy,PMP,GISP	Database Manager	ddms, Inc	M.S. Resource Analysis, B.S. Environmental Biology, 22 years of experience

QAPP Worksheet #7 Personnel Responsibilities and Qualifications

Name	Title	Organizational Affiliation	Education and Experience Qualifications
Mark Kelley, PE	Project Manager – Sitewide Soils and Sediment Remedy/Grouting Soil Structural Analysis Holding Basin Containment	Haley & Aldrich, Inc.	B.S., Civil Engineering, M.S. Civil Engineering, 31 years of experience
Tim Crowl, PE	Project Manager – Holding Basin Containment	Haley & Aldrich, Inc.	B.S., Civil and Environmental Engineering, M.S. Civil Engineering, 34 years of experience
Kenneth Alepidis	Field Team Coordinator – Holding Basin Containment/Sitewide Soils and Sediment Remedy	Haley & Aldrich, Inc.	B.S., Environmental Geology, 19 years of experience
Jean Louis Locsin, PE, PhD	Seismic and Slope Stability analysis – Holding Basin Containment	Haley & Aldrich, Inc.	B.S., Civil Engineering, M.S. Civil and Environmental Engineering, Ph.D., Civil and Environmental Engineering, 16 years of experience
Jay Peters	Cleanup Goal Compliance/Risk Assessment	Haley & Aldrich, Inc.	B.S., Toxicology, M.S. Environmental Engineering, over 26 years of experience
Nadia Glucksberg	TSCA/Radiological Regulatory/ARARs Support	Haley & Aldrich, Inc.	B.A., Geological Services, M.S. Environmental Science and Engineering, over 26 years of experience
Carl Elder, PE, PhD	Project Manager – ISS / 1,4-Dioxane and VOCs in Bedrock Groundwater	Geosyntec Consultants	BS, MS & Ph.D., Civil Engineering, 23 years of experience
David Adilman, PG	Project Manager – ISS / 1,4-Dioxane and VOCs in Bedrock Groundwater	Geosyntec Consultants	BS & MA, Geology and Petroleum Engineering, 40 years of experience

QAPP Worksheet #7 Personnel Responsibilities and Qualifications

Name	Title	Organizational Affiliation	Education and Experience Qualifications
Dariusz Chlebica, PG	Field Team Coordinator - ISS / 1,4-Dioxane and VOCs in Bedrock Groundwater	Geosyntec Consultants	BS & MS, Geologic Sciences, 12 years of experience
Chris Martin	Design Engineer - ISS	Geosyntec Consultants	BS & MS, Environmental and Water Resources Engineering, 9 years of experience
Nicholas Carabillo	Project Manager / Field Team Coordinator – Ex-situ Groundwater Treatment	O&M, Inc.	B.S, Chemical Engineering, 3 years of experience
Dave Sanford	Laboratory Project Manager	Alpha Analytical Westborough	B.A., Chemistry; over 33 years of experience
Jim Todaro	Laboratory QA Manager	Alpha Analytical Westborough	B.S., Biology; over 46 years of experience
Pete Henriksen	Laboratory Project Manager	Alpha Analytical Mansfield	BS Environmental Science; over 26 years of experience
Robert Pullano	Laboratory QA Manager	GEL	BS Biology and Marine Science; 35 years of experience with 32 years with GEL
Edie Kent	Laboratory Project Manager	GEL	BA History, MPA Public Administration; over 31 years of experience

QAPP Worksheet #7 Personnel Responsibilities and Qualifications

Notes:
The responsibilities of the various team members are summarized below by organization.

Project Coordinator
Responsibilities and duties include:
- Communicate with SRSNE Site Group, USEPA, CT DEEP, Project Team, and other stakeholders
- direct Project Team
- oversee all aspects of the RD/RA work
- provide approval for major project deliverables

Project Managers
The investigation components will be managed by various Task Managers. Duties of each Task Manager include, as appropriate:
- manage relevant day-to-day activities
- develop, establish and maintain files on relevant site activities
- review data reductions from the relevant site activities
- perform final data review of field data reductions and reports on relevant site activities
- verify that corrective actions are taken for deficiencies cited during audits of relevant site activities
- perform overall QA/QC of the relevant portions of the site activities
- review relevant field records and logs
- instruct personnel working on relevant site activities

Field team Coordinator
- coordinate field and laboratory schedules pertaining to relevant site activities
- request sample bottles from laboratory
- review field instrumentation, maintenance and calibration to meet quality objectives
- prepare reports pertaining to relevant field activities
- maintain field and laboratory files of notebooks/logs, data reductions and calculations; provide complete copies to the Project Coordinator.
- perform field procedures associated with the investigations as set forth in the Work Plan/Design Report
- perform field analyses and collect QA samples
- calibrate, operate and maintain field equipment
- reduce field data

QAPP Worksheet #7 Personnel Responsibilities and Qualifications

- maintain sample custody
- prepare field records and logs

QA Manager

Responsibilities and duties include:

- review laboratory data packages
- oversee and interface with the analytical laboratory
- coordinate field QA/QC procedures with Task Managers (including audits of field activities), concentrating on field analytical measurements and practices to meet data quality objectives (DQOs)
- review field reports
- perform and review audit reports as necessary and appropriate
- prepare interim QA/QC compliance reports as necessary and appropriate
- prepare a QA/QC report in accordance with USEPA guidelines, including an evaluation of field and laboratory data, and data usability reports
- five percent QA/QC check of imported field and laboratory results versus the hardcopy

Database Manager

- data collected in field will be transcribed from field form or notebooks and tabulated into database
- analytical results provided by the laboratory in the form of an electronic data deliverable (EDD) will imported into the database
- tabulation of the data to end users
- Report/Graphics preparation

QA Coordinator

Responsibilities and duties include:

- preparation of Quality Assurance Project Plan (QAPP)
- maintain QAPP

Analytical Laboratories

General responsibilities and duties of the analytical laboratories include:

- perform sample analyses and associated laboratory QA/QC procedures
- supply sampling containers and shipping cartons

QAPP Worksheet #7 Personnel Responsibilities and Qualifications

- maintain laboratory custody of sample
- adhere to all protocols in the QAPP

Project Manager
Responsibilities and duties include:
- serve as primary communication link between ARCADIS and laboratory technical staff
- monitor workloads and maintain availability of resources
- oversee preparation of analytical reports
- supervise in-house chain of custody

Laboratory QA Manager
Responsibilities and duties include:
- supervise personnel reviewing and inspecting all project-related laboratory activities
- conduct audits of all laboratory activities

United States Environmental Protection Agency

Remedial Project Manager
Responsibilities and duties include:
- provide the USEPA's reviews and approvals of submitted documents
- coordinate with other agency stakeholders
- monitor progress of site activities

QA Technical Staff
Responsibilities and duties include:
- review and approval of the QAPP
- review of the QA/QC portion of any submitted report
- perform field and laboratory audits, if necessary

QAPP Worksheet #8 Special Personnel Training Requirements

Project Function	Specialized Training	Training Provider	Training Date	Personnel/Groups Receiving Training	Personnel Titles/ Organizational Affiliation	Location of Training Records/Certificates
Field Activities	40-hour HAZWOPER	Certified Training Professionals	NA	Field operations personnel	All field personnel associated with RD/RA activities involving potential for exposure to site-related COCs	Project offices of respective employers
Analytical Chemistry	NELAP Accreditation	Primary Accrediting State	NA	NA	Alpha Analytical Westborough 8 Walkup Dr. Westborough, MA 01581 508.898.9220	Alpha Analytical Westborough 8 Walkup Dr. Westborough, MA 01581 508.898.9220
Analytical Chemistry	NELAP Accreditation	Primary Accrediting State	NA	NA	Alpha Analytical Mansfield 320 Forbes Blvd. Mansfield, MA 508.898.9220	Alpha Analytical Mansfield 320 Forbes Blvd. Mansfield, MA 508.898.9220
Analytical Chemistry	NELAP Accreditation	Primary Accrediting State	NA	NA	GEL 2040 Savage Road, Charleston, SC 29407 PO Box 30712, Charleston, SC 29417	GEL 2040 Savage Road, Charleston, SC 29407
Additional training/certification requirements are listed in the project HASP						

QAPP Worksheet #9 Project Team Planning Sessions Participants Sheet

Project Name: **NMI Remedial Design/Remedial Action**	Site Name: **Nuclear Metals, Inc. (NMI)**
Projected Date(s): of Sampling: Annual, starting June 2019	Site Location: 2229 Main Street, Concord, MA 01742-3897
Project Coordinator: Bruce Thompson	
January 8, 2020-Meeting	
Scoping Session Purpose: RD/RA planning meeting	

Name	Title	Affiliation	Phone	E-mail Address	Project Role
Chris Smith	Remedial Project Manager	USEPA	617.918.1339	Smith.christopher@epa.gov	Remedial Project Manager
Gary Waldeck	Project Manager	MassDEP	617.348.4017	Garry.waldeck@mass.gov	Project Manager
Bruce Thompson	Project Coordinator	de maximis, inc.	860.298.0541	brucet@demaximis.com	Project Coordinator
Jessie McCusker	Project Manager	de maximis, inc.	860.817.7544	jessie@demaximis.com	Project Manager
Nathan Hunt	Project Manager	de maximis, inc.	774.258.1015	nhunt@demaximis.com	Project Manager
Amy Hoffmann	Project Manager	de maximis, inc.	978.793.7163	ahoffmann@demaximis.com	Project Manager
Carl Elder	Project Manager	Geosyntec	978.206.5768	celder@geosyntec.com	Project Manager
Chris Martin	Engineer	Geosyntec	978.206.5711	cmartin@geosyntec.com	Design Engineer

QAPP Worksheet #9 Project Team Planning Sessions Participants Sheet

Name	Title	Affiliation	Phone	E-mail Address	Project Role
Dariusz Chlebica	Geologist	Geosyntec	978.206.5779	dchlebica@geosyntec.com	Geologist
David Adilman	Geologist	Geosyntec	978.206.5769	Dadilman@geosntec.com	Project Manager
Warren Diesl	Environmental	AECOM		warren.diesl@aecom.com	EPA Oversight Contractor
Andy Schkuta	Environmental	AECOM	781.290.9448	Andy.schkuta@aecom.com	EPA Oversight Contractor
Mark Kelly	Engineer	Haley and Aldrich	617.886.7338	mkelly@halyaldrich.com	Project Manager
Peter Zawadzkas	Engineer	Haley and Aldrich	617.886.7335	pzawadzkas@haleyaldrich.com	Project Manager

QAPP Worksheet #9 Project Team Planning Sessions Participants Sheet

Project Name: **NMI Remedial Design/Remedial Action**	Site Name: **Nuclear Metals, Inc. (NMI)**
Projected Date(s): bi-weekly	Site Location: 2229 Main Street, Concord, MA 01742-3897/conference call
Project Coordinator: Bruce Thompson	
February 4, 2020-Meeting	
Scoping Session Purpose: RD/RA planning meeting	

Name	Title	Affiliation	Phone	E-mail Address	Project Role
Chris Smith	Remedial Project Manager	USEPA	617.918.1339	Smith.christopher@epa.gov	Remedial Project Manager
Gary Waldeck	Project Manager	MassDEP	617.348.4017	Garry.waldeck@mass.gov	Project Manager
Bruce Thompson	Project Coordinator	de maximis, inc.	860.298.0541	brucet@demaximis.com	Project Coordinator
Jessie McCusker	Project Manager	de maximis, inc.	860.817.7544	jessie@demaximis.com	Project Manager
Nathan Hunt	Project Manager	de maximis, inc.	774.258.1015	nhunt@demaximis.com	Project Manager
Amy Hoffmann	Project Manager	de maximis, inc.	978.793.7163	ahoffmann@demaximis.com	Project Manager
Carl Elder	Project Manager	Geosyntec	978.206.5768	celder@geosyntec.com	Project Manager
Dariusz Chlebica	Geologist	Geosyntec	978.206.5779	dchlebica@geosyntec.com	Geologist

QAPP Worksheet #9 Project Team Planning Sessions Participants Sheet

Name	**Title**	**Affiliation**	**Phone**	**E-mail Address**	**Project Role**
David Adilman	Geologist	Geosyntec	978.206.5769	Dadilman@geosntec.com	Project Manager
Warren Diesl	Environmental	AECOM		warren.diesl@aecom.com	EPA Oversight Contractor
Mark Kelly	Engineer	Haley and Aldrich	617.886.7338	mkelly@halyaldrich.com	Project Manager
Peter Zawadzkas	Engineer	Haley and Aldrich	617.886.7335	pzawadzkas@haleyaldrich.com	Project Manager

QAPP Worksheet #10 Problem Definition — DQOs

Step 1: State the Problem:

As specified in the United States Environmental Protection Agency's (USEPA's) Record of Decision (ROD), the selected remedy for the NMI Site includes:

- Excavation and off-site disposal of approximately 82,500 cubic yards of contaminated sediments, underground drain lines and debris, and non-Holding Basin (HB) soils (contaminated with depleted uranium (DU), polychlorinated biphenyls (PCBs), and other contaminants of concern found in Tables L-2 through L-4 of the ROD) in various areas of the Site;
- In-situ sequestration (ISS) of DU contaminated soils in the HB via injection of a stabilization agent such as apatite (e.g., Apatite II™) or other comparable stabilization agent to prevent leaching of contaminants to groundwater, and ISS of DU in the overburden aquifer and of natural uranium in the bedrock aquifer;
- Containment of HB soils with a low-permeability vertical wall and horizontal sub-grade cover to isolate the stabilized soils and further limit mobility of contaminants by removing the flow of groundwater;
- Extraction and *ex-situ* treatment of volatile organic compounds (VOCs) and 1,4-dioxane in overburden and bedrock aquifers;
- Long-term monitoring to monitor effectiveness of *in-* and *ex-situ* treatment; and
- Institutional Controls to: 1) prevent unacceptable exposures to, and to prevent disturbance of, the HB area; 2) prohibit use of contaminated groundwater until cleanup levels are met; and 3) require installation of vapor mitigation systems should future structures be built above the VOC plume before groundwater cleanup levels are met, unless an evaluation of vapor intrusion risks is performed to show such systems are not required.

These remedial measures will mitigate uncontrolled migration of and exposure to NMI-related constituents of concern (COCs) and will allow for the restoration of the Site to beneficial uses including eventual use of the aquifer underlying the Site for drinking and other domestic uses in the long term.

Step 2: Identify the Goal of the Study:

Containment of HB- Containment of HB soils with a low-permeability vertical wall and horizontal sub-grade cover to isolate the stabilized soils and further limit mobility of contaminants by removing the flow of groundwater

ISS- In-situ sequestration (ISS) of DU contaminated soils in the HB via injection of a stabilization agent such as apatite (e.g., Apatite II™) or other

QAPP Worksheet #10 Problem Definition — DQOs

comparable stabilization agent to prevent leaching of contaminants to groundwater, and ISS of DU in overburden groundwater and natural uranium in bedrock groundwater

Excavation-Excavation and off-site disposal of approximately 82,500 cubic yards of contaminated sediments, underground drain lines and debris, and non-Holding Basin (HB) soils (contaminated with depleted uranium (DU), polychlorinated biphenyls (PCBs), and other contaminants of concern found in ROD Tables L-2 through L-4) in various areas of the Site

Extraction and *ex-situ* treatment of volatile organic compounds (VOCs) and 1,4-dioxane in overburden and bedrock aquifers

Long-term Monitoring - Purposes for Monitoring Well Network
The proposed monitoring network associated with the general plume characterization will initially consist of 130 monitoring wells to be used for sampling and water level measurements. The data obtained from monitoring this extensive monitoring network will be used to characterize the COC plume in terms of:

- Plume extent
- Temporal and spatial variations in plume chemistry and geometry
- Capture of all groundwater that exceeds federal drinking water standards and other risk-based levels.
- Progress in meeting the long-term remedial goal of groundwater restoration
- Effectiveness of institutional controls

The key goals of the Groundwater Monitoring Program are:
- Provide a groundwater sampling schedule, including frequency, analytical parameters and sampling methods.

QAPP Worksheet #10 Problem Definition — DQOs

Step 3: Identify Information Inputs:
Information inputs incorporate both the concentration and distribution of COCs in site media. A fundamental basis for decision-making is that a sufficient number of data points of acceptable quality are available from the specific RD investigation associated with the specific RD Work Plan to support the decision. Thus, the necessary input for the decision is the proportion of non-rejected (usable) data points.
Step 4: Define the Boundaries of the Sampling:
The NMI Site includes the property located at 2229 Main Street in Concord, MA and downgradient properties where groundwater contamination has migrated The NMI property is bordered by residential and woodland areas to the east and south, light commercial and industrial areas to the west, and Main Street (Route 62) and the Assabet River to the north. The NMI property consists of approximately 46.4 acres.
Step 5: Develop the Analytic Approach:
Typically, the decision on whether data can be used will be based on the validation results. Following validation, the data will be flagged, as appropriate, and any use restrictions will be noted. The RD Work Plans have been devised so that the loss of any single data point will not hinder description of the distribution of constituents of concern or the development of a risk assessment. A decision rule is adopted that 90 percent of the data points not be rejected or deemed unusable as a condition for use of the data set for decision-making purposes. The usable data will be evaluated versus the performance standards. The required reporting limits are also documented in Worksheets 15-1 through 15-3 so that the lowest achievable detection limit will be reported by the laboratory.
Step 6: Specify Performance or Acceptance Criteria:
Based on the potential uses of data in the decision-making process, performance and acceptance criteria are specified in the NMI Remedial Design/Remedial Action (RD/RA) Statement of Work (SOW). The work plans required by the SOW identified the various sampling and analysis programs that this QAPP addresses. Corrective actions are described within this document. The representative nature of the sampling design has been facilitated by discussions among professionals familiar with the site and the appropriate government agencies.

QAPP Worksheet #10 Problem Definition — DQOs

Step 7: Develop the Plan for Obtaining Data:
The overall QA objective is to develop and implement procedures for field sampling — Chain-of-Custody, laboratory analysis and reporting — that will provide results to support the evaluation of site data consistent with requirements identified in the NMI RD/RA SOW. Specific procedures for sampling, chain of custody procedures, laboratory instrument calibration, laboratory analysis, data reporting, internal QC, audits, preventive maintenance of field equipment and corrective action are described in other sections of this QAPP and FSP. A DQO summary for the sampling investigation efforts is presented in the following subsection. The summary consists of stated DQOs relative to data uses, data types, data quantity, sampling and analytical methods, and data measurement performance criteria.

QAPP Worksheet #11 Project Quality Objectives/Systematic Planning Process Statements

Who will use the data?
de maximis, ddms, Geosyntec Consultants, Haley & Aldrich, Inc., O&M, Inc., and their subcontractors and overseeing agencies
What will the data be used for?
Data generated during the RD will be used to: • Complete Draft Remedial Action Work Plan • Complete Remedial Action

QAPP Worksheet #11 Project Quality Objectives/Systematic Planning Process Statements

What type of data are needed? (target analytes, analytical groups, field screening, on-site analytical or off-site laboratory techniques, sampling techniques)

The primary COCs and cleanup levels are listed in Tables L-1, L-2 and L-3 of the Record of Decision (September 2015). Laboratory analyses will be conducted at an off-site laboratory. Standard protocols for sample collection and handling, sample preparation, and analytical methods will be followed. Standard operating procedures are provided in this document and in the FSP.

How "good" do the data need to be in order to support the environmental decision?

The data need comply with the QA/QC requirements of this QAPP to be consistent with the large historical RI database and all CERCLA regulations.

Data Categories

Three data categories have been defined to address various analytical data uses and the associated QA/QC effort and methods required to achieve the desired levels of quality. These categories are:

Screening Data: Screening data affords a quick assessment of site characteristics or conditions. This DQO is applicable to data collection activities that involve rapid, non-rigorous methods of analysis and quality assurance. This objective is generally applied to physical and/or chemical properties of samples, degree of contamination relative to concentration differences, and preliminary health and safety assessment.

Screening Data with Definitive Confirmation: Screening data allows rapid identification and quantitation, although the quantitation can be relatively imprecise. This DQO is available for data collection activities that require qualitative and/or quantitative verification of a select portion of sample findings (10% or more). This objective can also be used to verify less rigorous laboratory-based methods.

Definitive Data: Definitive data are generated using analytical methods such as approved USEPA reference methods. Data are analyte-specific, with confirmation of analyte identity and concentration. Methods produce raw data (e.g., chromatograms, spectra, digital values) in the form of paper printouts or computer-generated electronic files.

It is anticipated that both the screening and definitive data categories will be used during the investigation. Field screening data (e.g., pH, dissolved oxygen, turbidity, temperature, specific conductance, oxidation-reduction potential) will be obtained during sampling to provide real-time quantitative data that will assist in evaluating monitoring well placement. Groundwater samples will be collected from temporary monitoring wells and hydropunch sampling points and will be sent to the laboratory to obtain definitive pH and metals data.

QAPP Worksheet #11 Project Quality Objectives/Systematic Planning Process Statements

For this project, three levels of data reporting have been defined. They are as follows: Level 1 – Minimal Reporting: Minimal or "results only" reporting is used for analyses that, either due to their nature (i.e., field monitoring) or the intended data use (i.e., preliminary screening), do not generate or require extensive supporting documentation. Level 2 – Modified Reporting: Modified reporting is used for analyses that are performed following standard USEPA-approved methods and QA/QC protocols and that, based on the intended data use, require some supporting documentation but not, however, full "Contract Laboratory Program (CLP)-type" reporting. Level 3 – Full Reporting: Full "CLP-type" reporting is used for those analyses that, based on intended data use, require full documentation.
How much data are needed? (number of samples for each analytical group, matrix and concentration)
The number of samples and analyses for each media are summarized in Worksheet #20 and described in the various PDI WPs, TS WPs and SWMP.
Where, when, and how should the data be collected/generated?
Specific sample locations and collection methods are specified in the respective RD work plans. Samples will be sent to a lab for analytical evaluation and data results will be returned to de maximis.
Who will collect and generate the data?
de maximis, O&M Inc, Geosyntec Consultants, Haley & Aldrich, Inc. and their subcontractors.

QAPP Worksheet #11 Project Quality Objectives/Systematic Planning Process Statements

How will the data be reported?
The data will be reported by the laboratory in laboratory data packages, including PDF and electronic data deliverables (EDDs). Data will be validated and reported to the Agencies upon completion with annual reports. Data pertinent to a given activity will also be presented in the report summarizing the completion of that activity. In addition, data will be posted to a dedicated project web site (Project Portal) for use and querying by project-related personnel, including representatives of regulatory agencies.
How will the data be archived?
All data will be archived digitally by de maximis.

QAPP Worksheet #12-1 Measurement Performance Criteria (Volatile Organic Compounds)

Matrix	Aqueous/ Leachate
Analytical Group	VOCs
Concentration Level	All

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Laboratory Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)	Data Validation Criteria
NMI-GW-001 - NMI-GW-020	SW846 1311 (GL-LB-E-006) and/or SW846 8260D (GL-OA-E-038)	Accuracy/Bias Contamination	No target analytes detected greater than one-half RL and 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater). No laboratory common contaminants detected greater than RL.	Blanks (method, tumble, trip, field, equipment, method)	S&A	Sample concentrations ≤ blank concentration* will be qualified as not detected (U)
		Accuracy/Bias	Laboratory acceptance limits[5]	Surrogate	A	%R 70-130, %R<10 non-detects will be rejected (R)
		Accuracy/Bias/ Precision	Laboratory acceptance limits[5]	LCS/LCSD[4]	A	%R 70-130, RPD≤20
		Accuracy/Bias Precision	Laboratory acceptance limits[5]	MS/MSD[3]	S&A	%R 70-130, RPD≤30

QAPP Worksheet #12-1 Measurement Performance Criteria (Volatile Organic Compounds)

Matrix	Aqueous/ Leachate
Analytical Group	VOCs
Concentration Level	All

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Laboratory Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)	Data Validation Criteria
		Precision	RT within ±30 seconds from RT of initial calibration midpoint standard; area counts within -50% to +100% of initial calibration midpoint standard	Internal standard	A	RT within ±30 seconds from RT of initial calibration midpoint standard; area counts within -50% to +100% of initial calibration midpoint standard
		Precision	RPD ≤ 30%	Field duplicate	S&A	RPD ≤ 30% for concentrations ≥RL
		Sensitivity	RL < 1/3 PAL	RL	A	RL < 1/3 PAL, based on low standard in IC

NOTES:
*-Methylene chloride and toluene ≤2X blank concentration.
[1] See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]Reference number from QAPP Worksheet #23.
[3] Extra sample volume required for site-specific MS/MSD
[4] LCS/LCSD must be performed if site-specific MS/MSD is not performed.
PAL-Project Action Limit
[5]-laboratory acceptance limits based on Depart of Defense (DoD) quality Systems Manual (QSM)

QAPP Worksheet #12-1 Measurement Performance Criteria (Volatile Organic Compounds)
GEL

Matrix	Soil, Sediment
Analytical Group	VOCs
Concentration Level	All

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Laboratory Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)	Data Validation Criteria
NMI-S-001 - NMI-S-007	SW846 8260D / GL-OA-E-038	Accuracy/Bias Contamination	No target analytes detected greater than one-half RL and 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater). No laboratory common contaminants detected greater than RL.	Blanks (method)	S&A	Sample concentrations ≤ blank concentration* will qualified as not detected (U)
		Accuracy/Bias	Laboratory acceptance limits[5]	Surrogate	A	%R 70-130, %R<10 non-detects will be rejected (R)
		Accuracy/Bias/ Precision	Laboratory acceptance limits[5]	LCS/LCSD[4]	A	%R 70-130, RPD≤20
		Accuracy/Bias Precision	Laboratory acceptance limits[5]	MS/MSD[3]	S&A	%R 70-130, RPD≤30

QAPP Worksheet #12-1 Measurement Performance Criteria (Volatile Organic Compounds)
GEL

Matrix	Soil, Sediment
Analytical Group	VOCs
Concentration Level	All

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Laboratory Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)	Data Validation Criteria
		Precision	RT within ±30 seconds from RT of initial calibration midpoint standard; area counts within -50% to +100% of initial calibration midpoint standard	Internal standard	A	Area response 50-200% of CC +/- 0.06 RRT
		Precision	RPD < 30%	Field duplicate	S&A	RPD ≤ 50% for concentrations ≥RL

Notes:
*-Methylene chloride and toluene ≤2X blank concentration
[1] See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]Reference number from QAPP Worksheet #23.
[3] Extra sample volume required for site-specific MS/MSD
[4] LCS/LCSD must be performed if site-specific MS/MSD is not performed.
[5]-laboratory acceptance limits are based on Department of Defense (DoD) Quality Systems Manual (QSM)

QAPP Worksheet #12-1 Measurement Performance Criteria (Ammonia Nitrogen)

Matrix	Aqueous
Analytical Group	Ammonia Nitrogen
Concentration Level	All

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Laboratory Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)	Data Validation Criteria
NMI-GW-001 - NMI-GW-020	SM4500 NH3-BH / 2206	Accuracy/Bias Contamination	<RL	Blanks (Method, field, equipment)	S & A	Sample Concentrations ≤ blank concentration will be qualified not detected (U).
		Accuracy/Bias/ Precision	%R 90-110	LCS	A	%R 90-110
		Accuracy/Bias	%R 90-110	MS	S&A	%R 90-110
		Precision	RPD≤20	Lab Duplicates	A	RPD≤20
		Precision	RPD < 30% Results ≥ RL	Field Duplicates	S&A	RPD < 30% For concentrations ≥ RL

Notes:
[1]See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]Reference number from QAPP Worksheet #23.

QAPP Worksheet #12-2 Measurement Performance Criteria (Semivolatile Organic Compounds)

Matrix	Aqueous, Leachate
Analytical Group	SVOCs
Concentration Level	All

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Laboratory Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)	Data Validation Criteria
NMI-GW-001 - NMI-GW-020	SW826 1311 (GL-LB-E-006) and/or SW846 3510C (GL-OA-E-013) and SW846 8270E/ (GL-OA-E-009)	Accuracy/Bias Contamination	No target analytes detected greater than one-half RL and 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater). No laboratory common contaminants detected greater than RL.	Blanks (method, tumble, field, equipment, method)	S&A	Sample concentrations ≤ blank concentration will be qualified as not detected (U)
		Accuracy/Bias	Laboratory acceptance limits*	Surrogate	A	%R 70-130, %R<10 non-detects will be rejected (R)
		Accuracy/Bias/ Precision	Laboratory acceptance limits*	LCS/LCSD[4]	A	%R 70-130 RPD ≤20
		Accuracy/Bias Precision	Laboratory acceptance limits*	MS/MSD [3]	S&A	%R 70-130 RPD≤30

QAPP Worksheet #12-2 Measurement Performance Criteria (Semivolatile Organic Compounds)

Matrix	Aqueous, Leachate
Analytical Group	SVOCs
Concentration Level	All

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Laboratory Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)	Data Validation Criteria
		Precision	RT within ±30 seconds from RT of initial calibration midpoint standard; area counts within -50% to +100% of initial calibration midpoint standard	Internal standard	A	RT within ±30 seconds from RT of initial calibration midpoint standard; area counts within -50% to +100% of initial calibration midpoint standard
		Precision	RPD ≤ 30%	Field duplicate	S&A	RPD ≤ 30% for concentrations ≥RL
		Sensitivity	RL < 1/3 PAL	RL	A	RL < 1/3 PAL, based on low standard in IC

Notes:
[1] See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]Reference number from QAPP Worksheet #23.
[3] Extra sample volume required for site specific MS/MSD
[4] LCS/LCSD must be performed if site specific MS/MSD is not performed.
PAL-Project Action Limit
*-laboratory limits are based on Department of Defense (DoD) quality Systems Manual (QSM)

QAPP Worksheet #12-2 Measurement Performance Criteria (Semivolatile Organic Compounds)
GEL

Matrix	Soil, Sediment
Analytical Group	SVOCs
Concentration Level	All

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Laboratory Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)	Data Validation Criteria
NMI-S-001 - NMI-S-007	SW846 3541/ GL-OA-E-066 and SW846 8270E/ GL-OA-E-009	Accuracy/Bias Contamination	No target analytes detected greater than one-half RL and 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater). No laboratory common contaminants detected greater than RL.	Blanks (method)	S&A	Sample concentrations ≤ blank concentration will be qualified as not detected (U)
		Accuracy/Bias	Laboratory acceptance limits4	Surrogate	A	%R 70-130, %R<10 non-detects will be rejected (R)

QAPP Worksheet #12-2 Measurement Performance Criteria (Semivolatile Organic Compounds)
GEL

Matrix	Soil, Sediment
Analytical Group	SVOCs
Concentration Level	All

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Laboratory Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)	Data Validation Criteria
		Accuracy/Bias/ Precision	Laboratory acceptance limits4	LCS/LCSD[4]	A	%R 70-130 RPD ≤30
		Accuracy/Bias Precision	Laboratory acceptance limits4	MS/MSD	S&A	%R 70-130 RPD<50
		Precision	RT within ±30 seconds from RT of initial calibration midpoint standard; area counts within -50% to +100% of initial calibration midpoint standard	Internal standard	A	Area response 50-200% of CC +/- 0.06 RRT

QAPP Worksheet #12-2 Measurement Performance Criteria (Semivolatile Organic Compounds)
GEL

Matrix	Soil, Sediment
Analytical Group	SVOCs
Concentration Level	All

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Laboratory Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)	Data Validation Criteria
		Precision	RPD < 50%	Field duplicate	S&A	RPD < 50% for concentrations >RL
		Sensitivity	RL < 1/3 PAL	RL	A	RL < 1/3 PAL, based on low standard in IC

Notes:
[1] See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]Reference number from QAPP Worksheet #23.
[3] LCS/LCSD must be performed if site specific MS/MSD is not performed.
[4]-laboratory acceptance limits are based on Department of Defense (DoD) Quality Systems Manual (QSM)
PAL-Project Action Limit

QAPP Worksheet #12-2 Measurement Performance Criteria (Anions – bromide, chloride, fluoride, sulfate)

Matrix	Aqueous
Analytical Group	Anions
Concentration Level	All

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Laboratory Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)	Data Validation Criteria
NMI-GW-001 - NMI-GW-020	EPA 300.0 / 2214	Accuracy/Bias Contamination	<RL	Blanks (calibration, prep., field, equipment)	S&A	Sample concentration ≤ blank concentration will be qualified not detected (U)
		Accuracy/Bias/ Precision	%R 90-110	LCS	A	%R 90-110
		Accuracy/Bias/ Precision	%R 90-100	MS	S&A	%R 90-110
		Precision	RPD ≤20%	Laboratory duplicate	A	RPD ≤20%
		Accuracy/Precision	RPD ≤30%	Field Duplicate	S&A	RPD ≤30% for concentrations ≥RL

Notes:
[1]See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]Reference number from QAPP Worksheet #23.

QAPP Worksheet #12-3 Measurement Performance Criteria (Pesticides)
GEL

Matrix	Soil, Sediment
Analytical Group	Pesticides
Concentration Level	All

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Laboratory Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)	Data Validation Criteria
NMI-S-001 - NMI-S-007	SW846 3541 / GL-OA-E-066 and 8081B / GL-OA-E-041	Accuracy/Bias Contamination	< RL	Blanks (method)	S&A	Sample concentrations ≤ blank concentration will be qualified not detected (U)
		Accuracy/Bias	Laboratory acceptance limits[5]	Surrogate	A	%R 70-130, %R<10 non-detects will be rejected (R)
		Accuracy/Bias/Precision	Laboratory acceptance limits[5]	LCS/LCSD[4]	A	%R 70-130 RPD ≤20%
		Accuracy/Bias Precision	Laboratory acceptance limits[5]	MS/MSD[3]	S&A	%R 70-130 RPD≤30
		Accuracy	RPD≤40	Dual column agreement	A	RPD≤25
		Precision	-50 to +100% of CC	Internal Standard	A	-50 to +100% of CC
		Precision	RPD ≤ 50%	Field duplicate	S&A	RPD ≤ 50 for concentrations >RL

Notes:
[1] See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.[2]Reference number from QAPP Worksheet #23.
[2]-Reference number from QAPP Worksheet #23.
[3] -Extra sample volume required for site specific MS/MSD

[4]- LCS/LCSD required if site specific MS/MSD not performed.
[5]-laboratory acceptance limits are based on Department of Defense (DoD) Quality Systems Manual (QSM)

QAPP Worksheet #12-3 Measurement Performance Criteria (1,4-Dioxane)

Matrix	Aqueous
Analytical Group	SVOC (1,4-Dioxane)
Concentration Level	All

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Laboratory Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)	Data Validation Criteria
NMI-GW-001 - NMI-GW-020	SW846 3535A / L-OA-E-070 / SW824 8270E SIM / GL-OA-E-009	Accuracy/Bias Contamination	No target analytes detected greater than one-half RL and 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater). No laboratory common contaminants detected greater than RL.	Blanks (field, equipment, method)	S&A	Sample concentrations ≤ blank concentration will be qualified as not detected (U)
		Accuracy/Bias	Laboratory acceptance limits[5] ($1,4$-dioxane-d_8)	Surrogate	A	%R<10, non-detects will be rejected (R)
		Accuracy/Bias	%R 70-130%, RPD≤30	Laboratory Control Sample (LCS) / LCS Duplicate (LCSD)[4]	A	%R 70-130 RPD ≤20

QAPP Worksheet #12-3 Measurement Performance Criteria (1,4-Dioxane)

Matrix	Aqueous
Analytical Group	SVOC (1,4-Dioxane)
Concentration Level	All

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Laboratory Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)	Data Validation Criteria
		Precision	RT within ±30 seconds from RT of initial calibration midpoint standard; area counts within +/- 50% from the average are measured during initial calibration and within +/- 30% from the most recent CCV.	Internal standard	A	RT within ±30 seconds from RT of initial calibration midpoint standard; area counts within +/- 50% from the average are measured during initial calibration and within +/- 30% from the most recent CCV.
		Accuracy/Bias Precision	%R 70-130 RPD≤30	Matrix Spike (MS) / MS Duplicate (MSD) [3]	S&A	%R 70-130 RPD≤30
		Precision	RPD ≤30%	Field duplicate	S&A	RPD ≤ 30% For concentrations ≥RL.
		Sensitivity	RL < 1/3 PAL	RL	A	RL < 1/3 PAL, based on low standard in IC

Notes:

[1] See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP..
[2]Reference number from QAPP Worksheet #23.
[3] Extra sample volume required for site specific MS/MSD
[4] LCS/LCSD must be performed if site specific MS/MSD is not performed.
[5]-laboratory acceptance limit based on Department of Defense (DoD) Quality Systems Manual (QSM)

QAPP Worksheet #12-3 Measurement Performance Criteria (Perchlorate)

Matrix	Aqueous
Analytical Group	Perchlorate
Concentration Level	All

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Laboratory Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)	Data Validation Criteria
NMI-GW-001 - NMI-GW-020	EPA 332.0 / 2251	Accuracy/Bias Contamination	<RL	Blanks (calibration, prep., field, equipment)	A	Sample concentrations ≤ blank concentration will be qualified not detected (U)
		Precision	RPD≤30	Field Duplicates	S&A	RPD ≤ 30% Results ≥ RL
		Precision	RPD ≤20%	Laboratory duplicate	A	RPD ≤20%
		Accuracy/Bias/Precision	%R 80-120 RPD≤20	LCS/LCSD[4]	A	%R 80-120 RPD≤20
		Accuracy/Bias/Precision	%R 80 - 120 (50-150% if fortified at the RL) RPD≤30	MS/MSD[3]	S&A	%R 80 – 120 (50-150% if fortified at the RL) RPD≤30
		Precision	±30% of daily CCV	Internal Standard Area	A	±30% of daily CCV

Notes:
[1] See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]Reference number from QAPP Worksheet #23.
[3] Extra sample volume required for site specific MS/MSD
[4] LCS/LCSD required if site specific MS/MSD not performed.

QAPP Worksheet #12-4 Measurement Performance Criteria (PCBs as Aroclors)
GEL

Matrix	Soil, Sediment
Analytical Group	PCBs
Concentration Level	All

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Laboratory Measurement Performance Criteria[3]	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)	Data Validation Criteria
NMI-S-001 - NMI-S-007	SW846 3541A/ GL-OA-E-066 and 8082A GL-OA-E-040	Accuracy/Bias Contamination	No target analytes detected greater than one-half RL and 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater).	Blanks (method)	S&A	Sample concentrations ≤ blank concentration will be qualified not detected (U)
		Accuracy/Bias	Laboratory acceptance limits[4]	Surrogate	A	%R 70-130,%R<10 non-detects will be rejected (R)
		Accuracy/Bias/Precision	Laboratory acceptance limits[4]	LCS/LCSD[3]	A	%R 70-130 RPD ≤20%
		Accuracy/Bias Precision	Laboratory acceptance limits[4]	MS/MSD	S&A	%R 70-130 RPD≤30
		Accuracy	RPD≤40	Dual column agreement	A	RPD≤25

QAPP Worksheet #12-4 Measurement Performance Criteria (PCBs as Aroclors)
GEL

Matrix	Soil, Sediment
Analytical Group	PCBs
Concentration Level	All

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Laboratory Measurement Performance Criteria[3]	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)	Data Validation Criteria
		Precision	RPD $\leq$ 50%	Field duplicate	S&A	RPD $\leq$ 50 for concentrations $\geq$RL
		Sensitivity	RL < 1/3 PAL	RL	A	RL < 1/3 PAL, based on low standard in IC

Notes:
[1] See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.[2]Reference number from QAPP Worksheet #23.
[2]-Reference number from QAPP Worksheet #23.
[3]LCS/LCSD required if site specific MS/MSD not performed.
PAL-Project Action Limit
[4]-laboratory acceptance limits are based on Department of Defense (DoD) Quality Systems Manual (QSM)

QAPP Worksheet #12-5 Measurement Performance Criteria (Metals [ICPMS])
GEL

Matrix	Soil, Sediment
Analytical Group	Metals (ICPMS)
Concentration Level	All

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Laboratory Measurement Performance Criteria[3]	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)	Data Validation Criteria
NMI-S-001 - NMI-S-007	SW846 3050B/GL-MA-E-009 and SW846 6020A/ GL-MA-014	Accuracy/Bias Contamination	<RL	Blanks (calibration, prep.)	S&A	Sample concentrations ≤ blank concentration will be qualified as not detected (U)
		Precision	RPD <20%	Laboratory duplicate	A	RPD ≤20%
		Accuracy/Bias/Precision	%R 80-120 RPD≤20	LCS/LCSD[3]	A	%R 80-120 RPD<20
		Accuracy/Bias/Precision	%R 80-120, RPD<20	MS/MSD[4]	S&A	%R 80-120, RPD≤20
		Precision	%D ≤ 10% for concentrations >50X MDL	Serial dilution	A	%D ≤ 10% for concentrations ≥50X MDL

QAPP Worksheet #12-5 Measurement Performance Criteria (Metals [ICPMS])
GEL

Matrix	Soil, Sediment
Analytical Group	Metals (ICPMS)
Concentration Level	All

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Laboratory Measurement Performance Criteria[3]	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)	Data Validation Criteria
		Accuracy/Bias	%R 75-125	Post Spike	A	%R 75-125
		Precision	RPD<20 for concentrations ≥5X PQL	Field Duplicate	S&A	RPD<50 for concentrations ≥RL
		Sensitivity	PQL < 1/3 PAL	RL	A	PQL < 1/3 PAL, based on low standard in IC

Notes:
[1] See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]Reference number from QAPP Worksheet #23.
[3] LCS/LCSD must be performed if site specific MS/MSD is not performed.
[4-]MS and sample duplicate may be performed in lieu of MS/MSD.
PAL-Project Action Limit

QAPP Worksheet #12-6 Measurement Performance Criteria (Mercury)
GEL

Matrix	Aqueous, Soil, Sediment
Analytical Group	Mercury
Concentration Level	All

Sampling Procedure[1]	**Analytical Method/SOP[2]**	**Data Quality Indicators (DQIs)**	**Laboratory Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)**	**Data Validation Criteria**
NMI-GW-001 - NMI-GW-020 or NMI-S-001 - NMI-S-007	SW846 7470A, 7471B/ GL-MA-E-010	Accuracy/Bias Contamination	<RL	Blanks* (calibration, prep., field, equipment)	S&A	Sample concentrations ≤ blank concentration will be qualified as not detected (U)
		Precision	RPD ≤20%	Laboratory duplicate	A	RPD ≤ 20 for concentrations >RL
		Accuracy/Bias/ Precision	%R 80-120 RPD≤20	LCS/LCSD[4]	A	%R 80-120 RPD≤20
		Accuracy/Bias/ Precision	%R 80-120, RPD≤20	MS/MSD[5]	S&A	%R 80-120, RPD≤20
		Accuracy/Bias	%R 75-125	Post Spike	A	%R 75-125

QAPP Worksheet #12-6 Measurement Performance Criteria (Mercury)
GEL

Matrix	Aqueous, Soil, Sediment
Analytical Group	Mercury
Concentration Level	All

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Laboratory Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)	Data Validation Criteria
		Precision	RPD<20 for concentrations >5X RL	Field Duplicates	S&A	RPD<30 (aqueous), RPD<50 (soil,sed.) for concentrations ≥RL
		Sensitivity	RL < 1/3 PAL	RL	A	RL < 1/3 PAL, based on low standard in IC

Notes:
*-soil samples not qualified based on aqueous field or equipment blanks)
[1] See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.[2]Reference number from QAPP Worksheet #23.
[3] Extra sample volume required for site specific MS/MSD
[4] LCS/LCSD must be performed if site specific MS/MSD is not performed.
[5]-MS and sample duplicate may be performed in lieu of MS/MSD.
PAL-Project Action Limit

QAPP Worksheet #12-7 Measurement Performance Criteria (Bismuth-210, Actinium-227)
GEL

Matrix	Soil/Sediment
Analytical Group	Gamma Spec (Bismuth-210, Actinium-227)
Concentration Level	All

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Laboratory Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)	Data Validation Criteria
NMI-S-001 - NMI-S-007	EPA 901/1/GL-RAD-A-013	Accuracy/Bias Contamination	<MDC	Blanks (prep.)	S&A	Sample concentrations ≤ blank will be qualified as not detected (U)
		Precision	RPD ≤20% for activity >5X MDC	Laboratory duplicate	A	RPD ≤20% for activity >5X MDC
		Accuracy/Bias	%R 75-125	LCS	A	%R 75-125
		Precision	RPD<20 for activity >5X MDC	Field Duplicates	S&A	RPD<20 for activity >5X MDC

Notes:
[1] See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]Reference number from QAPP Worksheet #23.

QAPP Worksheet #12-8 Measurement Performance Criteria (Uranium-234)
GEL

Matrix	Soil/Sediment
Analytical Group	Radiochemistry (Uranium-234)
Concentration Level	All

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Laboratory Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)	Data Validation Criteria
NMI-S-001 - NMI-S-007	Modified HASL 300 / GL-RAD-A-015 and GL-RAD-A-011	Accuracy/Bias Contamination	<CRDL	Blanks (prep)	S&A	Sample concentrations ≤ blank concentration will be qualified as not detected (U)
		Precision	RPD ≤20% for activity >5 MDC	Laboratory duplicate	A	RPD ≤20% for activity >5 MDC
		Accuracy/Bias	%R 75-125	LCS	A	%R 75-125
		Accuracy/Bias	%R 15-125	Tracer	A	%R 70-130
		Precision	RPD ≤20% for activity >5 MDC	Field duplicate	S&A	RPD ≤20% for activity >5 MDC

Notes:
[1] See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]Reference number from QAPP Worksheet #23.

QAPP Worksheet #12-9 Measurement Performance Criteria (Lead-210)
GEL

Matrix	Soil/Sediment
Analytical Group	Radiochemistry (Lead-210)
Concentration Level	All

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Laboratory Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)	Data Validation Criteria
NMI-S-001 - NMI-S-007	Modified DOE RP280/GL-RAD-A-018	Accuracy/Bias Contamination	<CRDL	Blanks (prep.)	S&A	Sample concentrations ≤ blank concentration will be qualified as not detected (U)
		Precision	RPD ≤20% for activity >5 MDC	Laboratory duplicate	A	RPD ≤20% for activity >5 MDC
		Accuracy/Bias	%R 75-125	LCS	A	%R 75-125
		Accuracy/Bias	%R 75-125	MS	S&A	%R 75-125
		Accuracy/Bias	%R 15-125	Carrier	A	%R 70-130
		Precision	RPD ≤20% for activity >5 MDC	Field duplicate	S&A	RPD ≤20% for activity >5 MDC

Notes:
[1] See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.

[2]Reference number from QAPP Worksheet #23.

QAPP Worksheet #12-10 Measurement Performance Criteria (Thorium-232)
GEL

Matrix	Soil/Sediment
Analytical Group	Radiochemistry (Th-232)
Concentration Level	All

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Laboratory Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)	Data Validation Criteria
NMI-S-001 - NMI-S-007	Modified HASL 300 / GL-RAD-A-015 and GL-RAD-A-038	Accuracy/Bias Contamination	<CRDL	Blanks (prep.)	S&A	Sample concentrations ≤ blank concentration will be qualified as not detected (U)
		Precision	RPD ≤20% for activity >5 MDC	Laboratory duplicate	A	RPD ≤20% for activity >5 MDC
		Accuracy/Bias	%R 75-125	LCS	A	%R 75-125
		Accuracy/Bias	%R 15-125	Tracer	A	%R 70-130
		Precision	RPD ≤20% for activity >5 MDC	Field duplicate	S&A	RPD ≤20% for activity >5 MDC

Notes:
[1] See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]Reference number from QAPP Worksheet #23.

QAPP Worksheet #12-11 Measurement Performance Criteria (Reactive Cyanide and Reactive Sulfide)
GEL

Matrix	Soil
Analytical Group	RCN, RS
Concentration Level	All

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Laboratory Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)	Data Validation Criteria
NMI-S-001 - NMI-S-007	SW 846 7.3.3 / 7.3.4 / GL-GC-E-069	Accuracy/Bias Contamination	<RL	Blanks (method)	A	Sample concentration ≤ blank concentration will be qualified not detected (U)
		Accuracy	0-52 (RCN) 56.8-159 (RS)	LCS	A	%R 70-130
		Precision	RPD≤30 (RCN) RPD≤86.4	Lab Duplicates	A	RPD≤30 (aqueous) RPD≤50 (soil)
		Precision	RPD ≤ 50% Results ≥ RL	Field Duplicates	S&A	RPD ≤ 50% for concentrations ≥ RL

Notes:
[1] See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]Reference number from QAPP Worksheet #23.

QAPP Worksheet #12-12 Measurement Performance Criteria (Ignitability)
GEL

Matrix	Soil
Analytical Group	Ignitability
Concentration Level	All

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Laboratory Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)	Data Validation Criteria
NMI-S-001 - NMI-S-007	SW846 1020B / GL-GC-E-066	Precision	RPD≤9	Lab Duplicates	A	RPD≤15
		Accuracy	97-103 %R	LCS	A	%R 70-130

Notes:
[1] See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]Reference number from QAPP Worksheet #23.

QAPP Worksheet #12-13 Measurement Performance Criteria (pH)
GEL

Matrix	Soil, Sediment
Analytical Group	pH
Concentration Level	All

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Laboratory Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)	Data Validation Criteria
NMI-GW-001 - NMI-GW-020	SW846 9040C / GL-GC-E-008	Precision	RPD≤10	Sample Duplicates	A	±0.2 pH units
		Accuracy	%R 95-105	LCS	A	%R 95-105

Notes:
[1] See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]Reference number from QAPP Worksheet #23.

QAPP Worksheet #12-14 Measurement Performance Criteria (Volatile Organic Compounds)
Alpha Analytical

Matrix	Soil, Sediment
Analytical Group	VOCs
Concentration Level	All

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Laboratory Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)	Data Validation Criteria
NMI-S-001 - NMI-S-007	SW846 8260C/ 2108	Accuracy/Bias Contamination	< RL	Blanks (method)	S&A	Sample concentrations ≤ blank concentration* will qualified as not detected (U)
		Accuracy/Bias	%R 70-130	Surrogate	A	%R 70-130, %R<10 non-detects will be rejected (R)
		Accuracy/Bias/ Precision	%R 70-130, RPD≤20	LCS/LCSD[4]	A	%R 70-130, RPD≤20
		Accuracy/Bias Precision	%R 70-130, RPD≤30	MS/MSD[3]	S&A	%R 70-130, RPD≤30
		Precision	Area response 50-200% of CC +/- 0.06 RRT	Internal standard	A	Area response 50-200% of CC +/- 0.06 RRT
		Precision	RPD ≤ 30%	Field duplicate	S&A	RPD ≤ 50% for concentrations ≥RL

Notes:
*-Methylene chloride and toluene ≤2X blank concentration
[1] See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]Reference number from QAPP Worksheet #23.
[3] Extra sample volume required for site-specific MS/MSD

[4] LCS/LCSD must be performed if site-specific MS/MSD is not performed.

QAPP Worksheet #12-16 Measurement Performance Criteria (Semivolatile Organic Compounds)
Alpha Analytical

Matrix	Soil, Sediment
Analytical Group	SVOCs
Concentration Level	All

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Laboratory Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)	Data Validation Criteria
NMI-S-001 - NMI-S-007	SW846 3546/ 1959 and SW846 8270D/ 2111	Accuracy/Bias Contamination	< RL	Blanks (method)	S&A	Sample concentrations ≤ blank concentration will be qualified as not detected (U)
		Accuracy/Bias	%R 30-130 for base-neutral surrogates and 15-110% for acid surrogates	Surrogate	A	%R 70-130, %R<10 non-detects will be rejected (R)
		Accuracy/Bias/ Precision	%R 40 - 140 for base-neutral compounds, %R 30 - 130% for acid compounds except for "difficult" analytes which must be between 15-140%, RPD≤30	LCS/LCSD[4]	A	%R 70-130 RPD ≤30

QAPP Worksheet #12-16 Measurement Performance Criteria (Semivolatile Organic Compounds)
Alpha Analytical

Matrix	Soil, Sediment
Analytical Group	SVOCs
Concentration Level	All

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Laboratory Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)	Data Validation Criteria
		Accuracy/Bias Precision	%R 40 - 140 for base-neutral compounds, %R 30 - 130% for acid compounds except for "difficult" analytes which must be between 15-140%, RPD≤30	MS/MSD	S&A	%R 70-130 RPD≤50
		Precision	Area response 50-200% of CC +/- 0.06 RRT	Internal standard	A	Area response 50-200% of CC +/- 0.06 RRT
		Precision	RPD ≤ 50%	Field duplicate	S&A	RPD ≤ 50% for concentrations ≥RL
		Sensitivity	RL < 1/3 PAL	RL	A	RL < 1/3 PAL, based on low standard in IC

Notes:
[1] See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]Reference number from QAPP Worksheet #23.
[3] LCS/LCSD must be performed if site specific MS/MSD is not performed.
PAL-Project Action Limit

QAPP Worksheet #12-21 Measurement Performance Criteria (Pesticides)
Alpha Analytical

Matrix	Soil, Sediment
Analytical Group	Pesticides
Concentration Level	All

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Laboratory Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)	Data Validation Criteria
NMI-S-001 - NMI-S-007	SW846 3546/1959 and SW846 8081B/ 2116	Accuracy/Bias Contamination	< RL	Blanks (method)	S&A	Sample concentrations ≤ blank concentration will be qualified not detected (U)
		Accuracy/Bias	%R 30-150%	Surrogate	A	%R 70-130, %R<10 non-detects will be rejected (R)
		Accuracy/Bias/Precision	%R 40-140 RPD≤20	LCS/LCSD[4]	A	%R 70-130 RPD ≤20%
		Accuracy/Bias Precision	%R 30-150% RPD≤30	MS/MSD[3]	S&A	%R 70-130 RPD≤30
		Accuracy	RPD≤40	Dual column agreement	A	RPD≤25
		Precision	-50 to +100% of CC	Internal Standard	A	-50 to +100% of CC
		Precision	RPD ≤ 50%	Field duplicate	S&A	RPD ≤ 50 for concentrations >RL

Notes:
[1] See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.[2]Reference number from QAPP Worksheet #23.
[2]-Reference number from QAPP Worksheet #23.
[3] -Extra sample volume required for site specific MS/MSD

[4]- LCS/LCSD required if site specific MS/MSD not performed.

QAPP Worksheet #12-22 Measurement Performance Criteria (PCBs as Aroclors)
Alpha Analytical

Matrix	Soil, Sediment
Analytical Group	PCBs
Concentration Level	All

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Laboratory Measurement Performance Criteria[3]	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)	Data Validation Criteria
NMI-S-001 - NMI-S-007	SW846 3540C/1954 and SW846 8082A / 2129	Accuracy/Bias Contamination	< RL	Blanks (method)	S&A	Sample concentrations ≤ blank concentration will be qualified not detected (U)
		Accuracy/Bias	%R 30-150%	Surrogate	A	%R 70-130,%R<10 non-detects will be rejected (R)
		Accuracy/Bias/Precision	%R 40-140 RPD≤20	LCS/LCSD[3]	A	%R 70-130 RPD ≤20%
		Accuracy/Bias Precision	%R 40-140% RPD≤30	MS/MSD	S&A	%R 70-130 RPD≤30
		Accuracy	RPD≤40	Dual column agreement	A	RPD≤25
		Precision	-50 to +100% of CC	Internal Standard	A	-50 to +100% of CC
		Precision	RPD ≤ 50%	Field duplicate	S&A	RPD ≤ 50 for concentrations ≥RL

QAPP Worksheet #12-22 Measurement Performance Criteria (PCBs as Aroclors)
Alpha Analytical

Matrix	Soil, Sediment
Analytical Group	PCBs
Concentration Level	All

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Laboratory Measurement Performance Criteria[3]	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)	Data Validation Criteria
		Sensitivity	RL < 1/3 PAL	RL	A	RL < 1/3 PAL, based on low standard in IC

Notes:
[1] See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.[2]Reference number from QAPP Worksheet #23.
[2]-Reference number from QAPP Worksheet #23.
[3]LCS/LCSD required if site specific MS/MSD not performed.
PAL-Project Action Limit

QAPP Worksheet #12-23 Measurement Performance Criteria (Herbicides)
Alpha Analytical

Matrix	Leachate
Analytical Group	Herbicides
Concentration Level	All

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Laboratory Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)	Data Validation Criteria
NMI-GW-001 - NMI-GW-020	SW846 1311/2133 and/or SW846 3510C/1948 and SW846 8151B/ 2128	Accuracy/Bias Contamination	< RL	Blanks (field, equipment, method)	S&A	Sample concentrations ≤ blank concentration will be qualified not detected (U)
		Accuracy/Bias	%R 30-150%	Surrogate	A	%R 70-130. %R<10 non-detects will be rejected (R)
		Accuracy/Bias/Precision	%R 40-140 RPD≤20	LCS/LCSD	A	%R 70-130 RPD ≤20%
		Accuracy/Bias Precision	%R 30-150% RPD≤30	MS/MSD	S&A	%R 70-130 RPD≤30
		Accuracy	RPD≤40	Dual column agreement	A	RPD≤25
		Precision	-50 to +100% of CC	Internal Standard	A	-50 to +100% of CC
		Precision	RPD ≤ 30%	Field duplicate	S&A	RPD ≤ 30 for concentrations ≥RL

Notes:
[1] See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.[2]Reference number from QAPP Worksheet #23.
[2]-Reference number from QAPP Worksheet #23.

QAPP Worksheet #12-24 Measurement Performance Criteria (pH, Corrosivity)
Alpha Analytical

Matrix	Soil
Analytical Group	pH, Corrosivity
Concentration Level	All

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Laboratory Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)	Data Validation Criteria
NMI-S-001 - NMI-S-007	SW846 9045D / 2203	Precision	RPD≤5	Sample Duplicates	A	±0.5 pH unit

Notes:
[1] See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]Reference number from QAPP Worksheet #23.

QAPP Worksheet #12-25 Measurement Performance Criteria (Reactive Cyanide and Reactive Sulfide)
Alpha Analytical

Matrix	Soil
Analytical Group	RCN, RS
Concentration Level	All

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Laboratory Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)	Data Validation Criteria
NMI-S-001 - NMI-S-007	Laboratory Developed Method / 2228	Accuracy/Bias Contamination	<RL	Blanks (method)	A	Sample concentration ≤ blank concentration will be qualified not detected (U)
		Accuracy	%R 60-125 (RS) %R 30-125 (RCN)	LCS	A	%R 70-130
		Precision	RPD≤25 (aqueous) RPD≤40 (soil)	Lab Duplicates	A	RPD≤30 (aqueous) RPD≤50 (soil)
		Precision	RPD ≤ 50% Results ≥ RL	Field Duplicates	S&A	RPD ≤ 50% for concentrations ≥ RL

Notes:
[1] See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]Reference number from QAPP Worksheet #23.

QAPP Worksheet #12-12 Measurement Performance Criteria (Ignitability)
Alpha Analytical

Matrix	Soil
Analytical Group	Ignitability
Concentration Level	All

Sampling Procedure[1]	Analytical Method/SOP[2]	Data Quality Indicators (DQIs)	Laboratory Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)	Data Validation Criteria
NMI-S-001 - NMI-S-007	SW846 1030/ 2238	Precision	RPD≤15	Lab Duplicates[3]	A	RPD≤15

Notes:
[1] See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]Reference number from QAPP Worksheet #23.
[3]If any sample has a positive result; the sample must be analyzed in triplicate. All replicate tests must agree within 15%.

QAPP Worksheet #13 Secondary Data Criteria and Limitations

Secondary Data	Data Source (Originating Organization, Report Title and Date)	Data Generator(s) (Originating Organization, Data Types, Data Generation/Collection Dates)	How Data Will Be Used?	Limitations on Data Use
• Site geologic and hydrogeologic data • Groundwater quality data • Hydraulic gradients • Soil quality data • Wetland delineation • Human health and ecological risk assessments Site geologic and hydrogeologic data • Groundwater quality data • VOC, nitrate, DU and U regulatory plumes (above drinking water standards) • Hydraulic gradients • Soil quality data	RI Report	***de maximis, inc***.	Site characterization	None
• Supplemental soil data • Supplemental sediment data • Supplemental Groundwater data • Surface Complexation Model	FS Report	***de maximis, inc.***	Site characterization	None
• Groundwater containment and treatment system components	NTCRA 100% Ground-Water Containment and Treatment System Design Report	***de maximis, inc.***	Site characterization	None

QAPP Worksheet #13 Secondary Data Criteria and Limitations

Secondary Data	Data Source (Originating Organization, Report Title and Date)	Data Generator(s) (Originating Organization, Data Types, Data Generation/Collection Dates)	How Data Will Be Used?	Limitations on Data Use
• Groundwater containment and treatment system operating and monitoring data	Ongoing periodic Operation and Maintenance Reports prepared by O&M, Inc.	***de maximis, inc.***	Site characterization	None

QAPP Worksheet #14 Summary of Project Tasks

Sampling Tasks

- Groundwater monitoring; comprehensive events
- Groundwater sampling; as described in FSP, PDI and TSWPs
- Surface Water sampling; as described in FSP, PDI and TSWPs
- Soil Sampling; as described in FSP, PDI and TSWPs
- Sediment Sampling; as described in FSP, PDI and TSWPs

Analysis Tasks

- Groundwater, surface water, sediment, and soil samples will be processed, prepared and analyzed by:
    i. Alpha Analytical Westborough (AAW)
    ii. Alpha Analytical Mansfield (AAM)
    iii. GEL Laboratories (GEL)

Quality Control Tasks

The samples will be collected, processed and waste disposed of as documented in field SOPs provided in the Field Sampling Plan. The QC samples are described in Worksheet #20.

Secondary Data

See Worksheet #13.

Data Management Tasks

QAPP Worksheet #14 Summary of Project Tasks

The RDWP describes data management tasks and procedures.

Documentation and Records

Field sample identification — described in the Field Sampling Plan (FSP).

- **Field documentation** — Field personnel will provide comprehensive documentation covering various aspects of field sampling, field analysis and sample COC. This documentation consists of a record that allows reconstruction of field events and sampling handling to aid in the data review and interpretation process. Documents, records and information relating to the performance of the field work will be retained in the project file.

- **Laboratory project files** — The laboratory will establish a file for pertinent data. The file will include correspondence, faxed information, phone logs and COC forms. The laboratory will retain project files and data packages for a period not less than 5 years. de maximis and/or its designated representative will retain copies of the analytical data reports according to the requirements of the AOC.

- **Laboratory logbooks** — Workbooks, bench sheets, instrument logbooks and instrument printouts will be used to trace the history of samples through the analytical process and to document important aspects of the work, including the associated quality controls. As such, logbooks, bench sheets, instrument logs, and instrument printouts will be part of the permanent record of the laboratory. Each page or entry will be dated and initialed by the analyst at the time of entry. Errors in entry will be crossed out in indelible ink with one stroke, corrected without the use of white-out or by obliterating or writing directly over the erroneous entry, and initialed and dated by the individual making the correction. Pages of logbooks that are not used will be completed by lining out unused portions. Information regarding the sample, analytical procedures performed, and results of the testing will be recorded on laboratory forms or personal notebook pages by the analyst. These notes will be dated and will also identify the analyst, instrument used and instrument conditions. Laboratory notebooks will be periodically reviewed by the laboratory group leaders for accuracy, completeness and compliance with this QAPP. All entries and calculations will be verified by the laboratory group leader. If all entries on the pages are correct, the laboratory group leader will initial and date the pages. Corrective action will be taken for incorrect entries before the laboratory group leader signs.

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- **Computer and hard copy storage** — All electronic files and deliverables will be retained by the laboratory for not less than 5 years; hard copy data packages (or electronic copies) will also be retained for not less than 5 years. *de maximis* and/or its designated representative will retain copies of the analytical data.

- **Field data reporting** — Information collected in the field through visual observation, manual measurement and/or field instrumentation will be recorded in field notebooks or data sheets and/or on forms. Such data will be reviewed by the appropriate Field Program Manager for adherence to the Work Plan and for consistency. Concerns identified as a result of this review will be discussed with the field personnel, corrected if possible and (as necessary) incorporated into the data evaluation process. If applicable, field data forms and calculations will be processed and included in appendices to the appropriate reports (when generated). The original field logs documents, and data reductions will be kept in the project file at the de maxims-CT office.

- **Laboratory data reporting** — Data reports for all parameters will include, at a minimum, the following items:

  Narrative: Summary of activities that took place during sample analysis, including the following information:

  - laboratory name and address
  - date of sample receipt
  - cross reference of laboratory identification number to contractor sample identification
  - analytical methods used
  - deviations from specified protocol
  - corrective actions taken

  Included with the narrative will be any sample handling documents, including field and internal chain-of-custody forms, air bills, and shipping tags.

  Analytical Results: These will be reported according to analysis type and include the following information, as applicable:

  - sample identification (ID)
  - laboratory ID

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- date of collection
- date of receipt
- date of extraction
- date of analysis
- detection limits

Sample results on the report forms will be corrected for dilutions. Soil data will be reported on a dry weight basis. Unless otherwise specified, all results will be reported uncorrected for blank contamination.

The data associated with Contract Laboratory Program- (CLP-) equivalent reporting will be expanded to include supporting documentation necessary to provide a CLP-equivalent package. This additional documentation will include, but not be limited to, raw data required to recalculate any result, including instrument printouts and quantitation reports. The report also will include standards used in calibration and calculation of analytical results; sample extraction, digestion, and other preparation logs; standard preparation logs; instrument run logs; and moisture content calculations.

- **Data reporting level is as follows**:
    - **Level 2 — Modified Reporting**: Modified reporting is used for analyses that are performed following standard USEPA-approved methods and QA/QC protocols. Based on the intended data use, modified reporting may require some supporting documentation, but not full CLP or CLP-type reporting.

Assessment/Audit Tasks

Performance and systems audits will be completed in the field and laboratory during the site investigations, as described below and in Worksheets #31 and #32.

1. **Field Audits** — The following field performance and systems audits will be completed during this project.

   The appropriate Field Program Manager will monitor field performance. Field performance audit summaries will contain an evaluation of field activities to verify that the activities are performed according to established protocols. Field performance audits may be performed by the de

QAPP Worksheet #14 Summary of Project Tasks

maximis Coordinator (or his designee). The auditor(s) will review field reports and communicate concerns to the de maximis Project Manager and/or Field Program Managers, as appropriate.

The number and frequency of field performance audits conducted will be determined independently by the Project Coordinator or Field Program Manager. The de maximis Project Coordinator will conduct field performance audits at a frequency of approximately one per month during field activities. The observations made during field performance audits and any recommended changes/deviations to the field procedures will be recorded and documented.

In addition, the Data QA Manager will review the rinse and trip blank data to identify potential deficiencies in field sampling and cleaning procedures. In addition, systems audits comparing scheduled QA/QC activities from this QAPP with actual QA/QC activities completed will be performed. The appropriate Field Program Manager and Data QA Manager will periodically confirm that work is being performed consistent with this QAPP and the Work Plan.

2. Laboratory Audits

Internal laboratory audits are conducted by the Laboratory QA Manager periodically. As part of the audit, the overall performance of the laboratory staff is evaluated and compared to the performance criteria outlined in the laboratory QA manual and SOPs. Results of the audits are summarized and issued to each department supervisor, Laboratory Manager and Laboratory Director. A systems audit of each laboratory is also performed by the Data QA Manager to determine whether the procedures implemented by each laboratory comply with the QA manual and SOPs.

As a participant in state and federal certification programs, the laboratory is audited by representatives of the regulatory agency issuing certification, in addition to the laboratory's internal audits. Audits are usually conducted annually and focus on laboratory conformance to the specific program protocols for which the laboratory is seeking certification. The auditor reviews sample handling and tracking documentation, analytical methodologies, analytical supportive documentation and final reports. The audit findings are formally documented and submitted to the laboratory for corrective action, if necessary.

ddms, Inc. reserves the right to conduct an on-site audit of the laboratory prior to the start of analyses for the project. Additional audits may be performed during the project, as deemed necessary.

3. Corrective Action

Corrective actions are required when field or analytical data are not within the objectives specified in this QAPP. Corrective actions include procedures to promptly investigate, document, evaluate and correct data collection and/or analytical procedures. Field and laboratory corrective action procedures for the actions are described below.

a. Field Procedures

If, during field work, a condition is noted by the field crew that would have an adverse effect on data quality, corrective action will be taken so as not to repeat this condition. Condition identification, cause and corrective action implemented by the Field Team Coordinator or a designee will be documented on a Corrective Action Form and reported to the appropriate de maximis QA Coordinator, QA Manager and Project Coordinator.

Examples of situations that would require corrective actions are provided below:

- protocols as defined by the QAPP and/or Work Plan have not been followed
- equipment is not in proper working order or is not properly calibrated
- QC requirements have not been met
- issues resulting from performance or systems audits have not been resolved

Project personnel will continuously monitor ongoing work performance as part of daily responsibilities.

b. Laboratory Procedures

In the laboratory, when a condition is noted to have an adverse effect on data quality, corrective action will be taken so as not to repeat this condition. Condition identification, cause and corrective action taken will be documented and reported to the appropriate Project Manager, QA Coordinator and QA Manager.

Corrective action may be initiated, at a minimum, under the following conditions:

QAPP Worksheet #14 Summary of Project Tasks

- protocols as defined by this QAPP have not been followed
- predetermined data acceptance standards are not obtained
- equipment is not in proper working order or calibrated
- sample and test results are not completely traceable
- QC requirements have not been met
- issues resulting from performance or systems audits have not been resolved

Laboratory personnel will continuously monitor ongoing work performance as part of daily responsibilities. Corrective action is initiated at the point where the problem has been identified. At whatever level this occurs (analyst, supervisor, data review, or quality control), it is brought to the attention of the Laboratory QA Manager and, ultimately, the Laboratory Director. Final approval of any action deemed necessary is subject to the approval of the Laboratory Director.

Any corrective action deemed necessary based on system or performance audits, the analytical results of split samples, or the results of data review will be implemented. The corrective action may include sample re-extraction, re-preparation, re-analysis, cleanup, dilution, matrix modification, or other activities. The laboratory is to inform the QA Manager with in one business day as directed on Worksheet #6.

Data Review Tasks

See Worksheets #36 and #37.

Groundwater Cleanup Levels								
Method 8270D								
		Overburden Cleanup Level		Bedrock Cleanup Level		Laboratory		
Analyte	**CAS #**	**ug/L**	**Basis**	**ug/L**	**Basis**	**RL[1]**	**MDL**	**Units**
Acenaphthene	83-32-9	-	-	-	-	2.002	0.44408	ug/l
1,2,4-Trichlorobenzene	120-82-1	-	-	-	-	5.0232	0.49868	ug/l
Hexachlorobenzene	118-74-1	-	-	-	-	2.002	0.46592	ug/l
Bis(2-chloroethyl)ether	111-44-4	-	-	-	-	2.002	0.50596	ug/l
2-Chloronaphthalene	91-58-7	-	-	-	-	2.002	0.4368	ug/l
1,2-Dichlorobenzene	95-50-1	-	-	-	-	2.002	0.455	ug/l
1,3-Dichlorobenzene	541-73-1	-	-	-	-	2.002	0.4004	ug/l
1,4-Dichlorobenzene	106-46-7	-	-	-	-	2.002	0.43316	ug/l
3,3'-Dichlorobenzidine	91-94-1	-	-	-	-	5.0232	1.62344	ug/l
2,4-Dinitrotoluene	121-14-2	-	-	-	-	5.0232	1.1648	ug/l
2,6-Dinitrotoluene	606-20-2	-	-	-	-	5.0232	0.93184	ug/l
Azobenzene	103-33-3	-	-	-	-	2.002	0.36764	ug/l
Fluoranthene	206-44-0	-	-	-	-	2.002	0.27846	ug/l
4-Bromophenyl phenyl ether	101-55-3	-	-	-	-	2.002	0.37856	ug/l
Bis(2-chloroisopropyl)ether	108-60-1	-	-	-	-	2.002	0.5278	ug/l
Bis(2-chloroethoxy)methane	111-91-1	-	-	-	-	5.0232	0.50232	ug/l
Hexachlorobutadiene	87-68-3	-	-	-	-	2.002	0.97188	ug/l
Hexachloroethane	67-72-1	-	-	-	-	2.002	0.78624	ug/l
Isophorone	78-59-1	-	-	-	-	5.0232	1.20484	ug/l
Naphthalene	91-20-3	-	-	-	-	2.002	0.46592	ug/l
Nitrobenzene	98-95-3	-	-	-	-	2.002	0.77168	ug/l
Bis(2-Ethylhexyl)phthalate	117-81-7	6	MCL	6	MCL	3.003	1.6198	ug/l
Butyl benzyl phthalate	85-68-7	-	-	-	-	5.0232	1.17208	ug/l
Di-n-butylphthalate	84-74-2	-	-	-	-	5.0232	0.38948	ug/l
Di-n-octylphthalate	117-84-0	-	-	-	-	5.0232	1.274	ug/l

Diethyl phthalate	84-66-2	-	-	-	-	5.0232	0.3822	ug/l
Dimethyl phthalate	131-11-3	-	-	-	-	5.0232	1.82	ug/l
Benzo(a)anthracene	56-55-3	-	-	-	-	2.002	0.32578	ug/l
Benzo(a)pyrene	50-32-8	-	-	-	-	2.002	0.40768	ug/l
Benzo(b)fluoranthene	205-99-2	-	-	-	-	2.002	0.355264	ug/l
Benzo(k)fluoranthene	207-08-9	-	-	-	-	2.002	0.37492	ug/l
Chrysene	218-01-9	-	-	-	-	2.002	0.341068	ug/l
Acenaphthylene	208-96-8	-	-	-	-	2.002	0.46592	ug/l
Anthracene	120-12-7	-	-	-	-	2.002	0.32942	ug/l
Benzo(ghi)perylene	191-24-2	-	-	-	-	2.002	0.296296	ug/l
Fluorene	86-73-7	-	-	-	-	2.002	0.41496	ug/l
Phenanthrene	85-01-8	-	-	-	-	2.002	0.33124	ug/l
Dibenzo(a,h)anthracene	53-70-3	-	-	-	-	2.002	0.323232	ug/l
Indeno(1,2,3-cd)Pyrene	193-39-5	-	-	-	-	2.002	0.39676	ug/l
Pyrene	129-00-0	-	-	-	-	2.002	0.279552	ug/l
Aniline	62-53-3	-	-	-	-	2.002	0.67704	ug/l
4-Chloroaniline	106-47-8	-	-	-	-	5.0232	1.32132	ug/l
Dibenzofuran	132-64-9	-	-	-	-	2.002	0.49868	ug/l
2-Methylnaphthalene	91-57-6	-	-	-	-	2.002	0.455	ug/l
Acetophenone	98-86-2	-	-	-	-	5.0232	0.5278	ug/l
2,4,6-Trichlorophenol	88-06-2	-	-	-	-	5.0232	0.73528	ug/l
2-Chlorophenol	95-57-8	-	-	-	-	2.002	0.48048	ug/l
2,4-Dichlorophenol	120-83-2	-	-	-	-	5.0232	0.41132	ug/l
2,4-Dimethylphenol	105-67-9	-	-	-	-	5.0232	2.96296	ug/l
2-Nitrophenol	88-75-5	-	-	-	-	10.01	0.90636	ug/l
4-Nitrophenol	100-02-7	-	-	-	-	10.01	0.6734	ug/l
2,4-Dinitrophenol	51-28-5	-	-	-	-	20.02	6.6612	ug/l
Pentachlorophenol	87-86-5	-	-	-	-	10.01	1.79452	ug/l
Phenol	108-95-2	-	-	-	-	5.0232	0.56784	ug/l
2-Methylphenol	95-48-7	-	-	-	-	5.0232	0.64792	ug/l

3-/4-Methylphenol	108-39-4/106-44-5	-	-	-	-	5.0232	0.72436	ug/l

Groundwater Cleanup Levels								
Method 8270D SIM Isotope Dilution								
		Overburden Cleanup Level		**Bedrock Cleanup Level**		**Laboratory**		
Analyte	**CAS #**	**ug/L**	**Basis**	**ug/L**	**Basis**	**RL[1]**	**MDL**	**Units**
1,4-Dioxane	123-91-1	0.46	ILCR=10^{-6} Residential	0.46	ILCR=10^{-6} Residential	0.15	0.075	ug/l

MCL-Maximum contaminant level

ILCR-Incremental Lifetime Cancer Risk

Groundwater Cleanup Levels								
Method 8260C								
		Overburden Cleanup Level		Bedrock Cleanup Level		Laboratory		
Analyte	**CAS #**	**ug/L**	**Basis**	**ug/L**	**Basis**	**RL[1]**	**MDL**	**Units**
Methylene chloride	75-09-2	-	-	-	-	2	0.289	ug/l
1,1-Dichloroethane	75-34-3	NA	NA	2.7	ILCR=10^{-6} Residential	1	0.21	ug/l
Chloroform	67-66-3	-	-	-	-	1	0.162	ug/l
Carbon tetrachloride	56-23-5	-	-	-	-	1	0.134	ug/l
1,2-Dichloropropane	78-87-5	-	-	-	-	1	0.133	ug/l
Dibromochloromethane	124-48-1	-	-	-	-	1	0.149	ug/l
1,1,2-Trichloroethane	79-00-5	-	-	-	-	1	0.144	ug/l
Tetrachloroethene	127-18-4	5	MCL	5	MCL	1	0.181	ug/l
Chlorobenzene	108-90-7	-	-	-	-	1	0.178	ug/l
Trichlorofluoromethane	75-69-4	-	-	-	-	2	0.161	ug/l
1,2-Dichloroethane	107-06-2	-	-	-	-	1	0.132	ug/l
1,1,1-Trichloroethane	71-55-6	-	-	-	-	1	0.158	ug/l
Bromodichloromethane	75-27-4	-	-	-	-	1	0.192	ug/l
trans-1,3-Dichloropropene	10061-02-6	-	-	-	-	0.4	0.164	ug/l
cis-1,3-Dichloropropene	10061-01-5	-	-	-	-	0.4	0.144	ug/l
1,3-Dichloropropene, Total	542-75-6	-	-	-	-	0.4	0.144	ug/l
1,1-Dichloropropene	563-58-6	-	-	-	-	2	0.173	ug/l
Bromoform	75-25-2	-	-	-	-	2	0.248	ug/l
1,1,2,2-Tetrachloroethane	79-34-5	-	-	-	-	1	0.144	ug/l
Benzene	71-43-2	-	-	-	-	0.5	0.159	ug/l
Toluene	108-88-3	-	-	-	-	1	0.161	ug/l
Ethylbenzene	100-41-4	-	-	-	-	1	0.168	ug/l
Chloromethane	74-87-3	-	-	-	-	2	0.176	ug/l
Bromomethane	74-83-9	-	-	-	-	2	0.256	ug/l
Vinyl chloride	75-01-4	2	MCL	2	MCL	1	0.0699	ug/l

Chloroethane	75-00-3	-	-	-	-	2	0.134	ug/l
1,1-Dichloroethene	75-35-4	-	-	-	-	1	0.142	ug/l
trans-1,2-Dichloroethene	156-60-5	-	-	-	-	1	0.163	ug/l
Trichloroethene	79-01-6	5	MCL	5	MCL	1	0.175	ug/l
1,2-Dichlorobenzene	95-50-1	-	-	-	-	1	0.184	ug/l
1,3-Dichlorobenzene	541-73-1	-	-	-	-	1	0.186	ug/l
1,4-Dichlorobenzene	106-46-7	-	-	-	-	1	0.187	ug/l
Methyl tert butyl ether	1634-04-4	-	-	-	-	2	0.16	ug/l
p/m-Xylene	179601-23-1	-	-	-	-	2	0.332	ug/l
o-Xylene	95-47-6	-	-	-	-	1	0.33	ug/l
Xylene (Total)	1330-20-7	-	-	-	-	1	0.33	ug/l
cis-1,2-Dichloroethene	156-59-2	-	-	-	-	1	0.187	ug/l
1,2-Dichloroethene (total)	540-59-0	-	-	-	-	1	0.163	ug/l
Dibromomethane	74-95-3	-	-	-	-	2	0.363	ug/l
1,2,3-Trichloropropane	96-18-4	-	-	-	-	2	0.176	ug/l
Styrene	100-42-5	-	-	-	-	1	0.359	ug/l
Dichlorodifluoromethane	75-71-8	-	-	-	-	2	0.245	ug/l
Acetone	67-64-1	-	-	-	-	5	1.46	ug/l
Carbon disulfide	75-15-0	-	-	-	-	2	0.299	ug/l
2-Butanone	78-93-3	-	-	-	-	5	1.94	ug/l
4-Methyl-2-pentanone	108-10-1	-	-	-	-	5	0.416	ug/l
2-Hexanone	591-78-6	-	-	-	-	5	0.515	ug/l
Bromochloromethane	74-97-5	-	-	-	-	2	0.138	ug/l
Tetrahydrofuran	109-99-9	-	-	-	-	2	0.525	ug/l
2,2-Dichloropropane	594-20-7	-	-	-	-	2	0.204	ug/l
1,2-Dibromoethane	106-93-4	-	-	-	-	2	0.193	ug/l
1,3-Dichloropropane	142-28-9	-	-	-	-	2	0.212	ug/l
1,1,1,2-Tetrachloroethane	630-20-6	-	-	-	-	1	0.164	ug/l
Bromobenzene	108-86-1	-	-	-	-	2	0.152	ug/l

n-Butylbenzene	104-51-8	-	-	-	-	2	0.192	ug/l
sec-Butylbenzene	135-98-8	-	-	-	-	2	0.181	ug/l
tert-Butylbenzene	98-06-6	-	-	-	-	2	0.185	ug/l
o-Chlorotoluene	95-49-8	-	-	-	-	2	0.17	ug/l
p-Chlorotoluene	106-43-4	-	-	-	-	2	0.185	ug/l
1,2-Dibromo-3-chloropropane	96-12-8	-	-	-	-	2	0.327	ug/l
Hexachlorobutadiene	87-68-3	-	-	-	-	0.6	0.217	ug/l
Isopropylbenzene	98-82-8	-	-	-	-	2	0.187	ug/l
p-Isopropyltoluene	99-87-6	-	-	-	-	2	0.188	ug/l
Naphthalene	91-20-3	-	-	-	-	2	0.216	ug/l
n-Propylbenzene	103-65-1	-	-	-	-	2	0.173	ug/l
1,2,3-Trichlorobenzene	87-61-6	-	-	-	-	2	0.234	ug/l
1,2,4-Trichlorobenzene	120-82-1	-	-	-	-	2	0.22	ug/l
1,3,5-Trimethylbenzene	108-67-8	-	-	-	-	2	0.174	ug/l
1,2,4-Trimethylbenzene	95-63-6	-	-	-	-	2	0.191	ug/l
Ethyl ether	60-29-7	-	-	-	-	2	0.15	ug/l
Isopropyl Ether	108-20-3	-	-	-	-	2	0.425	ug/l
Ethyl-Tert-Butyl-Ether	637-92-3	-	-	-	-	2	0.179	ug/l
Tertiary-Amyl Methyl Ether	994-05-8	-	-	-	-	2	0.278	ug/l

MCL-Maximum contaminant level
ILCR-Incremental Lifetime Cancer Risk

Groundwater Cleanup Levels								
Method EPA 537 Modified								
		Overburden Cleanup Level		Bedrock Cleanup Level				
		ug/L	Basis	ug/L	Basis	Laboratory		
Analyte	**CAS #**	-	-	-	-	**RL[1]**	**MDL**	**Units**
Perfluorobutanoic Acid (PFBA)	375-22-4	-	-	-	-	2	0.408	ng/l
Perfluoropentanoic Acid (PFPeA)	2706-90-3	-	-	-	-	2	0.396	ng/l
Perfluorobutanesulfonic Acid (PFBS)	375-73-5	-	-	-	-	2	0.238	ng/l
1H,1H,2H,2H-Perfluorohexanesulfonic Acid (4:2FTS)	757124-72-4	-	-	-	-	2	0.452	ng/l
Perfluorohexanoic Acid (PFHxA)	307-24-4	-	-	-	-	2	0.328	ng/l
Perfluoropentanesulfonic Acid (PFPeS)	2706-91-4	-	-	-	-	2	0.2452	ng/l
Perfluoroheptanoic Acid (PFHpA)	375-85-9	-	-	-	-	2	0.2252	ng/l
Perfluorohexanesulfonic Acid (PFHxS)	355-46-4	-	-	-	-	2	0.376	ng/l
Perfluorooctanoic Acid (PFOA)	335-67-1	-	-	-	-	2	0.236	ng/l
1H,1H,2H,2H-Perfluorooctanesulfonic Acid (6:2FTS)	27619-97-2	-	-	-	-	2	1.332	ng/l
Perfluoroheptanesulfonic Acid (PFHpS)	375-92-8	-	-	-	-	2	0.688	ng/l
Perfluorononanoic Acid (PFNA)	375-95-1	-	-	-	-	2	0.312	ng/l
Perfluorooctanesulfonic Acid (PFOS)	1763-23-1	-	-	-	-	2	0.504	ng/l
Perfluorodecanoic Acid (PFDA)	335-76-2	-	-	-	-	2	0.304	ng/l
1H,1H,2H,2H-Perfluorodecanesulfonic Acid (8:2FTS)	39108-34-4	-	-	-	-	2	1.212	ng/l
Perfluorononanesulfonic Acid (PFNS)	68259-12-1	-	-	-	-	2	1.12	ng/l
N-Methyl Perfluorooctanesulfonamidoacetic Acid (NMeFOSAA)	2355-31-9	-	-	-	-	2	0.648	ng/l
Perfluoroundecanoic Acid (PFUnA)	2058-94-8	-	-	-	-	2	0.26	ng/l
Perfluorodecanesulfonic Acid (PFDS)	335-77-3	-	-	-	-	2	0.98	ng/l
Perfluorooctanesulfonamide (FOSA)	754-91-6	-	-	-	-	2	0.58	ng/l
N-Ethyl Perfluorooctanesulfonamidoacetic Acid (NEtFOSAA)	2991-50-6	-	-	-	-	2	0.804	ng/l
Perfluorododecanoic Acid (PFDoA)	307-55-1	-	-	-	-	2	0.372	ng/l
Perfluorotridecanoic Acid (PFTrDA)	72629-94-8	-	-	-	-	2	0.3272	ng/l
Perfluorotetradecanoic Acid (PFTA)	376-06-7	-	-	-	-	2	0.248	ng/l

Groundwater Cleanup Levels									
			Overburden Cleanup Level		Bedrock Cleanup Level				
			mg/L	Basis	mg/L	Basis	Laboratory		
Analyte	**Method**	**CAS #**					**RL[1]**	**MDL**	**Units**
Nitrogen, Nitrite	SM4500 NO_3 F/NO_2 B	NONE	1	MCL	1	MCL	0.05	0.0143	**mg/L**
Nitrogen, Nitrate	SM4500 NO_3 F/NO_2 B	14797-55-8	10	MCL	10	MCL	0.1	0.0328	**mg/L**
Nitrogen, Nitrate/Nitrite	SM4500 NO_3 F/NO_2 B	NONE	-	-	-	-	0.1	0.0328	**mg/L**
Nitrogen, Ammonia	SM4500-NH_3 BH	7664-41-7	-	-	-	-	0.075	0.023	**mg/L**
Sulfide	SM4500 S^2 D	18496-25-8	-	-	-	-	0.1	0.1	**mg/L**
Phosphorus, Orthophosphate	SM4500P-E	7723-14-0	-	-	-	-	0.005	0.001	**mg/L**
Phosphorus, Total	SM4500P-E	7723-14-0	-	-	-	-	0.01	0.004	**mg/L**
Alkalinity, Total	SM2320B	471-34-1	-	-	-	-	2	-	mg CaCO3/L
Perchlorate	EPA 332.0	14797-73-0	-	-	-	-	0.05	0.013	**mg/L**
Bromide	EPA 300.0	7726-95-6	-	-	-	-	0.05-	0.013	**mg/L**
Sulfate	EPA 300.0	14808-79-8	-	-	-	-	1	0.454	**mg/L**
Chloride	EPA 300.0	16887-00-6	-	-	-	-	0.5	0.0839	**mg/L**
Fluoride	EPA 300.0	16984-48-8	-	-	-	-	0.05	0.037	**mg/L**
Total Suspended Solids	SM2540D	NONE	-	-	-	-	5	-	**mg/L**
Total Dissolved Solids	SM2540C	NONE					10	-	**mg/L**

MCL-Maximum contaminant level

EPA Method TO-15				
Analyte	**CAS #**	**RL[1]**	**MDL**	**Units**
1,1,1-Trichloroethane	71-55-6	0.02	0.01	ppbV
1,1,1,2-Tetrachloroethane	630-20-6	0.02	0.01	ppbV
1,1,2,2-Tetrachloroethane	79-34-5	0.02	0.01	ppbV
1,1,2-Trichloroethane	79-00-5	0.02	0.01	ppbV
1,1-Dichloroethane	75-34-3	0.02	0.01	ppbV
1,1-Dichloroethene	75-35-4	0.02	0.01	ppbV
1,2,4-Trichlorobenzene	120-82-1	0.05	0.01	ppbV
1,2,4-Trimethylbenzene	95-63-6	0.02	0.01	ppbV
1,2-Dibromoethane	106-93-4	0.02	0.01	ppbV
1,2-Dichlorobenzene	95-50-1	0.02	0.01	ppbV
1,2-Dichloroethane	107-06-2	0.02	0.01	ppbV
1,2-Dichloropropane	78-87-5	0.02	0.01	ppbV
1,3,5-Trimethylbenzene	108-67-8	0.02	0.01	ppbV
1,3-Butadiene	106-99-0	0.02	0.01	ppbV
1,3-Dichlorobenzene	541-73-1	0.02	0.01	ppbV
1,4-Dichlorobenzene	106-46-7	0.02	0.01	ppbV
1,4-Dioxane	123-91-1	0.1	0.05	ppbV
Acetone	67-64-1	1	0.5	ppbV
Benzene	71-43-2	0.1	0.05	ppbV
Bromodichloromethane	75-27-4	0.02	0.01	ppbV
Bromoform	75-25-2	0.02	0.01	ppbV
Bromomethane	74-83-9	0.02	0.01	ppbV
Carbon tetrachloride	56-23-5	0.02	0.01	ppbV
Chlorobenzene	108-90-7	0.1	0.01	ppbV
Chloroethane	75-00-3	0.1	0.05	ppbV
Chloroform	67-66-3	0.02	0.01	ppbV
Chloromethane	74-87-3	0.2	0.1	ppbV

cis-1,2-Dichloroethene	156-59-2	0.02	0.01	ppbV
cis-1,3-Dichloropropene	10061-01-5	0.02	0.01	ppbV
Dibromochloromethane	124-48-1	0.02	0.01	ppbV
Dichlorodifluoromethane	75-71-8	0.2	0.1	ppbV
Ethylbenzene	100-41-4	0.02	0.01	ppbV
1,1,2-Trichloro-1,2,2-Trifluoroethane	76-13-1	0.05	0.025	ppbV
1,2-Dichloro-1,1,2,2-tetrafluoroethane	76-14-2	0.05	0.025	ppbV
Hexachlorobutadiene	87-68-3	0.05	0.025	ppbV
2-Butanone	78-93-3	0.5	0.25	ppbV
4-Methyl-2-pentanone	108-10-1	0.5	0.25	ppbV
Methylene chloride	75-09-2	0.5	0.25	ppbV
Methyl tert butyl ether	1634-04-4	0.2	0.01	ppbV
Naphthalene	91-20-3	0.05	0.025	ppbV
p/m-Xylene	179601-23-1	0.04	0.02	ppbV
o-Xylene	95-47-6	0.02	0.01	ppbV
Styrene	100-42-5	0.02	0.01	ppbV
Tetrachloroethene	127-18-4	0.02	0.01	ppbV
Toluene	108-88-3	0.05	0.025	ppbV
trans-1,2-Dichloroethene	156-60-5	0.02	0.01	ppbV
1,2-Dichloroethene (total)	540-59-0	0.02	0.01	ppbV
trans-1,3-Dichloropropene	10061-02-6	0.02	0.01	ppbV
Trichloroethene	79-01-6	0.02	0.01	ppbV
Trichlorofluoromethane	75-69-4	0.05	0.025	ppbV
Vinyl chloride	75-01-4	0.02	0.01	ppbV
Acrylonitrile	107-13-1	0.5	0.25	ppbV
n-Butylbenzene	104-51-8	0.2	0.1	ppbV
sec-Butylbenzene	135-98-8	0.2	0.1	ppbV
Isopropylbenzene	98-82-8	0.2	0.1	ppbV
p-Isopropyltoluene	99-87-6	0.2	0.1	ppbV

1,2,3-Trichlorobenzene	87-61-6	0.05	0.025	ppbV
sec-Butylbenzene	135-98-8	0.02	0.01	ppbV

Soil Cleanup Levels for the Protection of Human Health						
Method 8270D						
		Cleanup Level	**Basis**	**Laboratory**		
Analyte	**CAS #**	**ug/kg**		**RL[1]**	**MDL**	**Units**
Acenaphthene	83-32-9	-	-	133.6	17.3012	ug/kg
Benzidine	92-87-5	-	-	551.1	181.028	ug/kg
1,2,4-Trichlorobenzene	120-82-1	-	-	167	19.1048	ug/kg
Hexachlorobenzene	118-74-1	-	-	100.2	18.704	ug/kg
Bis(2-chloroethyl)ether	111-44-4	-	-	150.3	22.6452	ug/kg
2-Chloronaphthalene	91-58-7	-	-	167	16.5664	ug/kg
1,2-Dichlorobenzene	95-50-1	-	-	167	29.9932	ug/kg
1,3-Dichlorobenzene	541-73-1	-	-	167	28.724	ug/kg
1,4-Dichlorobenzene	106-46-7	-	-	167	29.1582	ug/kg
3,3'-Dichlorobenzidine	91-94-1	-	-	167	44.422	ug/kg
2,4-Dinitrotoluene	121-14-2	-	-	167	33.4	ug/kg
2,6-Dinitrotoluene	606-20-2	-	-	167	28.6572	ug/kg
Azobenzene	103-33-3	-	-	167	16.032	ug/kg
Fluoranthene	206-44-0	-	-	100.2	19.1716	ug/kg
4-Chlorophenyl phenyl ether	7005-72-3	-	-	167	17.869	ug/kg
4-Bromophenyl phenyl ether	101-55-3	-	-	167	25.4842	ug/kg
Bis(2-chloroisopropyl)ether	108-60-1	-	-	200.4	28.5236	ug/kg
Bis(2-chloroethoxy)methane	111-91-1	-	-	180.36	16.7334	ug/kg
Hexachlorobutadiene	87-68-3	-	-	167	24.4488	ug/kg
Hexachlorocyclopentadiene	77-47-4	-	-	477.62	151.302	ug/kg

Hexachloroethane	67-72-1	-	-	133.6	27.0206	ug/kg
Isophorone	78-59-1	-	-	150.3	21.6766	ug/kg
Naphthalene	91-20-3	-	-	167	20.3406	ug/kg
Nitrobenzene	98-95-3	-	-	150.3	24.716	ug/kg
Nitroso-di-phenyl amine(NDPA)/DPA	86-30-6	-	-	133.6	19.0046	ug/kg
n-Nitroso-di-n-propylamine	621-64-7	-	-	167	25.7848	ug/kg
Bis(2-Ethylhexyl)phthalate	117-81-7	-	-	167	57.782	ug/kg
Butyl benzyl phthalate	85-68-7	-	-	167	42.084	ug/kg
Di-n-butylphthalate	84-74-2	-	-	167	31.6632	ug/kg
Di-n-octyl phthalate	117-84-0	-	-	167	56.78	ug/kg
Diethyl phthalate	84-66-2	-	-	167	15.4642	ug/kg
Dimethyl phthalate	131-11-3	-	-	167	35.07	ug/kg
Benzo(a)anthracene	56-55-3	340	ILRC=10^{-6} Residential	100	18.8042	ug/kg
Benzo(a)pyrene	50-32-8	220	ILRC=10^{-6} Residential	134	40.748	ug/kg
Benzo(b)fluoranthene	205-99-2	340	ILRC=10^{-6} Residential	100.2	28.1228	ug/kg
Benzo(k)fluoranthene	207-08-9	-	-	100.2	26.72	ug/kg
Chrysene	218-01-9	-	-	100.2	17.368	ug/kg
Acenaphthylene	208-96-8	-	-	133.6	25.7848	ug/kg
Anthracene	120-12-7	-	-	100.2	32.565	ug/kg
Benzo(ghi)perylene	191-24-2	-	-	133.6	19.6392	ug/kg
Fluorene	86-73-7	-	-	167	16.2324	ug/kg
Phenanthrene	85-01-8	-	-	100.2	20.3072	ug/kg
Dibenzo(a,h)anthracene	53-70-3	-	-	100.2	19.3052	ug/kg
Indeno(1,2,3-cd)Pyrene	193-39-5	340	ILRC=10^{-6} Residential	133.6	23.2798	ug/kg
Pyrene	129-00-0	-	-	100.2	16.5998	ug/kg
Biphenyl	92-52-4	-	-	380.76	38.744	ug/kg
Aniline	62-53-3	-	-	200.4	78.824	ug/kg

4-Chloroaniline	106-47-8	-	-	167	30.394	ug/kg
1-Methylnaphthalene	90-12-0	-	-	167	19.372	ug/kg
2-Nitroaniline	88-74-4	-	-	167	32.1976	ug/kg
3-Nitroaniline	99-09-2	-	-	167	31.4962	ug/kg
4-Nitroaniline	100-01-6	-	-	167	69.138	ug/kg
Dibenzofuran	132-64-9	-	-	167	15.7982	ug/kg
2-Methylnaphthalene	91-57-6	-	-	200.4	20.1736	ug/kg
n-Nitrosodimethylamine	62-75-9	-	-	334	32.064	ug/kg
2,4,6-Trichlorophenol	88-06-2	-	-	100.2	31.6632	ug/kg
P-Chloro-M-Cresol	59-50-7	-	-	167	24.883	ug/kg
2-Chlorophenol	95-57-8	-	-	167	19.7394	ug/kg
2,4-Dichlorophenol	120-83-2	-	-	150.3	26.8536	ug/kg
2,4-Dimethylphenol	105-67-9	-	-	167	55.11	ug/kg
2-Nitrophenol	88-75-5	-	-	360.72	62.792	ug/kg
4-Nitrophenol	100-02-7	-	-	233.8	68.136	ug/kg
2,4-Dinitrophenol	51-28-5	-	-	801.6	77.822	ug/kg
4,6-Dinitro-o-cresol	534-52-1	-	-	434.2	80.16	ug/kg
Pentachlorophenol	87-86-5	-	-	133.6	36.74	ug/kg
Phenol	108-95-2	-	-	167	25.217	ug/kg
2-Methylphenol	95-48-7	-	-	167	25.885	ug/kg
3-Methylphenol/4-Methylphenol	108-39-4/106-44-5	-	-	240.48	26.1522	ug/kg
2,4,5-Trichlorophenol	95-95-4	-	-	167	31.9972	ug/kg
Benzoic Acid	65-85-0	-	-	541.08	169.004	ug/kg
Benzyl Alcohol	100-51-6	-	-	167	51.102	ug/kg
Carbazole	86-74-8	-	-	167	16.2324	ug/kg
Pyridine	110-86-1	-	-	180.36	63.46	ug/kg

ILCR-Incremental Lifetime Cancer Ris

Soil Cleanup Levels for the Protection of Human Health						
Method 8260C						
		Cleanup Level	**Basis**	**Laboratory**		
Analyte	**CAS #**			**RL[1]**	**MDL**	**Units**
Methylene chloride	75-09-2	-	-	5	2.29	ug/kg
1,1-Dichloroethane	75-34-3	-	-	1	0.145	ug/kg
Chloroform	67-66-3	-	-	1.5	0.14	ug/kg
Carbon tetrachloride	56-23-5	-	-	1	0.23	ug/kg
1,2-Dichloropropane	78-87-5	-	-	1	0.125	ug/kg
Dibromochloromethane	124-48-1	-	-	1	0.14	ug/kg
1,1,2-Trichloroethane	79-00-5	-	-	1	0.267	ug/kg
Tetrachloroethene	127-18-4	-	-	0.5	0.196	ug/kg
Chlorobenzene	108-90-7	-	-	0.5	0.127	ug/kg
Trichlorofluoromethane	75-69-4	-	-	4	0.695	ug/kg
1,2-Dichloroethane	107-06-2	-	-	1	0.257	ug/kg
1,1,1-Trichloroethane	71-55-6	-	-	0.5	0.167	ug/kg
Bromodichloromethane	75-27-4	-	-	0.5	0.109	ug/kg
trans-1,3-Dichloropropene	10061-02-6	-	-	1	0.273	ug/kg
cis-1,3-Dichloropropene	10061-01-5	-	-	0.5	0.158	ug/kg
1,3-Dichloropropene, Total	542-75-6	-	-	0.5	0.158	ug/kg
1,1-Dichloropropene	563-58-6	-	-	0.5	0.159	ug/kg
Bromoform	75-25-2	-	-	4	0.246	ug/kg
1,1,2,2-Tetrachloroethane	79-34-5	-	-	0.5	0.166	ug/kg
Benzene	71-43-2	-	-	0.5	0.166	ug/kg
Toluene	108-88-3	-	-	1	0.543	ug/kg

Ethylbenzene	100-41-4	-	-	1	0.141	ug/kg
Chloromethane	74-87-3	-	-	4	0.932	ug/kg
Bromomethane	74-83-9	-	-	2	0.581	ug/kg
Vinyl chloride	75-01-4	-	-	1	0.335	ug/kg
Chloroethane	75-00-3	-	-	2	0.452	ug/kg
1,1-Dichloroethene	75-35-4	-	-	1	0.238	ug/kg
trans-1,2-Dichloroethene	156-60-5	-	-	1.5	0.137	ug/kg
Trichloroethene	79-01-6	-	-	0.5	0.137	ug/kg
1,2-Dichlorobenzene	95-50-1	-	-	2	0.144	ug/kg
1,3-Dichlorobenzene	541-73-1	-	-	2	0.148	ug/kg
1,4-Dichlorobenzene	106-46-7	-	-	2	0.171	ug/kg
Methyl tert butyl ether	1634-04-4	-	-	2	0.201	ug/kg
p/m-Xylene	179601-23-1	-	-	2	0.56	ug/kg
o-Xylene	95-47-6	-	-	1	0.291	ug/kg
Xylene (Total)	1330-20-7	-	-	1	0.291	ug/kg
cis-1,2-Dichloroethene	156-59-2	-	-	1	0.175	ug/kg
1,2-Dichloroethene (total)	540-59-0	-	-	1	0.137	ug/kg
Dibromomethane	74-95-3	-	-	2	0.238	ug/kg
1,4-Dichlorobutane	110-56-5	-	-	10	0.226	ug/kg
1,2,3-Trichloropropane	96-18-4	-	-	2	0.127	ug/kg
Styrene	100-42-5	-	-	1	0.196	ug/kg
Dichlorodifluoromethane	75-71-8	-	-	10	0.915	ug/kg
Acetone	67-64-1	-	-	25	10	ug/kg
Carbon disulfide	75-15-0	-	-	10	4.55	ug/kg
2-Butanone	78-93-3	-	-	10	2.22	ug/kg
Vinyl acetate	108-05-4	-	-	10	2.15	ug/kg
4-Methyl-2-pentanone	108-10-1	-	-	10	1.28	ug/kg
2-Hexanone	591-78-6	-	-	10	1.18	ug/kg

Ethyl methacrylate	97-63-2	-	-	10	1.58	ug/kg
Acrylonitrile	107-13-1	-	-	4	1.15	ug/kg
Bromochloromethane	74-97-5	-	-	2	0.205	ug/kg
Tetrahydrofuran	109-99-9	-	-	4	1.59	ug/kg
2,2-Dichloropropane	594-20-7	-	-	2	0.202	ug/kg
1,2-Dibromoethane	106-93-4	-	-	1	0.279	ug/kg
1,3-Dichloropropane	142-28-9	-	-	2	0.167	ug/kg
1,1,1,2-Tetrachloroethane	630-20-6	-	-	0.5	0.132	ug/kg
Bromobenzene	108-86-1	-	-	2	0.145	ug/kg
n-Butylbenzene	104-51-8	-	-	1	0.167	ug/kg
sec-Butylbenzene	135-98-8	-	-	1	0.146	ug/kg
tert-Butylbenzene	98-06-6	-	-	2	0.118	ug/kg
o-Chlorotoluene	95-49-8	-	-	2	0.191	ug/kg
p-Chlorotoluene	106-43-4	-	-	2	0.108	ug/kg
1,2-Dibromo-3-chloropropane	96-12-8	-	-	3	0.998	ug/kg
Hexachlorobutadiene	87-68-3	-	-	4	0.169	ug/kg
Isopropylbenzene	98-82-8	-	-	1	0.109	ug/kg
p-Isopropyltoluene	99-87-6	-	-	1	0.109	ug/kg
Naphthalene	91-20-3	-	-	4	0.65	ug/kg
n-Propylbenzene	103-65-1	-	-	1	0.171	ug/kg
1,2,3-Trichlorobenzene	87-61-6	-	-	2	0.322	ug/kg
1,2,4-Trichlorobenzene	120-82-1	-	-	2	0.272	ug/kg
1,3,5-Trimethylbenzene	108-67-8	-	-	2	0.193	ug/kg
1,2,4-Trimethylbenzene	95-63-6	-	-	2	0.334	ug/kg
trans-1,4-Dichloro-2-butene	110-57-6	-	-	5	1.42	ug/kg
Ethyl ether	60-29-7	-	-	2	0.341	ug/kg

Soil Cleanup Levels for the Protection of Human Health						
Method 8082A						
		Cleanup Level	Basis	Laboratory		
Analyte	CAS #	ug/kg		RL[1]	MDL	Units
Aroclor 1016	12674-11-2	-	-	33.5	33.5	ug/kg
Aroclor 1221	11104-28-2	-	-	33.5	33.5	ug/kg
Aroclor 1232	11141-16-5	-	-	33.5	33.5	ug/kg
Aroclor 1242	53469-21-9	-	-	33.5	33.5	ug/kg
Aroclor 1248	12672-29-6	-	-	33.5	33.5	ug/kg
Aroclor 1254	11097-69-1	-	-	33.5	33.5	ug/kg
Aroclor 1260	11096-82-5	-	-	33.5	33.5	ug/kg
Aroclor 1262	37324-23-5	-	-	33.5	33.5	ug/kg
Aroclor 1268	11100-14-4	-	-	33.5	33.5	ug/kg
PCBs, Total	1336-36-3	1000	Policy	33.5	33.5	ug/kg

Soil Cleanup Levels for the Protection of Human Health						
Method 8081B						
		Cleanup Level	**Basis**	**Laboratory**		Units
		ug/kg		**RL[1]**	**MDL**	
Delta-BHC	319-86-8	-	-	1.6008	0.31349	ug/kg
Lindane	58-89-9	-	-	0.667	0.298149	ug/kg
Alpha-BHC	319-84-6	-	-	0.667	0.189428	ug/kg
Beta-BHC	319-85-7	-	-	1.6008	0.60697	ug/kg
Heptachlor	76-44-8	-	-	0.8004	0.358846	ug/kg
Aldrin	309-00-2	-	-	1.6008	0.563615	ug/kg
Heptachlor epoxide	1024-57-3	-	-	3.0015	0.90045	ug/kg
Endrin	72-20-8	-	-	0.667	0.27347	ug/kg
Endrin aldehyde	7421-93-4	-	-	2.001	0.70035	ug/kg
Endrin ketone	53494-70-5	-	-	1.6008	0.412206	ug/kg
Dieldrin	60-57-1	-	-	1.0005	0.50025	ug/kg
4,4'-DDE	72-55-9	-	-	1.6008	0.370185	ug/kg
4,4'-DDD	72-54-8	-	-	1.6008	0.570952	ug/kg
4,4'-DDT	50-29-3	-	-	3.0015	1.28731	ug/kg
Endosulfan I	959-98-8	-	-	1.6008	0.378189	ug/kg
Endosulfan II	33213-65-9	-	-	1.6008	0.534934	ug/kg
Endosulfan sulfate	1031-07-8	-	-	0.667	0.317492	ug/kg
Methoxychlor	72-43-5	-	-	3.0015	0.9338	ug/kg
Toxaphene	8001-35-2	-	-	30.015	8.4042	ug/kg
Chlordane	57-74-9	-	-	13.34	5.30265	ug/kg
cis-Chlordane	5103-71-9	-	-	2.001	0.557612	ug/kg
trans-Chlordane	5103-74-2	-	-	2.001	0.528264	ug/kg

Sediment Cleanup Levels for the Protection of Human Health						
Method 8082A						
		Cleanup Level	**Basis**	**Laboratory**		
Analyte	**CAS #**	**ug/kg**		**RL[1]**	**MDL**	**Units**
Aroclor 1016	12674-11-2	-	-	33.5	33.5	ug/kg
Aroclor 1221	11104-28-2	-	-	33.5	33.5	ug/kg
Aroclor 1232	11141-16-5	-	-	33.5	33.5	ug/kg
Aroclor 1242	53469-21-9	-	-	33.5	33.5	ug/kg
Aroclor 1248	12672-29-6	-	-	33.5	33.5	ug/kg
Aroclor 1254	11097-69-1	-	-	33.5	33.5	ug/kg
Aroclor 1260	11096-82-5	-	-	33.5	33.5	ug/kg
Aroclor 1262	37324-23-5	-	-	33.5	33.5	ug/kg
Aroclor 1268	11100-14-4	-	-	33.5	33.5	ug/kg
PCBs, Total	1336-36-3	2,7001	ILCR=10-6 (Abutting Resident/Recreational Visitor)	33.5	33.5	ug/kg
PCBs, Total	1336-36-3	1,0002	Policy	33.5	33.5	ug/kg

ILCR-Incremental Lifetime Cancer Risk

Sediment Cleanup Levels for the Protection of Ecological Receptors						
Method 8082A						
		Cleanup Level	**Basis**	**Laboratory**		
Analyte	**CAS #**	**ug/kg**		**RL[1]**	**MDL**	**Units**
Aroclor 1016	12674-11-2	-	-	33.5	33.5	ug/kg
Aroclor 1221	11104-28-2	-	-	33.5	33.5	ug/kg
Aroclor 1232	11141-16-5	-	-	33.5	33.5	ug/kg
Aroclor 1242	53469-21-9	-	-	33.5	33.5	ug/kg
Aroclor 1248	12672-29-6	-	-	33.5	33.5	ug/kg
Aroclor 1254	11097-69-1	-	-	33.5	33.5	ug/kg
Aroclor 1260	11096-82-5	-	-	33.5	33.5	ug/kg
Aroclor 1262	37324-23-5	-	-	33.5	33.5	ug/kg
Aroclor 1268	11100-14-4	-	-	33.5	33.5	ug/kg
PCBs, Total	1336-36-3	1,080	Site Specific MATC	33.5	33.5	ug/kg

MATC-Maximum Acceptable Toxic Concentration

Analyte	CAS #	RL[1]	MDL	Units
TCLP/VO – Method 1311/8260C				
Chloroform	67-66-3	7.5	1.62	ug/l
Carbon tetrachloride	56-23-5	5	1.34	ug/l
Tetrachloroethene	127-18-4	5	1.81	ug/l
Chlorobenzene	108-90-7	5	1.78	ug/l
1,2-Dichloroethane	107-06-2	5	1.32	ug/l
Benzene	71-43-2	5	1.59	ug/l
Vinyl chloride	75-01-4	10	0.699	ug/l
1,1-Dichloroethene	75-35-4	5	1.42	ug/l
Trichloroethene	79-01-6	5	1.75	ug/l
1,4-Dichlorobenzene	106-46-7	25	1.87	ug/l
2-Butanone	78-93-3	50	19.4	ug/l
TCLP/BNA – Method 1311/8270D				
2,4-Dinitrotoluene	121-14-2	25	1.91	ug/l
Hexachlorobutadiene	87-68-3	10	3	ug/l
Hexachlorocyclopentadiene	77-47-4	100	3.03	ug/l
Hexachloroethane	67-72-1	10	2.2	ug/l
Nitrobenzene	98-95-3	10	3.28	ug/l
2,4,6-Trichlorophenol	88-06-2	25	2.47	ug/l
Pentachlorophenol	87-86-5	50	9.75	ug/l
2-Methylphenol	95-48-7	25	5.5	ug/l
3-Methylphenol/4-Methylphenol	108-39-4/106-44-5	25	2.75	ug/l
2,4,5-Trichlorophenol	95-95-4	25	1.905	ug/l
Pyridine	110-86-1	17.5	4.525	ug/l

TCLP/Pesticides - SW846 1311/8081B				
Lindane	58-89-9	0.1	0.0217	ug/l
Heptachlor	76-44-8	0.1	0.0155	ug/l
Endrin	72-20-8	0.2	0.02145	ug/l
Methoxychlor	72-43-5	1	0.0342	ug/l
Toxaphene	8001-35-2	1	0.3135	ug/l
Chlordane	57-74-9	1	0.2315	ug/l
TCLP/Herbicides - SW846 1311/8151A				
2,4-D	94-75-7	0.025	0.001245	mg/l
2,4,5-TP (Silvex)	93-72-1	0.005	0.0013475	mg/l
TCLP/Metals - SW846 1311/ 6010D				
Arsenic, TCLP	7440-38-2	1	0.019	mg/l
Barium, TCLP	7440-39-3	0.5	0.021	mg/l
Cadmium, TCLP	7440-43-9	0.1	0.01	mg/l
Chromium, TCLP	7440-47-3	0.2	0.021	mg/l
Lead, TCLP	7439-92-1	0.5	0.027	mg/l
Selenium, TCLP	7782-49-2	0.5	0.035	mg/l
Silver, TCLP	7440-22-4	0.1	0.028	mg/l
Arsenic, TCLP	7440-38-2	1	0.019	mg/l
Waste Classification				
Method		**Laboratory**		
		RL[1]	**MDL**	**Units**
Sulfide, Reactive	Lab Developed	10	10	mg/kg
Cyanide, Reactive	Lab Developed	10	10	mg/kg
Ignitability	SW846 1030	na	na	na
pH/Corrosivity	SW846 9045D	na	na	na

Notes:

1-The laboratory reporting limit / quantitation limit cannot be lower than the lowest calibration standard.

Groundwater Cleanup Levels								
Method 8270D								
		Overburden Cleanup Level		Bedrock Cleanup Level		Laboratory		
Analyte	**CAS #**	**ug/L**	**Basis**	**ug/L**	**Basis**	**RL[1]**	**MDL**	**Units**
Bis(2-Ethylhexyl)phthalate	117-81-7	6	MCL	6	MCL	10	3	ug/l
1,1'-Biphenyl	92-52-4	-	-	-	-	10	3	ug/l
1,2,4-Trichlorobenzene	120-82-1	-	-	-	-	10	3	ug/l
1,2-Dichlorobenzene	95-50-1	-	-	-	-	10	3	ug/l
1,2-Diphenylhydrazine	122-66-7	-	-	-	-	10	3	ug/l
1,3-Dichlorobenzene	541-73-1	-	-	-	-	10	3	ug/l
1,4-Dichlorobenzene	106-46-7	-	-	-	-	10	3	ug/l
1,4-Dinitrobenzene	100-25-4	-	-	-	-	10	3	ug/l
1-Methylnaphthalene	90-12-0	-	-	-	-	1	0.3	ug/l
2,4,5-Trichlorophenol	95-95-4	-	-	-	-	10	3	ug/l
2,4,6-Trichlorophenol	88-06-2	-	-	-	-	10	3	ug/l
2,4-Dichlorophenol	120-83-2	-	-	-	-	10	3	ug/l
2,4-Dimethylphenol	105-67-9	-	-	-	-	10	3	ug/l
2,4-Dinitrophenol	51-28-5	-	-	-	-	20	5	ug/l
2,4-Dinitrotoluene	121-14-2	-	-	-	-	10	3	ug/l
2,6-Dinitrotoluene	606-20-2	-	-	-	-	10	3	ug/l
2-Chloronaphthalene	91-58-7	-	-	-	-	1	0.41	ug/l
2-Chlorophenol	95-57-8	-	-	-	-	10	3	ug/l
2-Methyl-4,6-dinitrophenol	534-52-1	-	-	-	-	10	3	ug/l
2-Methylnaphthalene	91-57-6	-	-	-	-	1	0.3	ug/l
2-Nitrophenol	88-75-5	-	-	-	-	10	3	ug/l
3,3'-Dichlorobenzidine	91-94-1					10	3	
4-Bromophenylphenylether	101-55-3	-	-	-	-	10	3	ug/l
4-Chloro-3-methylphenol	59-50-7	-	-	-	-	10	3	ug/l
4-Chloroaniline	106-47-8	-	-	-	-	10	3.3	ug/l
4-Chlorophenylphenylether	7005-72-3	-	-	-	-	10	3	ug/l

4-Nitrophenol	100-02-7	-	-	-	-	10	3	ug/l
Acenaphthene	83-32-9	-	-	-	-	1	0.3	ug/l
Acenaphthylene	208-96-8	-	-	-	-	1	0.3	ug/l
Aniline	62-53-3	-	-	-	-	10	4.2	ug/l
Anthracene	120-12-7	-	-	-	-	1	0.3	ug/l
Benzidine	92-87-5	-	-	-	-	10	3.9	ug/l
Benzo(a)anthracene	56-55-3	-	-	-	-	1	0.3	ug/l
Benzo(a)pyrene	50-32-8	-	-	-	-	1	0.3	ug/l
Benzo(b)fluoranthene	205-99-2	-	-	-	-	1	0.3	ug/l
Benzo(ghi)perylene	191-24-2	-	-	-	-	1	0.3	ug/l
Benzo(k)fluoranthene	207-08-9	-	-	-	-	1	0.3	ug/l
Benzoic acid	65-85-0	-	-	-	-	20	6	ug/l
Benzyl alcohol	100-51-6	-	-	-	-	10	3	ug/l
Butylbenzylphthalate	85-68-7	-	-	-	-	10	3	ug/l
Carbazole	86-74-8	-	-	-	-	1	0.3	ug/l
Chrysene	218-01-9	-	-	-	-	1	0.3	ug/l
Cresols (total)	1319-77-3	-	-	-	-			ug/l
Di-n-butylphthalate	84-74-2	-	-	-	-	10	3	ug/l
Di-n-octylphthalate	117-84-0	-	-	-	-	10	3	ug/l
Dibenzo(a,h)anthracene	53-70-3	-	-	-	-	1	0.3	ug/l
Diethylphthalate	84-66-2	-	-	-	-	10	3	ug/l
Dimethylphthalate	131-11-3	-	-	-	-	10	3	ug/l
Diphenylamine	122-39-4	-	-	-	-	10	3	ug/l
Fluoranthene	206-44-0	-	-	-	-	1	0.3	ug/l
Fluorene	86-73-7	-	-	-	-	1	0.3	ug/l
Hexachlorobenzene	118-74-1	-	-	-	-	10	3	ug/l
Hexachlorobutadiene	87-68-3	-	-	-	-	10	3	ug/l
Hexachlorocyclopentadiene	77-47-4	-	-	-	-	10	3	ug/l
Hexachloroethane	67-72-1	-	-	-	-	10	3	ug/l
Indeno(1,2,3-cd)pyrene	193-39-5	-	-	-	-	1	0.3	ug/l

Isophorone	78-59-1					10	3.5	ug/l
N-Methyl-N-nitrosomethylamine	62-75-9					10	3	ug/l
N-Nitrosodipropylamine	621-64-7					10	3	ug/l
Naphthalene	91-20-3					1	0.3	ug/l
Nitrobenzene	98-95-3					10	3	ug/l
Pentachlorophenol	87-86-5					10	3	ug/l
Phenanthrene	85-01-8					1	0.3	ug/l
Phenol	108-95-2					10	3	ug/l
Pyrene	129-00-0					1	0.3	ug/l
Pyridine	110-86-1					10	3	ug/l
bis(2-Chloro-1-methylethyl)ether	108-60-1					10	3	ug/l
bis(2-Chloroethoxy)methane	111-91-1					10	3	ug/l
bis(2-Chloroethyl) ether	111-44-4					10	3	ug/l
m,p-Cresols	65794-96-9					10	3.7	ug/l
m-Nitroaniline	99-09-2					10	3	ug/l
o-Cresol	95-48-7					10	3	ug/l
o-Nitroaniline	88-74-4					10	3	ug/l
p-Nitroaniline	100-01-6					10	3	ug/l

Groundwater Cleanup Levels								
Method 8270D SIM Isotope Dilution								
		Overburden Cleanup Level		**Bedrock Cleanup Level**		**Laboratory**		
Analyte	**CAS #**	**ug/L**	**Basis**	**ug/L**	**Basis**	**RL[1]**	**MDL**	**Units**
1,4-Dioxane	123-91-1	0.46	ILCR=10^{-6} Residential	0.46	ILCR=10^{-6} Residential	0.2	0.07	ug/l

MCL-Maximum contaminant level
ILCR-Incremental Lifetime Cancer Risk

Groundwater Cleanup Levels								
Method 8260C								
		Overburden Cleanup Level		Bedrock Cleanup Level		Laboratory		
Analyte	**CAS #**	**ug/L**	**Basis**	**ug/L**	**Basis**	**RL[1]**	**MDL**	**Units**
Methylene chloride	75-09-2	-	-	-	-	5	3.334	ug/l
1,1-Dichloroethane	75-34-3	NA	NA	2.7	ILCR=10-6 Residential	1	0.333	ug/l
1,1,1,2-Tetrachloroethane	630-20-6	-	-	-	-	1	0.333	ug/l
1,1,1-Trichloroethane	71-55-6	-	-	-	-	1	0.333	ug/l
1,1,2,2-Tetrachloroethane	79-34-5	-	-	-	-	1	0.333	ug/l
1,1,2-Trichloroethane	79-00-5	-	-	-	-	1	0.333	ug/l
1,1-Dichloroethylene -	75-35-4	-	-	-	-	1	0.333	ug/l
Tetrachloroethene	127-18-4	5	MCL	5	MCL	1	0.333	ug/l
1,1-Dichloropropene	563-58-6	-	-	-	-	1	0.333	ug/l
1,2,3-Trichlorobenzene	87-61-6	-	-	-	-	1	0.333	ug/l
1,2,3-Trichloropropane	96-18-4	-	-	-	-	1	0.333	ug/l
1,2,4-Trichlorobenzene	120-82-1	-	-	-	-	1	0.333	ug/l
1,2,4-Trimethylbenzene	95-63-6	-	-	-	-	1	0.333	ug/l
1,2-Dibromo-3-chloropropane	96-12-8	-	-	-	-	1	0.333	ug/l
1,2-Dibromoethane	106-93-4	-	-	-	-	1	0.333	ug/l
1,2-Dichlorobenzene	95-50-1	-	-	-	-	1	0.333	ug/l
1,2-Dichloroethane - also TCLP	107-06-2	-	-	-	-	1	0.333	ug/l
1,2-Dichloroethylene (total)	540-59-0	-	-	-	-	2	1.334	ug/l
1,2-Dichloropropane	78-87-5	-	-	-	-	1	0.333	ug/l
1,3,5-Trimethylbenzene	108-67-8	-	-	-	-	2	1.334	ug/l
1,3-Dichlorobenzene	541-73-1	-	-	-	-	1	0.333	ug/l
1,3-Dichloropropane	142-28-9	-	-	-	-	1	0.333	ug/l
1,3-Dichloropropylene	542-75-6	-	-	-	-	2	1.334	ug/l
1,4-Dichlorobenzene	106-46-7	-	-	-	-	1	0.333	ug/l
Vinyl chloride	75-01-4	2	MCL	2	MCL	1	0.333	ug/l

2,2-Dichloropropane	594-20-7	-	-	-	-	1	0.333	ug/l
2-Butanone	78-93-3	-	-	-	-	5	3.334	ug/l
2-Chlorotoluene	95-49-8	-	-	-	-	1	0.333	ug/l
Trichloroethene	79-01-6	5	MCL	5	MCL	1	0.333	ug/l
2-Hexanone	591-78-6	-	-	-	-	5	3.334	ug/l
4-Chlorotoluene	106-43-4	-	-	-	-	1	0.333	ug/l
4-Isopropyltoluene	99-87-6	-	-	-	-	1	0.333	ug/l
4-Methyl-2-pentanone	108-10-1	-	-	-	-	5	3.334	ug/l
Acetone	67-64-1	-	-	-	-	5	3.334	ug/l
Acrylonitrile	107-13-1	-	-	-	-	5	3.334	ug/l
Benzene - also TCLP	71-43-2	-	-	-	-	1	0.333	ug/l
Bromobenzene	108-86-1	-	-	-	-	1	0.333	ug/l
Bromochloromethane	74-97-5	-	-	-	-	1	0.333	ug/l
Bromodichloromethane	75-27-4	-	-	-	-	1	0.333	ug/l
Bromoform	75-25-2	-	-	-	-	1	0.333	ug/l
Bromomethane	74-83-9	-	-	-	-	1	0.333	ug/l
Carbon disulfide	75-15-0	-	-	-	-	5	3.334	ug/l
Carbon tetrachloride	56-23-5	-	-	-	-	1	0.333	ug/l
Chlorobenzene	108-90-7	-	-	-	-	1	0.333	ug/l
Chloroethane	75-00-3	-	-	-	-	1	0.333	ug/l
Chloroform -	67-66-3	-	-	-	-	1	0.333	ug/l
Chloromethane	74-87-3	-	-	-	-	1	0.333	ug/l
Dibromochloromethane	124-48-1	-	-	-	-	1	0.333	ug/l
Dibromomethane	74-95-3	-	-	-	-	1	0.333	ug/l
Dichlorodifluoromethane	75-71-8	-	-	-	-	1	0.333	ug/l
Ethyl ether	60-29-7	-	-	-	-	1	0.333	ug/l
Ethyl methacrylate	97-63-2	-	-	-	-	5	3.334	ug/l
Ethylbenzene	100-41-4	-	-	-	-	1	0.333	ug/l
Hexachlorobutadiene -	87-68-3	-	-	-	-	1	0.333	ug/l
Isopropylbenzene	98-82-8	-	-	-	-	1	0.333	ug/l

Methylene chloride	75-09-2	-	-	-	-	5	3.334	ug/l
Naphthalene	91-20-3	-	-	-	-	1	0.333	ug/l
Styrene	100-42-5	-	-	-	-	1	0.333	ug/l
Toluene	108-88-3	-	-	-	-	1	0.333	ug/l
Trichlorofluoromethane	75-69-4	-	-	-	-	1	0.333	ug/l
Vinyl acetate	108-05-4	-	-	-	-	5	3.334	ug/l
Xylenes (total)	1330-20-7	-	-	-	-	1	0.333	ug/l
cis-1,2-Dichloroethylene	156-59-2	-	-	-	-	1	0.333	ug/l
cis-1,3-Dichloropropylene	10061-01-5	-	-	-	-	1	0.333	ug/l
m,p-Xylenes	179601-23-1	-	-	-	-	2	1.334	ug/l
n-Butylbenzene	104-51-8	-	-	-	-	1	0.333	ug/l
n-Propylbenzene	103-65-1	-	-	-	-	1	0.333	ug/l
o-Xylene	95-47-6	-	-	-	-	1	0.333	ug/l
sec-Butylbenzene	135-98-8	-	-	-	-	1	0.333	ug/l
tert-Butyl methyl ether	1634-04-4	-	-	-	-	1	0.333	ug/l
tert-Butylbenzene	98-06-6	-	-	-	-	1	0.333	ug/l
trans-1,2-Dichloroethylene	156-60-5	-	-	-	-	1	0.333	ug/l
trans-1,3-Dichloropropylene	10061-02-6	-	-	-	-	1	0.333	ug/l
trans-1,4-Dichloro-2-butene	110-57-6					5	3.334	Ug/l

MCL-Maximum contaminant level
ILCR-Incremental Lifetime Cancer Risk

Groundwater Cleanup Levels								
Method 8081A								
		Overburden Cleanup Level		Bedrock Cleanup Level		Laboratory		
Analyte	**CAS #**	**ug/L**	**Basis**	**ug/L**	**Basis**	**RL[1]**	**MDL**	**Units**
4,4'-DDD	72-54-8	-	-	-	-	0.04	0.01	ug/l
4,4'-DDE	72-55-9	-	-	-	-	0.04	0.01	ug/l
4,4'-DDT	50-29-3	-	-	-	-	0.04	0.01	ug/l
Aldrin	309-00-2	-	-	-	-	0.02	0.00665	ug/l
Chlordane	57-74-9	-	-	-	-	0.25	0.0765	ug/l
Dieldrin	60-57-1	-	-	-	-	0.04	0.01	ug/l
Endosulfan I	959-98-8	-	-	-	-	0.02	0.00665	ug/l
Endosulfan II	33213-65-9	-	-	-	-	0.04	0.01	ug/l
Endosulfan sulfate	1031-07-8	-	-	-	-	0.04	0.01	ug/l
Endrin	72-20-8	-	-	-	-	0.04	0.01	ug/l
Endrin aldehyde	7421-93-4	-	-	-	-	0.04	0.00665	ug/l
Endrin ketone	53494-70-5	-	-	-	-	0.04	0.01	ug/l
Heptachlor	76-44-8	-	-	-	-	0.02	0.00665	ug/l
Hexachlorobenzene	118-74-1	-	-	-	-	0.02	0.00625	ug/l
Methoxychlor	72-43-5	-	-	-	-	0.2	0.05	ug/l
Toxaphene	8001-35-2	-	-	-	-	0.5	0.15	ug/l
alpha-BHC	319-84-6	-	-	-	-	0.02	0.00665	ug/l
beta-BHC	319-85-7	-	-	-	-	0.02	0.00665	ug/l
cis-Chlordane	5103-71-9	-	-	-	-	0.02	0.00665	ug/l
delta-BHC	319-86-8	-	-	-	-	0.02	0.00665	ug/l
gamma-BHC (Lindane)	58-89-9	-	-	-	-	0.02	0.00665	ug/l
trans-Chlordane	5103-74-2	-	-	-	-	0.02	0.00665	ug/l

Groundwater Cleanup Levels								
Method 6020A								
		Overburden Cleanup Level		Bedrock Cleanup Level				
		ug/L	Basis	ug/L	Basis	Laboratory		
Analyte	CAS #					PQL[1]	MDL	Units
Aluminum	7429-90-5	-	-	-	-	50	19.3	ug/L
Antimony	7440-36-0	-	-	-	-	3	1	ug/L
Arsenic	7440-38-2	10	MCL	10	MCL	5	2	ug/L
Barium	7440-39-3	NA	NA	2,000	MCL	4	0.67	ug/L
Beryllium	7440-41-7	-	-	-	-	0.5	0.2	ug/L
Cadmium	7440-43-9	-	-	-	-	1	0.3	ug/L
Calcium	7440-70-2	-	-	-	-	200	80	ug/L
Cesium	7440-46-2	-	-	-	-	0.5	0.167	ug/L
Chromium	7440-47-3	100	MCL	100	MCL	10	3	ug/L
Cobalt	7440-48-4	6.0	HI=1 Residential	6.0	HI=1 Residential	1	0.3	ug/L
Copper	7440-50-8	1,300	Action Level	NA	NA	2	0.3	ug/L
Iron	7439-89-6	14,000	HI=1 Residential	14,000	HI=1 Residential	100	33	ug/L
Lead	7439-92-1	-	-	-	-	2	0.5	ug/L
Magnesium	7439-95-4	-	-	-	-	30	10	ug/L
Manganese	7439-96-5	300	Health Advisory	300	Health Advisory	5	1	ug/L
Molybdenum	7439-98-7	100	HI=1 Residential	100	HI=1 Residential	1	0.2	ug/L
Nickel	7440-02-0	-	-	-	-	2	0.6	ug/L
Potassium	7440-09-7	-	-	-	-	300	80	ug/L
Selenium	7782-49-2	-	-	-	-	5	2	ug/L
Silver	7440-22-4	-	-	-	-	1	0.3	ug/L
Sodium	7440-23-5	-	-	-	-	250	80	ug/L
Thallium	7440-28-0	-	-	-	-	2	0.6	ug/L
Thorium	7440-29-1	0.33	ILRC=10^{-6}	0.33	ILRC=10^{-6} Residential	2	0.7	ug/L

Uranium	7440-61-1	30	MCL	30	MCL	0.2	0.067	ug/L
Uranium-235	15117-96-1	-	-	-	-	0.07	0.01	ug/L
Uranium-238	7440-61-1	-	-	-	-	0.2	0.067	ug/L
Vanadium	7440-62-2	-	-	-	-	20	3.3	ug/L
Zinc	7440-66-6	-	-	-	-	20	3.3	ug/L
Method 7470A								
Mercury	7439-97-6	-	-	-	-	0.2	0.067	ug/L
Laboratory Method RAD-A-016								
Polonium-210	-	-	-	-	-	n/a	1	pCi/L

HI-Hazard Index
MCL-Maximum Contaminant Level
ILCR-Incremental Lifetime Cancer Risk
NA-Not Applicable

Groundwater Cleanup Levels								
Method EPA 537 Modified								
		Overburden Cleanup Level		Bedrock Cleanup Level				
		ug/L	Basis	ug/L	Basis	Laboratory		
Analyte	**CAS #**	-	-	-	-	**RL[1]**	**MDL**	**Units**
Perfluorooctanesulfonate (PFOS)	1763-23-1	-	-	-	-	2	0.66	ng/l
Perfluoroundecanoic acid (PFUdA)	2058-94-8	-	-	-	-	2	0.72	ng/l
N-Methylperfluorooctane sulfonamido acetic acid (NMeFOSAA)	2355-31-9	-	-	-	-	4	1.32	ng/l
Perfluoropentanoic acid (PFPeA)	2706-90-3	-	-	-	-	2	0.66	ng/l
Perfluoropentanesulfonate (PFPeS)	2706-91-4	-	-	-	-	1.88	0.66	ng/l
1H, 1H, 2H, 2H-Perfluorooctane sulfonic acid (6:2 FTS)	27619-97-2	-	-	-	-	1.9	0.66	ng/l
N-Ethylperfluorooctane sulfonamido acetic acid (NEtFOSAA)	2991-50-6	-	-	-	-	4	1.32	ng/l
Perfluorohexanoic acid (PFHxA)	307-24-4	-	-	-	-	2	0.66	ng/l
Perfluorododecanoic acid (PFDOA)	307-55-1	-	-	-	-	2	0.66	ng/l
Perfluorooctanoic acid (PFOA)	335-67-1	-	-	-	-	2	0.66	ng/l
Perfluorodecanoic acid (PFDA)	335-76-2	-	-	-	-	2	0.66	ng/l
Perfluorodecane sulfonic acid (PFDS)	335-77-3	-	-	-	-	1.94	0.66	ng/l
Perfluorohexane sulfonic acid (PFHxS)	355-46-4	-	-	-	-	1.82	0.66	ng/l
Perfluorobutanoic acid (PFBA)	375-22-4	-	-	-	-	2	0.82	ng/l
Perfluorobutane sulfonic acid (PFBS)	375-73-5	-	-	-	-	1.78	0.8	ng/l
Perfluoroheptanoic acid (PFHpA)	375-85-9	-	-	-	-	2	0.66	ng/l
Perfluoroheptane sulfonic acid (PFHpS)	375-92-8	-	-	-	-	1.9	0.66	ng/l
Perfluorononanoic acid (PFNA)	375-95-1	-	-	-	-	2	0.66	ng/l
Perfluorotetradecanoic acid (PFTDA)	376-06-7	-	-	-	-	2	0.66	ng/l
1H, 1H, 2H, 2H-Perfluorodecane sulfonic acid (8:2 FTS)	39108-34-4	-	-	-	-	3.84	1.32	ng/l
Perfluorononane sulfonic acid (PFNS)	68259-12-1	-	-	-	-	1.92	0.7	ng/l
Perfluorotridecanoic acid (PFTrDA)	72629-94-8	-	-	-	-	2	0.66	ng/l
Perfluorooctane sulfonamide (PFOSAm)	754-91-6	-	-	-	-	1.86	0.66	ng/l
1H, 1H, 2H, 2H-Perfluorohexane sulfonic acid (4:2 FTS)	757124-72-4	-	-	-	-	1.88	0.66	ng/l

Groundwater Cleanup Levels									
			Overburden Cleanup Level		Bedrock Cleanup Level				
			mg/L	Basis	mg/L	Basis	Laboratory		
Analyte	**Method**	**CAS #**					**RL**[1]	**MDL**	**Units**
Nitrogen, Nitrite	300.0	14797-65-0	1	MCL	1	MCL	0.1	0.033	**mg/L**
Nitrogen, Nitrate	300.0	14797-55-8	10	MCL	10	MCL	0.1	0.033	**mg/L**
Nitrogen, Nitrate/Nitrite	353.2	NONE	-	-	-	-	0.05	0.017	**mg/L**
Nitrogen, Ammonia	SM4500-NH_3 BH	7664-41-7	-	-	-	-	0.05	0.017	**mg/L**
Sulfide	SM4500 S^2 D	NONE	-	-	-	-	0.1	0.033	**mg/L**
Phosphorus, Orthophosphate	300.0	264888-19-9	-	-	-	-	0.2	0.067	**mg/L**
Phosphorus, Total	SM4500P-E	NONE	-	-	-	-	0.05	0.02	**mg/L**
Alkalinity, Total	SM2320B	NONE	-	-	-	-	2	0.725	mg CaCO3/L
Perchlorate	EPA 332.0	14797-73-0	-	-	-	-			**mg/L**
Bromide	EPA 300.0	24959-67-9	-	-	-	-	0.2	0.067	**mg/L**
Sulfate	EPA 300.0	14808-79-8	-	-	-	-	0.4	0.133	**mg/L**
Chloride	EPA 300.0	16887-00-6	-	-	-	-	0.2	0.067	**mg/L**
Fluoride	EPA 300.0	16984-48-8	-	-	-	-	0.1	0.033	**mg/L**
Total Suspended Solids	SM2540D	NONE	-	-	-	-	5	1.14	**mg/L**
Total Dissolved Solids	SM2540C	NONE					14.3	3.4	**mg/L**
Hardness							2	1	**Mg/L**
Total Organic Carbon		7440-44-0					1000	330	**ug/L**
Dissolved Organic Carbon							1000	330	**ug/L**
Total Inorganic Carbon							1000	330	**ug/L**
Dissolved Inorganic Carbon							1000	330	**ug/L**

MCL-Maximum contaminant level

Notes:

1-The laboratory reporting limit / quantitation limit cannot be lower than the lowest calibration standard.

Soil Cleanup Levels for the Protection of Human Health						
Method 8270D						
		Cleanup Level	**Basis**	**Laboratory**		
Analyte	**CAS #**	**ug/kg**		**RL[1]**	**MDL**	**Units**
1,1'-Biphenyl	92-52-4	-	-	333	99.9	ug/kg
1,2,4-Trichlorobenzene	120-82-1	-	-	333	99.9	ug/kg
1,2-Dichlorobenzene	95-50-1	-	-	333	99.9	ug/kg
1,2-Diphenylhydrazine	122-66-7	-	-	333	99.9	ug/kg
1,3-Dichlorobenzene	541-73-1	-	-	333	99.9	ug/kg
1,4-Dichlorobenzene	106-46-7	-	-	333	99.9	ug/kg
1,4-Dinitrobenzene	100-25-4	-	-	330	99.9	ug/kg
1-Methylnaphthalene	90-12-0	-	-	33.3	9.99	ug/kg
2,4,5-Trichlorophenol	95-95-4	-	-	333	99.9	ug/kg
2,4,6-Trichlorophenol	88-06-2	-	-	333	99.9	ug/kg
2,4-Dichlorophenol	120-83-2	-	-	333	99.9	ug/kg
2,4-Dimethylphenol	105-67-9	-	-	333	99.9	ug/kg
2,4-Dinitrophenol	51-28-5	-	-	666	99.9	ug/kg
2,4-Dinitrotoluene	121-14-2	-	-	333	99.9	ug/kg
2,6-Dinitrotoluene	606-20-2	-	-	333	99.9	ug/kg
2-Chloronaphthalene	91-58-7	-	-	33.3	9.99	ug/kg
2-Chlorophenol	95-57-8	-	-	333	99.9	ug/kg
2-Methyl-4,6-dinitrophenol	534-52-1	-	-	333	99.9	ug/kg
2-Methylnaphthalene	91-57-6	-	-	33.3	9.99	ug/kg
2-Nitrophenol	88-75-5	-	-	333	99.9	ug/kg
3,3'-Dichlorobenzidine	91-94-1	-	-	333	99.9	ug/kg
4-Bromophenylphenylether	101-55-3	-	-	333	99.9	ug/kg
4-Chloro-3-methylphenol	59-50-7	-	-	333	133.2	ug/kg

4-Chloroaniline	106-47-8	-	-	333	99.9	ug/kg
4-Chlorophenylphenylether	7005-72-3	-	-	333	99.9	ug/kg
4-Nitrophenol	100-02-7	-	-	333	99.9	ug/kg
Acenaphthene	83-32-9	-	-	33.3	9.99	ug/kg
Acenaphthylene	208-96-8	-	-	33.3	9.99	ug/kg
Aniline	62-53-3	-	-	333	99.9	ug/kg
Anthracene	120-12-7	-	-	33.3	9.99	ug/kg
Benzidine	92-87-5	-	-	330	99.9	ug/kg
Benzo(a)anthracene	56-55-3	340	ILRC=10-6 Residential	33.3	9.99	ug/kg
Benzo(a)pyrene	50-32-8	220	ILRC=10-6 Residential	33.3	9.99	ug/kg
Benzo(b)fluoranthene	205-99-2	340	ILRC=10-6 Residential	33.3	9.99	ug/kg
Benzo(ghi)perylene	191-24-2	-	-	33.3	9.99	ug/kg
Benzo(k)fluoranthene	207-08-9	-	-	33.3	9.99	ug/kg
Benzoic acid	65-85-0	-	-	660	166.5	ug/kg
Benzyl alcohol	100-51-6	-	-	333	99.9	ug/kg
Butylbenzylphthalate	85-68-7	-	-	333	99.9	ug/kg
Carbazole	86-74-8	-	-	33.3	9.99	ug/kg
Chrysene	218-01-9	-	-	33.3	9.99	ug/kg
Cresols (total)	1319-77-3	-	-	33.3	9.99	ug/kg
Indeno(1,2,3-cd)Pyrene	193-39-5	340	ILRC=10-6 Residential	33.3	9.99	ug/kg
Di-n-butylphthalate	84-74-2	-	-	333	99.9	ug/kg
Di-n-octylphthalate	117-84-0	-	-	333	99.9	ug/kg
Dibenzo(a,h)anthracene	53-70-3	-	-	33.3	9.99	ug/kg
Diethylphthalate	84-66-2	-	-	333	99.9	ug/kg
Dimethylphthalate	131-11-3	-	-	333	99.9	ug/kg
Diphenylamine	122-39-4	-	-	333	99.9	ug/kg
Fluoranthene	206-44-0	-	-	33.3	9.99	ug/kg
Fluorene	86-73-7	-	-	33.3	9.99	ug/kg

Hexachlorobenzene	118-74-1	-	-	333	99.9	ug/kg
Hexachlorobutadiene	87-68-3	-	-	333	99.9	ug/kg
Hexachlorocyclopentadiene	77-47-4	-	-	333	99.9	ug/kg
Hexachloroethane	67-72-1	-	-	333	99.9	ug/kg
Isophorone	78-59-1	-	-	333	99.9	ug/kg
N-Methyl-N-nitrosomethylamine	62-75-9	-	-	330	99.9	ug/kg
N-Nitrosodipropylamine	621-64-7	-	-	333	99.9	ug/kg
Naphthalene	91-20-3	-	-	33.3	9.99	ug/kg
Nitrobenzene	98-95-3	-	-	333	99.9	ug/kg
Pentachlorophenol	87-86-5	-	-	333	99.9	ug/kg
Phenanthrene	85-01-8	-	-	33.3	9.99	ug/kg
Phenol	108-95-2	-	-	333	99.9	ug/kg
Pyrene	129-00-0	-	-	33.3	9.99	ug/kg
Pyridine	110-86-1	-	-	333	99.9	ug/kg
bis(2-Chloro-1-methylethyl)ether	108-60-1	-	-	333	99.9	ug/kg
bis(2-Chloroethoxy)methane	111-91-1	-	-	333	99.9	ug/kg
bis(2-Chloroethyl) ether	111-44-4	-	-	333	99.9	ug/kg
bis(2-Ethylhexyl)phthalate	117-81-7	-	-	333	99.9	ug/kg
m,p-Cresols	65794-96-9	-	-	333	99.9	ug/kg
m-Nitroaniline	99-09-2	-	-	333	99.9	ug/kg
o-Cresol	95-48-7	-	-	333	99.9	ug/kg
o-Nitroaniline	88-74-4			333	109.89	ug/kg
p-Nitroaniline	100-01-6			333	99.9	ug/kg

ILCR-Incremental Lifetime Cancer Risk

Soil Cleanup Levels for the Protection of Human Health						
Method 8260C						
		Cleanup Level	Basis	Laboratory		
Analyte	**CAS #**			**RL[1]**	**MDL**	**Units**
1,1,1,2-Tetrachloroethane	630-20-6	-	-	1	0.333	ug/kg
1,1,1-Trichloroethane	71-55-6	-	-	1	0.333	ug/kg
1,1,2,2-Tetrachloroethane	79-34-5	-	-	1	0.333	ug/kg
1,1,2-Trichloroethane	79-00-5	-	-	1	0.333	ug/kg
1,1-Dichloroethane	75-34-3	-	-	1	0.333	ug/kg
1,1-Dichloroethylene - also TCLP	75-35-4	-	-	1	0.333	ug/kg
1,1-Dichloropropene	563-58-6	-	-	1	0.333	ug/kg
1,2,3-Trichlorobenzene	87-61-6	-	-	1	0.333	ug/kg
1,2,3-Trichloropropane	96-18-4	-	-	1	0.333	ug/kg
1,2,4-Trichlorobenzene	120-82-1	-	-	1	0.333	ug/kg
1,2,4-Trimethylbenzene	95-63-6	-	-	1	0.333	ug/kg
1,2-Dibromo-3-chloropropane	96-12-8	-	-	2	0.5	ug/kg
1,2-Dibromoethane	106-93-4	-	-	1	0.333	ug/kg
1,2-Dichlorobenzene	95-50-1	-	-	1	0.333	ug/kg
1,2-Dichloroethane - also TCLP	107-06-2	-	-	1	0.333	ug/kg
1,2-Dichloroethylene (total)	540-59-0	-	-	2	0.333	ug/kg
1,2-Dichloropropane	78-87-5	-	-	1	0.333	ug/kg
1,3,5-Trimethylbenzene	108-67-8	-	-	1	0.333	ug/kg
1,3-Dichlorobenzene	541-73-1	-	-	1	0.333	ug/kg
1,3-Dichloropropane	142-28-9	-	-	1	0.333	ug/kg
1,3-Dichloropropylene	542-75-6	-	-	2	0.333	ug/kg

1,4-Dichlorobenzene - also TCLP	106-46-7	-	-	1	0.333	ug/kg
2,2-Dichloropropane	594-20-7	-	-	1	0.333	ug/kg
2-Butanone - also TCLP	78-93-3	-	-	5	1.667	ug/kg
2-Chlorotoluene	95-49-8	-	-	1	0.333	ug/kg
2-Hexanone	591-78-6	-	-	5	1.667	ug/kg
4-Chlorotoluene	106-43-4	-	-	1	0.333	ug/kg
4-Isopropyltoluene	99-87-6	-	-	1	0.333	ug/kg
4-Methyl-2-pentanone	108-10-1	-	-	5	1.667	ug/kg
Acetone	67-64-1	-	-	5	1.667	ug/kg
Acrylonitrile	107-13-1	-	-	5	1.667	ug/kg
Benzene - also TCLP	71-43-2	-	-	1	0.333	ug/kg
Bromobenzene	108-86-1	-	-	1	0.333	ug/kg
Bromochloromethane	74-97-5	-	-	1	0.333	ug/kg
Bromodichloromethane	75-27-4	-	-	1	0.333	ug/kg
Bromoform	75-25-2	-	-	1	0.333	ug/kg
Bromomethane	74-83-9	-	-	1	0.333	ug/kg
Carbon disulfide	75-15-0	-	-	5	1.667	ug/kg
Carbon tetrachloride - also TCLP	56-23-5	-	-	1	0.333	ug/kg
Chlorobenzene - also TCLP	108-90-7	-	-	1	0.333	ug/kg
Chloroethane	75-00-3	-	-	1	0.333	ug/kg
Chloroform - also TCLP	67-66-3	-	-	1	0.333	ug/kg
Chloromethane	74-87-3	-	-	1	0.333	ug/kg
Dibromochloromethane	124-48-1	-	-	1	0.333	ug/kg
Dibromomethane	74-95-3	-	-	1	0.333	ug/kg
Dichlorodifluoromethane	75-71-8	-	-	1	0.333	ug/kg
Ethyl ether	60-29-7	-	-	1	0.333	ug/kg
Ethyl methacrylate	97-63-2	-	-	5	1.667	ug/kg
Ethylbenzene	100-41-4	-	-	1	0.333	ug/kg
Hexachlorobutadiene - also TCLP	87-68-3	-	-	1	0.333	ug/kg

Isopropylbenzene	98-82-8	-	-	1	0.333	ug/kg
Methylene chloride	75-09-2	-	-	5	1.667	ug/kg
Naphthalene	91-20-3	-	-	1	0.333	ug/kg
Styrene	100-42-5	-	-	1	0.333	ug/kg
Tetrachloroethylene - also TCLP	127-18-4	-	-	1	0.333	ug/kg
Toluene	108-88-3	-	-	1	0.333	ug/kg
Trichloroethylene - also TCLP	79-01-6	-	-	1	0.333	ug/kg
Trichlorofluoromethane	75-69-4	-	-	1	0.333	ug/kg
Vinyl acetate	108-05-4	-	-	5	1.667	ug/kg
Vinyl chloride - also TCLP	75-01-4	-	-	1	0.333	ug/kg
Xylenes (total)	1330-20-7	-	-	3	1	ug/kg
cis-1,2-Dichloroethylene	156-59-2	-	-	1	0.333	ug/kg
cis-1,3-Dichloropropylene	10061-01-5	-	-	1	0.333	ug/kg
m,p-Xylenes	179601-23-1	-	-	2	0.667	ug/kg
n-Butylbenzene	104-51-8	-	-	1	0.333	ug/kg
n-Propylbenzene	103-65-1	-	-	1	0.333	ug/kg
o-Xylene	95-47-6	-	-	1	0.333	ug/kg
sec-Butylbenzene	135-98-8	-	-	1	0.333	ug/kg
tert-Butyl methyl ether	1634-04-4	-	-	1	0.333	ug/kg
tert-Butylbenzene	98-06-6	-	-	1	0.333	ug/kg
trans-1,2-Dichloroethylene	156-60-5	-	-	1	0.333	ug/kg
trans-1,3-Dichloropropylene	10061-02-6	-	-	1	0.333	ug/kg
trans-1,4-Dichloro-2-butene	110-57-6	-	-	5	1.667	ug/kg

Soil Cleanup Levels for the Protection of Human Health						
Method 8082A						
		Cleanup Level	Basis	Laboratory		
Analyte	CAS #	ug/kg		RL[1]	MDL	Units
Aroclor 1016	12674-11-2	-	-	3.3	1.0989	ug/kg
Aroclor 1221	11104-28-2	-	-	3.3	1.0989	ug/kg
Aroclor 1232	11141-16-5	-	-	3.3	1.0989	ug/kg
Aroclor 1242	53469-21-9	-	-	3.3	1.0989	ug/kg
Aroclor 1248	12672-29-6	-	-	3.3	1.0989	ug/kg
Aroclor 1254	11097-69-1	-	-	3.3	1.0989	ug/kg
Aroclor 1260	11096-82-5	-	-	3.3	1.0989	ug/kg
Aroclor 1262	37324-23-5	-	-	3.3	1.0989	ug/kg
Aroclor 1268	11100-14-4	-	-	3.3	1.0989	ug/kg
PCBs, Total	1336-36-3	1000	Policy	3.33	1.10889	ug/kg

Soil Cleanup Levels for the Protection of Human Health						
Method 8081B						
		Cleanup Level	**Basis**	**Laboratory**		Units
		ug/kg		**RL[1]**	**MDL**	
4,4'-DDD	72-54-8	-	-	1.336	0.334	ug/kg
4,4'-DDE	72-55-9	-	-	1.336	0.334	ug/kg
4,4'-DDT	50-29-3	-	-	1.336	0.334	ug/kg
Aldrin	309-00-2	-	-	0.668	0.167	ug/kg
Chlordane	57-74-9	-	-	8.35	1.67	ug/kg
Dieldrin	60-57-1	-	-	1.336	0.334	ug/kg
Endosulfan I	959-98-8	-	-	0.668	0.167	ug/kg
Endosulfan II	33213-65-9	-	-	1.336	0.334	ug/kg
Endosulfan sulfate	1031-07-8	-	-	1.336	0.334	ug/kg
Endrin	72-20-8	-	-	1.336	0.334	ug/kg
Endrin aldehyde	7421-93-4	-	-	1.336	0.334	ug/kg
Endrin ketone	53494-70-5	-	-	1.336	0.334	ug/kg
Heptachlor	76-44-8	-	-	0.668	0.167	ug/kg
Hexachlorobenzene	118-74-1	-	-	0.668	0.334	ug/kg
Methoxychlor	72-43-5	-	-	6.68	1.67	ug/kg
Toxaphene	8001-35-2	-	-	16.7	5.5611	ug/kg
alpha-BHC	319-84-6	-	-	0.668	0.167	ug/kg
beta-BHC	319-85-7	-	-	0.668	0.167	ug/kg
cis-Chlordane	5103-71-9	-	-	0.668	0.167	ug/kg
delta-BHC	319-86-8	-	-	0.668	0.167	ug/kg
gamma-BHC (Lindane)	58-89-9	-	-	0.668	0.167	ug/kg
trans-Chlordane	5103-74-2	-	-	0.668	0.167	ug/kg

Sediment Cleanup Levels for the Protection of Human Health						
Method 8082A						
		Cleanup Level	**Basis**	**Laboratory**		
Analyte	**CAS #**	**ug/kg**		**RL[1]**	**MDL**	**Units**
Aroclor 1016	12674-11-2	-	-	3.3	1.0989	ug/kg
Aroclor 1221	11104-28-2	-	-	3.3	1.0989	ug/kg
Aroclor 1232	11141-16-5	-	-	3.3	1.0989	ug/kg
Aroclor 1242	53469-21-9	-	-	3.3	1.0989	ug/kg
Aroclor 1248	12672-29-6	-	-	3.3	1.0989	ug/kg
Aroclor 1254	11097-69-1	-	-	3.3	1.0989	ug/kg
Aroclor 1260	11096-82-5	-	-	3.3	1.0989	ug/kg
Aroclor 1262	37324-23-5	-	-	3.3	1.0989	ug/kg
Aroclor 1268	11100-14-4	-	-	3.3	1.0989	ug/kg
PCBs, Total	1336-36-3	2,7001	ILCR=10-6 (Abutting Resident/Recreational Visitor)	3.33	1.10889	ug/kg
PCBs, Total	1336-36-3	1,0002	Policy	3.33	1.10889	ug/kg

ILCR-Incremental Lifetime Cancer Risk

Sediment Cleanup Levels for the Protection of Ecological Receptors						
Method 8082A						
		Cleanup Level	Basis	Laboratory		
Analyte	CAS #	ug/kg		RL[1]	MDL	Units
Aroclor 1016	12674-11-2	-	-	3.3	1.0989	ug/kg
Aroclor 1221	11104-28-2	-	-	3.3	1.0989	ug/kg
Aroclor 1232	11141-16-5	-	-	3.3	1.0989	ug/kg
Aroclor 1242	53469-21-9	-	-	3.3	1.0989	ug/kg
Aroclor 1248	12672-29-6	-	-	3.3	1.0989	ug/kg
Aroclor 1254	11097-69-1	-	-	3.3	1.0989	ug/kg
Aroclor 1260	11096-82-5	-	-	3.3	1.0989	ug/kg
Aroclor 1262	37324-23-5	-	-	3.3	1.0989	ug/kg
Aroclor 1268	11100-14-4	-	-	3.3	1.0989	ug/kg
PCBs, Total	1336-36-3	1,080	Site Specific MATC	3.3	1.0989	ug/kg

MATC-Maximum Acceptable Toxic Concentration

Soil Cleanup Levels for the Protection of Human Health							
Method 6020A							
		Cleanup Level			Laboratory		
Analyte	**CAS #**	**ug/kg**	**pCi/g**	**Basis**	**RL[1]**	**MDL**	**Units**
Aluminum	7429-90-5	-	-		10000	4550	ug/kg
Arsenic	7440-38-2	13,700	-	ILRC=10^{-6} Residential	1000	338	ug/kg
Barium	7440-39-3	-	-	-	800	100	ug/kg
Beryllium	7440-41-7	-	-	-	100	20	ug/kg
Cadmium	7440-43-9	-	-	-	200	20	ug/kg
Calcium	7440-70-2	-	-	-	40000	13400	ug/kg
Chromium	7440-47-3	-	-	-	600	200	ug/kg
Cobalt	7440-48-4	-	-	-	200	60	ug/kg
Copper	7440-50-8	-	-	-	400	66	ug/kg
Iron	7439-89-6	-	-	-	20000	6600	ug/kg
Lead	7439-92-1	-	-	-	400	100	ug/kg
Magnesium	7439-95-4	-	-	-	6000	2000	ug/kg
Manganese	7439-96-5	-	-	-	1000	200	ug/kg
Molybdenum	7439-98-7	-	-	-	200	80	ug/kg
Nickel	7440-02-0	-	-	-	400	100	ug/kg
Potassium	7440-09-7	-	-	-	60000	16000	ug/kg
Selenium	7782-49-2	-	-	-	1000	360	ug/kg
Sodium	7440-23-5	-	-	-	50000	16000	ug/kg
Strontium	7440-24-6	-	-	-	2000	600	ug/kg
Thallium	7440-28-0	-	-	-	400	140	ug/kg

Thorium	7440-29-1	7,400	0.81	Background	400	133.4	ug/kg
Uranium	744-61-1	2,700	1.1	ILRC=10^{-6} Residential	40	13.2	ug/kg
Uranium-235	15117-96-1	NA	0.01	ILRC=10^{-6} Residential	14	2	ug/kg
Uranium-238	7440-61-1	NA	0.90	ILRC=10^{-6} Residential	40	13.2	ug/kg
Vanadium	7440-62-2	-	-	-	4000	300	ug/kg
Zinc	7440-66-6	-	-	-	4000	800	ug/kg
Alpha Spectroscopy Method							
Thorium-232	7440-29-1	NA	0.81	Background	n/a	0.81	pCi/g
Uranium-234	13966-29-5	NA	0.15	ILRC=10^{-6} Residential	n/a	0.15	pCi/g
Method DOE RP280 Modified							
Lead-210	-	-	-	-	n/a	5	pCi/g
Method EPA 901.1							
Bismuth-210	-	-	-	-	n/a	5	pCi/g
Actinium-227	-	-	-		n/a	0.5	pCi/g

ILCR - Incremental Lifetime Cancer Risk;

Sediment Cleanup Levels for the Protection of Ecological Receptors						
Method 6020A						
		Protective Level				
Analyte	**CAS #**	**ug/kg**	**Basis**	**RL[1]**	**MDL**	**Units**
Aluminum	7429-90-5	-		10000	4550	ug/kg
Arsenic	7440-38-2			1000	338	ug/kg
Barium	7440-39-3	-	-	800	100	ug/kg
Beryllium	7440-41-7	-	-	100	20	ug/kg
Cadmium	7440-43-9	-	-	200	20	ug/kg
Calcium	7440-70-2	-	-	40000	13400	ug/kg
Chromium	7440-47-3	-	-	600	200	ug/kg
Cobalt	7440-48-4	-	-	200	60	ug/kg
Copper	7440-50-8	176,000	Site Specific MATC	400	66	ug/kg
Iron	7439-89-6	-	-	20000	6600	ug/kg
Lead	7439-92-1	97,300	Site Specific MATC	400	100	ug/kg
Magnesium	7439-95-4	-	-	6000	2000	ug/kg
Manganese	7439-96-5	-	-	1000	200	ug/kg
Mercury	7439-97-6	1,300	Site Specific MATC	24	8.04	ug/kg
Molybdenum	7439-98-7	-	-	200	80	ug/kg
Nickel	7440-02-0	-	-	400	100	ug/kg
Potassium	7440-09-7	-	-	60000	16000	ug/kg
Selenium	7782-49-2	-	-	1000	360	ug/kg

Sodium	7440-23-5	-	-	50000	16000	ug/kg
Strontium	7440-24-6	-	-	2000	600	ug/kg
Thallium	7440-28-0	-	-	400	140	ug/kg
Thorium	7440-29-1			400	133.4	ug/kg
Uranium	744-61-1			40	13.2	ug/kg
Uranium-235	15117-96-1			14	2	ug/kg
Uranium-238	7440-61-1			40	13.2	ug/kg
Vanadium	7440-62-2			4000	300	ug/kg
Zinc	7440-66-6	-	-	4000	800	ug/kg
Alpha spectroscopy method						
Thorium-232	7440-29-1	-	-	-	0.8	pCi/g
Uranium-234	13966-29-5	-	-	-	0.15	pCi/g

Analyte	CAS #	RL[1]	MDL	Units
TCLP/VO – Method 1311/8260C				
Chloroform	67-66-3	10	3.33	ug/l
Carbon tetrachloride	56-23-5	10	3.33	ug/l
Tetrachloroethene	127-18-4	10	3.33	ug/l
Chlorobenzene	108-90-7	10	3.33	ug/l
1,2-Dichloroethane	107-06-2	10	3.33	ug/l
Benzene	71-43-2	10	3.33	ug/l
Vinyl chloride	75-01-4	10	3.33	ug/l
1,1-Dichloroethene	75-35-4	10	3.33	ug/l
Trichloroethene	79-01-6	10	3.33	ug/l
1,4-Dichlorobenzene	106-46-7	10	3.33	ug/l
2-Butanone	78-93-3	10	3.33	ug/l
TCLP/BNA – Method 1311/8270D				
2,4-Dinitrotoluene	121-14-2	50	15	ug/l
Hexachlorobutadiene	87-68-3	50	15	ug/l
Hexachlorocyclopentadiene	77-47-4	50	15	ug/l
Hexachloroethane	67-72-1	50	15	ug/l
Nitrobenzene	98-95-3	50	15	ug/l
2,4,6-Trichlorophenol	88-06-2	50	15	ug/l
Pentachlorophenol	87-86-5	50	15	ug/l
2-Methylphenol	95-48-7	100	15	ug/l
3-Methylphenol/4-Methylphenol	108-39-4/106-44-5	100	15	ug/l
2,4,5-Trichlorophenol	95-95-4	50	15	ug/l
Pyridine	110-86-1	50	15	ug/l

TCLP/Pesticides - SW846 1311/8081A				
Chlordane	57-74-9	2.5	0.765	ug/l
Endrin	72-20-8	0.4	0.1	ug/l
Heptachlor	76-44-8	0.2	0.0665	ug/l
Methoxychlor	72-43-5	2	0.5	ug/l
Toxaphene	8001-35-2	5	1.5	ug/l
gamma-BHC (Lindane)	58-89-9	0.2	0.0665	ug/l
TCLP/Herbicides - SW846 1311/8151A				
2,4,5-TP	93-72-1	50	16.666	ug/l
2,4-D	94-75-7	50	16.666	ug/l
TCLP/Metals - SW846 1311/ 6010D				
Arsenic, TCLP	7440-38-2	0.3	0.05	mg/l
Barium, TCLP	7440-39-3	0.05	0.01	mg/l
Cadmium, TCLP	7440-43-9	0.05	0.01	mg/l
Chromium, TCLP	7440-47-3	0.1	0.01	mg/l
Lead, TCLP	7439-92-1	0.2	0.033	mg/l
Selenium, TCLP	7782-49-2	0.3	0.06	mg/l
Silver, TCLP	7440-22-4	0.05	0.01	mg/l
Waste Classification				
Method		**Laboratory**		
		RL[1]	**MDL**	**Units**
Sulfide, Reactive	Lab Developed	10	10	mg/kg
Cyanide, Reactive	Lab Developed	10	10	mg/kg
Ignitability	SW846 1030	na	na	na
pH/Corrosivity	SW846 9045D	na	na	na

Notes:

1-The laboratory reporting limit / quantitation limit cannot be lower than the lowest calibration standard.

Groundwater Cleanup Levels								
Ozark Underground Laboratory Develop Method								
		Overburden Cleanup Level		Bedrock Cleanup Level				
		ug/L	Basis	ug/L	Basis	Laboratory		
Analyte	CAS #					PQL	MDL	Units
Sodium Fluorescein	2321-07-5	-	-	-	-	0.0075	0.0025	ug/L

QAPP Worksheet #16 Project Schedule/Timeline

Activities	Organization	Anticipated Date(s) of Initiation	Anticipated Date of Completion	Deliverable	Deliverable Due Date
RDWP/RD POP	de maximis	November 2019	February 2020		March 2020
QAPP/FSP, HASP	de maximis	November 2019	February 2020		March 2020
PDIWPs	de maximis, H&A, Geosyntec, ddms	November 2019	February 2020		March 2020
TSWPs	de maximis, H&A, Geosyntec, ddms	November 2019	February 2020		March 2020
Sitewide Monitoring Program	de maximis, O&M, Geosyntec, ddms	November 2019	November 2019		March 2020

NOTE: Additional detail regarding project schedule is provided in the RDWP.

QAPP Worksheet #17 Sampling Design and Rationale

Describe and provide a rationale for choosing the sampling approach (e.g., grid system, biased statistical approach):

RD/RA Monitoring Well Network

Groundwater sampling results obtained from the monitoring well network will be used to monitor the NMI-related COC plume. Periodic sampling of wells within the plume will provide additional data to supplement historical data – the composite data set will demonstrate the temporal and spatial variations in plume chemistry and geometry, and progress in meeting long-term groundwater restoration goals. Plume delineation results will also provide a basis to confirm that the institutional controls relating to groundwater remain protective. The monitoring well network will also be used to demonstrate that all groundwater that exceeds federal drinking water standards and other risk-based levels is being captured.

1,4-dioxane in overburden and bedrock aquifers

Existing overburden and bedrock wells were sampled in November 2019 to provide a baseline for 1,4-dioxane concentration in groundwater. A description of the sampling program and results from the event are provided in Appendix B as PDI-ISS-1. Based on results of the November 2019 event, additional wells for the assessment of 1,4-dioxane will be installed. The rationale for selecting the location for new wells as well as the sampling program for the new wells is described in Appendix D.

In-situ sequestration (ISS)

ISS predesign will involve collection of soils, laboratory treatability testing and field pilot testing. In summary, laboratory studies include testing ISS for overburden soils inside and outside the Holding Basin as well as bedrock from outside of the Holding Basin. The rationale for which soils are being tested, what groundwater is used for the testing and the series of treatability tests being performed are described in Appendix E. The location for soils to collect for treatability testing is provided in Appendix B.

QAPP Worksheet #17 Sampling Design and Rationale

Excavation and off-site disposal
Biased sampling is proposed to help establish the limits of the proposed excavation of impacted soils and the extent of contamination at depth. Proposed sample locations were selected based on explorations which previously indicated elevated concentrations of site chemicals of concern (primarily polychlorinated biphenyls and uranium), and where there was not a nearby sample at the same depth with concentrations that met site cleanup levels. These proposed locations were placed approximately 5 to 10 feet away from samples with elevated concentrations of site COCs, and they were approximately oriented on magnetic North-South-East-West. Where there was insufficient data at depth, samples are also proposed to be collected beneath samples with concentrations of site COCs above site cleanup levels to delineate the vertical extent of contamination. Samples are also proposed to be collected at depth (up to 26 feet) in the former building slab to evaluate the presence of COCs related to incidental leaks from drainage utilities beneath the building. Additional surficial samples are also proposed around the Cooling Water Recharge Pond to evaluate surficial concentrations of site COCs. If necessary, delineation samples may be collected if the limits of the excavation are not fully defined following the initial round of soil and sediment sampling. If performed, delineation samples will be collected approximately 10 feet away from a sample with concentrations of COCs above site RAOs and oriented on magnetic North-South-East-West. Sampling of sediment and groundwater within the footprint of the Cooling Pond will be conducted to determine the proposed sequence of excavation of the sediment and the proposed restoration plans to match the hydrology of the existing cooling pond functions and values. The stability of the Gabion wall will also be taken into consideration and information on the soils above and below the wall will be collected to conduct a slope stability analysis of the proposed excavations needed for removal of sediment from the cooling pond, and peat bog.
HB Containment
The proposed test borings are spaced approximately every 100 ft. along the alignment of the containment wall to collect soil density data, subsurface strata thickness, and rock characteristics for purposes of designing an effective low permeability containment wall. The location and number of borings is intended to capture the variability in depth of overburden soils, the variability to the soil density, and rock quality and fracturing. Continuous monitoring tubes (CMTs) will be installed within up to six of the boreholes along with couplet observation wells to characterize the hydraulic properties of each strata within the alignment of the low permeability containment wall. The hydraulic conductivity of each strata along with the chemistry of each strata will be analyzed in order to determine the depth the low permeability wall needs to extend. The goal is to install the low permeability wall into a strata that minimizes the transport of the COCs into or out of the Holding Basin (HB).Up to three test pits will be conducted along the wall alignment to identify potential obstructions to the wall construction such as building foundations and utilities. Additional explorations are proposed that include two sets of three borings drilled within 10 to 20 ft. of each other, in order to conduct seismic cross-hole testing. These tests are necessary to determine the soil and rock response to ground motions that would be induced by a design earthquake

QAPP Worksheet #17 Sampling Design and Rationale

to be determined by the seismic analysis. These tests are needed ultimately to determine the design load the wall will need to withstand under static and earthquake loading conditions. Finally, borings are planned within the bottom of the existing HB to determine the soil density and soil characteristics to design a low permeability cap. The construction of the cap will involve filling the low portion of the HB with soil that will induce a load to the underlying soil. The data collected is necessary to determine potential future settlement of underlying soils from the cap and design the finished grades accordingly. Supplemental testing of the cooling pond and peat bog sediment strength and thickness will be conducted in order to evaluate the stability of slopes during excavation and following the establishment of finished grades at the Site.

QAPP Worksheet #18 Sampling Locations and Methods/SOP Requirements

Sampling Area/ID Number	Matrix	Sample Type and Number: Depth Intervals	Analytical Group	Concentration Level	Number of Samples (plus field duplicates)[1]	Sampling SOP Reference Number[2]	Rationale for Sampling Location
Site-Wide Soils and Sediments	Soils and Sediment	Refer to Appendix A of the RDWP- Pre-Design Investigation Work Plan – Site-Wide Soils and Sediments					
In-Situ Sequestration of DU & U	Soils	Refer to Appendix B of the RDWP-Pre-Design Investigation Work Plan – In-Situ Sequestration of DU & U					
Holding Basin Containment	Soils and Sediment	Refer to Appendix C of the RDWP-Pre-Design Investigation Work Plan – Holding Basin Containment					
1,4-dioxane and VOCs in Bedrock Groundwater	Soil and Sediment	Refer to Appendix D- Pre-Design Investigation Work Plan – 1,4-dioxane and VOCs in Bedrock Groundwater					
Groundwater Monitoring	Water	Refer to Appendix K of RDWP-Site Wide Monitoring Plan (SWMP)- Appendix K of RDWP					

Notes: 1. One blind duplicate sample will be collected for every 20 samples.
2. Sampling SOP reference number from QAPP Worksheet #21.

Worksheet 19 *(Alpha Aqueous, Air) and (OUL Aqueous)*

Matrix	# of Samples*	Parameter (Lab)	Analytical and Preparation Method/SOP Reference	Method	Minimum Required Sample Volume and Container Type	Preservation	Holding Time [1]
Groundwater and/or IDW		VOCs	2108	SW846 8260C	3 x 40 ml amber glass vials	HCl to pH<2, Cool to 4°C±2°C	14 days to analysis
		1,4-Dioxane	1948/2164	SW8463510C / 8270D Isotope Dilution	2 x 1 L amber glass bottles	HCl to pH<2, Cool to 4°C±2°C	7 days to extraction/ 40 to analysis
		SVOCs	1948/2111	SW846 3510C/ 8270D	2 x 1 L amber glass bottles with Teflon®-lined lid	Cool to 4°C±2°C	7 days to extraction/ 40 to analysis
		Pesticides	1948/2116	SW846 3510C/ 8081B	2 x 1 L amber glass bottles with Teflon®-lined lid	Cool to 4°C±2°C	7 days to extraction/ 40 to analysis
		PFAS	23528	Modified EPA 537.1 Isotope Dilution	two (2) 250-mL high density polyethylene (HDPE)container with an unlined plastic screw cap	Cool to 4°C+2°C	14 days to extraction/28 days to analysis after extraction
		Alkalinity, Total	2213	SM2320B	1 x 250 ml plastic bottle	Cool to 4°C+2°C	14 days to analysis
		Bromide	2214	EPA 300.0			
		Chloride			1 x 500 ml plastic bottle	Cool to 4°C+2°C	28 days to analysis
		Sulfate					
		Fluoride					
		ortho-Phosphate	2225	SM 4500P-E	1 x 250 ml plastic bottle	Cool to 4°C+2°C	48 hours to analysis
		Total Phosphorous	2225	EPA 365.4	1 x 250 ml plastic bottle	Cool to 4°C+2°C	28 days to analysis
		Nitrate-N	2217	4500NO3-F, 4500NO2-B	1 L amber glass	Cool to 4°C+2°C	48 hours to analysis
		Nitrite-N					
		Nitrate/Nitrite Nitrogen			1 L amber glass	1:1 H2SO4	48 hours to analysis
		Ammonia Nitrogen	2206	SM4500 NH3-BH	1 x 250 ml plastic bottle	H2SO4 conc.	28 days to analysis
		pH	2202	SW846 9040C	125-mL plastic	Cool to 4°C+2°C	15 minutes
		Total Dissolved Solids	2219	SM2540C	1 x 250 ml glass bottle	Cool to 4°C+2°C	7 day to analysis
		Total Suspended Solids	2220	SM2540D	1 x 250 ml glass bottle	Cool to 4°C+2°C	7 day to analysis
		Perchlorate	2251	EPA 332.0	4-oz glass jar w/Teflon lid	Cool to 4°C+2°C	28 days to analysis
		Total Sulfide	2221	SM 4500-S2− D	1 x 250 ml plastic bottle	ZnAc2 & NaOH & 4°C	7 day to analysis
		Sodium Fluorescein	'Analysis of Fluorescent Dyes and Water and Charcoal Samplers'	Laboratory Developed Method	1 x 250 ml glass or plastic bottle	Cool to 4°C	30 days to analysis
Air		VOCs	2186	EPA TO-15	6 liter canister	NA	30 days to analysis

Notes:
***-Refer to RDWP**

1. All holding times are measured from date of collection.

°C = Degrees Celsius.
PCB = Polychlorinated biphenyls.
TOC = Total organic carbon.
DOC - Disolved organic carbon
TIC - Total inorganic carbon
DIC - Dissolved inorganic carbon
TSS = Total suspended solids.
VOCs = Volatile organic compounds.
SVOCs - Semivolatile organic compounds
* - DOC and DIC must be field filtered using 0.45 micron filter

Worksheet 19 *(GEL Aqueous)*

Matrix	# of Samples*	Parameter (Lab)	Analytical and Preparation Method/SOP Reference	Method	Minimum Required Sample Volume and Container Type	Preservation	Holding Time [1]
Groundwater and/or IDW		VOCs	GL-OA-E-039 / GL-OA-E-038	SW846 8260D	3, 40-ml VOA vials w/ PTFE-faced silicone septum	Cool to 4°C±2°C, HCl pH<2	14 days to analysis
		1,4-Dioxane	GL-OA-E-013 / GL-OA-E-009	SW846 3510C/ 8270E SIM	2 x 1 L amber glass bottles with Teflon®-lined lid	Cool to 4°C±2°C	7 days to extraction/ 40 to analysis
		SVOCs	GL-OA-E-013 / GL-OA-E-009	SW846 3510C/ 8270E SIM	2 x 1 L amber glass bottles with Teflon®-lined lid	Cool to 4°C±2°C	7 days to extraction/ 40 to analysis
		Pesticides	GL-OA-E-013 / GL-OA-E-041	SW846 3535A / 8081B	2 x 1 L amber glass bottles with Teflon®-lined lid	Cool to 4°C±2°C	7 days to extraction/ 40 to analysis
		PFAS	GL-OA-E-076	EPA Modified Method 537	two (2) 250-mL high density polyethylene (HDPE)container with an unlined plastic screw cap	Cool to 4°C+2°C	14 days to extraction/28 days to analysis after extraction
		Metals including Uranium, Uranium-235, Uranium-238, and Thorium	GL-MA-E-006/GL-MA-E-014	SW846 3005A/ 6020A	1 x 250 ml plastic bottle	HNO_3 to pH<2	180 days to analysis
		Mercury	GL-MA-E-010	SW846 7470A	1 x 250 ml plastic bottle	HNO_3 to pH<2	28 days to analysis
		Polonium-210	GL-RAD-A-016	Lab Developed Method	1L Plastic	HNO_3 to pH<2	180 days to analysis
		Alkalinity, Total	GL-GC-E-033	SM 2320B	1 x 250 ml plastic bottle	Cool to 4°C+2°C	14 days to analysis
		Bromide	GL-GC-E-086	Mrthod 300.0	1 x 125 ml plastic bottle**	Cool to 4°C+2°C	28 days to analysis
		Chloride					
		Sulfate					
		Fluoride					
		ortho-Phosphate					48 hours to analysis
		Nitrate-N					
		Nitrite-N					
		Total Phosphorous	GL-GC-E-103	Method 365.4	1x125 ml plastic bottle	Cool to 4°C±2°C	28 days to analysis
		Nitrate/Nitrite Nitrogen	GL-GC-E-128	Method 353.2	1x125 ml plastic bottle	Cool to 4°C+2°C, H2SO4	28 days to analysis
		Ammonia Nitrogen	GL-GC-E-072	SM 4500 NH3 B	1 x 250 ml plastic bottle	Cool to 4°C+2°C, H2SO4	28 days to analysis
		pH	GL-GC-E-008	SM4500-H B	125-mL plastic	Cool to 4°C+2°C	15 minutes
		Total Dissolved Solids	GL-GC-E-001	SM2540C	1 x 250 ml plastic bottle	Cool to 4°C+2°C	7 day to analysis
		Total Suspended Solids	GL-GC-E-012	SM2540D	2 x 1000 ml plastic bottle	Cool to 4°C+2°C	7 day to analysis
		Perchlorate	GL-OA-E-067	SW846 6850 Modified	125 ml polyethylene	Cool to 4°C+2°C	28 days to analysis
		Hardness, Total as CaCO3	GL-GC-E-100	SM2340C	250-mL HDPE	HNO3 to pH<2; Cool to 4°C+2°C	180 days to analysis
		Total Sulfide	GL-GC-E-052	SM 4500-S(2-) D	1 x 250 ml plastic bottle	ZnAc2 & NaOH & 4°C	7 day to analysis
		TOC/TIC	GL-GC-E-093	SW846 9060/2215	250 mL amber glass	H_2SO_4 to pH<2, Cool to 4°C	28 days to analysis
		DOC/DIC*			250 mL amber glass	H_2SO_4 to pH<2, Cool to 4°C after field filtration; Cool to 4°C if lab	24 hours to filtration; 28 days to analysis

Notes:
***-Refer to RDWP**
1. All holding times are measured from date of collection.
°C = Degrees Celsius.
PCB = Polychlorinated biphenyls.
TOC = Total organic carbon.
DOC - Disolved organic carbon
TIC - Total inorganic carbon
DIC - Dissolved inorganic carbon
TSS = Total suspended solids.
VOCs = Volatile organic compounds.
SVOCs - Semivolatile organic compounds
* - DOC and DIC must be field filtered using 0.45 micron filter
**-one bottle for all analytes

Worksheet 19 *(Alpha Soil/Sediments)*

Matrix	# of Samples*	Parameter (Lab)	Analytical and Preparation Method/SOP Reference	Method	Minimum Required Sample Volume and Container Type	Preservation	Holding Time [1]
Groundwater and/or IDW		VOCs	2108	SW846 8260C	3 x 40 ml amber glass vials	HCl to pH<2, Cool to 4°C±2°C	14 days to analysis
		1,4-Dioxane	1948/2164	SW8463510C / 8270D Isotope Dilution	2 x 1 L amber glass bottles	HCl to pH<2, Cool to 4°C±2°C	7 days to extraction/ 40 to analysis
		SVOCs	1948/2111	SW846 3510C/ 8270D	2 x 1 L amber glass bottles with Teflon®-lined lid	Cool to 4°C±2°C	7 days to extraction/ 40 to analysis
		Pesticides	1948/2116	SW846 3510C/ 8081B	2 x 1 L amber glass bottles with Teflon®-lined lid	Cool to 4°C±2°C	7 days to extraction/ 40 to analysis
		PFAS	23528	Modified EPA 537.1 Isotope Dilution	two (2) 250-mL high density polyethylene (HDPE)container with an unlined plastic screw cap	Cool to 4°C+2°C	14 days to extraction/28 days to analysis after extraction
		Alkalinity, Total	2213	SM2320B	1 x 250 ml plastic bottle	Cool to 4°C+2°C	14 days to analysis
		Bromide	2214	EPA 300.0			
		Chloride			1 x 500 ml plastic bottle	Cool to 4°C+2°C	28 days to analysis
		Sulfate					
		Fluoride					
		ortho-Phosphate	2225	SM 4500P-E	1 x 250 ml plastic bottle	Cool to 4°C+2°C	48 hours to analysis
		Total Phosphorous	2225	EPA 365.4	1 x 250 ml plastic bottle	Cool to 4°C+2°C	28 days to analysis
		Nitrate-N	2217	4500NO3-F, 4500NO2-B	1 L amber glass	Cool to 4°C+2°C	48 hours to analysis
		Nitrite-N					
		Nitrate/Nitrite Nitrogen			1 L amber glass	1:1 H2SO4	48 hours to analysis
		Ammonia Nitrogen	2206	SM4500 NH3-BH	1 x 250 ml plastic bottle	H2SO4 conc.	28 days to analysis
		pH	2202	SW846 9040C	125-mL plastic	Cool to 4°C+2°C	15 minutes
		Total Dissolved Solids	2219	SM2540C	1 x 250 ml glass bottle	Cool to 4°C+2°C	7 day to analysis
		Total Suspended Solids	2220	SM2540D	1 x 250 ml glass bottle	Cool to 4°C+2°C	7 day to analysis
		Perchlorate	2251	EPA 332.0	4-oz glass jar w/Teflon lid	Cool to 4°C+2°C	28 days to analysis
		Total Sulfide	2221	SM 4500-S2− D	1 x 250 ml plastic bottle	ZnAc2 & NaOH & 4°C	7 day to analysis
		Sodium Fluorescein	'Analysis of Fluorescent Dyes and Water and Charcoal Samplers'	Laboratory Developed Method	1 x 250 ml glass or plastic bottle	Cool to 4°C	30 days to analysis
Air		VOCs	2186	EPA TO-15	6 liter canister	NA	30 days to analysis

Worksheet 19 *(Alpha Soil/Sediments)*

Matrix	# of Samples*	Parameter (Lab)	Analytical and Preparation Method/SOP Reference	Method	Minimum Required Sample Volume and Container Type	Preservation	Holding Time [1]
Soil/Sediments		VOCs	2108	SW846 8260C	3-EnCore™ samplers	Cool to 4°C±2°C	48 hours to preservation
					One 100ml plastic bottle for Moisture		14 days to analysis
		SVOCs	1959/2111	SW846 3546/8270D	1 x 8-oz glass jar with Teflon®-lined lid	Cool to 4°C+2°C	14 days to extraction;40days to analysis
		PCBs	1954/2129	SW846 3540C/8082A	1 x 4-oz glass jar with Teflon®-lined lid	Cool to 4°C+2°C	14 days to extraction;40days to analysis
		Pesticides	1959/2116	SW846 3546/8081B	1 x 8-oz glass jar with Teflon®-lined lid	Cool to 4°C+2°C	14 days to extraction;40days to analysis
Wasre Classification		PCBs	1954/2129	SW846 3540C/8082A	1 x4-oz glass jar with Teflon®-lined lid	Cool to 4°C+2°C	14 days to extraction; 14 days to analysis
		TCLP VOCs	2112/2108	SW846 1311/8260C	1 x4-oz glass jar with Teflon®-lined lid	Cool to 4°C+2°C	14 days to leach; 14 days to analysis
		TCLP SVOCs	2133/1948/2111	SW846 1311/3510C/8270D	1 x4-oz glass jar with Teflon®-lined lid	Cool to 4°C+2°C	14 days to leach; 7 days to extraction; 40 days to analysis
		TCLP Pesticides	2133/1948/2116	SW846 1311/3510C/8081B	1 x4-oz glass jar with Teflon®-lined lid	Cool to 4°C+2°C	14 days to leach; 7 days to extraction; 40 days to analysis
		TCLP Herbicides	2133/1948 /2128	SW846 1311/3510C/8151B	1 x4-oz glass jar with Teflon®-lined lid	Cool to 4°C+2°C	14 days to leach; 7 days to extraction; 40 days to analysis
		TCLP Metals	2133/26796	SW846 1311/6010B	1 x4-oz glass jar with Teflon®-lined lid	Cool to 4°C+2°C	14 days to leach/128 days to analysis
		Ignitability	2238	SW846 1030	1 x4-oz glass jar with Teflon®-lined lid	Cool to 4°C+2°C	14 day to analysis
		Reactive Cyanide	2228	Lab Developed Method	1 x4-oz glass jar with Teflon®-lined lid	Cool to 4°C+2°C	7 day to analysis
		Reactive Sulfide			1 x4-oz glass jar with Teflon®-lined lid	Cool to 4°C+2°C	7 day to analysis
		pH / Corrosivity	2203	SW846 9045D	1 x4-oz glass jar with Teflon®-lined lid	Cool to 4°C+2°C	15 minutes

Notes:
***-Refer to RDWP**

1. All holding times are measured from date of collection.

°C = Degrees Celsius.
PCB = Polychlorinated biphenyls.
TOC = Total organic carbon.
DOC - Disolved organic carbon
TIC - Total inorganic carbon
DIC - Dissolved inorganic carbon
TSS = Total suspended solids.
VOCs = Volatile organic compounds.
SVOCs - Semivolatile organic compounds
* - DOC and DIC must be field filtered using 0.45 micron filter

Worksheet 19 *(GEL Soils and Waste Classification)*

Matrix	# of Samples*	Parameter (Lab)	Analytical and Preparation Method/SOP Reference	Method	Minimum Required Sample Volume and Container Type	Preservation	Holding Time [1]
Soil/Sediments		VOCs	GL-OA-E-039 / GL-OA-E-039	SW846 5035A / 8260D	4 ± 2°C, 2 vials with de-I water preservative and 1 vial with MeOH preservative	Cool to 4°C±2°C	48 hours to preservation 14 days to analysis
		SVOCs	GL-OA-E-066 / GL-OA-E-009	SW846 3541 / 8270E	250 mL glass with Teflon®-lined lid	Cool to 4°C+2°C	14 days to extraction;40days to analysis
		Metals including Uranium, Uranium-235, Uranium-238, and Thorium	GL-MA-E-009 / GL-MA-E-014	SW846 3050B/6020A	1 x 4-oz plastic	NA	180 days to analysis
		Mercury	GL-MA-E-010	SW846 7471B		Cool to 4°C+2°C	28 days to analysis
		Bismuth-210, Actinium-227	GL-RAD-A-013	EPA 901.1	1 x 16-oz plastic	NA	180 days to analysis
		Uranium-234	GL-RAD-A-011	HASL 300	1 x 8-oz plastic**	NA	180 days to analysis
		Thorium-232	GL-RAD-A-038	HASL 300	1 x 8-oz plastic**	NA	180 days to analysis
		Lead-210	GL-RAD-A-018	Modified DOE RP280	1 x 8-oz plastic**	NA	180 days to analysis
		PCBs	GL-OA-E-066 / GL-OA-E-040	SW846 3541 / 8082A	250 mL glass with Teflon®-lined lid	Cool to 4°C+2°C	365 days to extraction;40days to analysis
		Pesticides	GL-OA-E-066 / GL-OA-E-041	SW846 3541 / 8081B	250 mL glass with Teflon®-lined lid	Cool to 4°C+2°C	14 days to extraction;40days to analysis
Waste Classification		PCBs	GL-OA-E-066 / GL-OA-E-040	SW846 3541 / 8082A	250 mL glass with Teflon®-lined lid	Cool to 4°C+2°C	365 days to extraction; 14 days to analysis
		TCLP VOCs	GL-LB-E-006 / GL-OA-E-038	SW846 1311/8260D	250 mL glass with Teflon®-lined lid	Cool to 4°C+2°C	14 days to leach; 7 days to analysis
		TCLP SVOCs	GL-LB-E-006 / GL-OA-E-013 / GL-OA-E-009	SW846 1311 / 3541/8270E	250 mL glass with Teflon®-lined lid	Cool to 4°C+2°C	14 days to leach; 7 days to extraction; 40 days to analysis
		TCLP Pesticides	GL-LB-E-006 / GL-OA-E-070 / GL-OA-E-041	SW846 1311/3535A/8081B	250 mL glass with Teflon®-lined lid	Cool to 4°C+2°C	14 days to leach; 7 days to extraction; 40 days to analysis
		TCLP Herbicides	GA-LB-E-006 / GL-OA-E-015 / GL-OA-E-011	SW846 1311/8151A	250 mL glass with Teflon®-lined lid	Cool to 4°C+2°C	14 days to leach; 7 days to extraction; 40 days to analysis
		TCLP Metals	GL-MA-E-013	SW846 1311/3010A/6010D	250 mL glass with Teflon®-lined lid	Cool to 4°C+2°C	14 days to leach/128 days to analysis
		Ignitability	GL-GC-E-066	SW846 1020B	250 mL glass with Teflon®-lined lid	Cool to 4°C+2°C	14 day to analysis
		Reactive Cyanide	GL-GC-E-068	SW846 7.3.3/7.3.4	250 mL glass with Teflon®-lined lid	Cool to 4°C+2°C	7 day to analysis
		Reactive Sulfide			250 mL glass with Teflon®-lined lid	Cool to 4°C+2°C	7 day to analysis
		pH / Corrosivity	GL-GC-E-008	SW846 9040C	250 mL glass with Teflon®-lined lid	Cool to 4°C+2°C	15 minutes

Notes:
***-Refer to RDWP**
1. All holding times are measured from date of collection.
°C = Degrees Celsius.
PCB = Polychlorinated biphenyls.
TOC = Total organic carbon.
DOC - Disolved organic carbon
TIC - Total inorganic carbon
DIC - Dissolved inorganic carbon
TSS = Total suspended solids.
VOCs = Volatile organic compounds.
SVOCs - Semivolatile organic compounds
* - DOC and DIC must be field filtered using 0.45 micron filter
** - only one container required for all three analytes

QAPP Worksheet #20 Sample Quantities and Control Frequencies

Parameter	Laboratory [1]	Analytical and Preparation SOP[2,5]	Estimated Environ. Sample Quantity[3]	Field QC Analyses[5]						Laboratory QC Sample[6]						Total
				Trip Blank		Rinse Blank		Field Duplicate		Matrix Spike		Matrix Spike Duplicate		Lab Duplicate		
				Freq.	No.	Freq.	No.	Freq.	No.	Freq.	No.	Freq.	No.	Freq.	No.	
Groundwater																
VOCs	AAW	2108		1/cooler/day	TBD	1/day	TBD	1/20	0	1/20	0	1/20	0	NA	--	0
SVOCs	AAW	2111		NA	--	1/day	TBD	1/20	0	1/20	0	1/20	0	NA	--	0
PFAS	AAM	23528		NA	--	1/day	TBD	1/20	0	1/20	0	NA	--	NA	0	0
1,4-Dioxane	AAW	2164		NA	--	1/day	TBD	1/20	0	1/20	0	NA	--	NA	0	0
Nitrogen, Ammonia	AAW	2206		NA	--	NA	--	1/20	0	1/20	0	NA	--	NA	0	0
Nitrogen, Nitrate	AAW	2217		NA	--	NA	--	1/20	0	1/20	0	NA	--	NA	0	0
Nitrogen, Nitrite	AAW	2217		NA	--	NA	--	1/20	0	1/20	0	NA	--	NA	0	0
Nitrogen, Nitrate/Nitrite	AAW	2217		NA	--	1/day	TBD	1/20	0	1/20	0	NA	--	NA	0	0
Sulfide, Total	AAW	2221		NA	--	NA	--	1/20	0	1/20	0	NA	--	NA	0	0
Phosphorus, Orthophosphate	AAW	2225		NA	--	NA	--	1/20	0	1/20	0	NA	--	NA	0	0
Phosphorus, Total	AAW	2225		NA	--	NA	--	1/20	0	1/20	0	NA	--	NA	0	0
Alkalinity, Total	AAW	2213		NA	--	1/day	TBD	1/20	0	1/20	0	NA	--	NA	0	0
Perchlorate	AAW	2251		NA	--	NA	--	1/20	0	1/20	0	NA	--	NA	0	0
pH	AAW	2202		NA	--	NA	--	1/20	0	0	0	NA	--	NA	0	0
Bromide	AAW	2214		NA	-	NA		1/20	0	0	0	NA	--	NA	0	0
Sulfate	AAW	2214		NA	--	NA	--	1/20	0	1/20	0	NA	--	NA	0	0
Chloride	AAW	2214		NA	--	NA	--	1/20	0	1/20	0	NA	--	NA	0	0
Fluoride	AAW	2214		NA	--	NA	--	1/20	0	1/20	0	NA	--	NA	0	0
Aluminum	GEL	GL-MA-E-014		NA	--	1/day	--	1/20	0	1/20	0	NA	--	NA	0	0
Antimony	GEL	GL-MA-E-014		NA	--	1/day	--	1/20	0	1/20	0	NA	--	NA	0	0
Arsenic	GEL	GL-MA-E-014		NA	--	1/day	--	1/20	0	1/20	0	NA	--	NA	0	0
Barium	GEL	GL-MA-E-014		NA	--	1/day	--	1/20	0	1/20	0	NA	--	NA	0	0
Beryllium	GEL	GL-MA-E-014		NA	--	1/day	--	1/20	0	1/20	0	NA	--	NA	0	0
Cadmium	GEL	GL-MA-E-014		NA	--	1/day	--	1/20	0	1/20	0	NA	--	NA	0	0
Calcium	GEL	GL-MA-E-014		NA	--	1/day	--	1/20	0	1/20	0	NA	--	NA	0	0
Cesium	GEL	GL-MA-E-014		NA	--	1/day	--	1/20	0	1/20	0	NA	--	NA	0	0
Chromium	GEL	GL-MA-E-014		NA	--	1/day	--	1/20	0	1/20	0	NA	--	NA	0	0
Cobalt	GEL	GL-MA-E-014		NA	--	1/day	--	1/20	0	1/20	0	NA	--	NA	0	0
Copper	GEL	GL-MA-E-014		NA	--	1/day	--	1/20	0	1/20	0	NA	--	NA	0	0
Iron	GEL	GL-MA-E-014		NA	--	1/day	--	1/20	0	1/20	0	NA	--	NA	0	0
Lead	GEL	GL-MA-E-014		NA	--	1/day	--	1/20	0	1/20	0	NA	--	NA	0	0
Magnesium	GEL	GL-MA-E-014		NA	--	1/day	--	1/20	0	1/20	0	NA	--	NA	0	0
Manganese	GEL	GL-MA-E-014		NA	--	1/day	--	1/20	0	1/20	0	NA	--	NA	0	0
Molybdenum	GEL	GL-MA-E-014		NA	--	1/day	--	1/20	0	1/20	0	NA	--	NA	0	0
Nickel	GEL	GL-MA-E-014		NA	--	1/day	--	1/20	0	1/20	0	NA	--	NA	0	0
Potassium	GEL	GL-MA-E-014		NA	--	1/day	--	1/20	0	1/20	0	NA	--	NA	0	0
Selenium	GEL	GL-MA-E-014		NA	--	1/day	--	1/20	0	1/20	0	NA	--	NA	0	0
Silver	GEL	GL-MA-E-014		NA	--	1/day	--	1/20	0	1/20	0	NA	--	NA	0	0
Sodium	GEL	GL-MA-E-014		NA	--	1/day	--	1/20	0	1/20	0	NA	--	NA	0	0
Thallium	GEL	GL-MA-E-014		NA	--	1/day	--	1/20	0	1/20	0	NA	--	NA	0	0
Thorium	GEL	GL-MA-E-014		NA	--	1/day	--	1/20	0	1/20	0	NA	--	NA	0	0
Total Uranium	GEL	GL-MA-E-014		NA	--	1/day	--	1/20	0	1/20	0	NA	--	NA	0	0
Uranium-235	GEL	GL-MA-E-014		NA	--	1/day	--	1/20	0	1/20	0	NA	--	NA	0	0
Uranium-238	GEL	GL-MA-E-014		NA	--	1/day	--	1/20	0	1/20	0	NA	--	NA	0	0
Vanadium	GEL	GL-MA-E-014		NA	--	1/day	--	1/20	0	1/20	0	NA	--	NA	0	0
Zinc	GEL	GL-MA-E-014		NA	--	1/day	--	1/20	0	1/20	0	NA	--	NA	0	0
Mercury	GEL	GL-MA-E-010		NA	--	1/day	--	1/20	0	1/20	0	NA	--	NA	0	0
Polonium-210	GEL	GL-RAD-A016		NA	--	NA	--	1/20	0	1/20	0	NA	--	NA	0	0
Hardness	GEL	GL-GC-E-100		NA	--	NA	--	1/20	0	1/20	0	NA	--	NA	0	0
Total Organic Carbon	GEL	GL-GC-E-093		NA	--	NA	--	1/20	0	1/20	0	NA	--	NA	0	0
Total Inorganic Carbon	GEL	GL-GC-E-093		NA	--	NA	--	1/20	0	1/20	0	NA	--	NA	0	0
Dissolved Inorganic Carbon	GEL	GL-GC-E-093		NA	--	NA	--	1/20	0	1/20	0	NA	--	NA	0	0
Dissolved Organic Carbon	GEL	GL-GC-E-093		NA	--	NA	--	1/20	0	1/20	0	NA	--	NA	0	0
TDS	AAW	2219		NA	--	NA	--	1/21	0	1/20	0	NA	--	NA	0	0
TSS	AAW	2220		NA	--	NA	--	1/20	0	1/20	0	NA	--	NA	0	0
Sodium Fluorescein	OUL	Analysis of Fluorescent Dyes in Water and Charcoal Samples		NA	--	NA	--	1/20	0	1/20	0	NA	--	NA	0	0

Parameter	Laboratory [1]	Analytical and Preparation SOP[2,5]	Estimated Environ. Sample Quantity[3]	Field QC Analyses[5]						Laboratory QC Sample[6]						Total
				Trip Blank		Rinse Blank		Field Duplicate		Matrix Spike		Matrix Spike Duplicate		Lab Duplicate		
				Freq.	No.	Freq.	No.	Freq.	No.	Freq.	No.	Freq.	No.	Freq.	No.	
Soil/sedment																
VOCs	AAW	2108		1/cooler	TBD	1/day	TBD	1/20	0	1/20	0	1/20	0	NA	--	0
SVOCs	AAW	2111		NA	--	1/day	TBD	1/20	0	1/20	0	1/20	0	NA	--	0
Aluminum	GEL	GL-MA-E-014		NA	--	NA	--	1/20	0	1/20	0	NA	--	1/20	0	0
Arsenic	GEL	GL-MA-E-014		NA	--	NA	--	1/20	0	1/20	0	NA	--	1/20	0	0
Antimony	GEL	GL-MA-E-014		NA	--	NA	--	1/20	0	1/20	0	NA	--	1/20	0	0
Barium	GEL	GL-MA-E-014		NA	--	NA	--	1/20	0	1/20	0	NA	--	1/20	0	0
Beryllium	GEL	GL-MA-E-014		NA	--	NA	--	1/20	0	1/20	0	NA	--	1/20	0	0
Cadmium	GEL	GL-MA-E-014		NA	--	NA	--	1/20	0	1/20	0	NA	--	1/20	0	0
Calcium	GEL	GL-MA-E-014		NA	--	NA	--	1/20	0	1/20	0	NA	--	1/20	0	0
Chromium	GEL	GL-MA-E-014		NA	--	NA	--	1/20	0	1/20	0	NA	--	1/20	0	0
Cobalt	GEL	GL-MA-E-014		NA	--	NA	--	1/20	0	1/20	0	NA	--	1/20	0	0
Copper	GEL	GL-MA-E-014		NA	--	NA	--	1/20	0	1/20	0	NA	--	1/20	0	0
Iron	GEL	GL-MA-E-014		NA	--	NA	--	1/20	0	1/20	0	NA	--	1/20	0	0
Lead	GEL	GL-MA-E-014		NA	--	NA	--	1/20	0	1/20	0	NA	--	1/20	0	0
Magnesium	GEL	GL-MA-E-014		NA	--	NA	--	1/20	0	1/20	0	NA	--	1/20	0	0
Manganese	GEL	GL-MA-E-014		NA	--	NA	--	1/20	0	1/20	0	NA	--	1/20	0	0
Molybdenum	GEL	GL-MA-E-014		NA	--	NA	--	1/20	0	1/20	0	NA	--	1/20	0	0
Nickel	GEL	GL-MA-E-014		NA	--	NA	--	1/20	0	1/20	0	NA	--	1/20	0	0
Potassium	GEL	GL-MA-E-014		NA	--	NA	--	1/20	0	1/20	0	NA	--	1/20	0	0
Selenium	GEL	GL-MA-E-014		NA	--	NA	--	1/20	0	1/20	0	NA	--	1/20	0	0
Sodium	GEL	GL-MA-E-014		NA	--	NA	--	1/20	0	1/20	0	NA	--	1/20	0	0
Strontium	GEL	GL-MA-E-014		NA	--	NA	--	1/20	0	1/20	0	NA	--	1/20	0	0
Thallium	GEL	GL-MA-E-014		NA	--	NA	--	1/20	0	1/20	0	NA	--	1/20	0	0
Thorium	GEL	GL-MA-E-014		NA	--	NA	--	1/20	0	1/20	0	NA	--	1/20	0	0
Uranium	GEL	GL-MA-E-014		NA	--	NA	--	1/20	0	1/20	0	NA	--	1/20	0	0
Uranium-235	GEL	GL-MA-E-014		NA	--	NA	--	1/20	0	1/20	0	NA	--	1/20	0	0
Uranium-238	GEL	GL-MA-E-014		NA	--	NA	--	1/20	0	1/20	0	NA	--	1/20	0	0
Vanadium	GEL	GL-MA-E-014		NA	--	NA	--	1/20	0	1/20	0	NA	--	1/20	0	0
Zinc	GEL	GL-MA-E-014		NA	--	NA	--	1/20	0	1/20	0	NA	--	1/20	0	0
Mercury	GEL	GL-MA-E-010		NA	--	NA	--	1/20	0	1/20	0	NA	--	1/20	0	0
Bismuth-210, Actinium-227	GEL	GL-RAD-A-013		NA	--	NA	--	1/20	0	1/20	0	NA	--	1/20	0	0
Lead-210	GEL	GL-RAD-A-018		NA	--	NA	--	1/20	0	1/20	0	NA	--	1/20	0	0
Uranium-234	GEL	GL-RAD-A-015/ GL-RAD-A-011		NA	--	NA	--	1/20	0	1/20	0	NA	--	1/21	0	0
Thorium-232	GEL	GL-RAD-A-015GL-RAD-A-038		NA	--	NA	--	1/20	0	1/20	0	NA	--	1/22	0	0
PCBs, Total	AAM	2129		NA	--	NA	--	1/20	0	1/20	0	NA	--	1/20	0	0
Pesticides	AAW	2116		NA	--	NA	--	1/20	0	1/20	0	NA	--	1/20	0	0
Sulfide, Reactive	AAW	2228		NA	--	NA	--	1/20	0	1/20	0	NA	--	1/20	0	0
Cyanide, Reactive	AAW	2228		NA	--	NA	--	1/20	0	1/20	0	NA	--	1/20	0	0
Air																
VOCs	AAW	2186		1/cooler	NA	NA	NA	1/20	0	1/20	0	NA	0	NA	--	0
Waste Classification																
PCBs	AAW	2186		NA	--	NA	--	NA	--	NA	--	NA	--	NA	0	0
TCLP VOCs	AAW	2112/2108		NA	--	NA	--	NA	--	NA	--	NA	--	NA	0	0
TCLP SVOCs	AAW	2133/1948/2111		NA	--	NA	--	NA	--	NA	--	NA	--	NA	0	0
TCLP Pesticides	AAW	2133/1948/2116		NA	--	NA	--	NA	--	NA	--	NA	--	NA	0	0
TCLP Herbicides	AAW	2133/1948 /2128		NA	--	NA	--	NA	--	NA	--	NA	--	NA	0	0
TCLP Metals	AAW	2133/26796		NA	--	NA	--	NA	--	NA	--	NA	--	NA	0	0
Ignitability	AAW	2238		NA	--	NA	--	NA	--	NA	--	NA	--	NA	0	0
Reactive Cyanide	AAW	2228		NA	--	NA	--	NA	--	NA	--	NA	--	NA	0	0
Reactive Sulfide				NA	--	NA	--	NA	--	NA	--	NA	--	NA	0	0
pH / Corrosivity	AAW	2203		NA	--	NA	--	NA	--	NA	--	NA	--	NA	0	0

1. See Worksheet #30 for contact information.

2. See Worksheet #23 for SOP details.

3. Sample counts are an approximation, See RDWP Appendicies (Site-Wide Soils and Sediments-A, In-Situ Sequestration of DU & U-B, Holding Basin Containment-C, 1,4-dioxane and VOCs in Bedrock Groundwater-D) for for PDIs and Appendix I-FSP.

4. Field duplicates will consist of co-located samples for air analysis.

5. Laboratories are required to run the latest State-certified, industry-standard version of the specified method.

6. Frequency applicable to Design phase of project only.

1/day = One rinse blank per day or one per 20 samples, whichever is more frequent. Rinse blanks not required when dedicated sampling equipment is used.
Freq = Frequency.

QAPP Worksheet #21 Field Sampling SOP References

Reference Number	Title, Revision Date and/or Number	Originating Organization	Equipment Type	Modified for Project Work? (Y/N)	Comments
See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP					

QAPP Worksheet #22 Field Equipment Calibration, Maintenance, Testing and Inspection

Field Equipment	Calibration Activity/ Frequency	Maintenance Activity	Testing Activity	Inspection Activity	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[1]
Refer to Appendix A of the RDWP- Pre-Design Investigation Work Plan – Site-Wide Soils and Sediments								
Refer to Appendix B of the RDWP-Pre-Design Investigation Work Plan – In-Situ Sequestration of DU & U								
Refer to Appendix C of the RDWP-Pre-Design Investigation Work Plan – Holding Basin Containment								
Refer to Appendix D- Pre-Design Investigation Work Plan – 1,4-dioxane and VOCs in Bedrock Groundwater								
Refer to Appendix K of RDWP-Site Wide Monitoring Plan (SWMP)- Appendix K of RDWP								

Note: [1]SOPs are found in the Field Sampling Plan (FSP)-Appendix I of the RDWP.

QAPP Worksheet #23 Analytical SOP References

SOP Reference Number[1,2]	Title, Revision Date, and/or Number[3]	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
NMI-GW-007	FSP– 'Down-Hole Groundwater Field Parameter Measurement' SOP #NMI-GW-007	Definitive	Temperature, pH, turbidity, conductivity, DO	Combination water quality meter	de maximis, O&M, Geosyntec and H&A Field Team	N
NMI-GW-009	FSP – 'Water-Level Measurement Procedures' SOP #NMI-GW-009	Screening	Water Level measurements	Staff gauge / tape	de maximis, O&M, Geosyntec and H&A Field Team	N
See PDIs	Chlorine, Total Residual (4500CL-D)	Definitive	Chlorine in Water	HACH K17 ON-70T	(FIELD)	N
1948	Separatory Funnel Liquid-Liquid Extraction (3510C)	Definitive	Semi-volatiles preparation of waters	NA	AAW	N
1954	Soxhlet Extraction	Definitive	Aroclors in Soil preparation	GC/ECD	AAM	N
1959	Microwave Extraction (3546)	Definitive	Semi-Volatiles Preparation of soils	Microwave Accelerated Reaction System	AAW	N
2108	Volatile Organic Compounds by GC/MS (8260C)	Definitive	Volatile Organic Compounds in water, soils and sediments	Gas Chromatogram/ Mass Spectrometry (GC/MS)	AAW	N
2111	SVOCs (8270D)	Definitive	Semi-volatile organic compounds in water, soils and sediments	Gas Chromatogram/ Mass Spectrometry (GC/MS)	AAW	N
2116	Pesticides (8081B)	Definitive	Organochlorine Pesticides	GC / ECD	AAW	N

QAPP Worksheet #23 Analytical SOP References

SOP Reference Number[1,2]	Title, Revision Date, and/or Number[3]	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
2128	Herbicides (TCLP)	Definitive	Chlorinated Herbicides	GC / ECD	AAW	N
2129	PCBs (8082A)	Definitive	Aroclors in Soil	GC/ECD	AAM	N
2164	1,4-Dioxane By Gas Chromatography/Mass Spectrometry in Selected Ion Mode (GC/MS-SIM) with Isotope Dilution Modification (8270D – SIM)	Definitive	1,4-Dioxane in water	Gas Chromatogram/ Mass Spectrometry (GC/MS)	AAM	N
2186	VOCs – Soil Vapor (TO-15)	Definitive	Volatile Organics in Air	Gas Chromatogram/ Mass Spectrometry (GC/MS)	AAM	N
2206	Ammonia Nitrogen (SM4500 NH_3-BH)	Definitive	Ammonia Nitrogen	Lachat Autoanalyzer	AAW	N
2213	Alkalinity (Carb/Bicarb) (SM2320B)	Definitive	Alkalinity	pH meter	AAW	N
2214	Determination of Inorganic Anions by Ion Chromatography (300.0)	Definitive	Bromide, chloride, sulfate, fluoride in water	IC	AAW	N
2217	Nitrate, Nitrite and Nitrate/Nitrite Nitrogen (SM4500 NO_2 B, NO_3 F)	Definitive	Nitrate, Nitrite in water	Lachat Autoanalyzer	AAW	N
2219	Total Dissolved Solids	Definitive	Solids	NA	AAW	N
2220	Total Suspended Solids (SM2540D)	Definitive	Solids	NA	AAW	N
2202	pH	Definitive	pH	pH Meter	AAW	N

QAPP Worksheet #23 Analytical SOP References

SOP Reference Number[1,2]	Title, Revision Date, and/or Number[3]	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
2221	Total Sulfide, Methylene Blue	Definitive	Sulfide in water	Colorimetric	AAW	N
2225	Ortho-Phosphate , Total Phosphorous (SMP4500 P-E)	Definitive	ortho-Phosphate	Spectrophotometer	AAW	N
2251	Perchlorate (332.0)	Definitive	Perchlorate	Ion Chromatography with Suppressed Conductivity and Electrospray Ionization Mass Spectrometry	AAW	N
2228	RCN, RS	Definitive	Waste Characterization	Spectrophotometer	AAW	N
2238	Ignitability	Definitive	Waste Characterization	NA	AAW	N
2203	pH, Corrosivity	Definitive	Waste Characterization	pH Meter	AAW	N
2112, 2133	TCLP Preparation	Definitive	TCLP	(Various)	AAW	N
26796	ICP-AES	Definitive	Metals (TCLP)	ICP AES	AAM	N

QAPP Worksheet #23 Analytical SOP References

SOP Reference Number[1,2]	Title, Revision Date, and/or Number[3]	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
23528	Per- and Poly-fluorinated Substances (PFAS) in Drinking Water and Non-Potable Water (Modified 537.1 Isotope Dilution)	Definitive	PFAS in water	Liquid Chromatography/ tandem Mass Spectrometry (LC/MS/MS)	AAM	N
GL-MA-E-006	Hot Block Digestion for Aqueous Samples	Definitive	Preparation of waters for metals, except mercury	NA	GEL	N
GL-MA-E-009	Acid Digestion	Definitive	Preparation of soil, sludges ad sediments	NA	GEL	N
GL-MA-E-010	Mercury Analysis Using Perkin Elmer Automated Mercury Analyzer	Definitive	Mercury	Perkin Elmer FIMS100	GEL	N
GL-MA-E-014	Inductivity Coupled Plasma – Atomic Emission Spectrometry (6020A)	Definitive	Metals, except mercury. Incl U-235/U-238 and Thorium	ICP-AES	GEL	N
GL-RAD-A-011	The Isotopic Determination Of Americium, Curium, Plutonium, And Uranium	Definitive	Radiochemistry	Alpha Spec	GEL	N
GL-RAD-A-013	Bismuth-210, Actinium-227	Definitive	Radiochemistry	Gamma Spec	GEL	N
GL-RAD-A-015	Soil Preparation	Definitive	Radiochemistry	NA	GEL	N

QAPP Worksheet #23 Analytical SOP References

SOP Reference Number[1,2]	Title, Revision Date, and/or Number[3]	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
GL-RAD-A-016	Polonium-210	Definitive	Radiochemistry	NA	GEL	N
GL-RAD-A-018	Lead -210	Definitive	Radiochemistry	Gas Flow Proportional Counter	GEL	N
GL-RAD-A-038	Standard Operating Procedure for The Isotopic Determination of Thorium	Definitive	Radiochemistry	Alpha Spec	GEL	N
GL-GC-E-100	Hardness, Total as CaCO3 (SM2340B)	Definitive	Hardness in water	NA	GEL	N
GL-GC-E-093	Total and Dissolved Organic Carbon (TOC/DOC) Total and Dissolved Inorganic Carbon (TIC/DIC) Persulfate – Ultraviolet Oxidation Method	Definitive	TOC/TIC in water	TOC Analyzer	GEL	N
GL-OE-E-067	Definitive Low Level Perchlorate Analysis Utilizing Liquid Chromatography/Mass Spectrometry/ Mass Spectrometry (LC/MS/MS) By EPA Method 6850 Modified (6850M)	Definitive	Water	LC/MS/MS	GEL	N

QAPP Worksheet #24/25: Analytical Instrument Calibration/Analytical Instrument and Equipment Maintenance, Testing and Inspection

Instrument	Calibration Procedure/Range	Frequency of Calibration	Acceptance Criteria	CA by Analyst
ICP - AES	Initial Calibration (ICAL) – minimum one high standard and a calibration blank	Daily initial calibration prior to sample analysis	3 standards and a blank. Correlation Coefficient of ≥ 0.998	Recalibrate
	ICV	Once after each initial calibration, prior to sample analysis	Value of second source for all analyte(s) within ± 10% of expected	Recalibrate
	ICB	Immediately after the ICV	< absolute value of PQL	Positive result: accept sample results >10X the ICB or < 1/2 RL. Negative result: accept results >10x ICB. All other samples must be reanalyzed with compliant ICB
	Continuing Calibration Verification (CCV)	After every 10 samples and at the end of the analysis sequence	All analytes within + 10% of expected value	Recalibrate – rerun 10 samples previous to failed CCV.
	Continuing Calibration Blank (CCB)	Immediately after the CCV and at a frequency of every 10 samples	±PQL	Positive result: accept sample results >10X the ICB or < 1/2 RL. Negative result: accept results >10x ICB. All other samples must be reanalyzed with compliant CCB
	Interference Check Sample	At the beginning of each run immediately following the low level check (LLC)	± 20% of the true value for each analyte	Recalibrate.

QAPP Worksheet #24/25: Analytical Instrument Calibration/Analytical Instrument and Equipment Maintenance, Testing and Inspection

Instrument	Calibration Procedure/Range	Frequency of Calibration	Acceptance Criteria	CA by Analyst
ICPMS	Tuning	Daily	No atomic mass unit (amu) diff. of > 0.1 peak width ≥ 0.64 and ≥ 0.66; % RSD <5 for masses used for tuning	Perform mass cal. for amu. Adjust mass cal. for peak width
	ICAL consists of Blank and 1 point:	Daily initial calibration prior to sample analysis	3 standards and a blank. Correlation Coefficient of ≥ 0.998	Recalibrate, perform instrument maintenance if cal. cannot conform to criteria, recalibrate.
	ICV	After each cal.	± 10% of true value	Reanalyze
	ICB	Immediately after the ICV	No analytes detected > 1/2 LOQ	Positive result: accept sample results >10X the ICB or < 1/2 RL. Negative result: accept results > 10x ICB. All other samples must be reanalyzed with compliant ICB
	CCV	Immediately after the ICSAB and every 10 samples	± 10% of true value	Reanalyze
	CCB	Immediately after the CCV and every 10 samples	±PQL	Positive result: accept sample results > 10X the ICB or < 1/2 PQL. Negative result: accept results > 10x ICB. All other samples must be reanalyzed with compliant CCB
	Interference Check Sample	At the beginning of each run immediately following the LLC	± 20% of the true value for each analyte	Recalibrate.
	Linear Range	Quarterly	± 10% of true value	Samples > 90% of the linear range must be reanalyzed as a dilution

QAPP Worksheet #24/25: Analytical Instrument Calibration/Analytical Instrument and Equipment Maintenance, Testing and Inspection

Instrument	Calibration Procedure/Range	Frequency of Calibration	Acceptance Criteria	CA by Analyst
CVAA	ICAL consists of a Blank and 5 points	Daily initial calibration prior to sample analysis	Correlation coefficient (r) > 0.995	Recalibrate., perform instrument maintenance if cal. cannot conform to criteria, recalibrate.
	ICV	After each cal.	± 10% of true value	If the ICV is out of specification high report the elements that are < RL. For elements > RL, data cannot be reported. Reanalyze the ICV, if the recovery is acceptable, proceed with sample analysis. If the acceptance criteria are not met after reanalysis, re-digest and reanalyze the curve and all associated samples
	ICB	Immediately after the ICV	No analytes > RL	Reanalyze
	CCV	Immediately after the detection limit standard (CRA) and every 10 samples	± 20% of true value	"If the CCV is out of specification high and the sample is not < - RL, accept samples that report as non-detect. Data bracketing the CCV cannot be reported from other samples on the run (reanalyze). If the CCV is out of specification, it is read in duplicate. If both CCVs are within specification, the data from the last good CCV is reanalyzed. If one or both CCVs are still out of specification, then the run is terminated and the samples after the last good CCV must be analyzed on a new run
	CCB	Immediately after the CCV and at a frequency of every 10 samples	No analytes > RL	Data bracketing the CCB cannot be reported from the run (reanalyze). If the CCB is out of specification, it can be read in duplicate. If both CCBs are within specification, the data from the last good CCB is reanalyzed. If one or both CCBs are still out of specification, then the run is terminated and the samples after the last good CCB must be reanalyzed on a new run

QAPP Worksheet #24/25: Analytical Instrument Calibration/Analytical Instrument and Equipment Maintenance, Testing and Inspection

Instrument	Calibration Procedure/Range	Frequency of Calibration	Acceptance Criteria	CA by Analyst
GC/MS	GC/MS Tuning (VO)	Tune with bromofluorobenzene (BFB) before an ICAL or CCV	Per ion abundance criteria in method;	Retune, clean the source and/or change the column
	GC/MS Tuning (SVO)	Tune with decafluoro-triphenylphosphine (DFTPP) before an ICAL or CCV	Per ion abundance criteria in method; tailing factors: benzidine ≤ 2; pentachlorophenol ≤ 2	Retune, clean the source and/or change the column
	ICAL	After continuing calibration fails and beginning of analysis on instrument following major maintenance.	Minimum 5 standards. RSD for each compound should be ≤ 20%. A linear fit can be used if Correlation Coefficient (CC) is ≥ 0.99. A quadratic fit (6 points) can be used if the Coefficient of Determination (COD) is ≥ 0.99. For linear cal., the low cal. is re-fitted onto the curve; the recalculated conc. of the low cal. point must be ± 30% of the standard's true conc.to verify the linearity of the curve. If more than 10% of the compounds in the ICAL exceed 20% RSD and/or also do not meet the minimum CC or COD criteria, the analysis cannot proceed.	Perform more aggressive instrument maintenance and recalibrate.
	Retention Time Window Establishment	Once per ICAL and at beginning of the analytical sequence	Position is set using the midpoint standard of the ICAL curve when ICAL is performed. On days with no ICAL use the initial CCV	Not applicable
	Evaluation of Relative Retention Times (RRT)	With each sample	RRT of each reported analyte within ± 0.06 RRT units	Correct problem, then recalibrate

QAPP Worksheet #24/25: Analytical Instrument Calibration/Analytical Instrument and Equipment Maintenance, Testing and Inspection

	Ion ratio (SIM analysis)	Performed during ICAL	At least two ions monitored for each target analyte, and the mid-point of the calibration curve is used to establish ion ratio criteria for each compound. All monitored ions must be correctly integrated in order to achieve accurate ion ratios. The primary/secondary ion ratios and the reference mass spectrum should be updated from the mid-point ICAL standard.	Correct problem, then recalibrate
	ICV standard	Immediately following initial calibration.	Concentration level near midpoint of curve. Prepared using standard source different than used for initial calibration. Must contain all target analytes. Percent recoveries must be between 70-130% for target analytes except for "difficult" analytes which must exhibit percent recoveries between 40-160%.	Reanalyze the ICV and samples associated with the non-compliant ICV. If ICV fails again do system maintenance, recalibrate., and reanalyze samples
	CCV standard	Every 12 hours prior to the analysis of samples	Concentration level near midpoint of curve. Must contain all target analytes. %D must be $\leq$20 for each target analyte except for "difficult" analytes which must exhibit %Ds $\leq$60. Minimum RFs as per Table 4 of SW-846 8260C. Area counts of internal standards in continuing calibration must be between 50 – 200% of the area counts in the associated mid-level initial calibration standard.	Reanalyze CCV in duplicate immediately. If both pass, samples can be reported. If either fails or if immediate reanalysis of CCV in duplicate cannot be performed all samples since acceptable CCV must be reanalyzed. If the CCV fails high any associated samples that are non-detect (ND) can be reported

QAPP Worksheet #24/25: Analytical Instrument Calibration/Analytical Instrument and Equipment Maintenance, Testing and Inspection

Instrument	Calibration Procedure/Range	Frequency of Calibration	Acceptance Criteria	CA by Analyst
GC	Retention Time Windows	Prior to initial calibration and when a new GC column is installed	± 0.07 minutes.	Perform instrument maintenance and recalibrate.
	ICAL with a minimum 5 points.	After continuing cal. fails	5-point calibration. (A minimum of 5 unique peaks must be evaluated for PCBs). % RSD ≤ 20%, if > 20% then alternate fit linear with correlation coefficient of ≥ 0.99. If linear or non-linear regression used, verify the RL by recalculating concentrations in lowest calibration standard using the final calibration curve; recoveries must be 70-130%.	Perform more aggressive instrument maintenance and recalibrate.
	Breakdown Standard	Before ICAL and ongoing in the sequence after every 12 hours	DDT Breakdown ≤15%; Endrin Breakdown ≤15%	Perform instrument maintenance and recalibrate.
	ICV Standard	Immediately following initial calibration.	**C**oncentration level near midpoint of curve. Prepared using standard source different than used for initial calibration. Target analytes +/- 20% of the nominal concentration and within established retention time windows	Reanalyze the ICV and samples associated with the non-compliant ICV. If ICV fails again do system maintenance, recalibrate, and reanalyze samples after acceptable IC and ICV are established.
	CCV Standard	Prior to sample analysis, after every 20 injections or 12 hours, whichever comes first and at the end of the sequence	%D must be ≤20 for each analyte. Verify that all analytes fall within retention time windows. Area count of internal standard in continuing calibration must be within +50% of the average area count in the associated initial calibration.	All samples since last acceptable CCV must be reanalyzed. If the CCV fails high, any associated samples that are ND can be reported

QAPP Worksheet #24/25: Analytical Instrument Calibration/Analytical Instrument and Equipment Maintenance, Testing and Inspection

	Confirmation of positive results	All positive results	Cal. and QC criteria are the same for the confirmation analysis as for primary analysis. RPD for results between primary and confirmation is ≤ 40% D.	Flag outliers

QAPP Worksheet #24/25: Analytical Instrument Calibration/Analytical Instrument and Equipment Maintenance, Testing and Inspection

Instrument	Calibration Procedure/Range	Frequency of Calibration	Acceptance Criteria	CA by Analyst
LC/MS/MS	Tuning	Prior to ICAL	Calibrate the mass scale of the MS with the calibration compounds and procedures prescribed by the manufacturer.	Retune, perform maintenance and recalibrate
	Establish initial calibration with a minimum of five standards.	After continuing cal. fails	The LC/MS/MS system is calibrated using the IS technique. Must be forced through zero. A linear fit is acceptable if the coefficient of determination (r2) is greater than 0.99. When quantitated using the initial calibration curve, each calibration point, except the lowest point, for each analyte should calculate to be within 70-130% of its true value. The lowest CAL point should calculate to be within 50-150% of its true value.	Recalibrate., perform instrument maintenance. If cal. does not meet method criteria, recalibrate.
	ICV	Immediately following each initial calibration	Target compounds ≤ 20% drift	Reanalyze the ICV. If ICV fails again do system maintenance and recalibrate.
	RL Standard	Beginning of each sequence	50-150%R	Reanalyze. If fails again do system maintenance and recalibrate.
	CCV	Verify the initial calibration at the beginning and end of each group of analyses, and after every tenth sample during analyses.	The calculated amount for each analyte for medium level CCVs must be within ± 30% of the true value with an allowance of 10% of the reported analytes to be greater than 30%, but less than 40%. The calculated amount for each extracted internal standards (EIS) must be within ± 50% of the true value. The calculated amount for the lowest calibration point for each analyte must be within ± 50%. If the CCV fails because the calculated concentration is greater than 130% (150% for the low-level CCV) for a particular method analyte, and samples show no detection for that method analyte, nondetects may be reported without re-analysis.	Reanalyze the CCV. If CCV fails again do system maintenance and recalibrate.

QAPP Worksheet #24/25: Analytical Instrument Calibration/Analytical Instrument and Equipment Maintenance, Testing and Inspection

Instrument	Calibration Procedure/Range	Frequency of Calibration	Acceptance Criteria	CA by Analyst
pH electrode	Cal. using at least 3 points, sloped with pH 4, 7, and 10 buffers	Daily	percent slope between 92% and 102%	Correct the problem and recalibrate.
	CCV Standard	After each cal., every 10 samples, end of batch or if idle for four or more hours.	± 10% drift	Correct the problem, recalibrate. and reanalyze affected samples
UV Spectrophotometer	Cal. using at least 5 points	Every 3 months or when a new reagent is prepared	Correlation coefficient must be ≥0.995	Correct the problem and recalibrate.
	CCV Standard	Beginning of each batch, every 10 samples, and end of batch	± 10% drift	Correct the problem, recalibrate. and reanalyze affected samples
IC	ICAL with a minimum of 5 points.	Monthly or when CCV fails	r > 0.995; Level 1 standard must recover ≥ 50% of the true value	Perform more aggressive instrument maintenance and recalibrate.
	ICB	Immediately following each initial calibration	No analytes detected > MDL	Correct problem and reanalyze the ICB. Recalibrate. if needed
	ICV	Immediately following each initial calibration	Within ± 10% of the nominal concentration	Reanalyze.If ICV fails again do system maintenance and recalibrate.
	CCV	Beginning of each sequence, after every 10 field samples, and at the end of the sequence	Within ± 10% of the nominal concentration	Recalibrate.; reanalyze affected samples
	CCB	Beginning of each sequence, after every 10 field samples, and at the end of the sequence	No analytes detected > MDL	Recalibrate.; reanalyze affected samples

QAPP Worksheet #24/25: Analytical Instrument Calibration/Analytical Instrument and Equipment Maintenance, Testing and Inspection

Instrument	Calibration Procedure/Range	Frequency of Calibration	Acceptance Criteria	CA by Analyst
Total Organic Carbon Analyzer	ICAL with a minimum 5 points	Monthly or after CCV and/or CCB fails	$r^2 \geq 0.995$	Perform more aggressive instrument maintenance and recalibrate.
	ICB Standard	Immediately following each initial calibration	No analytes detected > RL	Perform more aggressive instrument maintenance and recalibrate.
	ICV Standard	Immediately following each initial calibration	Within ± 10% of the nominal concentration	Reanalyze the ICV. If ICV fails again do system maintenance and recalibrate.
	Total Inorganic Check Standard (TIC)	Daily	Within ± 20% of the nominal concentration	All affected samples are reanalyzed
	CCV Standard	If instrument is idle > 4 hours, after every 10 field samples, and at the end of the sequence	Within ± 10% of the nominal concentration	All affected samples are reanalyzed
	CCB Standard	If instrument is idle > 4 hours, after every 10 field samples, and at the end of the sequence	No analytes detected > RL	All affected samples are reanalyzed
	Tuning/Mass calibration Verification	Prior to ICAL		Retune and recalibrate.

QAPP Worksheet #24/25: Analytical Instrument Calibration/Analytical Instrument and Equipment Maintenance, Testing and Inspection

IC/MS/MS	ICAL	After continuing cal. fails.	A minimum of five calibration standards are required for a curve that extends over a single order of magnitude and a minimum of seven calibration standards are required if the curve covers two orders of magnitude. A correlation coefficient (r2) of 0.995 or better is acceptable. The calculated value for each standard equal to or less than the MRL should be within 50-150% of true value. All other calibration points should have a calculated value 80-120% within true value.	
	ICV Standard	Immediately following each initial calibration	%D≤20	Reanalyze the ICV. If ICV fails again do system maintenance and recalibrate.
	CCV	Beginning of each sequence, prior to sample analysis	%R 80-120	
	RL standard	Each day, prior to sample analyses	%R 50-150	
	Transition Ratio	Each standard, sample and QC sample.	3.08. A ratio less than 2.31 or greater than 3.85 should never be used for quantitation samples	Reanalyze sample, retune and/or recalibrate.

QAPP Worksheet #24/25: Analytical Instrument Calibration/Analytical Instrument and Equipment Maintenance, Testing and Inspection

Instrument	Calibration Procedure/Range	Frequency of Calibration	Acceptance Criteria	CA by Analyst
Gamma Spectroscopy	Initial: multi-point (8-12 isotopes across energy range) for each geometry	Initially and as required per method	For the energy calibration, the absolute value of the difference must be less than 1.0 and for the FWHM calibration, the absolute value of the difference must be less than 0.5. Verification source counted must be within 10% of known values for all isotopes.	Re-run initial calibration verification (ICV). Corrective action for out-of-control data might require instrument maintenance, re-analysis, using a new spike mix, or a more complex set of actions.
	Continuing: multi-point	Daily	Peak centroid energy ± 2 keV Peak centroid channel ± 4 channels FWHM based on manufacturers specifications Activity 10% of known or 3sigma	Immediately rerun check. If the check fails again, mark the detector out of service and make a note in the instrument logbook. The instrument can be returned to service following a successful instrument check.
Alpha Spectrometer	Initial: multi-point	Initially and then monthly	Energy Calibration Offset: 2300-2450 kiloelectron volt (keV) Energy Calibration Slope: 4.7-5.3 keV/channel Constant full width at half maximum (FWHM): 3-25 channels Average Efficiency: 3s standard deviation	The status board will be updated to reflect the “out of service” condition for that instrument and a logbook entry will be made. The condition will be investigated, and the instrument may be returned to service following a successful instrument calibration.
	Continuing: single point	Daily	FWHM 1-35 keV Peak centroid ± 15 channels of mean Peak energy ± 50 keV of mean Count rate ± 3% of mean	The failed check should be immediately rerun. If the check fails a second time the instrument is locked out of service and the cause will be investigated. The instrument status board will be updated to reflect the lockout condition and a logbook entry will be made. The instrument will be returned to service following a successful instrument check.

QAPP Worksheet #24/25: Analytical Instrument Calibration/Analytical Instrument and Equipment Maintenance, Testing and Inspection

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	CA	Responsible Person	SOP Reference
ICP - AES	As needed replacement of components	Cal. checks	Visual inspection of components	As needed maintenance	90-110% for the cal. checks	Re-cal.	GEL Analyst	GL-MA-E-013
CVAA	As needed replacement of components	Cal. checks	Visual inspection of components	As needed maintenance	90-110% for the cal. checks	Re-cal.	GEL Analyst t	GL-MA-E-010
ICPMS	As needed replacement of components	Cal. checks	Visual inspection of components	As needed maintenance	90-110% for the cal. checks	Re-cal.	GEL Analyst	GL-MA-E-014
Gas Flow Proportional Counter	Computer back up, print control charts, cleaning	Cal. checks	Visual inspection of components	As needed maintenance	As specified in SOP	Re-cal.	GEL Analyst	GL-RAD-A-018
GC/MS	Daily items may include septa replacement, injection port items, solvent replenishment, instrument tuning adjustment, etc.	CCV and tune standard per 12-hour tune period	Instrument resolving power, GC performanc e, and isomer specificity are monitored daily.	Maintenance is ongoing and performed as needed. Preventative maintenance such as septa replacement and solvent replenishment is performed daily.	ICV or CCV passes method criteria	Perform maintenance again; re-cal. if necessary. Documentatio n of item addressed is located in the instruments maintenance logbook.	Analysts or group leaders	2108, 2111

QAPP Worksheet #24/25: Analytical Instrument Calibration/Analytical Instrument and Equipment Maintenance, Testing and Inspection

GC with Electron Capture Detector (ECD)	Injection port maintenance; ECD maintenance	Cal. Check All analytes within +/- 20% of the nominal conc. And within established retention time windows	Visual inspection of components	As needed	ICAL after maintenance is within specifications	Perform maintenance again	Alpha GC analyst	2116, 2128, 2129

QAPP Worksheet #24/25: Analytical Instrument Calibration/Analytical Instrument and Equipment Maintenance, Testing and Inspection

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptanc e Criteria	CA	Responsible Person	SOP Reference
HRGC/ HRMS	Injection port, column, ion source, others as needed	Cal. Check	Visual inspection of components	As needed	ICAL or cal. verification passes method specifications	Perform additional maintenance prior to instrument cal. or cal. verification	Alpha Analysts	23528
pH Meter	As needed replacement of components	Cal. checks	Visual inspection of components	As needed maintenance/cal. checks every 10 injections	90-110% for cal. checks	Re-cal.	Alpha Analysts	2202
UV Spectrophoto meter	As needed replacement of components	Cal. checks	Visual inspection of components	As needed maintenance/cal. checks every 10 injections	90-110% for cal. checks	Re-cal.	Alpha Analysts	2212, 2225
IC	As needed replacement of components	Cal. checks	Visual inspection of components	As needed maintenance/ cal. checks every 10 injections	90-110% for cal. checks	Re-cal.	Alpha Analysts	2251
TOC Analyzer	As needed replacement of components	Cal. checks	Visual inspection of components	As needed maintenance/cal. checks every 10 injections	90-110% for cal. checks	Re-cal.	GEL Analysts	GL-GC-E-093

QAPP Worksheet #24/25: Analytical Instrument Calibration/Analytical Instrument and Equipment Maintenance, Testing and Inspection

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	CA	Responsible Person	SOP Reference
Gamma Spectroscopy	Liquid Nitrogen fill	NA, testing not performed	NA	Weekly	NA	NA	Laboratory Analyst or certified instrument technician	GL-RAD-I-010
Alpha Spectrometer	Vacuum Pump Oil replacement	NA, testing not performed	NA	semi-annually	NA	NA	Laboratory Analyst or certified instrument technician	GL-RAD-I-010
Alpha Spectrometer	Filter cleaning on the air intake of the instrument cabinet	NA, testing not performed	NA	quarterly	NA	NA	Laboratory Analyst or certified instrument technician	GL-RAD-I-010

QAPP Worksheet #26 Sample Handling System

Sample Collection, Packaging and Shipment
Sample Collection (Personnel/Organization): TBD/de maximis, O&M, Geosyntec, H&A
Sample Packaging (Personnel/Organization): TBD/de maximis, O&M, Geosyntec, H&A
Coordination of Shipment (Personnel/Organization): TBD/de maximis, O&M, Geosyntec, H&A
Type of Shipment/Carrier: AAW courier to AAW, Courier to GEL
Sample Receipt and Analysis
Sample Receipt (Personnel/Organization): Kim Bailey/AAW; Sample Receiving/GEL
Sample Custody and Storage (Personnel/Organization): Kim Bailey/AAW; Joanne Harley/GEL
Sample Preparation (Personnel/Organization): Jon Zygmuntowicz/AAW; Reference GEL Organizational Chart/GEL
Sample Determinative Analysis (Personnel/Organization): Lisa Westerlind/AAW; Reference GEL Organizational Chart /GEL
Sample Archiving
Field Sample Storage (no. of days from sample collection): 30 days from submittal of final report
Sample Extract/Digestate Storage (no. of days from extraction/digestion): 40 days
Biological Sample Storage (no. of days from sample collection): N/A
Sample Disposal
Personnel/Organization: Melissa White/AAW; Lonnie Morris/GEL
Number of Days from Analysis: 30-day minimum from final report.

Note: AAW and GEL will be responsible for all sample receipt, analysis, archiving, and disposal of samples.

QAPP Worksheet #27 Sample Custody Requirements

Sample Handling and Custody Requirements

Field and laboratory personnel will always be aware of the need to maintain all samples, whether in the field or in the laboratory, under strict chain of custody and in a manner to retain physical properties and chemical composition. This Worksheet details sample handling and custody requirements from collection to ultimate disposal.

Sample Handling (Sample Packaging, Shipping Containers and Sample Shipment, Sample Custody)

Samples for analysis by AAW will be picked up by AAW couriers and brought back to the laboratory. Samples for analysis by GEL will be packages and shipped by FedEx. Samples will be packaged for shipment as outlined below:

- Securely affix the sample label to the container with clear packing tape.
- Check the cap on the sample container to confirm that it is properly sealed.
- Wrap the sample container cap with clear packing tape to prevent the cap from becoming loose.
- Complete the chain-of-custody form with the required sampling information and confirm that the recorded information matches the sample labels. NOTE: If the designated sampler relinquishes the samples to other sampling or field personnel for packing or other purposes, the sampler will complete the chain-of-custody prior to this transfer. The appropriate personnel will sign and date the chain-of-custody form to document the sample custody transfer.
- Using duct tape, secure the outside drain plug at the bottom of the cooler.
- Wrap sample containers in bubble wrap or other cushioning material.
- Place 1 to 2 inches of cushioning material at the bottom of the cooler.
- Place the sealed sample containers into the cooler.
- Place ice in plastic bags and seal. Place loosely in the cooler. (Note: ice not needed for process vapor samples)
- Fill the remaining space in the cooler with cushioning material.
- Place chain-of-custody forms in a plastic bag and seal. Tape the forms to the inside of the cooler lid.
- Close the lid of the cooler, lock and secure with duct tape.
- Wrap strapping tape around both ends of the cooler at least twice.
- Mark the cooler on the outside with the shipping address and return address, affix “Fragile” labels and draw (or affix) arrows indicating “this side up.” Cover the labels with clear plastic tape.
- Place a signed custody seal over the sample cooler lid.

Samples will be packaged by the field personnel and transported as low-concentration environmental samples. Other samples will be hand delivered or delivered by an express carrier within 24- hours of the time of collection. Shipments will be accompanied by the chain-of-custody form identifying the contents. The original form will accompany the shipment; copies will be retained by the sampler for the sampling office records. If the samples are sent by common carrier, a bill of lading will be used. Receipts or bills of lading will be retained as part of the permanent project documentation. Commercial carriers are not required to sign off on

QAPP Worksheet #27 Sample Custody Requirements

the chain-of-custody form as long as the forms are sealed inside the sample cooler, and the custody seals remain intact.

Sample custody seals and packing materials for filled sample containers will be provided by the analytical laboratory. The filled, labeled and sealed containers will be placed in a cooler on ice and carefully packed to eliminate the possibility of container breakage.

Additional procedures for packing, handling and shipping environmental samples are presented in the Remedial Design Work Plan.

Field Custody Procedures

The objective of field sample custody is to protect samples from tampering from the time of sample collection through time of transport to the analytical laboratory. Persons will have custody of samples when the samples are in their physical possession, in their view after being in their possession, or in their physical possession and secured so they cannot be tampered with. In addition, when samples are secured in a restricted area accessible only to authorized personnel, they will be deemed to be in the custody of such authorized personnel.

Field custody documentation consists of both field logbooks and field chain-of-custody forms.

Field logbooks will provide the means of recording the data collecting activities that are performed. As such, entries will be described in as much detail as possible so that persons going to the site could reconstruct a particular situation without reliance on memory. Field logbooks will be bound field survey books or notebooks. Logbooks will be assigned to field personnel but will be stored in a secure location when not in use. Each logbook will be identified by the project-specific document number. The title page of each logbook will contain the following:

- person to whom the logbook is assigned
- logbook number
- project name
- project start date
- end date

Field Custody Procedures Continued

Entries into the logbook will contain a variety of information. At the beginning of each entry, the date, start time, weather conditions, names of all sampling team members present, level of personal protection being used and signature of the person making the entry will be provided. The names of visitors to the site and field sampling or investigation team personnel, as well as the purpose of their visit, will also be recorded in the field logbook.

Measurements made and samples collected will be recorded. Entries will be made in ink, with no erasures. If an incorrect entry is made, the information will be

QAPP Worksheet #27 Sample Custody Requirements

crossed out with one strike mark. The initials of the person making the correction and the date of the correction will be noted next to the correction. Whenever a sample is collected or a measurement is made, a detailed description of the location of the station will be recorded. The number of the photographs taken, if any, will also be noted. All equipment used to make measurements will be identified, along with the date of calibration.

Samples will be collected following the sampling procedures documented in FSP. The equipment used to collect samples will be noted, along with the time of sampling, sample description, depth at which the sample was collected, volume and number of containers. Sample identification numbers will be assigned prior to sample collection. Field duplicate samples, which will receive an entirely separate sample identification number, will be noted under sample description.

Sample Labels

Preprinted sample labels will be affixed to sample bottles prior to delivery at the sampling site. The following information is required on each sample label:

- project name
- date collected
- time collected
- location
- sampler
- analysis to be performed
- preservative
- sample number

Chain of Custody Record

Completed chain-of-custody forms will be required for all samples to be analyzed. Chain-of-custody forms will be initiated by the sampling crew in the field. The chain-of-custody forms will contain the unique sample identification number, sample date and military time, sample description, sample type, preservation (if any) and analyses required. The original chain-of-custody form will accompany the samples to the laboratory. Copies of the chain-of-custody will be made prior to shipment (or multiple copy forms will be used) for field documentation. The chain-of-custody forms will remain with the samples at all times. The samples and signed chain-of-custody forms will remain in the possession of the sampling crew until the samples are delivered to the express carrier (e.g., Federal Express), hand delivered to a mobile or permanent laboratory, or placed in secure storage.

Sample labels will be completed for each sample using waterproof ink. The labels will include the information listed in the section above. The completed sample

QAPP Worksheet #27 Sample Custody Requirements

labels will be affixed to each sample bottle and covered with clear tape.

Whenever samples are split with a government agency or other party, a separate chain-of-custody will be prepared for those samples and marked to identify the party with whom the samples are being split. The person relinquishing the samples to the facility or agency should request the representative's signature acknowledging sample receipt. If the representative is unavailable or refuses, this is noted in the "Received By" space.

Laboratory Custody Procedures

Upon sample receipt, laboratory personnel will be responsible for sample custody. The original field chain-of-custody form will accompany all samples requiring laboratory analysis. The laboratory will use chain-of-custody guidelines described in the USEPA guidance documents. Samples will be kept secured in the laboratory until all stages of analysis are complete. All laboratory personnel having samples in their custody will be responsible for documenting and maintaining sample integrity.

Immediately upon sample receipt, the laboratory sample custodian will verify the integrity of the cooler seal, open the cooler, record cooler temperature on chain of custody, and compare the contents against the field chain-of-custody. If a sample container is missing, a sample container is received broken, the sample is in an inappropriate container, or the sample has not been preserved by appropriate means, de maximis will be notified. The laboratory sample custodian will be responsible for logging the samples in, assigning a unique laboratory identification number to each sample, labeling the sample bottle with the laboratory identification number, and moving the sample to an appropriate storage location to await analysis. The project name, field sample code, date sampled, date received, analysis required, storage location and date, and action for final disposition will be recorded in the laboratory tracking system. Relevant custody documentation will be placed in the project file.

QAPP Worksheet #28-1 QC Samples — Ammonia Nitrogen

Matrix	Aqueous	**Analytical Method/ SOP Reference**	SM4500 NH3-BH / 2206	**No. of Sample Locations**	See Footnote 2 below	
Analytical Group	Ammonia Nitrogen	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020	**Analytical Organization**	AAW			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Field duplicate	1 per 20 field samples	RPD ≤ 30%	Qualify data as needed	Polly Newbold or designee, ddms	Precision	RPD < 30% Results ≥ RL
Method blanks	1 per analytical batch	< RL	Qualify data as needed or reanalysis of batch	Lab personnel	Accuracy/bias contamination	Sample concentrations ≤ blank concentration will be qualified not detected (U)
Field/Equipment blanks	1 per 20 field samples	< RL	Qualify data as needed	Polly Newbold or designee, ddms	Accuracy/bias contamination	Sample concentrations ≤ blank concentration will be qualified not detected (U)
MS	1 per analytical batch	%R 90-110	If the LCS meets acceptance criteria, no corrective action required. Qualify data as needed.	Lab and/or Polly Newbold or designee, ddms	Accuracy/bias	%R 90-110
LCS	1 per analytical batch	%R 90-110	If sufficient sample volume, reanalyze entire batch. Otherwise, qualify data as needed	Lab and/or Polly Newbold or designee, ddms	Accuracy/bias	%R 90-110%

QAPP Worksheet #28-1 QC Samples — Ammonia Nitrogen

Matrix	Aqueous	**Analytical Method/ SOP Reference**	SM4500 NH3-BH / 2206	**No. of Sample Locations**	See Footnote 2 below	
Analytical Group	Ammonia Nitrogen	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020	**Analytical Organization**	AAW			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Lab Duplicate	1 per analytical batch	RPD≤20	Qualify data as needed	Lab personnel	Precision	RPD≤20

[1]- See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]-See RDWP Appendices (Site-Wide Soils and Sediments-A, In-Situ Sequestration of DU & U-B, Holding Basin Containment-C, 1,4-dioxane and VOCs in Bedrock Groundwater-D) for PDIs, IPs and Appendix I-FSP.

QAPP Worksheet #28-1 QC Samples — VOCs

Matrix	Aqueous, Leachate	**Analytical Method/ SOP Reference**	SW846 8260D /GL-OA-E-038	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	VOCs	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	H&A			
Sampling SOP[1]	NMI-S-001 - NMI-S-007	**Analytical Organization**	GEL			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Field duplicate	1 per 20 field samples	RPD < 30%	Qualify data as needed	Polly Newbold or designee, ddms	Precision	RPD ≤ 30% for concentrations ≥RL
Surrogates	3 per sample	Laboratory acceptance limits[3]	Reanalyze sample, surrogate high sample ND, report data and qualify	Lab personnel	Accuracy/bias	%R, 70-130%, %R<10 non-detects will be rejected (R)
Method blanks	1 per analytical batch	One per preparation batch	No target analytes detected greater than one-half RL and 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater). No laboratory common contaminants detected greater than RL.	Correct problem, then re- reanalyze method blank and all samples processed with the contaminated blank	Accuracy/bias contamination	Sample concentrations ≤ blank concentration* will be qualified not detected (U)

QAPP Worksheet #28-1 QC Samples — VOCs

Matrix	Aqueous, Leachate	**Analytical Method/ SOP Reference**	SW846 8260D /GL-OA-E-038	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	VOCs	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	H&A			
Sampling SOP[1]	NMI-S-001 - NMI-S-007	**Analytical Organization**	GEL			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Tumble Blanks	One per tumble batch	No target analytes detected greater than one-half RL and 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater).	Correct problem, then re- reanalyze method blank and all samples processed with the contaminated blank	Correct problem, then re- reanalyze method blank and all samples processed with the contaminated blank	Accuracy/bias contamination	Sample concentrations ≤ blank concentration* will be qualified not detected (U)
LCS/LCSD*	1/batch	Laboratory acceptance limits[3]	If sufficient sample volume, reanalyze entire batch. Otherwise, qualify data as needed	Lab personnel	Accuracy/Bias/ Precision	%R 70-130, RPD≤20
Internal standard	At least three/sample	RT within ±30 seconds from RT of initial calibration midpoint standard; area counts within -50% to +100% of initial calibration midpoint standard	Reanalyze sample	Lab personnel	Precision	Area response 50-200% of CC +/- 0.06 RRT

QAPP Worksheet #28-1 QC Samples — VOCs

Matrix	Aqueous, Leachate	**Analytical Method/ SOP Reference**	SW846 8260D /GL-OA-E-038	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	VOCs	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	H&A			
Sampling SOP[1]	NMI-S-001 - NMI-S-007	**Analytical Organization**	GEL			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
MS/MSD	1/batch	Laboratory acceptance limits[3]	If the LCS meets acceptance criteria, no corrective action required. Qualify data as needed.	Lab and/or Polly Newbold or designee, ddms	Accuracy/Bias/ Precision	%R 70-130, RPD≤50

*-Methylene chloride and toluene ≤2X blank concentration
[1]- See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]-See RDWP Appendices (Site-Wide Soils and Sediments-A, In-Situ Sequestration of DU & U-B, Holding Basin Containment-C, 1,4-dioxane and VOCs in Bedrock Groundwater-D) for PDIs, IPs and Appendix I-FSP.
[3]-laboratory acceptance limits are based on Department of Defense (DoD) Quality Systems Manual (QSM)

QAPP Worksheet #28-2 QC Samples — Anions – chloride, fluoride, sulfate

Matrix	Aqueous	**Analytical Method/ SOP Reference**	EPA 300.0 / 2214	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	Anions – bromide, chloride, fluoride, sulfate	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020	**Analytical Organization**	AAW			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Field duplicate	1 per 20 field samples	RPD $\leq$ 30%	Qualify data as needed	Polly Newbold or designee, ddms	Precision	RPD $\leq$ 30% for concentrations ≥ RL
Method blanks	1 per analytical batch	< RL	Qualify data as needed or reanalysis of batch	Lab personnel	Accuracy/bias contamination	Sample concentrations $\leq$ blank concentration will be qualified not detected (U)
Field, Equipment blanks	1 per 20 field samples	< RL	Qualify data as needed	Polly Newbold or designee, ddms	Accuracy/bias contamination	Sample concentrations $\leq$ blank concentration will be qualified not detected (U)
MS	1 per 20 field samples of similar matrix	%R 90-110	If LCS acceptable no corrective action required; qualify data as needed.	Lab and/or Polly Newbold or designee, ddms	Accuracy/bias	%R 90-110
LCS	1 per analytical batch	%R 90-110	If sufficient sample volume, re-extract entire batch. Otherwise, qualify data as needed	Lab and/or Polly Newbold or designee, ddms	Accuracy/bias	%R 90-100

QAPP Worksheet #28-2 QC Samples — Anions – chloride, fluoride, sulfate

Matrix	Aqueous	**Analytical Method/ SOP Reference**	EPA 300.0 / 2214	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	Anions – bromide, chloride, fluoride, sulfate	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020	**Analytical Organization**	AAW			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Lab duplicate	1 per analytical batch	RPD≤20	Qualify data as needed	Lab personnel	Precision	RPD≤20

[1]- See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP
[2]-See RDWP Appendices (Site-Wide Soils and Sediments-A, In-Situ Sequestration of DU & U-B, Holding Basin Containment-C, 1,4-dioxane and VOCs in Bedrock Groundwater-D) for PDIs, IPs and Appendix I-FSP.

QAPP Worksheet #28-2 QC Samples — SVOCs

Matrix	Aqueous, Leachate	**Analytical Method/ SOP Reference**	SW846 8270E / GL-OA-E-009	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	SVOCs	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	H&A			
Sampling SOP[1]	NMI-S-001 - NMI-S-007	**Analytical Organization**	GEL			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Field duplicate	1 per 20 field samples	RPD $\leq$ 30%	Qualify data as needed	Polly Newbold or designee, ddms	Precision	RPD $\leq$ 50% for concentrations $\geq$RL
Surrogates	3 BN/3 AC per sample	Laboratory acceptance limits[3]	Reanalyze and/or re-extract sample, surrogate high sample ND, report data and qualify	Lab personnel	Accuracy/bias	%R, 70-130%, %R<10 non-detects will be rejected (R)
Method blanks	1 per analytical batch	No target analytes detected greater than one-half RL and 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater). No laboratory common contaminants detected greater than RL.	Correct problem, then re- reanalyze method blank and all samples processed with the contaminated blank	Lab personnel	Accuracy/bias contamination	Sample concentrations < blank concentration will be qualified not detected (U)

QAPP Worksheet #28-2 QC Samples — SVOCs

Matrix	Aqueous, Leachate	**Analytical Method/ SOP Reference**	SW846 8270E / GL-OA-E-009	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	SVOCs	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	H&A			
Sampling SOP[1]	NMI-S-001 - NMI-S-007	**Analytical Organization**	GEL			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Tumble Blanks	One per tumble batch	No target analytes detected greater than one-half RL and 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater).	Correct problem, then re- reanalyze method blank and all samples processed with the contaminated blank	Lab personnel	Accuracy/bias	Sample concentrations < blank concentration will be qualified not detected (U)
LCS/LCSD*	1/batch	Laboratory acceptance limits[3]	If sufficient sample volume, re-extract entire batch. Otherwise, qualify data as needed	Lab personnel	Accuracy/Bias/ Precision	%R 70-130, RPD≤20
Internal standard	At least three/sample	RT within ±30 seconds from RT of initial calibration midpoint standard; area counts within -50% to +100% of initial calibration midpoint standard	Correct problem, then re- reanalyze affected samples	Lab Manager/ Analyst	Precision	Area response 50-200% of CC +/- 0.06 RRT

QAPP Worksheet #28-2 QC Samples — SVOCs

Matrix	Aqueous, Leachate	**Analytical Method/ SOP Reference**	SW846 8270E / GL-OA-E-009	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	SVOCs	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	H&A			
Sampling SOP[1]	NMI-S-001 - NMI-S-007	**Analytical Organization**	GEL			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
MS/MSD	1/batch	Laboratory acceptance limits[3]	Identify problem; if not related to matrix interference, re-reanalyze MS/MSD and all associated batch samples	Lab and/or Polly Newbold or designee, ddms	Accuracy/Bias/ Precision	%R 70-130, RPD≤20

[1]- See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]-See RDWP Appendices (Site-Wide Soils and Sediments-A, In-Situ Sequestration of DU & U-B, Holding Basin Containment-C, 1,4-dioxane and VOCs in Bedrock Groundwater-D) for PDIs, IPs and Appendix I-FSP.
[3]- laboratory acceptance limits are based on Department of Defense (DoD) Quality Systems Manual (QSM)

QAPP Worksheet #28-3 QC Samples — SVOCs-1,4-Dioxane (SIM)

Matrix	Aqueous	**Analytical Method/ SOP Reference**	SW846 8270E SIM GL-OA-E-009	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	SVOCs 1,4-dioxane only	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020	**Analytical Organization**	GEL			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Field duplicate	1 per 20 field samples	RPD < 20%	Qualify data as needed	Polly Newbold or designee, ddms	Precision — overall	RPD < 30% For compounds >RL.
Surrogate[4]	1 per sample	Laboratory acceptance limit3 (1,4-dioxane-d8)	Re-extract and/or analyze sample	Lab personnel	Accuracy/bias	%R<10 non-detects will be rejected (R)
Method blanks	1 per analytical batch	No target analytes detected greater than one-half RL and 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater). No laboratory common contaminants detected greater than RL.	Correct problem, then re- reanalyze method blank and all samples processed with the contaminated blank	Lab and/or Polly Newbold or designee, ddms	Accuracy/bias contamination	Sample concentrations < blank concentration will be qualified not detected (U)

QAPP Worksheet #28-3 QC Samples — SVOCs-1,4-Dioxane (SIM)

Matrix	Aqueous	**Analytical Method/ SOP Reference**	SW846 8270E SIM GL-OA-E-009	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	SVOCs 1,4-dioxane only	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020	**Analytical Organization**	GEL			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Field, Equipment blanks	1 per 20 field samples	< RL	Qualify data as needed	Polly Newbold or designee, ddms	Accuracy/bias contamination	Sample concentrations < blank concentration will be qualified not detected (U)
LCS/LCSD*	1 per analytical batch	70-130%. RPD <30	If sufficient sample volume, re-extract entire batch. Otherwise, qualify data as needed	Lab personnel	Accuracy/bias/ Precision	70-130%. RPD <20
MS/MSD	One MS/MSD pair per preparation batch per matrix	70-130%. RPD <30	Identify problem; if not related to matrix interference, re-reanalyze MS/MSD and all associated batch samples	Lab personnel	Accuracy/bias/ Precision	70-130%. RPD <30

QAPP Worksheet #28-3 QC Samples — SVOCs-1,4-Dioxane (SIM)

Matrix	Aqueous	**Analytical Method/ SOP Reference**	SW846 8270E SIM GL-OA-E-009	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	SVOCs 1,4-dioxane only	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020	**Analytical Organization**	GEL			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Internal standard	Every field sample and QC samples	RT within ±30 seconds from RT of initial calibration midpoint standard; area counts within +/- 50% from the average are measured during initial calibration and within +/- 30% from the most recent CCV.	Correct problem, then re- reanalyze affected samples	Lab personnel	Precision	RT within ±30 seconds from RT of initial calibration midpoint standard; area counts within +/- 50% from the average are measured during initial calibration and within +/- 30% from the most recent CCV.

[1]- See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]-See RDWP Appendices (Site-Wide Soils and Sediments-A, In-Situ Sequestration of DU & U-B, Holding Basin Containment-C, 1,4-dioxane and VOCs in Bedrock Groundwater-D) for PDIs, IPs and Appendix I-FSP.
[3]-laboratory acceptance limits are based on Department of Defense (DoD) Quality Systems Manual (QSM)
[4]- laboratory does not perform isotope dilution. Isotope dilution calculation will be performed during validation using 1,4-dioxane-d8

QAPP Worksheet #28-3 QC Samples — Perchlorate

Matrix	Aqueous	**Analytical Method/ SOP Reference**	EPA 332.0 / 2251	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	Perchlorate	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020	**Analytical Organization**	AAW			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Field duplicate	1 per 20 field samples	RPD ≤ 30%	Qualify data as needed	Polly Newbold or designee, ddms	Precision	RPD ≤ 30% For concentrations ≥ RL
Method blanks	1 per analytical batch	< RL	Qualify data as needed or reanalysis of batch	Lab personnel	Accuracy/bias contamination	Sample concentrations ≤ blank concentration will be qualified not detected (U)
Field, Equipment blanks	1 per 20 field samples	< RL	Qualify data as needed	Polly Newbold or designee, ddms	Accuracy/bias contamination	Sample concentrations ≤ blank concentration will be qualified not detected (U)
MS/MSD	1 per 20 field samples of similar matrix	80-120%, 50-150% if spiked at the RL RPD≤30	If the LCS meets acceptance criteria, no corrective action required. Qualify data as needed.	Lab personnel	Accuracy/Bias/ Precision	80-120%, 50-150% if spiked at the RL RPD<30
LCS/LCSD	1 per analytical batch	%R 80-120 RPD≤20	If sufficient sample volume, re-extract entire batch. Otherwise, qualify data as needed	Lab personnel	Accuracy/Bias/ Precision	%R 80-120 RPD ≤ 20%
Internal Standard	Each standard, sample and QC sample	±30% of daily CCV	Reanalyze; qualify as needed	Lab personnel	Precision	±30% of daily CCV

QAPP Worksheet #28-3 QC Samples — Perchlorate

Matrix	Aqueous	**Analytical Method/ SOP Reference**	EPA 332.0 / 2251	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	Perchlorate	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020	**Analytical Organization**	AAW			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Lab duplicate	1 per analytical batch if MSD not performed	RPD≤20	Qualify data as needed	Lab personnel	Precision	RPD≤20

1- See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP
2-See RDWP Appendices (Site-Wide Soils and Sediments-A, In-Situ Sequestration of DU & U-B, Holding Basin Containment-C, 1,4-dioxane and VOCs in Bedrock Groundwater-D) for PDIs, IPs and Appendix I-FSP.

QAPP Worksheet #28-4 QC Samples —Pesticides

Matrix	Aqueous, Leachate	**Analytical Method/ SOP Reference**	SW846 8081B GL-OA-E-041	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	Pesticides	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	H&A			
Sampling SOP[1]	NMI-S-001 – NMI-S-007	**Analytical Organization**	GEL			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Field Duplicates	1 per 20 field samples	RPD ≤ 50%	Qualify data as required	Polly Newbold or designee, ddms	Precision	RPD < 50% for concentrations >RL
Surrogate Spike	2 per sample	Laboratory acceptance limits[3]	If sufficient sample volume is available, re-extract and reanalyze the samples	Lab and/or Polly Newbold or designee, ddms	Accuracy/Bias	%R, 70-130%, %R<10 non-detects will be rejected ®
Method Blank	1 per extraction batch	No target analytes detected greater than one-half RL and 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater).	Correct problem, then re- reanalyze method blank and all samples processed with the contaminated blank	Lab personnel	Accuracy/Bias/ Contamination	Sample concentrations ≤ blank concentration will be qualified not detected (U)
Tumble Blanks	One per tumble batch	No target analytes detected greater than one-half RL and 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater).	Correct problem, then re- reanalyze method blank and all samples processed with the contaminated blank	Lab personnel	Accuracy/Bias/ Contamination	Sample concentrations < blank concentration will be qualified not detected (U)

QAPP Worksheet #28-4 QC Samples —Pesticides

Matrix	Aqueous, Leachate	**Analytical Method/ SOP Reference**	SW846 8081B GL-OA-E-041	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	Pesticides	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	H&A			
Sampling SOP[1]	NMI-S-001 – NMI-S-007	**Analytical Organization**	GEL			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
LCS/LCSD	1 per extraction batch	Laboratory acceptance limits[3]	If sufficient sample volume is available, re-extract and reanalyze the samples; otherwise qualify data as needed	Lab and/or Polly Newbold or designee, ddms	Accuracy/Bias/ Precision	70-130% RPD< 20
MS/MSD	1 per extraction batch	Laboratory acceptance limits[3]	If the LCS meets acceptance criteria, no corrective action required. Qualify data as needed.	Lab and/or Polly Newbold or designee, ddms	Accuracy/Bias/ Precision	70-130% RPD $\leq$ 30%
Identification and Quantitation	Each Sample	Minimum3 peaks used for quantitation. RPD<45 between columns	Reanalyze sample if RPD>100 at a dilution. Both runs must be reported	Lab Personnel	Accuracy	Minimum3 peaks used for quantitation. RPD<25 between columns.

[1]- See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]-See RDWP Appendices (Site-Wide Soils and Sediments-A, In-Situ Sequestration of DU & U-B, Holding Basin Containment-C, 1,4-dioxane and VOCs in Bedrock Groundwater-D) for PDIs, IPs and Appendix I-FSP.
[3]- laboratory acceptance limits are based on Department of Defense (DoD) Quality Systems Manual (QSM)

QAPP Worksheet #28-4 QC Samples — Alkalinity

Matrix	Alkalinity	**Analytical Method/ SOP Reference**	SM2320B / 2213	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	Alkalinity	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec			
Sampling SOP	NMI-GW-001 - NMI-GW-020	**Analytical Organization**	AAW			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Field Duplicates	1 per 20 field samples	RPD≤30	Qualify data as required	Polly Newbold or designee, ddms	Precision	RPD≤30 for concentrations ≥RL
Method Blank	1 per batch	<RL	If sufficient sample volume is available, reanalyze the samples	Lab and/or Polly Newbold or designee, ddms	Accuracy/ contamination	Sample concentrations ≤ blank concentration will be qualified not detected (U)
Field/Equipment blanks	1 per 20 field samples	<RL	If sufficient sample volume is available, reanalyze the samples	Lab and/or Polly Newbold or designee, ddms	Accuracy/ contamination	Sample concentrations ≤ blank concentration will be qualified not detected (U)
LCS	1 per batch	%R 90-110	If sufficient sample volume, reanalyze entire batch. Otherwise, qualify data as needed	Lab personnel	Accuracy/Bias	%R 980-110
MS	1 per batch	%R 80-120	If the LCS meets acceptance criteria, no corrective action required. Qualify data as needed.	Lab personnel	Accuracy/Bias	%R 80-120

QAPP Worksheet #28-4 QC Samples — Alkalinity

Matrix	Alkalinity	**Analytical Method/ SOP Reference**	SM2320B / 2213	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	Alkalinity	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec			
Sampling SOP	NMI-GW-001 - NMI-GW-020	**Analytical Organization**	AAW			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Lab Duplicates	1 per batch	RPD≤20	Qualify data as required	Lab personnel	Precision	RPD≤20

[1]- See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]-See RDWP Appendices (Site-Wide Soils and Sediments-A, In-Situ Sequestration of DU & U-B, Holding Basin Containment-C, 1,4-dioxane and VOCs in Bedrock Groundwater-D) for PDIs, IPs and Appendix I-FSP.

QAPP Worksheet #28-5 QC Samples —Herbicides

Matrix	Leachate	**Analytical Method/ SOP Reference**	SW846 8151A / GL-OA-E-011	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	Pesticides	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	H&A			
Sampling SOP[1]	NMI-S-001 – NMI-S-007	**Analytical Organization**	GEL			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Field Duplicates	1 per 20 field samples	RPD ≤ 50%	Qualify data as required	Polly Newbold or designee, ddms	Precision	RPD < 50% for concentrations >RL
Surrogate Spike	2 per sample	Laboratory acceptance limits[3]	If sufficient sample volume is available, re-extract and reanalyze the samples	Lab and/or Polly Newbold or designee, ddms	Accuracy/Bias	%R, 70-130%, %R<10 non-detects will be rejected ®
Method Blank	One per preparation batch	No target analytes detected greater than one-half RL and 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater).	Correct problem, then re- reanalyze method blank and all samples processed with the contaminated blank	Lab personnel	Accuracy/Bias/ Contamination	Sample concentrations < blank concentration will be qualified not detected (U)
Tumble Blanks	One per tumble batch	No target analytes detected greater than one-half RL and 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater).	Correct problem, then re- reanalyze method blank and all samples processed with the contaminated blank	Lab personnel	Accuracy/Bias/ Contamination	Sample concentrations < blank concentration will be qualified not detected (U)

QAPP Worksheet #28-5 QC Samples —Herbicides

Matrix	Leachate	**Analytical Method/ SOP Reference**	SW846 8151A / GL-OA-E-011	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	Pesticides	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	H&A			
Sampling SOP[1]	NMI-S-001 – NMI-S-007	**Analytical Organization**	GEL			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
LCS/LCSD	1 per extraction batch	Laboratory acceptance limits[3]	If sufficient sample volume is available, re-extract and reanalyze the samples; otherwise qualify data as needed	Lab and/or Polly Newbold or designee, ddms	Accuracy/Bias/ Precision	70-130% RPD< 20
MS/MSD	1 per extraction batch	Laboratory acceptance limits[3]	If the LCS meets acceptance criteria, no corrective action required. Qualify data as needed.	Lab and/or Polly Newbold or designee, ddms	Accuracy/Bias/ Precision	70-130% RPD $\leq$ 30%
Identification and Quantitation	Each Sample	Minimum3 peaks used for quantitation. RPD<45 between columns	Reanalyze sample if RPD>100 at a dilution. Both runs must be reported	Lab Personnel	Accuracy	Minimum3 peaks used for quantitation. RPD<25 between columns.

[1]- See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]-See RDWP Appendices (Site-Wide Soils and Sediments-A, In-Situ Sequestration of DU & U-B, Holding Basin Containment-C, 1,4-dioxane and VOCs in Bedrock Groundwater-D) for PDIs, IPs and Appendix I-FSP.
[3]- laboratory acceptance limits are based on Department of Defense (DoD) Quality Systems Manual (QSM)

QAPP Worksheet #28-5 QC Samples — Nitrate/Nitrite

Matrix	Aqueous	**Analytical Method/ SOP Reference**	SM4500 NO_3 F NO^2 B /2217	**No. of Sample Locations**	See footnote2 below	
Analytical Group	Nitrate, Nitrite, Nitrate/Nitrite	**Sampler's Name**	NTBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020	**Analytical Organization**	AAW			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Field duplicate	1 per 20 field samples	RPD ≤ 30%	Qualify data as needed	Polly Newbold or designee, ddms	Precision - overall	RPD ≤ 30% For concentrations ≥ RL
Method blanks	1 per analytical batch	< RL	Qualify data as needed or reanalysis of batch	Lab personnel	Accuracy/bias contamination	Sample concentrations ≤ blank concentration will be qualified not detected (U)
Field, Equipment blanks	1 per 20 field samples	< RL	Qualify data as needed	Polly Newbold or designee, ddms	Accuracy/bias contamination	Sample concentrations ≤ blank concentration will be qualified not detected (U)
MS	1 per analytical batch	Laboratory acceptance limit	Qualify data as needed	Lab personnel	Accuracy/bias	%R 75-125
LCS	1 per analytical batch	%R 90-110	If sufficient sample is available, reanalyze samples; otherwise qualify data as needed	Lab personnel	Accuracy/bias	%R 90-110

QAPP Worksheet #28-5 QC Samples — Nitrate/Nitrite

Matrix	Aqueous	**Analytical Method/ SOP Reference**	SM4500 NO_3 F NO^2 B /2217	**No. of Sample Locations**	See footnote2 below	
Analytical Group	Nitrate, Nitrite, Nitrate/Nitrite	**Sampler's Name**	NTBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020	**Analytical Organization**	AAW			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Lab duplicate	1 per analytical batch	Laboratory acceptance limit	Qualify data as needed	Lab personnel	Precision	RPD $\leq$ 30% Results ≥ RL

Note: *An analytical batch is defined as no more than 20 field samples, blanks, MS and LCS.
[1]-See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]-See RDWP Appendices (Site-Wide Soils and Sediments-A, In-Situ Sequestration of DU & U-B, Holding Basin Containment-C, 1,4-dioxane and VOCs in Bedrock Groundwater-D) for PDIs, IPs and Appendix I-FSP.

QAPP Worksheet #28-6 QC Samples — o-Phosphate, Total Phosphorous

Matrix	Aqueous	**Analytical Method/ SOP Reference**	SM4500 OP-E/2225	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	o-Phosphate, Total Phosphorous	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020	**Analytical Organization**	AAW			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Field duplicate	1 per 20 field samples	RPD $\leq$ 30%	Qualify data as needed	Polly Newbold or designee, ddms	Precision	RPD $\leq$ 30% For concentrations ≥ RL
Method blanks	1 per analytical batch	< RL	Qualify data as needed or reanalyze batch	Lab personnel	Accuracy/bias contamination	Sample concentrations $\leq$ blank concentration will be qualified not detected (U)
Field, Equipment blanks	1 per 20 field samples	< RL	Qualify data as needed	Polly Newbold or designee, ddms	Accuracy/bias contamination	Sample concentrations $\leq$ blank concentration will be qualified not detected (U)
MS	1 per analytical batch	%R 80-120	Qualify data as needed	Lab personnel	Accuracy/bias	%R 80-120

QAPP Worksheet #28-6 QC Samples — o-Phosphate, Total Phosphorous

Matrix	Aqueous	**Analytical Method/ SOP Reference**	SM4500 OP-E/2225	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	o-Phosphate, Total Phosphorous	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020	**Analytical Organization**	AAW			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
LCS	1 per analytical batch	%R 90-110	If sufficient sample is available, reanalyze samples; otherwise qualify data as needed.	Lab personnel	Accuracy/bias	%R 90-110

[1]-See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP
[2]-See RDWP Appendices (Site-Wide Soils and Sediments-A, In-Situ Sequestration of DU & U-B, Holding Basin Containment-C, 1,4-dioxane and VOCs in Bedrock Groundwater-D) for PDIs, IPs and Appendix I-FSP.

QAPP Worksheet #28-6 QC Samples — Total Phosphorous

Matrix	Aqueous	**Analytical Method/ SOP Reference**	EPA 365.4 / GL-GC-E-103	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	o-Phosphate, Total Phosphorous	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020	**Analytical Organization**	AAW			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Field duplicate	1 per 20 field samples	RPD $\leq$ 30%	Qualify data as needed	Polly Newbold or designee, ddms	Precision	RPD $\leq$ 30% For concentrations ≥ RL
Method blanks	1 per analytical batch	< RL	Qualify data as needed or reanalyze batch	Lab personnel	Accuracy/bias contamination	Sample concentrations $\leq$ blank concentration will be qualified not detected (U)
Field, Equipment blanks	1 per 20 field samples	< RL	Qualify data as needed	Polly Newbold or designee, ddms	Accuracy/bias contamination	Sample concentrations $\leq$ blank concentration will be qualified not detected (U)
MS	1 per analytical batch	%R 70-13-	Qualify data as needed	Lab personnel	Accuracy/bias	%R 80-120

QAPP Worksheet #28-6 QC Samples — Total Phosphorous

Matrix	Aqueous	**Analytical Method/ SOP Reference**	EPA 365.4 / GL-GC-E-103	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	o-Phosphate, Total Phosphorous	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020	**Analytical Organization**	AAW			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
LCS	1 per analytical batch	%R 80-120	If sufficient sample is available, reanalyze samples; otherwise qualify data as needed.	Lab personnel	Accuracy/bias	%R 90-110

[1]-See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP
[2]-See RDWP Appendices (Site-Wide Soils and Sediments-A, In-Situ Sequestration of DU & U-B, Holding Basin Containment-C, 1,4-dioxane and VOCs in Bedrock Groundwater-D) for PDIs, IPs and Appendix I-FSP.

QAPP Worksheet #28-7 QC Samples — pH

Matrix	Aqueous	**Analytical Method/ SOP Reference**	SM4500 H-B / GL-GC-E-008	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	pH	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020	**Analytical Organization**	GEL			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Lab Duplicates	1 per 20 field samples	RPD≤5	Qualify data as needed	Lab and/or Polly Newbold or designee, ddms	Precision	±0.2 pH units
LCS	1 per 20 field samples	%R 99-101	Repeat LCS, Reanalyze all samples in batch	Lab personnel	Accuracy	%R 99-101

[1] See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]--See RDWP Appendices (Site-Wide Soils and Sediments-A, In-Situ Sequestration of DU & U-B, Holding Basin Containment-C, 1,4-dioxane and VOCs in Bedrock Groundwater-D) for PDIs, IPs and Appendix I-FSP.

QAPP Worksheet #28-7A QC Samples — pH

Matrix	Aqueous	**Analytical Method/ SOP Reference**	SW846 9040C / 2202	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	pH	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020	**Analytical Organization**	AAW			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Lab Duplicates	1 per 20 field samples	RPD≤5	Qualify data as needed	Lab and/or Polly Newbold or designee, ddms	Precision	±0.2 pH units

[1] See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]--See RDWP Appendices (Site-Wide Soils and Sediments-A, In-Situ Sequestration of DU & U-B, Holding Basin Containment-C, 1,4-dioxane and VOCs in Bedrock Groundwater-D) for PDIs, IPs and Appendix I-FSP.

QAPP Worksheet #28-8 QC Samples — Perchlorate

Matrix	Aqueous	**Analytical Method/ SOP Reference**	EPA 6850M / GL-OE-067	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	Perchlorate	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020	**Analytical Organization**	GEL			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Field duplicate	1 per 20 field samples	RPD < 30%	Qualify data as needed	Polly Newbold or designee, ddms	Precision	RPD < 30% For concentrations ≥ RL
Method blanks	One per batch of 20 or fewer samples per matrix	No analytes detected >½ LOQ or >1/10th the amount measured in any sample or 1/10th the regulatory limit (whichever is greater).	Correct problem; if required, re-prepare and/or reanalyze method blank and all QC samples and samples processed with the contaminated blank. If reanalysis cannot be performed, data must be qualified and explained in the case narrative.	Lab personnel	/bias contamination	Sample concentrations < blank concentration will be qualified not detected (U)
Field, Equipment blanks	1 per 20 field samples	< RL	Qualify data as needed	Polly Newbold or designee, ddms	Accuracy/bias contamination	Sample concentrations < blank concentration will be qualified not detected (U)
MS/MSD	1 per 20 field samples of similar matrix	70-130%, RPD<30	If the LCS meets acceptance criteria, no corrective action required. Qualify data	Lab personnel	Accuracy/Bias/ Precision	70-130%, RPD<30

QAPP Worksheet #28-8 QC Samples — Perchlorate

Matrix	Aqueous	**Analytical Method/ SOP Reference**	EPA 6850M / GL-OE-067	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	Perchlorate	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020	**Analytical Organization**	GEL			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
			as needed.			
LCS/LCSD	One per batch of 20 or fewer samples per matrix	%R 70-130%. RPD ≤30	If sufficient sample volume, re-extract entire batch. Otherwise, qualify data as needed	Lab personnel	Accuracy/Bias/ Precision	%R 70-130 RPD ≤ 30%
Internal Standard	Each standard, sample and QC sample	Measured 18O IS area within ± 50% of the value from the average of the IS area counts of the ICAL. RRT of the perchlorate ion must be 1.0 ± 2% (0.98 – 1.02).	Rerun the sample at increasing dilutions until the ± 50% acceptance criteria are met. If criteria cannot be met with dilution, the interference is suspected, and the sample must be re-prepped using additional pretreatment steps.	Lab personnel	Accuracy Bias	±50% of average from ICAL

QAPP Worksheet #28-8 QC Samples — Perchlorate

Matrix	Aqueous	**Analytical Method/ SOP Reference**	EPA 6850M / GL-OE-067	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	Perchlorate	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020	**Analytical Organization**	GEL			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Interference Check Sample (ICS)	One per batch of 20 or fewer samples per matrix	Perchlorate concentration must be within ± 20% of its true value	Correct problem. Reanalyze all samples and QC samples in the batch. If poor recovery from the cleanup filters is suspected, a different lot of filters must be used to re-extract all samples in the batch.	Lab personnel	"Bias Contamination"	± 20% of true value

1- See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP
2-See RDWP Appendices (Site-Wide Soils and Sediments-A, In-Situ Sequestration of DU & U-B, Holding Basin Containment-C, 1,4-dioxane and VOCs in Bedrock Groundwater-D) for PDIs, IPs and Appendix I-FSP.

QAPP Worksheet #28-8 QC Samples — Total Sulfide

Matrix	Aqueous	**Analytical Method/ SOP Reference**	SM4500S2-D/2221	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	Total Sulfide	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020	**Analytical Organization**	AAW			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Field duplicate	1 per 20 field samples	RPD $\leq$ 30%	Qualify data as needed	Poly Newbold or designee, ddms	Precision	RPD $\leq$ 30% For concentrations ≥ RL
Method blanks	1 per analytical batch	< RL	Qualify data as needed or reanalysis of batch	Lab personnel	Accuracy/bias contamination	Sample Concentrations $\leq$ RL will be qualified not detected (U).
Field/Equipment blanks	1 per 20 field samples	< RL	Qualify data as needed	Poly Newbold or designee, ddms	Accuracy/bias contamination	Sample Concentrations $\leq$ RL will be qualified not detected (U).
MS	1 per analytical batch	Laboratory acceptance limits	If LCS acceptable no corrective action; qualify data as needed.	Lab personnel	Accuracy/bias	%R 75-125%
LCS	1 per analytical batch	%R 75-125	If sufficient sample volume, re-analyze entire batch. Otherwise, qualify data as needed	Lab personnel	Accuracy/bias	%R 85-115
Lab duplicate	1 per analytical batch	RPD$\leq$20	Qualify data as needed	Lab personnel	Precision	RPD$\leq$15

[1]- See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]-See RDWP Appendices (Site-Wide Soils and Sediments-A, In-Situ Sequestration of DU & U-B, Holding Basin Containment-C, 1,4-dioxane and VOCs in Bedrock Groundwater-D) for PDIs, IPs and Appendix I-FSP.

QAPP Worksheet #28-9 QC Samples — Polonium-210						
Matrix	Aqueous	**Analytical Method/ SOP Reference**	Lab Developed Method/GL-RAD-A-016	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	Radiochemistry (Polonium-210)	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020	**Analytical Organization**	GEL			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Field duplicate	1 per 20 field samples	RPD ≤20% for activity >5 MDC	Qualify data as needed	Lab and/or Polly Newbold or designee, ddms	Precision	RPD ≤20% for activity >5 MDC
Method blanks	1/batch	< RL	Qualify data as needed or reanalysis of batch	Lab personnel	Accuracy/Bias contamination	Samples ≤ blank will be qualified as not detected (U)
Field, Equipment blanks	1 per 20 field samples	< RL	Qualify data as needed	Lab and/or Polly Newbold or designee, ddms	Accuracy/Bias contamination	Samples ≤ blank will be qualified as not detected (U)
Laboratory duplicate	1/batch	RPD ≤20% for activity >5 MDC	Qualify data as needed	Lab personnel	Precision	RPD ≤20% for activity >5 MDC.
Tracer	Each sample	%R 15-125	Qualify data as needed	Lab personnel	Accuracy/Bias	%R 70-130. %R<10 non-detects will be rejected (R)

[1]- See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]-See RDWP Appendices (Site-Wide Soils and Sediments-A, In-Situ Sequestration of DU & U-B, Holding Basin Containment-C, 1,4-dioxane and VOCs in Bedrock Groundwater-D) for PDIs, IPs and Appendix I-FSP.

QAPP Worksheet #28-9 QC Samples — Total Suspended Solids

Matrix	Aqueous	**Analytical Method/ SOP Reference**	SM 2540 D/2220	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	Total Suspended Solids	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020	**Analytical Organization**	AAW			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Field duplicate	1 per 20 field samples	RPD ≤ 30%	Qualify data as needed	Polly Newbold or designee, ddms	Precision	RPD < 30% For concentrations ≥ RL
Method blank	1 per analytical batch	< RL	Qualify data as needed or reanalysis of batch	Lab personnel	Accuracy/bias contamination	Sample concentrations ≤ blank concentration will be qualified not detected (U)
Field/Equipment blanks	1 per 20 field samples	< RL	Qualify data as needed	Polly Newbold or designee, ddms	Accuracy/bias contamination	Sample concentrations ≤ blank concentration will be qualified not detected (U)
Lab Duplicates	1 per analytical batch	5% RPD	Qualify data as needed	Lab and/or Polly Newbold or designee, ddms	Precision	RPD ≤ 5%

[1]- See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]-See RDWP Appendices (Site-Wide Soils and Sediments-A, In-Situ Sequestration of DU & U-B, Holding Basin Containment-C, 1,4-dioxane and VOCs in Bedrock Groundwater-D) for PDIs, IPs and Appendix I-FSP.

QAPP Worksheet #28-9A QC Samples — Total Dissolved Solids

Matrix	Aqueous	**Analytical Method/ SOP Reference**	SM 2540C / 2220	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	Total Dissolved Solids	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020	**Analytical Organization**	AAW			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Field duplicate	1 per 20 field samples	RPD ≤ 30%	Qualify data as needed	Polly Newbold or designee, ddms	Precision	RPD < 30% For concentrations ≥ RL
Method blank	1 per analytical batch	< RL	Qualify data as needed or reanalysis of batch	Lab personnel	Accuracy/bias contamination	Sample concentrations ≤ blank concentration will be qualified not detected (U)
Field/Equipment blanks	1 per 20 field samples	< RL	Qualify data as needed	Polly Newbold or designee, ddms	Accuracy/bias contamination	Sample concentrations ≤ blank concentration will be qualified not detected (U)
Lab Duplicates	1 per analytical batch	5% RPD	Qualify data as needed	Lab and/or Polly Newbold or designee, ddms	Precision	RPD ≤ 5%

[1]-See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]-See RDWP Appendices (Site-Wide Soils and Sediments-A, In-Situ Sequestration of DU & U-B, Holding Basin Containment-C, 1,4-dioxane and VOCs in Bedrock Groundwater-D) for PDIs, IPs and Appendix I-FSP.

QAPP Worksheet #28-10 QC Samples — Anions – chloride, fluoride, sulfate

Matrix	Aqueous	**Analytical Method/ SOP Reference**	EPA 300.0 / GL-GC-E-086	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	Anions – bromide, chloride, fluoride, sulfate, nitrate, nitrite, o-phosphate	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020	**Analytical Organization**	GEL			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Field duplicate	1 per 20 field samples	RPD $\leq$ 30%	Qualify data as needed	Polly Newbold or designee, ddms	Precision	RPD $\leq$ 30% for concentrations ≥ RL
Method blanks	1 per analytical batch	< RL	Qualify data as needed or reanalysis of batch	Lab personnel	Accuracy/bias contamination	Sample concentrations $\leq$ blank concentration will be qualified not detected (U)
Field, Equipment blanks	1 per 20 field samples	< RL	Qualify data as needed	Polly Newbold or designee, ddms	Accuracy/bias contamination	Sample concentrations $\leq$ blank concentration will be qualified not detected (U)
MS	1 per 20 field samples of similar matrix	%R 90-110	If LCS acceptable no corrective action required; qualify data as needed.	Lab and/or Polly Newbold or designee, ddms	Accuracy/bias	%R 90-110
LCS	1 per analytical batch	%R 90-110	If sufficient sample volume, re-extract entire batch. Otherwise, qualify data as needed	Lab and/or Polly Newbold or designee, ddms	Accuracy/bias	%R 90-100

QAPP Worksheet #28-10 QC Samples — Anions – chloride, fluoride, sulfate

Matrix	Aqueous	**Analytical Method/ SOP Reference**	EPA 300.0 / GL-GC-E-086	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	Anions – bromide, chloride, fluoride, sulfate, nitrate, nitrite, o-phosphate	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020	**Analytical Organization**	GEL			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Lab duplicate	1 per analytical batch	RPD≤20	Qualify data as needed	Lab personnel	Precision	RPD≤20

[1]- See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP
[2]-See RDWP Appendices (Site-Wide Soils and Sediments-A, In-Situ Sequestration of DU & U-B, Holding Basin Containment-C, 1,4-dioxane and VOCs in Bedrock Groundwater-D) for PDIs, IPs and Appendix I-FSP.

QAPP Worksheet #28-10 QC Samples — VOCs

Matrix	Aqueous, Leachate	**Analytical Method/ SOP Reference**	SW846 8260C/2108	**See footnote 2 below**	Numerous	
Analytical Group	VOCs	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec, H&A			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020 or NMI-S-001 – NMI-S-007	**Analytical Organization**	AAW			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Field duplicate	1 per 20 field samples	RPD ≤ 30%	Qualify data as needed	Polly Newbold or designee, ddms	Precision	RPD ≤ 30% for concentrations ≥RL
Surrogates	3 per sample	%R 70-130%	Reanalyze sample, surrogate high sample ND, report data and qualify	Lab personnel	Accuracy/bias	%R, 70-130%, %R<10 non-detects will be rejected (R)
Method blanks	1 per analytical batch	< RL	Qualify data as needed or reanalyze batch	Lab personnel	Accuracy/bias contamination	Sample concentrations ≤ blank concentration* will be qualified not detected (U)
Trip blanks	1 per cooler containing VOC samples	< RL	Qualify data as needed	Polly Newbold or designee, ddms	Accuracy/bias contamination	Sample concentrations ≤ blank concentration* will be qualified not detected (U)
Equipment blanks	1 per 20 field samples	< RL	Qualify data as needed	Polly Newbold or designee, ddms	Accuracy/bias contamination	Sample concentrations ≤ blank concentration* will be qualified not detected (U)

QAPP Worksheet #28-10 QC Samples — VOCs

Matrix	Aqueous, Leachate	**Analytical Method/ SOP Reference**	SW846 8260C/2108	**See footnote 2 below**	Numerous	
Analytical Group	VOCs	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec, H&A			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020 or NMI-S-001 – NMI-S-007	**Analytical Organization**	AAW			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
LCS/LCSD*	1/batch	%R 70-130, RPD≤20	If sufficient sample volume, reanalyze entire batch. Otherwise, qualify data as needed	Lab personnel	Accuracy/Bias/ Precision	%R 70-130, RPD≤20
Internal standard	At least three/sample	Area response 50-200% of CC +/- 0.06 RRT	Reanalyze sample	Lab personnel	Precision	Area response 50-200% of CC +/- 0.06 RRT
MS/MSD	1/batch	%R 70-130, RPD≤20	If the LCS meets acceptance criteria, no corrective action required. Qualify data as needed.	Lab and/or Polly Newbold or designee, ddms	Accuracy/Bias/ Precision	%R 70-130, RPD≤20

*-Methylene chloride and toluene ≤2X blank conentration
[1]- See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]-See RDWP Appendices (Site-Wide Soils and Sediments-A, In-Situ Sequestration of DU & U-B, Holding Basin Containment-C, 1,4-dioxane and VOCs in Bedrock Groundwater-D) for PDIs, IPs and Appendix I-FSP.

QAPP Worksheet #28-11 QC Samples — Metals						
Matrix	Aqueous, Leachate	**Analytical Method/ SOP Reference**	SW846 6010D/ GL-MA-E-013	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	Metals	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	H&A			
Sampling SOP[1]	NMI-S-001 – NMI-S-007	**Analytical Organization**	GEL			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Field duplicate	1 per 20 field samples	RPD $\leq$ 30%	Qualify data as needed	Polly Newbold or designee, ddms	Precision	RPD $\leq$ 30% for concentrations $\geq$RL
Method blanks	1/batch	No target analytes detected greater than one-half RL and 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater).	Qualify data as needed or reanalysis of batch	Lab personnel	Accuracy/Bias contamination	Sample concentrations $\leq$ blank concentration will be qualified as not detected (U)
Calibration blanks	Before beginning a sample run, after every 10 samples, and at end of the analysis sequence	No analytes detected > 2 × MDL	Any sample associated with a blank that fails the criteria checks will be reprocessed in a subsequent preparation batch, except when the sample analysis resulted in a non-detect. If no sample volume remains for reprocessing, the results will be	Lab personnel	Accuracy/Bias contamination	Sample concentrations $\leq$ blank concentration will be qualified as not detected (U)

QAPP Worksheet #28-11 QC Samples — Metals						
Matrix	Aqueous, Leachate	**Analytical Method/ SOP Reference**	SW846 6010D/ GL-MA-E-013	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	Metals	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	H&A			
Sampling SOP[1]	NMI-S-001 – NMI-S-007	**Analytical Organization**	GEL			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
			reported with appropriate data qualifying codes.			
Tumble Blank	One per tumble batch	No target analytes detected greater than one-half RL and 1/10 the amount measured in any sample or 1/10 regulatory limit (whichever is greater).	Correct problem, then re- reanalyze method blank and all samples processed with the contaminated blank	Lab personnel	Accuracy/Bias contamination	Sample concentrations ≤ blank concentration will be qualified as not detected (U)
Field, Equipment blanks	1 per 20 field samples	< RL	Qualify data as needed	Lab and/or Polly Newbold or designee, ddms	Accuracy/Bias contamination	Sample concentrations ≤ blank concentration will be qualified as not detected (U)
MS/MSD	1/batch	Laboratory acceptance limits[3]	If the LCS meets acceptance criteria, no corrective action required. Qualify data as needed.	Lab personnel	Accuracy/Bias/ Precision	%R 80-120, RPD≤20
Laboratory duplicate	1/batch	Laboratory acceptance limits[3]	Qualify data as needed	Lab personnel	Precision	RPD ≤ 20% for results > RL.
LCS/LCSD	1/batch	Laboratory	If sufficient sample volume, redigest	Lab and/or Polly Newbold or designee,	Accuracy/Bias/	%R 80-120

QAPP Worksheet #28-11 QC Samples — Metals						
Matrix	Aqueous, Leachate	**Analytical Method/ SOP Reference**	SW846 6010D/ GL-MA-E-013	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	Metals	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	H&A			
Sampling SOP[1]	NMI-S-001 – NMI-S-007	**Analytical Organization**	GEL			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
		acceptance limits[3]	entire batch. Otherwise, qualify data as needed	ddms	Precision	RPD≤20
Post Spike	When serial dilution or matrix spike fails	%R 75-125%	Qualify data as needed	Lab personnel	Accuracy/Bias	%R 75-125%
Serial dilution	Each new sample matrix	1:5 dilution must agree within ±10% of original determination.	Perform post-digestion spike if serial dilution does not meet criteria	Lab personnel	Precision/Bias	%D ≤ 10% for concentrations ≥50X MDL < 10%

[1]- See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]-See RDWP Appendices (Site-Wide Soils and Sediments-A, In-Situ Sequestration of DU & U-B, Holding Basin Containment-C, 1,4-dioxane and VOCs in Bedrock Groundwater-D) for PDIs, IPs and Appendix I-FSP.
[3]-laboratory acceptance limits are based on Department of Defense (DoD) Quality Systems Manual (QSM)

QAPP Worksheet #28-11 QC Samples — SVOCs

Matrix	Aqueous, Leachate	**Analytical Method/ SOP Reference**	SW846 8270D/2111	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	SVOCs	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec, H&A			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020 or NMI-S-001-NMI-S-007	**Analytical Organization**	AAW			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Field duplicate	1 per 20 field samples	RPD $\leq$ 30%	Qualify data as needed	Polly Newbold or designee, ddms	Precision	RPD $\leq$ 30% for concentrations $\geq$RL
Surrogates	3 BN/3 AC per sample	%R 30-130 for base-neutral surrogates and 15-110% for acid surrogates	Reanalyze and/or re-extract sample, surrogate high sample ND, report data and qualify	Lab personnel	Accuracy/bias	%R, 70-130%, %R<10 non-detects will be rejected (R)
Method blanks	1 per analytical batch	< RL	Qualify data as needed or reanalyze batch	Lab personnel	Accuracy/bias contamination	Sample concentrations < blank concentration will be qualified not detected (U)
Field, Equipment blanks	1 per 20 field samples	< RL	Qualify data as needed	Polly Newbold or designee, ddms	Accuracy/bias contamination	Sample concentrations < blank concentration will be qualified not detected (U)
LCS/LCSD*	1/batch	%R 40 - 140 for base-neutral compounds, %R 30 - 130% for acid compounds except for "difficult" analytes which must	If sufficient sample volume, re-extract entire batch. Otherwise, qualify data as needed	Lab personnel	Accuracy/Bias/ Precision	%R 70-130, RPD$\leq$20

QAPP Worksheet #28-11 QC Samples — SVOCs

Matrix	Aqueous, Leachate	**Analytical Method/ SOP Reference**	SW846 8270D/2111	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	SVOCs	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec, H&A			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020 or NMI-S-001-NMI-S-007	**Analytical Organization**	AAW			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
		be between 15-140%, RPD≤20				
Internal standard	At least three/sample	Area response 50-200% of CC +/- 0.06 RRT	Reanalyze sample	Lab personnel	Precision	Area response 50-200% of CC +/- 0.06 RRT
MS/MSD	1/batch	%R 40 - 140 for base-neutral compounds, %R 30 - 130% for acid compounds except for "difficult" analytes which must be between 15-140%, RPD≤20	If the LCS meets acceptance criteria, no corrective action required. Qualify data as needed.	Lab and/or Polly Newbold or designee, ddms	Accuracy/Bias/ Precision	%R 70-130, RPD≤20

[1]- See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]-See RDWP Appendices (Site-Wide Soils and Sediments-A, In-Situ Sequestration of DU & U-B, Holding Basin Containment-C, 1,4-dioxane and VOCs in Bedrock Groundwater-D) for PDIs, IPs and Appendix I-FSP.

QAPP Worksheet #28-12 QC Samples — PFAS

Matrix	Aqueous	**Analytical Method/ SOP Reference**	Modified 537 Isotope Dilution / GL-OA-E-076	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	PFAS	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020	**Analytical Organization**	GEL			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Field duplicate	1 per 20 field samples	RPD ≤ 30%	Qualify data as needed	Polly Newbold or designee, ddms	Precision	RPD<30 for concentrations >RL
Method Blank	One blank per prep batch not to exceed 20 samples; whichever is more frequent	No analytes detected >½ LOQ or >1/10th the amount measured in any sample or 1/10th the regulatory limit (whichever is greater).	Correct problem; if required, re-prepare and/or reanalyze method blank and all QC samples and samples processed with the contaminated blank. If reanalysis cannot be performed, data must be qualified and explained in the case narrative.	Lab personnel	Accuracy/bias contamination	Sample concentrations ≤ blank concentration will be qualified not detected (U)
Field, Equipment blanks	1 per 20 field samples	< RL	Qualify data as needed	Polly Newbold or designee, ddms	Accuracy/bias	Sample concentrations <5X blank concentration will be qualified not detected (U)

QAPP Worksheet #28-12 QC Samples — PFAS

Matrix	Aqueous	**Analytical Method/ SOP Reference**	Modified 537 Isotope Dilution / GL-OA-E-076	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	PFAS	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020	**Analytical Organization**	GEL			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Labeled Analogs/Extracted Internal Standards	Every field sample, standard, and QC sample	%R 50-150	If recoveries are unacceptable for QC samples, correct problem, and reanalyze all associated failed field samples. Discuss in the case narrative only if reanalysis confirms failures in exactly the same manner.	Lab personnel	Accuracy/bias	%R 40-140; %R<10 non-detects will be rejected
Internal Standard Analytes	Every field sample, standard, and QC sample	Peak areas must be within -50% to +50% of the area measured in the initial calibration midpoint standard. On days when initial calibration is not performed, the peak areas must be within -50% to +50% of the peak area measured in daily continuing calibration verification.	If peak areas are unacceptable, analyze a second aliquot of the extract or sample if enough extract remains. If there is not enough extract, reanalyze the first aliquot. If second analysis meets acceptance criteria, report the second analysis. If it fails, either analysis may be reported with the appropriate flags.	Lab personnel	Accuracy/bias	Peak areas must be within -50% to +50% of the area measured in the initial calibration midpoint standard. On days when initial calibration is not performed, the peak areas must be within -50% to +50% of the peak area measured in daily continuing calibration verification.

QAPP Worksheet #28-12 QC Samples — PFAS

Matrix	Aqueous	**Analytical Method/ SOP Reference**	Modified 537 Isotope Dilution / GL-OA-E-076	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	PFAS	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020	**Analytical Organization**	GEL			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
LCS/LCSD	One per prep batch up to 20 samples	Blank spiked with all analytes at a concentration ≥LOQ and ≤ the mid-level calibration concentration. %R — 70-130%. RPD ≤30	Correct the problem, then reprepare and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes if sufficient sample material is available. If reanalysis cannot be performed, data must be qualified and explained in the case narrative.	Lab personnel	Accuracy/bias/ Precision	%R 70-130 (50-150% for low-level spikes) RPD<30
MS/MSD	1 per batch of 20 samples	70-130% recovery (50-150% for low-level spikes); RPD≤30 % for duplicate spikes	If the LCS meets acceptance criteria, no corrective action required. Qualify data as needed.	Lab personnel	Accuracy/bias/ Precision	%R 70-130 Recovery RPD<30

1- See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
2- See RDWP Appendices (Site-Wide Soils and Sediments-A, In-Situ Sequestration of DU & U-B, Holding Basin Containment-C, 1,4-dioxane and VOCs in Bedrock Groundwater-D) for PDIs, IPs and Appendix I-FSP.

QAPP Worksheet #28-12 QC Samples — SVOCs-1,4-Dioxane

Matrix	Aqueous	**Analytical Method/ SOP Reference**	SW846 8270 SIM/ID 1948, 2164	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	SVOCs 1,4-dioxane only	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020	**Analytical Organization**	AAW			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Field duplicate	1 per 20 field samples	RPD $\leq$ 20%	Qualify data as needed	Polly Newbold or designee, ddms	Precision — overall	RPD $\leq$ 30% For compounds $\geq$RL.
Surrogate	1 per sample	%R 30-130% (1,4-dioxane-d_8)	Re-extract and/or analyze sample	Lab personnel	Accuracy/bias	%R<10 non-detects will be rejected (R)
Method blanks	1 per analytical batch	< RL	Qualify data as needed or reanalyze batch	Lab and/or Polly Newbold or designee, ddms	Accuracy/bias contamination	Sample concentrations $\leq$ blank concentration will be qualified not detected (U)
Field, Equipment blanks	1 per 20 field samples	< RL	Qualify data as needed	Polly Newbold or designee, ddms	Accuracy/bias contamination	Sample concentrations $\leq$ blank concentration will be qualified not detected (U)
LCS/LCSD*	1 per analytical batch	%R 40-140%, RPD$\leq$20	If sufficient sample volume, re-extract entire batch. Otherwise, qualify data as needed	Lab personnel	Accuracy/bias/ Precision	70-130% RPD$\leq$20

QAPP Worksheet #28-12 QC Samples — SVOCs-1,4-Dioxane

Matrix	Aqueous	**Analytical Method/ SOP Reference**	SW846 8270 SIM/ID 1948, 2164	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	SVOCs 1,4-dioxane only	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020	**Analytical Organization**	AAW			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Internal standard	Every field sample and QC samples	Area response 50-200% of CC +/- 0.06 RRT	Reanalyze sample	Lab personnel	Precision	Area response 50-200% of CC +/- 0.06 RRT
MS/MSD	1/batch	%R 40-140, RPD≤20	If the LCS meets acceptance criteria, no corrective action required. Qualify data as needed.	Lab and/or Polly Newbold or designee, ddms	Accuracy/Bias /Precision	%R 70-130 RPD≤20

[1]- See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]-See RDWP Appendices (Site-Wide Soils and Sediments-A, In-Situ Sequestration of DU & U-B, Holding Basin Containment-C, 1,4-dioxane and VOCs in Bedrock Groundwater-D) for PDIs, IPs and Appendix I-FSP.

QAPP Worksheet #28-13 QC Samples —Pesticides

Matrix	Aqueous, Leachate	**Analytical Method/ SOP Reference**	SW846 8081B/2116	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	Pesticides	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec, H&A			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020 or NMI-S-001 - NMI-S-007	**Analytical Organization**	AAW			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Field Duplicates	1 per 20 field samples	RPD $\leq$ 30%	Qualify data as required	Polly Newbold or designee, ddms	Precision	RPD < 30% for concentrations >RL
Surrogate Spike	2 per sample	%R 30-150	If sufficient sample volume is available, re-extract and reanalyze the samples	Lab and/or Polly Newbold or designee, ddms	Accuracy/Bias	%R, 70-130%, %R<10 non-detects will be rejected (R)
Method Blank	1 per extraction batch	<RL	Qualify data as needed or re-extract and reanalyze batch	Lab personnel	Accuracy/Bias/ Contamination	Sample concentrations $\leq$ blank concentration will be qualified not detected (U)
Field, Equipment Blank	1 per 20 field samples	<RL	Qualify data as required	Polly Newbold or designee, ddms	Accuracy/Bias/ Contamination	Sample concentrations $\leq$ blank concentration will be qualified not detected (U)

QAPP Worksheet #28-13 QC Samples —Pesticides

Matrix	Aqueous, Leachate	**Analytical Method/ SOP Reference**	SW846 8081B/2116	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	Pesticides	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec, H&A			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020 or NMI-S-001 - NMI-S-007	**Analytical Organization**	AAW			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
LCS/LCSD	1 per extraction batch	%R 40-140 RPD≤20	If sufficient sample volume is available, re-extract and reanalyze the samples; otherwise qualify data as needed	Lab and/or Polly Newbold or designee, ddms	Accuracy/Bias/ Precision	70-130% RPD≤ 20
MS/MSD	1 per extraction batch	%R 30-150 RPD≤30	If the LCS meets acceptance criteria, no corrective action required. Qualify data as needed.	Lab and/or Polly Newbold or designee, ddms	Accuracy/Bias/ Precision	70-130% RPD ≤ 30%
Internal Standard	Each sample	-50 to +100% of CC	If internal standard is outside of limits, reanalyze sample unless chromatographic interference is present.	Lab personnel	Precision	-50 to +100% of CC

QAPP Worksheet #28-13 QC Samples —Pesticides

Matrix	Aqueous, Leachate	**Analytical Method/ SOP Reference**	SW846 8081B/2116	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	Pesticides	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec, H&A			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020 or NMI-S-001 - NMI-S-007	**Analytical Organization**	AAW			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Identification and Quantitation	Each Sample	Minimum3 peaks used for quantitation. RPD<45 between columns	Reanalyze sample if RPD>100 at a dilution. Both runs must be reported	Lab Personnel	Accuracy	Minimum3 peaks used for quantitation. RPD<25 between columns.

1- See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
2-See RDWP Appendices (Site-Wide Soils and Sediments-A, In-Situ Sequestration of DU & U-B, Holding Basin Containment-C, 1,4-dioxane and VOCs in Bedrock Groundwater-D) for PDIs, IPs and Appendix I-FSP.

QAPP Worksheet #28-13 QC Samples — Total Organic Carbon, Dissolved Organic Carbon, Total Inorganic Carbon, Dissolved Inorganic Carbon)

Matrix	Aqueous	**Analytical Method/ SOP Reference**	SW846 9060A/ GC-E-093	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	TOC, DOC, TIC, DIC	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020	**Analytical Organization**	GEL			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Field duplicate	1 per 20 field samples	RPD $\leq$ 30%	Qualify data as needed	Polly Newbold or designee, ddms	Precision	RPD $\leq$ 30% for concentrations $\geq$RL
Method blanks	1 per analytical batch	< RL	Qualify data as needed or reanalysis of batch	Lab personnel	Accuracy/Bias contamination	Sample concentrations $\leq$ blank concentration will be qualified as not detected (U)
Field, Equipment blanks	1 per 20 field samples	< RL	Qualify data as needed	Polly Newbold or designee, ddms	Accuracy/Bias contamination	Sample concentrations $\leq$ blank concentration will be qualified as not detected (U)
LCS	1 per analytical batch	%R 90-110	If sufficient sample reanalyze entire batch. Otherwise, qualify data as needed	Lab personnel	Accuracy/Bias	90-110%R
MS/MSD	1 per analytical batch	%R 85-115 RPD$\leq$20	If the LCS meets acceptance criteria, no corrective action required. Qualify data as needed	Lab personnel	Accuracy/Bias/ Precision	%R 85-115 RPD$\leq$20

QAPP Worksheet #28-13 QC Samples — Total Organic Carbon, Dissolved Organic Carbon, Total Inorganic Carbon, Dissolved Inorganic Carbon)

Matrix	Aqueous	**Analytical Method/ SOP Reference**	SW846 9060A/ GC-E-093	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	TOC, DOC, TIC, DIC	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020	**Analytical Organization**	GEL			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
Quadruplicate analysis	Each sample	RPD≤20 when values >5mg/L	If samples were not analyzed in quadruplicate, reanalyze	Lab personnel	Precision	RPD<20 when values >5mg/L

[1]-See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]-See RDWP Appendices (Site-Wide Soils and Sediments-A, In-Situ Sequestration of DU & U-B, Holding Basin Containment-C, 1,4-dioxane and VOCs in Bedrock Groundwater-D) for PDIs, IPs and Appendix I-FSP.

QAPP Worksheet #28-14 QC Samples — Hardness

Matrix	Aqueous	**Analytical Method/ SOP Reference**	SM2340C/ GEL-GC-E-100	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	Hardness	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020	**Analytical Organization**	GEL			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Field duplicate	1 per 20 field samples	RPD $\leq$ 30%	Qualify data as needed	Polly Newbold or designee, ddms	Precision	RPD $\leq$ 30% Results $\geq$ RL
Method blanks	1/ batch	< RL	Qualify data as needed or reanalysis of batch	Lab personnel	Accuracy/Bias contamination	Sample concentrations $\leq$ blank concentration will be qualified as not detected (U)
Field, Equipment blanks	1 per 20 field samples	< RL	Qualify data as needed	Lab and/or Polly Newbold or designee, ddms	Accuracy/Bias contamination	Sample concentrations $\leq$ blank concentration will be qualified as not detected (U)
Laboratory control sample	1/ batch	%R 90-110	If sufficient sample volume, reanalyze entire batch. Otherwise, qualify data as needed	Lab personnel	Accuracy/Bias	%R 90-110
MS	1/ batch	%R 90-110	If the LCS meets acceptance criteria, no corrective action required. Qualify data as needed.	Lab personnel	Accuracy/bias	%R 90-110

QAPP Worksheet #28-14 QC Samples — Hardness

Matrix	Aqueous	**Analytical Method/ SOP Reference**	SM2340C/ GEL-GC-E-100	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	Hardness	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020	**Analytical Organization**	GEL			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Laboratory duplicate	1/ batch	RPD $\leq$10%	Qualify data as needed	Lab personnel	Precision	RPD $\leq$10%

[1]- See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]-See RDWP Appendices (Site-Wide Soils and Sediments-A, In-Situ Sequestration of DU & U-B, Holding Basin Containment-C, 1,4-dioxane and VOCs in Bedrock Groundwater-D) for PDIs, IPs and Appendix I-FSP.

QAPP Worksheet #28-14 QC Samples — PFAS

Matrix	Aqueous	**Analytical Method/ SOP Reference**	Modified 537 Isotope Dilution/23528	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	PFAS	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020	**Analytical Organization**	AAM			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Field duplicate	1 per 20 field samples	RPD $\leq$ 50%	Qualify data as needed	Polly Newbold or designee, ddms	Precision	RPD<30 for concentrations >RL
Method Blank	One blank per prep batch not to exceed 20 samples; whichever is more frequent	<RL	Re-inject the blank. If failure continues, sample analysis is terminated; corrective action is needed and may involve re-extraction of the batch after source of contamination is determined and corrected	Lab personnel	Accuracy/bias contamination	Sample concentrations $\leq$ blank concentration will be qualified not detected (U)

QAPP Worksheet #28-14 QC Samples — PFAS

Matrix	Aqueous	**Analytical Method/ SOP Reference**	Modified 537 Isotope Dilution/23528	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	PFAS	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020	**Analytical Organization**	AAM			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Field, Equipment blanks	1 per 20 field samples	< RL	Qualify data as needed	Polly Newbold or designee, ddms	Accuracy/bias	Sample concentrations <5X blank concentration will be qualified not detected (U)
Labeled Analogs	Added to each sample	%R 40-140 Reportable if >10% and S/N >10	Reanalyze sample, if high recovery and sample ND, report data and qualify	Lab personnel	Accuracy/bias	%R 40-140; %R<10 non-detects will be rejected
LCS/LCSD	One per prep batch up to 20 samples	%R 50-150 RPD≤30	If sufficient sample volume, re-extract entire batch. Otherwise, qualify data as needed	Lab personnel	Accuracy/bias/ Precision	%R 70-130 (50-150% for low-level spikes) RPD<30

QAPP Worksheet #28-14 QC Samples — PFAS

Matrix	Aqueous	**Analytical Method/ SOP Reference**	Modified 537 Isotope Dilution/23528	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	PFAS	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020	**Analytical Organization**	AAM			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
MS/MSD	1 per batch of 20 samples	70-130% recovery (50-150% for low-level spikes); RPD≤30 % for duplicate spikes	If the LCS meets acceptance criteria, no corrective action required. Qualify data as needed.	Lab personnel	Accuracy/bias/ Precision	%R 70-130 Recovery RPD<30
Laboratory Duplicate	1 per 20. Can be performed on a field sample, MS or LCS	RPD≤30	Reanalyze if analytical error is suspected. Note in the narrative. Narrate interferences if observed.	Lab personnel	Precision	RPD<30 for compounds >RL

1- See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
2- See RDWP Appendices (Site-Wide Soils and Sediments-A, In-Situ Sequestration of DU & U-B, Holding Basin Containment-C, 1,4-dioxane and VOCs in Bedrock Groundwater-D) for PDIs, IPs and Appendix I-FSP.

QAPP Worksheet #28-15 QC Samples — Ammonia Nitrogen

Matrix	Aqueous	**Analytical Method/ SOP Reference**	SM4500 NH3-BH / GL-GC-E-072	**No. of Sample Locations**	See Footnote 2 below	
Analytical Group	Ammonia Nitrogen	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020	**Analytical Organization**	GEL			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Field duplicate	1 per 20 field samples	RPD ≤ 30%	Qualify data as needed	Polly Newbold or designee, ddms	Precision	RPD < 30% Results ≥ RL
Method blanks	1 per analytical batch	< RL	Qualify data as needed or reanalysis of batch	Lab personnel	Accuracy/bias contamination	Sample concentrations ≤ blank concentration will be qualified not detected (U)
Field/Equipment blanks	1 per 20 field samples	< RL	Qualify data as needed	Polly Newbold or designee, ddms	Accuracy/bias contamination	Sample concentrations ≤ blank concentration will be qualified not detected (U)
MS	1 per analytical batch	%R 90-110	If the LCS meets acceptance criteria, no corrective action required. Qualify data as needed.	Lab and/or Polly Newbold or designee, ddms	Accuracy/bias	%R 90-110
LCS	1 per analytical batch	%R 90-110	If sufficient sample volume, reanalyze entire batch. Otherwise, qualify data as needed	Lab and/or Polly Newbold or designee, ddms	Accuracy/bias	%R 90-110%

QAPP Worksheet #28-15 QC Samples — Ammonia Nitrogen

Matrix	Aqueous	**Analytical Method/ SOP Reference**	SM4500 NH3-BH / GL-GC-E-072	**No. of Sample Locations**	See Footnote 2 below	
Analytical Group	Ammonia Nitrogen	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020	**Analytical Organization**	GEL			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Lab Duplicate	1 per analytical batch	RPD≤20	Qualify data as needed	Lab personnel	Precision	RPD≤20

[1]- See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]-See RDWP Appendices (Site-Wide Soils and Sediments-A, In-Situ Sequestration of DU & U-B, Holding Basin Containment-C, 1,4-dioxane and VOCs in Bedrock Groundwater-D) for PDIs, IPs and Appendix I-FSP.

QAPP Worksheet #28-21 QC Samples — VOCs

Matrix	Soil Vapor	**Analytical Method/ SOP Reference**	TO-15/2186	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	VOCs	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	TBD			
Sampling SOP[1]	NMI-A-001	**Analytical Organization**	AAM			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Field duplicate	1 per 20 field samples	RPD<25	Qualify data as needed	Polly Newbold or designee, ddms	Precision	RPD <25% for concentrations ≥RL.
Surrogates*	3 per sample	%R, 70-130	Reanalyze sample	Lab and/or Polly Newbold or designee, ddms	Accuracy/bias	%R, 70-130%, %R<10 non-detects will be rejected (R)
Method blanks	1 per analytical batch	< RL	Qualify data as needed or reanalyze batch	Lab and/or Polly Newbold or designee, ddms	Accuracy/bias contamination	Sample concentrations ≤ blank concentration will be qualified not detected (U)
LCS/LCSD	1 per analytical batch	%R 70-130, RPD≤20	If sufficient sample volume, reanalyze entire batch. Otherwise, qualify data as needed	Lab and/or Polly Newbold or designee, ddms	Accuracy/Bias/ Precision	%R 70-130, RPD<20

QAPP Worksheet #28-21 QC Samples — VOCs

Matrix	Soil Vapor	**Analytical Method/ SOP Reference**	TO-15/2186	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	VOCs	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	TBD			
Sampling SOP[1]	NMI-A-001	**Analytical Organization**	AAM			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Internal standard	At least three/sample	60-140% of CC, ±0.33 min	Reanalyze sample	Lab personnel	Precision	60-140% of CC, ±0.33 min

Notes: *-surrogates are not a requirement of TO-15 but may be added by the laboratory.
[1]- See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]-See RDWP Appendices (Site-Wide Soils and Sediments-A, In-Situ Sequestration of DU & U-B, Holding Basin Containment-C, 1,4-dioxane and VOCs in Bedrock Groundwater-D) for PDIs, IPs and Appendix I-FSP.

QAPP Worksheet #28-16 QC Samples —Herbicides

Matrix	Leachate	**Analytical Method/ SOP Reference**	SW846 8081B/2116	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	Herbicides	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	H&A			
Sampling SOP	NMI-S-001 - NMI-S-007	**Analytical Organization**	AAW			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Field Duplicates	1 per 20 field samples	RPD $\leq$ 30%	Qualify data as required	Polly Newbold or designee, ddms	Accuracy	RPD < 30% for concentrations >RL
Surrogate Spike	2 per sample	%R 30-150	If sufficient sample volume is available, re-extract and reanalyze the samples	Lab and/or Polly Newbold or designee, ddms	Accuracy	%R, 70-130%, %R<10 non-detects will be rejected (R)
Method Blank	1 per extraction batch	<RL	Qualify data as needed or re-extract and reanalyze batch	Lab personnel	Accuracy	Sample concentrations $\leq$ blank concentration will be qualified not detected (U)
Field, Equipment Blank	1 per 20 field samples	<RL	Qualify data as required	Polly Newbold or designee, ddms	Accuracy	Sample concentrations $\leq$ blank concentration will be qualified not detected (U)
LCS/LCSD	1 per extraction batch	%R 40-140 RPD$\leq$20	If sufficient sample volume, re-extract entire batch. Otherwise, qualify data as needed	Lab and/or Polly Newbold or designee, ddms	Accuracy/Bias/ Precision	70-130% RPD< 20

QAPP Worksheet #28-16 QC Samples —Herbicides

Matrix	Leachate	**Analytical Method/ SOP Reference**	SW846 8081B/2116	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	Herbicides	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	H&A			
Sampling SOP	NMI-S-001 - NMI-S-007	**Analytical Organization**	AAW			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
MS/MSD	1 per extraction batch	%R 30-150 RPD≤30	If the LCS meets acceptance criteria, no corrective action required. Qualify data as needed.	Lab and/or Polly Newbold or designee, ddms	Accuracy/Bias/ Precision	70-130% RPD < 30%
Internal Standard	Each sample	-50 to +100% of CC	If internal standard is outside of limits, reanalyze sample unless chromatographic interference is present.	Lab personnel	Precision	-50 to +100% of CC
Identification and Quantitation	Each Sample	Minimum3 peaks used for quantitation. RPD<45 between columns	Reanalyze sample if RPD>100 at a dilution. Both runs must be reported	Lab Personnel	Accuracy	Minimum3 peaks used for quantitation. RPD<25 between columns.

[1]- See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]-See RDWP Appendices (Site-Wide Soils and Sediments-A, In-Situ Sequestration of DU & U-B, Holding Basin Containment-C, 1,4-dioxane and VOCs in Bedrock Groundwater-D) for PDIs, IPs and Appendix I-FSP.

QAPP Worksheet #28-16 QC Samples — Alkalinity

Matrix	Alkalinity	**Analytical Method/ SOP Reference**	SM2320B / GL-GC-E-033	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	Alkalinity	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec			
Sampling SOP	NMI-GW-001 - NMI-GW-020	**Analytical Organization**	GEL			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Field Duplicates	1 per 20 field samples	RPD≤30	Qualify data as required	Polly Newbold or designee, ddms	Precision	RPD≤30 for concentrations ≥RL
Method Blank	1 per batch	<RL	If sufficient sample volume is available, reanalyze the samples	Lab and/or Polly Newbold or designee, ddms	Accuracy/ contamination	Sample concentrations ≤ blank concentration will be qualified not detected (U)
Field/Equipment blanks	1 per 20 field samples	<RL	If sufficient sample volume is available, reanalyze the samples	Lab and/or Polly Newbold or designee, ddms	Accuracy/ contamination	Sample concentrations ≤ blank concentration will be qualified not detected (U)
LCS	1 per batch	%R 90-110	If sufficient sample volume, reanalyze entire batch. Otherwise, qualify data as needed	Lab personnel	Accuracy/Bias	%R 90-110
MS	1 per batch	%R 80-120	If the LCS meets acceptance criteria, no corrective action required. Qualify data as needed.	Lab personnel	Accuracy/Bias	%R 80-120

QAPP Worksheet #28-16 QC Samples — Alkalinity

Matrix	Alkalinity	**Analytical Method/ SOP Reference**	SM2320B / GL-GC-E-033	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	Alkalinity	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec			
Sampling SOP	NMI-GW-001 - NMI-GW-020	**Analytical Organization**	GEL			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Lab Duplicates	1 per batch	RPD≤20	Qualify data as required	Lab personnel	Precision	RPD≤20

[1]- See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]-See RDWP Appendices (Site-Wide Soils and Sediments-A, In-Situ Sequestration of DU & U-B, Holding Basin Containment-C, 1,4-dioxane and VOCs in Bedrock Groundwater-D) for PDIs, IPs and Appendix I-FSP.

QAPP Worksheet #28-17 QC Samples

Matrix	Aqueous	**Analytical Method/ SOP Reference**	Lab SOP 'Fluorescent Tracer Dye Analysis'	**No. of Sample Locations**	See footnote below	
Analytical Group	Fluorescent Tracer Dye Analysis	**Sampler's Name**	NA			
Concentration Level	All	**Field Sampling Organization**	Geosyntec			
Sampling SOP	Lab SOP 'Fluorescent Tracer Dye Analysis'	**Analytical Organization**	Ozark Underground Laboratory			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Field duplicate	TBD	RPD $\leq$ 30%	Qualify data as needed	Polly Newbold or designee, ddms	Precision	RPD<30%
Laboratory Blank	1/ batch	< DL	Qualify data as needed or reanalysis of batch	Lab personnel	Accuracy/Bias contamination	Sample concentrations $\leq$ blank concentration will be qualified not detected (U)
Laboratory duplicate	1/ batch	RPD $\leq$30%	Qualify data as needed	Lab personnel	Precision	RPD<30%

See RDWP Appendices (Site-Wide Soils and Sediments-A, In-Situ Sequestration of DU & U-B, Holding Basin Containment-C, 1,4-dioxane and VOCs in Bedrock Groundwater-D) for PDIs, IPs and Appendix I-FSP.

QAPP Worksheet #28-17 QC Samples — Total Suspended Solids

Matrix	Aqueous	**Analytical Method/ SOP Reference**	SM 2540D / GL-GC-E-012	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	Total Suspended Solids	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec			
Sampling SOP[1]	NMI-GW-001 – NMI-GW-020	**Analytical Organization**	GEL			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Field duplicate	1 per 20 field samples	RPD ≤ 30%	Qualify data as needed	Polly Newbold or designee, ddms	Precision	RPD < 30% For concentrations ≥ RL
Method blank	1 per analytical batch	< RL	Qualify data as needed or reanalysis of batch	Lab personnel	Accuracy/bias contamination	Sample concentrations < blank concentration will be qualified not detected (U)
LCS	1 per analytical batch	%r 95-105	Repeat LCS; reanalyze all samples in batch	Lab personnel	Accuracy	%R 95-105
Field/Equipment blanks	1 per 20 field samples	< RL	Qualify data as needed	Polly Newbold or designee, ddms	Accuracy/bias contamination	Sample concentrations < blank concentration will be qualified not detected (U)
Lab Duplicates	1 per analytical batch	5% RPD	Qualify data as needed	Lab and/or Polly Newbold or designee, ddms	Precision	RPD ≤ 5%

[1]- See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]-See RDWP Appendices (Site-Wide Soils and Sediments-A, In-Situ Sequestration of DU & U-B, Holding Basin Containment-C, 1,4-dioxane and VOCs in Bedrock Groundwater-D) for PDIs, IPs and Appendix I-FSP.

QAPP Worksheet #28-18 QC Samples — Metals						
Matrix	Leachate	**Analytical Method/ SOP Reference**	SW846 6010B/ 26796	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	Metals	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	H&A			
Sampling SOP[1]	NMI-S-001 – NMI-S-007	**Analytical Organization**	AAM			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Field duplicate	1 per 20 field samples	RPD ≤ 30%	Qualify data as needed	Polly Newbold or designee, ddms	Precision	RPD ≤ 30% for concentrations ≥RL
Method blanks	1/batch	< RL	Qualify data as needed or reanalysis of batch	Lab personnel	Accuracy/Bias contamination	Sample concentrations ≤ blank concentration will be qualified as not detected (U)
Field, Equipment blanks	1 per 20 field samples	< RL	Qualify data as needed	Lab and/or Polly Newbold or designee, ddms	Accuracy/Bias contamination	Sample concentrations ≤ blank concentration will be qualified as not detected (U)
MS/MSD	1/batch	%R 80-120, RPD<20	If the LCS meets acceptance criteria, no corrective action required. Qualify data as needed.	Lab personnel	Accuracy/Bias/ Precision	%R 80-120, RPD≤20
Laboratory duplicate	1/batch	RPD ≤ 20%	Qualify data as needed	Lab personnel	Precision	RPD ≤ 20% for results > RL.
LCS/LCSD	1/batch	%R 80-120 RPD≤20	If sufficient sample volume, redigest entire batch. Otherwise, qualify data as needed	Lab and/or Polly Newbold or designee, ddms	Accuracy/Bias/ Precision	%R 80-120 RPD≤20

QAPP Worksheet #28-18 QC Samples — Metals						
Matrix	Leachate	**Analytical Method/ SOP Reference**	SW846 6010B/ 26796	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	Metals	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	H&A			
Sampling SOP[1]	NMI-S-001 – NMI-S-007	**Analytical Organization**	AAM			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Post Spike	1/batch	%R 75-125%	Qualify data as needed	Lab personnel	Accuracy/Bias	%R 75-125%
Serial dilution	1/batch	%D $\leq$ 10% for concentrations $\geq$50X MDL	Qualify data as needed	Lab personnel	Precision/Bias	%D $\leq$ 10% for concentrations $\geq$50X MDL < 10%

[1]- See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]-See RDWP Appendices (Site-Wide Soils and Sediments-A, In-Situ Sequestration of DU & U-B, Holding Basin Containment-C, 1,4-dioxane and VOCs in Bedrock Groundwater-D) for PDIs, IPs and Appendix I-FSP.

QAPP Worksheet #28-18 QC Samples — Total Dissolved Solids

Matrix	Aqueous	**Analytical Method/ SOP Reference**	SM 2540C / GL-GC-E--1	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	Total Dissolved Solids	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020	**Analytical Organization**	GEL			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Field duplicate	1 per 20 field samples	RPD ≤ 30%	Qualify data as needed	Polly Newbold or designee, ddms	Precision	RPD < 30% For concentrations ≥ RL
Method blank	1 per analytical batch	< RL	Qualify data as needed or reanalysis of batch	Lab personnel	Accuracy/bias contamination	Sample concentrations ≤ blank concentration will be qualified not detected (U)
Field/Equipment blanks	1 per 20 field samples	< RL	Qualify data as needed	Polly Newbold or designee, ddms	Accuracy/bias contamination	Sample concentrations ≤ blank concentration will be qualified not detected (U)
Lab Duplicates	1 per analytical batch	5% RPD	Qualify data as needed	Lab and/or Polly Newbold or designee, ddms	Precision	RPD ≤ 5%
LCS	1 per analytical batch	%R 95-105	Repeat LCS; reanalyze all samples in batch	Lab personnel	Accuracy	%R 95-105

[1]-See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]-See RDWP Appendices (Site-Wide Soils and Sediments-A, In-Situ Sequestration of DU & U-B, Holding Basin Containment-C, 1,4-dioxane and VOCs in Bedrock Groundwater-D) for PDIs, IPs and Appendix I-FSP.

QAPP Worksheet #28-19 QC Samples — Nitrate/Nitrite

Matrix	Aqueous	**Analytical Method/ SOP Reference**	EPA 353.2 / GL-GC-E-128	**No. of Sample Locations**	See footnote2 below	
Analytical Group	Nitrate/Nitrite	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020	**Analytical Organization**	GEL			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Field duplicate	1 per 20 field samples	RPD $\leq$ 30%	Qualify data as needed	Polly Newbold or designee, ddms	Precision - overall	RPD $\leq$ 30% For concentrations ≥ RL
Method blanks	1 per analytical batch	< RL	Qualify data as needed or reanalysis of batch	Lab personnel	Accuracy/bias contamination	Sample concentrations $\leq$ blank concentration will be qualified not detected (U)
Field, Equipment blanks	1 per 20 field samples	< RL	Qualify data as needed	Polly Newbold or designee, ddms	Accuracy/bias contamination	Sample concentrations $\leq$ blank concentration will be qualified not detected (U)
MS	1 per analytical batch	Laboratory acceptance limit	Qualify data as needed	Lab personnel	Accuracy/bias	%R 75-125
LCS	1 per analytical batch	%R 90-110	If sufficient sample is available, reanalyze samples; otherwise qualify data as needed	Lab personnel	Accuracy/bias	%R 90-110

QAPP Worksheet #28-19 QC Samples — Nitrate/Nitrite

Matrix	Aqueous	**Analytical Method/ SOP Reference**	EPA 353.2 / GL-GC-E-128	**No. of Sample Locations**	See footnote2 below	
Analytical Group	Nitrate/Nitrite	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020	**Analytical Organization**	GEL			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Lab duplicate	1 per analytical batch	RPD≤20	Qualify data as needed	Lab personnel	Precision	RPD ≤ 20% Results ≥ RL

Note: *An analytical batch is defined as no more than 20 field samples, blanks, MS and LCS.
[1]-See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]-See RDWP Appendices (Site-Wide Soils and Sediments-A, In-Situ Sequestration of DU & U-B, Holding Basin Containment-C, 1,4-dioxane and VOCs in Bedrock Groundwater-D) for PDIs, IPs and Appendix I-FSP.

QAPP Worksheet #28-19 QC Samples — VOCs

Matrix	Soil, Sediment	**Analytical Method/ SOP Reference**	SW846 8260C/2108	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	VOCs	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	H&A			
Sampling SOP[1]	NMI-S-001 - NMI-S-007	**Analytical Organization**	AAW			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Field duplicate	1 per 20 field samples	RPD < 30%	Qualify data as needed	Polly Newbold or designee, ddms	Precision	RPD ≤ 30% for concentrations ≥RL
Surrogates	3 per sample	%R, 70-130%	Reanalyze sample, surrogate high sample ND, report data and qualify	Lab personnel	Accuracy/bias	%R, 70-130%, %R<10 non-detects will be rejected (R)
Method blanks	1 per analytical batch	< RL	Qualify data as needed or reanalyze batch	Lab personnel	Accuracy/bias contamination	Sample concentrations ≤ blank concentration* will be qualified not detected (U)
LCS/LCSD*	1/batch	%R 70-130, RPD≤20	If sufficient sample volume, reanalyze entire batch. Otherwise, qualify data as needed	Lab personnel	Accuracy/Bias/ Precision	%R 70-130, RPD≤20
Internal standard	At least three/sample	Area response 50-200% of CC +/- 0.06 RRT	Reanalyze sample	Lab personnel	Precision	Area response 50-200% of CC +/- 0.06 RRT

QAPP Worksheet #28-19 QC Samples — VOCs

Matrix	Soil, Sediment	**Analytical Method/ SOP Reference**	SW846 8260C/2108	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	VOCs	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	H&A			
Sampling SOP[1]	NMI-S-001 - NMI-S-007	**Analytical Organization**	AAW			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
MS/MSD	1/batch	%R 70-130, RPD≤30	If the LCS meets acceptance criteria, no corrective action required. Qualify data as needed.	Lab and/or Polly Newbold or designee, ddms	Accuracy/Bias/ Precision	%R 70-130, RPD≤50

*-Methylene chloride and toluene ≤2X blank concentration
[1]- See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]-See RDWP Appendices (Site-Wide Soils and Sediments-A, In-Situ Sequestration of DU & U-B, Holding Basin Containment-C, 1,4-dioxane and VOCs in Bedrock Groundwater-D) for PDIs, IPs and Appendix I-FSP.

QAPP Worksheet #28-20 QC Samples — Total Sulfide

Matrix	Aqueous	**Analytical Method/ SOP Reference**	SM4500S2-D / GL-GC-E-052	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	Total Sulfide	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	Geosyntec			
Sampling SOP[1]	NMI-GW-001 - NMI-GW-020	**Analytical Organization**	GEL			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Field duplicate	1 per 20 field samples	RPD ≤ 30%	Qualify data as needed	Poly Newbold or designee, ddms	Precision	RPD ≤ 30% For concentrations ≥ RL
Method blanks	1 per analytical batch	< RL	Qualify data as needed or reanalysis of batch	Lab personnel	Accuracy/bias contamination	Sample Concentrations ≤ RL will be qualified not detected (U).
Field/Equipment blanks	1 per 20 field samples	< RL	Qualify data as needed	Poly Newbold or designee, ddms	Accuracy/bias contamination	Sample Concentrations ≤ RL will be qualified not detected (U).
MS	1 per analytical batch	%R 75-125	If LCS acceptable no corrective action; qualify data as needed.	Lab personnel	Accuracy/bias	%R 75-125%
LCS	1 per analytical batch	%R 85-115	If sufficient sample volume, re-analyze entire batch. Otherwise, qualify data as needed	Lab personnel	Accuracy/bias	%R 85-115
Lab duplicate	1 per analytical batch	RPD≤15	Qualify data as needed	Lab personnel	Precision	RPD≤15

[1]- See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]-See RDWP Appendices (Site-Wide Soils and Sediments-A, In-Situ Sequestration of DU & U-B, Holding Basin Containment-C, 1,4-dioxane and VOCs in Bedrock Groundwater-D) for PDIs, IPs and Appendix I-FSP.

QAPP Worksheet #28-20 QC Samples — SVOCs

Matrix	Soil, Sediment	**Analytical Method/ SOP Reference**	SW846 8270D/2111	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	SVOCs	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	H&A			
Sampling SOP[1]	NMI-S-001 - NMI-S-007	**Analytical Organization**	AAW			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Field duplicate	1 per 20 field samples	RPD ≤ 30%	Qualify data as needed	Polly Newbold or designee, ddms	Precision	RPD ≤ 50% for concentrations ≥RL
Surrogates	3 BN/3 AC per sample	%R 30-130 for base-neutral surrogates and 15-110% for acid surrogates	Reanalyze and/or re-extract sample, surrogate high sample ND, report data and qualify	Lab personnel	Accuracy/bias	%R, 70-130%, %R<10 non-detects will be rejected (R)
Method blanks	1 per analytical batch	< RL	Qualify data as needed or re-extract and reanalyze batch	Lab personnel	Accuracy/bias contamination	Sample concentrations ≤ blank concentration will be qualified not detected (U)
LCS/LCSD*	1/batch	%R 40 - 140 for base-neutral compounds, %R 30 - 130% for acid compounds except for "difficult" analytes which must be between 15-140%, RPD≤30	If sufficient sample volume, re-extract entire batch. Otherwise, qualify data as needed	Lab personnel	Accuracy/Bias/ Precision	%R 70-130, RPD≤20

QAPP Worksheet #28-20 QC Samples — SVOCs

Matrix	Soil, Sediment	**Analytical Method/ SOP Reference**	SW846 8270D/2111	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	SVOCs	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	H&A			
Sampling SOP[1]	NMI-S-001 - NMI-S-007	**Analytical Organization**	AAW			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Internal standard	At least three/sample	Area response 50-200% of CC +/- 0.06 RRT	Reanalyze sample	Lab personnel	Precision	Area response 50-200% of CC +/- 0.06 RRT
MS/MSD	1/batch	%R 40 - 140 for base-neutral compounds, %R 30 - 130% for acid compounds except for "difficult" analytes which must be between 15-140%, RPD≤30	If the LCS meets acceptance criteria, no corrective action required. Qualify data as needed.	Lab and/or Polly Newbold or designee, ddms	Accuracy/Bias/ Precision	%R 70-130, RPD≤20

[1]- See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]-See RDWP Appendices (Site-Wide Soils and Sediments-A, In-Situ Sequestration of DU & U-B, Holding Basin Containment-C, 1,4-dioxane and VOCs in Bedrock Groundwater-D) for PDIs, IPs and Appendix I-FSP.

QAPP Worksheet #28-21 QC Samples —Pesticides

Matrix	Soil, Sediment	**Analytical Method/ SOP Reference**	SW846 8081B/2116	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	Pesticides	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	H&A			
Sampling SOP[1]	NMI-S-001 - NMI-S-007	**Analytical Organization**	AAW			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Field Duplicates	1 per 20 field samples	RPD ≤ 50%	Qualify data as required	Polly Newbold or designee, ddms	Precision	RPD < 50% for concentrations >RL
Surrogate Spike	2 per sample	%R 30-150	If sufficient sample volume is available, re-extract and reanalyze the samples	Lab and/or Polly Newbold or designee, ddms	Accuracy/Bias	%R, 70-130%, %R<10 non-detects will be rejected (R)
Method Blank	1 per extraction batch	<RL	Qualify data as needed or re-extract and reanalyze batch	Lab personnel	Accuracy/Bias/ Contamination	Sample concentrations ≤ blank concentration will be qualified not detected (U)
LCS/LCSD	1 per extraction batch	%R 40-140 RPD≤20	If sufficient sample volume is available, re-extract and reanalyze the samples; otherwise qualify data as needed	Lab and/or Polly Newbold or designee, ddms	Accuracy/Bias/ Precision	70-130% RPD< 20

QAPP Worksheet #28-21 QC Samples —Pesticides

Matrix	Soil, Sediment	**Analytical Method/ SOP Reference**	SW846 8081B/2116	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	Pesticides	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	H&A			
Sampling SOP[1]	NMI-S-001 - NMI-S-007	**Analytical Organization**	AAW			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
MS/MSD	1 per extraction batch	%R 30-150 RPD≤30	If the LCS meets acceptance criteria, no corrective action required. Qualify data as needed.	Lab and/or Polly Newbold or designee, ddms	Accuracy/Bias/ Precision	70-130% RPD ≤ 30%
Internal Standard	Each sample	-50 to +100% of CC	If internal standard is outside of limits, reanalyze sample unless chromatographic interference is present.	Lab personnel	Precision	-50 to +100% of CC
Identification and Quantitation	Each Sample	Minimum3 peaks used for quantitation. RPD<45 between columns	Reanalyze sample if RPD>100 at a dilution. Both runs must be reported	Lab Personnel	Accuracy	Minimum3 peaks used for quantitation. RPD<25 between columns.

[1]- See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]-See RDWP Appendices (Site-Wide Soils and Sediments-A, In-Situ Sequestration of DU & U-B, Holding Basin Containment-C, 1,4-dioxane and VOCs in Bedrock Groundwater-D) for PDIs, IPs and Appendix I-FSP.

QAPP Worksheet #28-22 QC Samples —PCBs

Matrix	Soil, Sediment	**Analytical Method/ SOP Reference**	SW846 8082A/2129	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	PCBs	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	H&A			
Sampling SOP[1]	NMI-S-001 - NMI-S-007	**Analytical Organization**	AAW			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Field Duplicates	1 per 20 field samples	RPD ≤ 50%	Qualify data as required	Polly Newbold or designee, ddms	Accuracy	RPD < 50% for concentrations >RL
Surrogate Spike	2 per sample	%R 30-150	If sufficient sample volume is available, re-extract and reanalyze the samples	Lab and/or Polly Newbold or designee, ddms	Accuracy	%R, 70-130%, %R<10 non-detects will be rejected (R)
Method Blank	1 per extraction batch	<RL	Qualify data as needed or re-extract and reanalyze batch	Lab personnel	Accuracy	Sample concentrations ≤ blank concentration will be qualified not detected (U)
LCS/LCSD	1 per extraction batch	%R 40-140 RPD≤20	If sufficient sample volume, re-extract entire batch. Otherwise, qualify data as needed	Lab and/or Polly Newbold or designee, ddms	Accuracy	70-130% RPD< 20
MS/MSD	1 per extraction batch	%R 40-140 RPD≤30	If the LCS meets acceptance criteria, no corrective action required. Qualify data as needed.	Lab and/or Polly Newbold or designee, ddms	Accuracy	70-130% RPD ≤ 30%

QAPP Worksheet #28-22 QC Samples —PCBs

Matrix	Soil, Sediment	**Analytical Method/ SOP Reference**	SW846 8082A/2129	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	PCBs	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	H&A			
Sampling SOP[1]	NMI-S-001 - NMI-S-007	**Analytical Organization**	AAW			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Internal Standard	Each sample	-50 to +100% of CC	If internal standard is outside of limits, reanalyze sample unless chromatographic interference is present.	Lab personnel	Precision	-50 to +100% of CC
Identification and Quantitation	Each Sample	Minimum3 peaks used for quantitation. RPD<45 between columns	Reanalyze sample if RPD>100 at a dilution. Both runs must be reported	Lab Personnel	Accuracy	Minimum3 peaks used for quantitation. RPD<25 between columns.

[1]- See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]-See RDWP Appendices (Site-Wide Soils and Sediments-A, In-Situ Sequestration of DU & U-B, Holding Basin Containment-C, 1,4-dioxane and VOCs in Bedrock Groundwater-D) for PDIs, IPs and Appendix I-FSP.

QAPP Worksheet #28-23 QC Samples —Herbicides

Matrix	Leachate	**Analytical Method/ SOP Reference**	SW846 8081B/2116	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	Herbicides	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	H&A			
Sampling SOP	NMI-S-001 - NMI-S-007	**Analytical Organization**	AAW			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Field Duplicates	1 per 20 field samples	RPD $\leq$ 30%	Qualify data as required	Polly Newbold or designee, ddms	Accuracy	RPD < 30% for concentrations >RL
Surrogate Spike	2 per sample	%R 30-150	If sufficient sample volume is available, re-extract and reanalyze the samples	Lab and/or Polly Newbold or designee, ddms	Accuracy	%R, 70-130%, %R<10 non-detects will be rejected (R)
Method Blank	1 per extraction batch	<RL	Qualify data as needed or re-extract and reanalyze batch	Lab personnel	Accuracy	Sample concentrations <5X blank concentration will be qualified not detected (U)
Field, Equipment Blank	1 per 20 field samples	<RL	Qualify data as required	Polly Newbold or designee, ddms	Accuracy	Sample concentrations <5X blank concentration will be qualified not detected (U)
LCS/LCSD	1 per extraction batch	%R 40-140 RPD$\leq$20	If sufficient sample volume, re-extract entire batch. Otherwise, qualify data as needed	Lab and/or Polly Newbold or designee, ddms	Accuracy/Bias/ Precision	70-130% RPD< 20

QAPP Worksheet #28-23 QC Samples —Herbicides

Matrix	Leachate	**Analytical Method/ SOP Reference**	SW846 8081B/2116	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	Herbicides	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	H&A			
Sampling SOP	NMI-S-001 - NMI-S-007	**Analytical Organization**	AAW			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
MS/MSD	1 per extraction batch	%R 30-150 RPD≤30	If the LCS meets acceptance criteria, no corrective action required. Qualify data as needed.	Lab and/or Polly Newbold or designee, ddms	Accuracy/Bias/ Precision	70-130% RPD < 30%
Internal Standard	Each sample	-50 to +100% of CC	If internal standard is outside of limits, reanalyze sample unless chromatographic interference is present.	Lab personnel	Precision	-50 to +100% of CC
Identification and Quantitation	Each Sample	Minimum3 peaks used for quantitation. RPD<45 between columns	Reanalyze sample if RPD>100 at a dilution. Both runs must be reported	Lab Personnel	Accuracy	Minimum3 peaks used for quantitation. RPD<25 between columns.

[1]- See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]-See RDWP Appendices (Site-Wide Soils and Sediments-A, In-Situ Sequestration of DU & U-B, Holding Basin Containment-C, 1,4-dioxane and VOCs in Bedrock Groundwater-D) for PDIs, IPs and Appendix I-FSP.

QAPP Worksheet #28-24 QC Samples — pH

Matrix	Soil	**Analytical Method/ SOP Reference**	SW846 9045D / 2203	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	pH, Corrosivity	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	H&A			
Sampling SOP[1]	NMI-S-001 - NMI-S-007	**Analytical Organization**	AAW			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Lab Duplicates	1 per 20 field samples	RPD≤5	Qualify data as needed	Lab and/or Polly Newbold or designee, ddms	Precision	±0.5 pH units

[1]- See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]-See RDWP Appendices (Site-Wide Soils and Sediments-A, In-Situ Sequestration of DU & U-B, Holding Basin Containment-C, 1,4-dioxane and VOCs in Bedrock Groundwater-D) for PDIs, IPs and Appendix I-FSP.

QAPP Worksheet #28-25 QC Samples — Reactive Cyanide and Reactive Sulfide

Matrix	Soil	**Analytical Method/ SOP Reference**	Laboratory Developed Method / 2228	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	Reactive Cyanide and Reactive Sulfide	**Sampler's Name**	TBD			
Concentration Level	All	**Field Sampling Organization**	H&A			
Sampling SOP[1]	NMI-S-001 - NMI-S-007	**Analytical Organization**	AAW			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Method blanks	1 per analytical batch	< RL	Qualify data as needed or reanalysis of batch	Lab personnel	Accuracy/Bias contamination	Sample Concentrations ≤ RL will be qualified not detected (U).
Lab Duplicates	1 per analytical batch	RPD≤25 (aqueous) RPD≤40 (soil)	Qualify data as needed	Lab personnel	Precision	RPD≤25 (aqueous) RPD≤40 (soil)
LCS	1 per analytical batch	%R 60-125 (RS) %R 30-125 (RCN)	If sufficient sample is available, reanalyze batch	Lab and/or Polly Newbold or designee, ddms	Accuracy/Bias	%R 70-130

[1]- See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]-See RDWP Appendices (Site-Wide Soils and Sediments-A, In-Situ Sequestration of DU & U-B, Holding Basin Containment-C, 1,4-dioxane and VOCs in Bedrock Groundwater-D) for PDIs, IPs and Appendix I-FSP.

QAPP Worksheet #28-12 QC Samples — Ignitability

Matrix	Soil	**Analytical Method/ SOP Reference**	SW846 1030/ 2238	**No. of Sample Locations**	See footnote 2 below	
Analytical Group	Ignitability	**Sampler's Name**	NATBD			
Concentration Level	All	**Field Sampling Organization**	H&A			
Sampling SOP[1]	NMI-S-001 - NMI-S-007	**Analytical Organization**	AAW			
QC Sample	**Frequency/Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (Data Validation/Review)**
Triplicate Analysis	If any sample has a positive result, the sample must be analyzed in triplicate.	RPD≤15	Qualify data as needed	Lab personnel	Precision	RPD≤15

[1]- See Table 3-1: Summary of Standard Operating Procedures Required by Pre-Design Investigation, Appendix I, Field Sampling Plan of the RDWP.
[2]-See RDWP Appendices (Site-Wide Soils and Sediments-A, In-Situ Sequestration of DU & U-B, Holding Basin Containment-C, 1,4-dioxane and VOCs in Bedrock Groundwater-D) for PDIs, IPs and Appendix I-FSP.

QAPP Worksheet #29 Project Documents and Records

Sample Collection Documents and Records	On-site Analysis Documents and Records[1]	Off-site Analysis Documents and Records	Data Assessment Documents and Records	Other
- Field Notes - Sampling Logs - Chain-of-Custody Records - Air Bills - Custody Seals	- Equipment Calibration Logs - Field Data Records - Field Instrument Maintenance Logs	- Sample Receipt, Custody and Tracking Records - Standard Traceability Logs - Equipment Calibration Logs - Sample Prep Logs - Run Logs - Equipment Maintenance, Testing and Inspection Logs - Corrective Action Forms - Reported Field Sample Results - Reported Results for Standards, QC Checks and QC Samples - Instrument Printouts (raw data) for Field Samples, Standards, QC Checks and QC Samples - Data Package Completeness Checklists - Sample Disposal Records - Extraction/Cleanup Records - Raw Data (stored on disk or CD-R) - Analytical Reports to Polly Newbold, ddms	- Data Validation Checklists/reports - Data Quality Assessments	- FSP - HASP - QAPP - Data Management Plan - Treatability Study Summary Reports - Risk Assessments - Routine Monitoring Reports - Record of Decision documents - Proposed Plans - Feasibility Studies - Long-Term Monitoring Reports

QAPP Worksheet #29 Project Documents and Records

Sample Collection Documents and Records	On-site Analysis Documents and Records[1]	Off-site Analysis Documents and Records	Data Assessment Documents and Records	Other
- Field Notes - Sampling Logs - Chain-of-Custody Records - Air Bills - Custody Seals	- Equipment Calibration Logs - Field Data Records - Field Instrument Maintenance Logs	- Sample Receipt, Custody and Tracking Records - Standard Traceability Logs - Equipment Calibration Logs - Sample Prep Logs - Run Logs - Equipment Maintenance, Testing and Inspection Logs - Corrective Action Forms - Reported Field Sample Results - Reported Results for Standards, QC Checks and QC Samples - Instrument Printouts (raw data) for Field Samples, Standards, QC Checks and QC Samples - Data Package Completeness Checklists - Sample Disposal Records - Extraction/Cleanup Records - Raw Data (stored on disk or CD-R) - Analytical Reports to Polly Newbold, ddms	- Data Validation Checklists/reports - Data Quality Assessments	- FSP - HASP - QAPP - Data Management Plan - Treatability Study Summary Reports - Risk Assessments - Routine Monitoring Reports - Record of Decision documents - Proposed Plans - Feasibility Studies - Long-Term Monitoring Reports

Worksheet 30
Analytical Services Table

Matrix	***Analytical Group***	**Concentration Level**	**Analytical SOP**	**Data Package Turnaround Time**	**Laboratory/Organization (Name and Address, Contact Person and Telephone Number)**	**Backup Laboratory/Organization (Name and Address, Contact Person and Telephone Number)**
Soil Gas	*VOCs*	All	2186	10 days for EDD and PDF of Level 2, and 20 days for Level 4 Data Packages	Alpha Analytical 320 Forbes Blvd, Mansfield, MA 02048	NA
Groundwater and/or IDW	*PFAS*	All	23528			
	VOCs	All	23639		Alpha Analytical 8 Walkup Dr. Westborough, MA 01581 Dave Sanford 508.898.9220	
	SVOCs	All	1948/2111			
	1,4-Dioxane	All	1948/2164			
	Pesticides	All	1948/2116			
	Perchlorate	All	2251			
	Metals	All	GL-MA-E-006/ GL-MA-E-014	20 days for EDD and PDF of Level 4 Data Packages	GEL Laboratories 2040 Savage Road, Charleston, SC 29407 Edie Kent 843.769.7385	NA
	Mercury	All	GL-MA-E-010			
	Polonium-210	All	RAD-A-016			
	Total Hardness	All	GL-GE-E-100			

Worksheet 30
Analytical Services Table

	TOC, DOC TIC, DIC	All	GC-E-093			
	Alkalinity, Total	All	2213	10 days for EDD and PDF of Level 2, and 20 days for Level 4 Data Packages	Alpha Analytical 8 Walkup Dr. Westborough, MA 01581 Dave Sanford 508.898.9220	NA
	Bromide, Chloride, Sulfate, Fluoride	All	2214			
	ortho-Phosphate	All	2225			
	Total Phosphorous	All	2225			
	Ammonia, Nitrogen	All	2206			
	Nitrogen, Nitrite Nitrogen, Nitrate Nitrate/Nitrite, Nitrogen	All	2217			
	Total Dissolved Solids	All	2219			
	Total Suspended Solids	All	2220			
	pH	All	2202			
	Total Sulfide	All	2221			
Soil/Sediment	VOCs	All	32639	10 days for EDD and PDF of Level 2, and 20 days for Level 4 Data Packages	Alpha Analytical 8 Walkup Dr. Westborough, MA 01581 Dave Sanford 508.898.9220	NA
	SVOCs	All	1959/2111			

Worksheet 30
Analytical Services Table

	Metals	All	GL-MA-E-009/ GL-MA-E-014	20 days for EDD and PDF of Level 4 Data Packages	GEL Laboratories 2040 Savage Road, Charleston, SC 29407 Edie Kent 843.769.7385	
	Lead-210	All	RAD-A-018			
	Bismouth-210, Actinium-227	All	RAD-A-013			
	Uranium-234	All	GL-RAD-A-011			
	Thorium-232	All	GL-RAD-A-038			
	Mercury	All	GL-MA-E-010			
	PCBs	All	1954/2129	10 days for EDD and PDF of Level 2, and 20 days for Level 4 Data Packages	Alpha Analytical 8 Walkup Dr. Westborough, MA 01581 Dave Sanford 508.898.9220	
	Pesticides	All	1959/2116			
Waste Characterization	TCLP/VOC	All	2112/2108	10 days for EDD and PDF of Level 2, and 20 days for Level 4 Data Packages	Alpha Analytical 8 Walkup Dr. Westborough, MA 01581 and Alpha Analytical 320 Forbes Blvd, Mansfield, MA 02048 Dave Sanford 508.898.9220	NA
	TCLP/SVOC	All	2133/1948/2111			
	TCLP/Pesticides	All	2133/1948/2116			
	TCLP/Herbicides	All	2133/1948/2128			
	TCLP/Metals	All	2133/26796			
	PCBs	All	1954/2129			
	RCN	All	2228			
	RS	All	2228			
	Ignitability	All	2238			
	pH/Corrosivity	All	2203			

QAPP Worksheet #31 Planned Project Assessments

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) Responsible for Performing Assessment (Title and Organizational Affiliation)	Person(s) Responsible for Responding to Assessment Findings (title and organizational affiliation)	Person(s) Responsible for Identifying and Implementing Corrective Actions (CAs) (title and organizational affiliation)	Person(s) Responsible for Monitoring Effectiveness of CA (title and organizational affiliation)
Field Audit (Each Sampling Program)	1 per event	Internal	de maximis, O&M, H&A, GS	Field Project Manager	Field Team Coordinator	Project Manager	Bruce Thompson, Project Coordinator, de maximis
Laboratory Audit of Alpha Analytical Westborough	As necessary based on laboratory performance	External	ddms	Polly Newbold QA Manager/Validator ddms	Jim Todaro QA Officer Alpha Analytical	Jim Todaro QA Officer Alpha Analytical	Bruce Thompson, Project Coordinator, de maximis
Laboratory Audit of GEL	As necessary based on laboratory performance	External	ddms	Polly Newbold QA Manager/Validator ddms	Nancy Mattern QA Officer GEL	Nancy Mattern QA Officer GEL	Bruce Thompson, Project Coordinator, de maximis
Field Inspections	Intermittent	Internal	de maximis	Task Manager, de maximis	Field Team Coordinator, H&A or GS	Project Manager, H&A or GS	Bruce Thompson, Project Coordinator, de maximis
Safety Audits	Annually and/or Field Assessment to determine frequency (based on > 400 field hours/ quarter)	Internal	de maximis	Corporate H&S Staff, Regional H&S Manager or Regional H&S Specialist	Field Team Coordinator, H&A or GS	Project Manager, H&A or GS	Bruce Thompson, Project Coordinator, de maximis

QAPP Worksheet #32 Assessment Findings and Corrective Action Responses

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (name, title, organization)	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response (name, title, organization)	Timeframe for Response
Field Sampling Technical Systems Audit	Written Audit Report	Project Manager, H&A or GS, Task Manager, de maximis, and Bruce Thompson, Project Coordinator, de maximis	72 hours after audit	Letter	Field Team Coordinators	48 hours after notification
Contract Laboratory Technical Audit (external)	Written Audit Report	Project Manager, H&A or GS, Bruce Thompson, Project Coordinator, de maximis, Polly Newbold QA Manager/Validator, ddms, inc.	1 week after audit	Letter	Dave Sanford, Alpha Analytical, and Edie Kent, GEL Laboratory QA Manager	48 hours after notification
Field Inspection	Memorandum	Field Team Coordinators, H&A or GS and Task Manager, de maximis	2 days	Memorandum	Project Manager, H&A or GS and Bruce Thompson, Project Coordinator, de maximis	48 hours after notification
Contract Laboratory Technical Audit (internal)	Memorandum	Polly Newbold QA Manager/Validator, **ddms, inc.**	2 days	Memorandum	Dave Sanford, Alpha Analytical and Edie Kent, GEL, Laboratory QA Manager	48 hours after notification

QAPP Worksheet #33 QA Management Reports

Type of Report	Frequency (e.g., daily, weekly monthly, quarterly, annually)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (title and organizational affiliation)	Report Recipient(s) (title and organizational affiliation)
Field Sampling Technical Systems Audit Report	As necessary during project	NA	(Project Manager/Field Program Coordinator), de maximis, O&M, H&A or GS	Bruce Thompson, Project Coordinator, de maximis
Contract Laboratory Technical Audit Report	As necessary during project	NA	Polly Newbold QA Manager/Validator, ddms	Bruce Thompson, Project Coordinator, de maximis
Data Validation Reports	As specified in the Data Assessment section based on intended use of the data and required percentage to meet project DQOs	As generated throughout project	Polly Newbold QA Manager/Validator, ddms	Bruce Thompson, Project Coordinator, de maximis, Project Manager/Field Program Coordinators, H&A or GS or de maximis
Data Quality Summary Reports	As appropriate for data use and as required for report completion	As generated throughout project	Polly Newbold QA Manager/Validator, ddms	Bruce Thompson, Project Coordinator, de maximis, Project Managers/Field Program Coordinators, de maximis or H&A or GS

QAPP Worksheet #34: Data Verification and Validation Inputs

Item/Description	Verification (Completeness)	Validation (Conformance to specifications)
Planning Documents/Records		
1. Approved QAPP	x	
2. Contract	x	
3. Field SOPs	x	
4. Laboratory SOPs	x	
Field Records		
5. Field logbooks	x	x
6. Equipment calibration records	x	x
7. Chain of custody forms	x	x
8. Sampling diagrams/surveys	x	x
9. Drilling logs	x	x
10. Relevant correspondence	x	x
11. Deviations	x	x
12. Field audit reports	x	x
13. Field CA reports	x	x
Analytical Data Package		
14. Cover sheet (laboratory identifying information)	x	x
15. Case narrative	x	x
16. Inter-laboratory chain-of-custody documents	x	x
17. Sample receipt records	x	x
18. Sample chronology (i.e., dates and times of receipt, prep, analysis)	x	x
19. Laboratory bench sheets and instrument logs	x	
20. MDL/RL establishment and verification	x	x
21. Standards traceability	x	x
22. Instrument calibration records	x	x
23. Definition of laboratory qualifiers	x	x
24. Results reporting forms	x	x

QAPP Worksheet #34: Data Verification and Validation Inputs (Continued)

Item/Description	Verification (Completeness)	Validation (Conformance to specifications)
25. QC sample results	x	x
26. CA reports (summarized in Case Narrative)	x	x
27. Raw data for tunes, calibrations, QC samples, and field samples	x	x
28. Electronic data deliverable	x	x
29. Certification Summary	x	

QAPP Worksheet #35: Data Verification

Records Reviewed	Requirement Documents	Process Description	Responsible Person, Organization
Field forms	QAPP, field SOPs	Verify that records are present and complete for each day of field activities. Verify that all planned samples including field QC samples were collected and that sample collection locations are documented. Verify that meteorological data were provided for each day of field activities. Verify that changes/exceptions are documented and were reported in accordance with requirements. Verify that any required field monitoring was performed, and results are documented.	Project QA Officer / Geosyntec / H&A, O&M, ddms
COC forms	QAPP	Verify the completeness of COC records. Examine entries for consistency with the field logbook. Check that appropriate methods and sample preservation have been recorded. Verify that the required volume of sample has been collected and that sufficient sample volume is available for QC samples (e.g. MS/MSD). Verify that all required signatures and dates are present. Check for transcription errors. Verify that laboratory receipt and log-in conform to field COC requests and to the QAPP. Contact Jessie McCusker, Polly Newbold and Jeri Ross if samples have been lost upon receipt, or if holding times will not be met.	Project QA Officer / Geosyntec / H&A, O&M, ddms
Laboratory deliverable	QAPP	Verify that the laboratory deliverable contains all records specified in the QAPP. Check sample receipt records to ensure sample condition upon receipt was noted, and any missing/broken sample containers were noted and reported according to plan. Compare the data package with the COCs to verify that results were provided for all collected samples. Review the narrative to ensure all QC exceptions are described. Establish that all QAPP-required QC samples were run and met required criteria. Determine that all sample results met the project quantitation limits specified in QAPP Worksheet #15. Check for evidence that any required notifications were provided to project personnel as specified in the QAPP. Verify that necessary signatures and dates are present.	ddms
Audit Reports, CA Reports (if required)	QAPP	Verify that all planned audits were conducted. Examine audit reports. For any deficiencies noted, verify that CA was implemented according to plan.	Not requried

QAPP Worksheet #36: Data Validation Procedures

Matrix	Analytical Group	Concentration Level	Validation Criteria*	Data Validator (title and organizational affiliation)
All	All	Low	Validation will be performed according to the data validation procedures specified in the EPA Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use, ddms' SOPs, the analytical method and laboratory SOPs. The National Functional Guidelines may be consulted. Professional judgment with be applied as necessary and appropriate. All data packages for each method and matrix and sampling round excluding waste characterization will receive a Stage 2B data verification/validation.	Project QA Officer/ddms

* These guidelines will be modified to reflect any differences in analytical methodology and to incorporate the project-specific acceptance criteria defined per this QAPP.

The following data qualifiers will be applied during data validation. Potential impacts on project-specific DQOs will be discussed in the data usability report (refer to Worksheet #37).

Qualifier	Definition
U	The analyte was analyzed for, but was not detected above the level of the reported sample QL.
J	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
J+	The result is an estimated quantity, but the result may be biased high.
J-	The result is an estimated quantity, but the result may be biased low.
NJ	The analyte has been “tentatively identified” or “presumptively” as present and the associated numerical value is the estimated concentration in the sample.
UJ	The analyte was not detected above the reported concentration. The reported QL is approximate and may or may be inaccurate or imprecise.
R	The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet QC criteria. The presence or absence of the analyte cannot be verified.

QAPP Worksheet #37 Usability Assessment

The Data Usability Assessment will be performed by ddms for data associated with delineation/investigation and data collected to inform design of remedies at the Site (e.g., changes in groundwater concentration over time during a pilot test). Laboratory data to be collected as part of the RDWP includes concentrations of chemicals in soil, groundwater, surface water and air. Appendices A through E of the RDWP provide detailed descriptions of the pre-design testing that will be performed; these work plans include tables listing where samples are collected, what they are analyzed for and the analytical methods. Documentation generated during the usability assessment will consist of data validation checklists with a brief summary of overall data usability.

The Data Usability Assessment process involves data verification and data validation. Data verification is the process by which laboratory results are checked to provide that the proper quality control steps were performed and key items have met QC objectives (both analytical and contractual). The key items checked in a ddms data verification include:

- sample collection, handling and analysis procedures
- field sampling, handling and analysis activities will be documented (e.g., QC signatures in field logs, QC checklist)
- sampling, handling, on-site analytical and off-site laboratory data will be verified internally at the data generator level
- laboratory data (e.g., laboratory-qualified data)
- sampling, on-site analytical and off-site laboratory data
- data package deliverable completeness
- review of case narrative
- present all analytical results
- QC sample data summaries
- applicable raw data

All required data deliverables must be present in the data package in order to proceed to the next step of data validation.

Data validation entails a review of the sample collection, handling, field analysis and QC data, and the raw data to verify that the laboratory was operating within required limits; analytical results were correctly transcribed from the instrument read-outs; and which (if any) environmental samples were related to out-of-control QC samples. The objective of data validation is to identify any questionable or invalid laboratory measurements.

QAPP Worksheet #37 Usability Assessment

The data quality indicators (DQIs) used to evaluate conformance with the project DQOs are presented below.

DQIs are generally defined in terms of six parameters:

1. representativeness
2. comparability
3. completeness
4. precision
5. accuracy
6. sensitivity

Each parameter is defined below. Specific objectives for the site actions are presented in other sections of this QAPP, as referenced below.

Representativeness

Representativeness is the degree to which sampling data accurately and precisely represent site conditions and is dependent on sampling and analytical variability and the variability of environmental media at the site. Actions have been designed to assess the presence of chemical constituents at the time of sampling, for example, the EPA low-flow minimal drawdown procedure is used to collect water samples representative of groundwater. The QAPP presents the rationale for sample quantities and location. This QAPP presents field sampling and laboratory analytical methodologies. Use of the prescribed field and laboratory analytical methods with associated holding times and preservation requirements are intended to provide representative data.

Comparability

Comparability is the degree of confidence with which one data set can be compared to another. Comparability between phases of the actions (if additional phases are required) will be maintained through consistent use of the sampling and analytical methodologies set forth in this QAPP, established QA/QC procedures and use of appropriately trained personnel.

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Completeness

Completeness for a task or event is defined as a measure of the amount of valid data obtained from an event and/or investigation compared to the total amount that was obtained. This will be determined upon final assessment of the analytical results. Completeness of a field or laboratory data set will be calculated by comparing the number of valid sample results generated to the total number of results generated.

$$\text{Completeness} = \frac{\text{Number valid results}}{\text{Total number of results generated}} \times 100$$

As a general guideline, overall project completeness is expected to be at least 90 percent. The assessment of completeness will require professional judgment to determine data usability for intended purposes.

Precision

Precision is a measure of the reproducibility of sample results. The goal is to maintain a level of analytical precision consistent with the objectives of the action. To maximize precision, sampling and analytical procedures will be followed. All work for the site actions will adhere to established protocols presented in the QAPP. Checks for analytical precision will include the analysis of MS/MSDs, laboratory duplicates and field duplicates. Checks for field measurement precision will include duplicate field measurements.

The precision of data will be measured by calculating the Relative Percent Difference (RPD) by the following equation:

$$\text{RPD} = \frac{(A-B)}{(A+B)/2} \times 100$$

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Where:

A = Analytical result from one of two duplicate measurements

B = Analytical result from the second measurement

Accuracy

Accuracy is a measure of how close a measured result is to the true value. Both field and analytical accuracy will be monitored through initial and continuing calibration of instruments. In addition, reference standards, MSs, blank spikes and surrogate standards will be used to assess the accuracy of the analytical data.

Accuracy will be calculated in terms of percent recovery as follows:

$$\% \text{ Recovery} = \frac{A-X}{B} \times 100$$

Where:

A = Value measured in spiked sample or standard

X = Value measured in original sample

B = True value of amount added to sample or true value of standard

Sensitivity

Sensitivity is a quantitative measurement to determine if the analytical laboratory's procedures/methodologies and their associated MDLs can satisfy the project requirements as they relate to the project clean-up levels. MDLs are updated annually by the laboratory. The current MDLs for the analytical laboratories are presented in Worksheet #15.

Data Validation and Usability

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ddms will validate all data generated using the USEPA's National Functional Guidelines (EPA-540-R-2017-001, 2017), EPA New England Environmental Data Review Supplement for Region 1: Data Review Elements and Superfund Specific Guidance/Procedures (EQADR-Supplement 1, Final #1, June 2018) and MassDEP protocols contained in the Massachusetts Contingency Plan (M.G.L c 21E, 310 CMR 40.00: Massachusetts Contingency Plan, revision dated March 2020) as needed for ARARs and as long as they do not conflict with EPA guidance. These procedures and criteria may be modified, as necessary, to address project-specific and method-specific criteria, control limits and procedures. Data validation will consist of data screening, checking, reviewing, editing and interpretation to document analytical data quality and to determine whether the quality is sufficient to meet the DQOs.

The data validator will verify that reduction of laboratory measurements and laboratory reporting of analytical parameters is in accordance with the procedures specified for each analytical method and/or as specified in this QAPP. Any deviations from the analytical method or any special reporting requirements apart from those specified in this QAPP will be detailed on COC forms.

Upon receipt of laboratory data, the following procedures will be executed by the data validator:

- Evaluate completeness of data package.
- Verify that field COC forms were completed and that samples were handled properly.
- Verify that holding times were met for each parameter. Holding time exceedances, should they occur, will be documented. Data for all samples exceeding holding time requirements will be flagged as either estimated or rejected. The decision as to which qualifier is more appropriate will be made on a case-by-case basis.
- Verify that parameters were analyzed according to the methods specified.
- Review QA/QC data (i.e., confirm that duplicates, blanks and spikes were analyzed on the required number of samples, as specified in the method and verify that results for duplicate and MS are within acceptable ranges).
- Review the laboratory narrative to understand any potential concerns about data quality and/or deviations in the analysis expressed by the laboratory.
- Investigate anomalies identified during review. When anomalies are identified, they will be discussed with the Project Manager and/or Laboratory Manager, as appropriate.
- If data appear suspect, investigate the specific data of concern. Calculations will be traced back to raw data. If calculations do not agree, the cause will be determined and corrected.

Deficiencies discovered as a result of the data review, as well as the corrective actions implemented in response, will be documented and submitted in the form of

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a written report addressing the following topics, as applicable to each method:

- assessment of the data package
- description of any protocol deviations
- failures to reconcile reported and/or raw data
- assessment of any compromised data
- overall appraisal of the analytical data
- table of site name, sample quantities, matrix and fractions analyzed

It should be noted that qualified results do not necessarily invalidate data. The goal to produce the best possible data does not necessarily mean that data must be produced without QC qualifiers. Qualified data can provide useful information.

During the review process, laboratory qualified and unqualified data are verified against the supporting documentation. Based on this evaluation, qualifier codes may be added, deleted, or modified by the data reviewer. Results will be qualified with the following codes in accordance with the USEPA National Functional Guidelines:

Validation qualifiers:

U The analyte/compound was analyzed for, but not detected. The associated value is the compound quantitation limit.

UJ The compound was not detected above the reported sample quantitation limit. However, the reported limit is approximate and may or may not represent the actual limit of quantitation.

J The compound was positively identified; however, the associated numerical value is an estimated concentration only.

J+ and J- may be used to indicate the estimated concentration (indicated by a J-qualifier) has a positive or negative bias, respectively, but only if QA/QC data provide an unambiguous direction for the bias.

JN The analysis indicates the presence of a compound for which there is presumptive evidence to make a tentative identification. The associated numerical value is an estimated concentration only.

R The sample results are rejected.

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Typical laboratory Qualifiers

Concentration (C) qualifiers:

U The analyte/compound was analyzed for but not detected. The associated value is the compound quantitation limit.

Inorganics:

B The reported value was obtained from a reading less than the required reporting limit (RL), but greater than or equal to the instrument.

Quantitation (Q) qualifiers:

Inorganics:

E The reported value is estimated due to the presence of interference.

N Spiked sample recovery not within control limits.

* Duplicate analysis not within control limits.

Organics:

J The compound was positively identified; however, the associated numerical value is an estimated concentration only.

B The compound has been found in the sample as well as its associated blank; its presence in the sample may be suspect.

N The analysis indicates the presence of a compound for which there is presumptive evidence to make a tentative identification.

P The lower of the two values is reported when the percent difference between the results of two GC columns is greater than 25 percent.

E The compound was quantitated above the calibration range.

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D Concentration is based on a diluted sample analysis.

C Identification confirmed by gas chromatography/mass spectrometry (GC/MS).

Two facts will be noted to all data users. First, the "R" flag means that the associated value is unusable. In other words, due to significant QC problems, the analysis is invalid and provides no information as to whether the compound is present or not. "R" values should not appear on data tables because they cannot be relied upon, even as a last resort. The second fact is that no compound concentration, even if it has passed all QC tests, is guaranteed to be accurate. Strict QC serves to increase confidence in data, but any value potentially contains error.

Resolution of any issues regarding laboratory performance or deliverables will be handled between the laboratory and the data validator. Suggestions for reanalysis may be made by the QAC at this point.

Validation Reports

The data validation reports will identify all deficiencies and the potential impact on the results. The Validation and Database Project Team will amend qualifiers generated during the validation process to the database. The validation checklists and the database will be the primary location of all applicable data qualifiers. The original values and laboratory qualifiers are archived and accessible. Qualifiers will not be applied to the hard copy analytical reports.

Field Data Review

Field data are generated from in-field measurement, which may include a geophysical survey, turbidity measurements from a field probe during well development, groundwater geochemical parameters (e.g., temperature) during groundwater sampling and surface-water sampling. The quality objective for the in-field measurement activities is to obtain accurate estimations of sample characteristics, including pH, conductivity, temperature, turbidity, dissolved oxygen and/or redox potential, using appropriate equipment. Data are recorded in field logbooks or on field sampling sheets and calibration logs. Calibration logs will be reviewed with other field documentation to identify any potential impacts to data quality and usability. Field logbooks are reviewed as part of the QC inspections. Instruments used to collect these measurements are calibrated, typically at the beginning of each field day at a minimum using calibrations standards and per the manufacturer's SOP. Calibration is recorded in field forms. Further information on calibration and monitoring of field parameter is provided in the FSP. Field data will be deemed usable (i.e., precise, accurate, and representative) when it is collected following the appropriate standard operating procedures (SOPs), using the proper equipment that has been correctly calibrated. The Field Sampling Plan contains SOPs that will be followed by field staff during execution of the work. SOPs are included for calibration of field probes as well as collection of field data. In many cases, the SOPs include forms to be completed by field staff which support and document proper calibration of field instruments and data collection. These forms will be reviewed by field team leaders, who will then make the judgement

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regarding subsequent data usability.

Reconciliation with Data Usability Requirements

The data results will be examined to determine the performance that was achieved for each data need. The performance will then be compared with the project objectives and DQOs. Deviations from objectives will be noted. Additional action may be warranted when performance does not meet performance objectives for critical data. Options for corrective action relating to incomplete information, questionable results, or inconsistent data may include any or all of the following:

- retrieval of missing information
- request for additional explanation or clarification
- reanalysis of sample from extract (when appropriate)
- recalculation or reinterpretation of results by the laboratory

These actions may improve the data quality, reduce uncertainty and eliminate the need to qualify or reject data.

If these actions do not improve the data quality to an acceptable level, the following additional actions may be taken:

- extrapolation of missing data from existing data points
- use of historical data
- evaluation of the critical/noncritical nature of the sample

If the data gap cannot be resolved by these actions, the data bias and potential for false negatives and positives can be evaluated. If the resultant uncertainty level is unacceptable, the following action must be taken:

- additional sample collection and analysis

Appendices

1,4-Dioxane

By Gas Chromatography / Mass Spectrometry in Selected Ion Mode (GC/MS-SIM) with Isotope Dilution Modification

References:

Method 8270D, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Revision 4, February 2007, Test Methods for Evaluating Solid Waste, SW-846.

EPA 8000C, SW-846,Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846. Update III, March 2003.

1,4-Dioxane Analytical Notes, Appendix II-B-4, WSC-CAM-II-B, Revision 1, July 2010, (Massachusetts Department of Environmental Protection Bureau of Waste Site Cleanup).

1. Scope and Application

Matrices: Aqueous, soil/sediment and non-aqueous waste matrices

Definitions: Refer to Alpha Analytical Quality Manual.

This method is applicable to the quantification of 1,4-Dioxane extracted from aqueous samples in methylene chloride and analyzed by GC/MS-SIM. The extraction method is listed below and should be referenced for more details. Detection limits will vary with instrument calibration range, and volume of sample analyzed. 1,4-Dioxane detected over the calibration ranges of the instrument it is being analyzed on will be diluted and re-analyzed for accurate quantification.

The following extraction method applies:

- *Extraction of Water Samples by Separatory Funnel* (SOP 2165)
- *Microscale Solvent Extraction (MSE)* (SOP 2172)
- *Organic Waste Dilution Extraction* (SOP 2265)

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the GC/MS-SIM and in the interpretation of GC/MS-SIM data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

250mL, 500mL or 1000mL of aqueous samples are serially extracted with methylene chloride in a 2 Liter Separatory Funnel (Method 3510) at a neutral pH. The extract is concentrated in MeCl2 to a 2.5mL to 10mL final volume respectively depending on the volume of sample extracted. Soil/sediment samples are extracted by Microscale Solvent Extraction (MSE Method 3570). Approximately 5g of sample is extracted and concentrated to a 4mL final volume. Non-aqueous

waste samples are extracted by Organic Waste Dilution Extraction (Method 3580). Approximately 1g is diluted to 10mL final volume.

Analytes are introduced into the GC/MS using a large volume injector and injecting 3ul of the calibration standards, quality control samples, and sample extracts into the GC equipped with a narrow-bore capillary column. The GC column is temperature programmed to separate the analytes, which are then detected with a mass spectrometer (MS) in selective ion mode. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact spectra of the calibration standards. Concentrations are determined using mean relative response factors from a multi-level calibration curve. Response factors for target analytes and surrogate compounds are determined relative to the internal standards.

Isotope dilution quantification is achieved by spiking 1,4-Dioxane-d8 at extraction which is then in turn used as both an internal standard (IS) and surrogate. For quantification, the 1,4-Dioxane-d8 IS quantifies 1,4-Dioxane in samples. An additional IS added prior to analysis, 1,4-Dichlorobenzene-d4, quantifies 1,4-Dioxane-d8 as a surrogate.

2.1 Method Modifications from Reference

SIM option with Isotope dilution is utilized to increase sensitivity for this analyte. Note that while this method may be used for the evaluation of 1,4-Dioxane in soil/sediment samples, the MA DEP CAM does not approve of this method for determining presumptive certainty for MA DEP cleanup sites. Method 8260 should be employed in these cases.

3. Reporting Limits

Concentrations for 1,4-Dioxane can be detected in water samples in the range of 150ng/L to 100,000ng/L. Soil/sediment samples can be detected in the range of 8ug/Kg to 8,000ug/Kg. Standard reporting limits for aqueous samples are 150ng/L and for soil/sediment samples 8ug/kg respectively.

4. Interferences

4.1 Phthalate esters can be a major source of contamination if any material containing plasticizers (phthalates) comes in contact with the sample during the extraction process. Use of plastic or any material containing plasticizers (phthalates) should be avoided during extraction or analysis.

4.2 The injection port of the gas chromatograph can become contaminated with high boiling compounds resulting in the loss of sensitivity. It may be necessary to replace the injection port liner routinely to prevent this loss of sensitivity. Clipping off approximately four inches of the column at the injection end may also increase sensitivity. Low instrument response can be detected during the daily tuning procedure by including pentachlorophenol and benzidine in the daily tuning mix.

4.3 Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences or carryover. Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed.

4.4 Solvents, reagents and glassware may introduce interferences. These must be demonstrated to be free of interferences by the analysis of a method blank. See the SOP *Reagent, Solvent and Standard Control* (G-008) and *Laboratory Glassware Cleaning* (G-002), for additional details.

4.5 It should be noted that there are some chromatographic consequences observed due to the acetone used in the MSE extraction process. This is characterized by a shift in retention time for 1,4-Dioxane and the 1,4-Dioxane-d8 surrogate (~0.4minutes), as well as the presence of a large peak (likely acetone) in all chromatograms.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Solid samples; A minimum of 100grams of sample must be collected in a glass jar with a Teflon lined screw cap.

Water samples: A minimum of 0.25 to 1 liters of sample must be collected in amber glass bottles.

6.2 Sample Preservation

Solid samples: The sample must be refrigerated and maintained at 4±2 °C until extraction and analysis. Sediment samples can be frozen at -20±5 °C until extraction to extend hold time. The extracts must be refrigerated and maintained at 4±2 °C until analysis.

Water samples: The samples must not be preserved except by refrigeration at 4±2 °C until extraction and analysis. The extracts must be refrigerated and maintained at 4±2 °C until analysis.

Non-aqueous waste samples: Concentrated sample extracts must be stored in contaminant-free containers and preserved in a refrigerator when not used for more than four hours.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

Solid samples: All solid samples must be extracted within 14 days from the date of collection. Frozen sample hold times are monitored up to 14 days from the date removed from freezer. The extracts must be refrigerated and maintained at 4±2 °C until analysis. Sample extracts must be analyzed within 40 days from date of extraction.

Water samples: All water samples must be extracted within 7 days from the date of collection. Sample extracts must be analyzed within 40 days from date of extraction.

Non-aqueous waste samples: Hold times do not apply to neat oils/NAPL/product samples.

7. Equipment and Supplies

7.1 Gas chromatograph – Programmable, heating range from 40C to 350C; splitless-type

inlet system, (Hewlett Packard 6890N Series II or similar); mass selective detector (Hewlett Packard 5973, or similar); automatic injector (Hewlett Packard 7683B or similar).

7.2 Chromatography Column – Fused silica capillary column, 0.25mm ID x 60m length, 0.25um film thickness RTX-5, Restek Corporation, 5% diphenyl-95% dimethyl polysiloxane, Fused silica capillary column, 0.18mm ID x 60m length, 0.18um film thickness RTX-PCB, Restek Corporation, Fused silica capillary column, 0.25mm ID x 30m length, .25um film thickness (Zebron ZB-SemiVolatiles, Phenomenex Corporation, 5% Polysilarylene - 95% Polydimethylsiloxane) , or equivalent.

7.3 Gerstel Large Volume Injection System – Temperature programmable range from 0C to 350C; pressure programmable; capable of split or splitless injection; Injection volumes range from 1 to 50uL and Cryo cooling availability to allow for cold injections.

7.4 Agilent Split/Splitless injector System

7.5 Data Acquisition System - Computerized system for collecting, storing, and processing detector output (Hewlett Packard Enviroquant target software) or equivalent.

7.6 Gases - BIP Ultra high purity helium (99.9995%); Compressed nitrogen for N-Evap. Carbon dioxide (siphon type) for Gerstel.

7.7 Syringes – 10uL to 1.0mL

7.8 Vials- including 2ml, 4ml, 10ml, 40ml and other sizes as necessary.

7.9 Hamilton Gas tight Syringes - varying sizes

7.10 Gerstel Single baffle injection port liners- packed lightly with glass wool.

7.11 GC Injection Port Liner: Phenomenex Direct Connect Top Hole

7.12 Class A Volumetric flasks: Including 10ml, 20 ml, 50 ml, 100ml and other sizes as necessary

8. Reagents and Standards

Use reagent grade chemicals for all reagents. Deionized (DI) water is ASTM Type II laboratory reagent grade water.

8.1 Solvents: All solvent expirations determined as indicated by manufacturer guidelines

8.1.1 Methylene Chloride, ACS approved, Pesticide grade, see SOP *Reagent, Solvent and Standard Control* (SOP 1816) for additional details regarding solvent purity. Used to extract samples and prepare instrument/analytical standards.

8.1.2 Acetone, ACS approved, Pesticide grade, see SOP *Reagent, Solvent and Standard Control* (SOP 1816) for additional details regarding solvent purity. This water soluble solvent is used for surrogate and LCS/MS preparation.

8.1.3 Methanol, ACS approved, Pesticide grade, *Reagent, Solvent and Standard Control SOP* (SOP 1816) for additional details regarding solvent purity.

8.2 Analytical Standards: Standards should be stored at –10C or less, away from light when not in use. They should be discarded after 1 year unless the vendor expiration date states otherwise or, if degradation is observed. Stock standards are given a 1 year expiration from the preparation date or the expiration of the primary vendor solution, whichever occurs first. Working standards are given six month expiration from the preparation date or the expiration of the primary solution whichever occurs first. All analytical standards are made up in Methylene Chloride. All prep standards are made up in Acetone.

8.3 Surrogate/Internal Standard (IS):

8.3.1 A 1,4-Dioxane-d8 Primary neat standard is commercially obtained from Cambridge Isotope (Cat #DLM-28-10 or equivalent). A stock surrogate/internal standard solution is prepared by weighing 0.1 g of the primary neat standard and diluting volumetrically in 10 mls methylene chloride (or equivalent preparation) to obtain a concentration of ~10,000 ug/ml.

8.3.2 From this stock (Section 8.3.1), the Surrogate spiking solution is made by a serial dilution (1 ml diluted up in a 50 ml volumetric flask, followed by a 0.5 ml of this solution diluted up in a 20 ml volumetric flask or equivalent preparations) in Acetone to achieve a concentration of 5 ug/mL. Of this surrogate solution, 1 mL is spiked into each water sample, and 0.4 mL is spiked into each soil/sediment sample for a surrogate/IS concentration of 500 ng/mL in samples.

8.3.3 The initial stock solution (Section 8.3.1) is diluted volumetrically (250 uL diluted up in a 100 ml volumetric flask or equivalent preparation) in methylene chloride to obtain an IS solution at a concentration of 25 ug/ml for the spiking of only the analytical standards (i.e. calibration curve and continuing calibration).

8.4 Internal Standard: 1,4-Dichlorobenzene-d4 commercially obtained from Restek (Cat #31206 or equivalent). This primary solution is at 2000 ug/mL and contains other Semivolatile Internal standards, however this method only utilizes the 1,4-Dichlorobenzene-d4. This solution is diluted volumetrically (250 uL diluted up in a 50 ml volumetric flask or equivalent preparation) in methylene chloride to obtain a SIM-IS solution at a concentration of 25 ug/ml. All samples and standards are spiked with 20uL of internal standard before analysis. This IS is intended to be used for both quantitation (of the surrogate 1,4-Dioxane-d8) and the establishment of relative retention times.

8.5 Matrix Spike/Laboratory Control Spike Solutions (MS/LCS) – 1,4-Dioxane primary spike solution is commercially obtained from Restek at 2000 ug/mL (Cat #31853 or equivalent). To prepare the working spike solution, syringe measure 0.25 mL of stock and bring to 100 mL in acetone (or equivalent preparation) for a 5 ug/mL concentration. From this solution, 1 mL is spiked into each MS/LCS QC water sample and 0.4 mL is spiked into each soil/sediment sample. The final concentration in a 10 mL water extract and in a 4 mL soil/sediment sample will be 500 ng/mL.

8.6 Stock Calibration Standards are prepared from a primary standard commercially obtained from Restek at a concentration of 2000 ug/ml (Cat #31853 or equivalent). The primary standard is used to appropriately prepare a stock standard at a concentration of 10,000 ng/ml (125 uL diluted up in a 25 ml volumetric flask or equivalent preparation). The stock solution is generally made up in 25 ml increments. The stock solution then doubles as a high level standard for the calibration curve, as well as a stock solution by which to serially dilute to prepare the other calibration standards. Except for the high level standard, the calibration curve levels are generally each made at 10 ml. Refer to the table below for example calibration

curve levels. All the calibration curve levels, once aliquoted at 1 ml increments for injection, have 20 ul of 1,4 Dichlorobenzene IS (SIM IS – Section 8.4) added as well as 20 uL of 1,4 Dioxane-d8 IS/Surrogate (Section 8.3.3) added.

8.7 Curve Preparation: The following 7 levels are the possible levels that can be analyzed for an ICAL.

Calibration Level	*Volume of Stock Std Added Into 10 ml of DCM*
Level 1 (10 ng/mL)	10 uL
Level 2 (50 ng/mL)	50 uL
Level 3 (100 ng/mL)	100 uL
Level 4 (500 ng/mL)	500 uL
Level 5 (1000 ng/mL) - CCV	1.0 mL
Level 6 (5000 ng/mL)	5.0 mL
Level 7 (10,000 ng/mL)	NA

8.8 The Independent Check Verification Standard is prepared from a Primary standard solution commercially obtained from Ultra (Cat NV-150-1 or equivalent) at a concentration of 100 ug/ml. The primary solution is diluted volumetrically (100 ul diluted up in a 10 ml volumetric flask or equivalent preparation) in methylene chloride to create a working ICV solution at a concentration of 1000 ng/ml. The working ICV solution is aliquoted into 1 ml increments as needed to inject with each calibration curve. Additionally, 20 ul of SIM IS (Section 8.4) and 20 ul of 1,4-Dioxane-d8 IS (Section 8.3.3) is added into the 1 ml aliquot.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

A method blank must be prepared once per every 20 samples or per extraction batch, whichever is more frequent.

Organic compounds of interest must not be detectable in the method blank at a concentration greater than the reporting limit.

Corrective Action: For contaminated blanks, all efforts must be made to identify and eliminate the source of contamination. The presence of analytes at concentrations at or above the reporting limit will warrant application of a “B” qualifier to that target compound(s) on all associated report forms, and perhaps re-extraction of all associated samples. Re-extraction of the method blank and all associated samples must be performed until the blank is in control. Surrogate recoveries must meet the QC limits for the method blank. Re-extraction must be initiated immediately so that minimum time is wasted before re-extraction can occur - if at all possible-this re-extraction should take place within holding time. Re-extraction *corrective action* that would exceed the sample holding time criteria should be discussed with the Organics Supervisor, Project Manager, client, and Operations Manager prior to implementation. Exceptions may be made with approval of the Organics Section Supervisor if the samples associated with an out of control method blank are non-detect for the affected compound(s) or if the concentration of the affected compound(s)in the sample is greater than 10x the blank level. In such cases, the sample results are accepted without corrective action for the high

method blank result. The client must be notified, via the project narrative, of any method blank non-compliance associated with sample results

9.2 Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD)

Laboratory control samples (LCS/LCSD) must be prepared once per every 20 samples or per extraction batch, whichever is more frequent, and spiked with 1,4 Dioxane spike solution (Section 8.5) and surrogate (Section 8.3.2) before extraction. The IS (Section 8.4) is spiked after extraction and before the analysis.

Acceptable Recovery limits are 40% - 140%. The relative percent difference (RPD) between the LCS/LCSD is 30%. Limits are adapted from MCP protocol and are generally monitored and documented in-house through control charts.

Corrective Action: Analysis must be repeated if an analytical error is suspected. If the LCS/LCSD recoveries and/or %RPD are still out of control, re-extract and re-analyze the LCS/LCSD and all associated samples. Samples cannot be reported until an acceptable LCS is obtained.

9.3 Initial Calibration Verification (ICV)

Refer to Section 10.2.

9.4 Continuing Calibration Verification (CCV)

Refer to Section 10.4.

9.5 Matrix Spike / Matrix Spike Duplicate (MS/MSD)

Matrix spike / matrix spike duplicate (MS/MSD) samples are performed upon project specifications. They are performed per client request. The sample is spiked with 1,4-Dioxane spike solution (Section 8.5) and surrogate (Section 8.3.2) before extraction. The IS (Section 8.4) is spiked after extraction and before the analysis. The recovery limits are 40% - 140% and the RPD limit is 30%. Limits are adapted from MCP protocol and are generally monitored and documented in-house through control charts.

Corrective Action: Analysis must be repeated if an analytical error is suspected. If the % recovery and/or %RPD still exceeds the control limits and the LCS/LCSD is compliant; include a project narrative with the results to client noting that there may be potential matrix effects on the accuracy or precision of the reported results as evidenced by MS/MSD recoveries and/or %RPD outside of QC limits.

9.6 Laboratory Duplicate

Duplicate analyses are performed upon client and/or workplan request. *For Organic analyses, the matrix duplicate is usually in the form of the matrix spike duplicate, see Section 9.5.*

Acceptable relative percent difference (RPD) of duplicates is 30%. Acceptance criterion is not applicable to sample concentrations less than 5 times the reporting limit. Calculate the RPD as follows:

$$RPD = \frac{R1 - R2}{\frac{[R1 + R2]}{2}} \times 100$$

where:

R1 = sample Replicate #1
R2 = sample Replicate #2

The RPD limits should be monitored and documented in-house through control charts and updated as needed.

Corrective Action: Analysis must be repeated if an analytical error is suspected.. If the % RPD still exceeds the control limits; include a project narrative with the results to client noting that there may be potential matrix effects on the precision of the reported results as evidenced by the matrix duplicate % RPD exceedence.

9.7 Method-specific Quality Control Samples

9.7.1 Surrogates

Surrogate spikes (Section 8.3.2 and Section 8.3.3) must be added to QC and field samples to evaluate the extraction method performance.

The acceptable surrogate recovery limits are 15% - 110%. Limits are adapted from MCP protocol.

Corrective Action: Analysis must be repeated if an analytical error is suspected. If the % recovery still exceeds the control limits the sample must be re-extracted and re-analyzed to confirm the sample matrix. If *obvious* matrix interferences are noted, consultation with the Organic Supervisor or Operations Manager may be in order to confirm the need for sample re-extraction. If no re-extraction occurs, the surrogate results and reasons for the decision not to re-extract must be discussed in the project narrative to the client. Due to the isotope dilution nature of this method, the analyst must pay close attention to surrogate recoveries and areas as this recovery is then used to surrogate correct (as an Internal standard) the response of 1,4-Dioxane.

9.7.2 Internal Standards

Internal standards must be added to all sample extracts, QC samples and standards for quantitation purposes. For sample extracts, only the 1,4-Dichlorbenzene-d4 IS (SIM IS – Section 8.4) should be added since the extraction surrogate is then used as an Internal Standard upon analysis. However, all Calibration standards must be spiked with both 1,4-Dichlorobenzene-d4 as well as 1,4-Dioxane-d8 (Section 8.3.3). In the sample extracts the area counts for 1,4-Dioxane-d8 will vary based on the extraction, however the 1,4-Dichlorobenzene-d4 area counts should remain fairly constant. The 1,4-Dichlorobenzene-d4 internal standard should also remain constant with respect to the continuing calibration analyzed at the beginning of the run. Sample IS areas must be –50% to +100% of the Internal Standards in the Continuing Calibration – applies only to 1,4-Dichlorobenzene-d4. Additionally, the IS retention times should not differ more than 30 seconds from the Continuing Calibration.

Corrective Action: Analysis must be repeated once unless there are obvious samples matrix interferences, i.e., the sample extract was very colored and viscous, or there are obvious chromatographic interferences. If *obvious* matrix interferences are noted, consultation with the Organic Supervisor or Operations Manager may be in order to confirm the need for sample re-analysis or re-extraction.

9.8 Method Sequence

- Tune
- CCV
- Method Blank
- LCS
- LCSD
- Samples

10. Procedure

10.1 Equipment Set-up

10.1.1 The instrument used for the analysis is a HP 6890N Series gas chromatograph. The HP system is equipped with a Gerstel large volume injection system, and a 7683B-type autosampler or equivalent. The mass spectrometer is an HP 5973 or 5975 with the HP Enviroquant data system. The method is modified for selective ion monitoring. The table below lists the ions monitored in one SIM window. This method must only be set up and analyzed by an experienced mass spectrometrist.

Compound	*Primary Ion*	*Secondary Ion*
1,4-Dioxane	88	58, 43
1,4-Dioxane-d8	64	96
1,4-Dichlorobenzene-d4	152	115

10.1.2 The basic GC parameters are as follows for the Gerstel Large Volume Injection System:

Oven Equib Time: 0.10 min
Oven Max: 325
Initial Temp.: 45°C
Initial Time: 6.00 min

Level	Rate (°C/min)	Final Temp. (°C)	Final Time (min)
1	19.00	120	1.0
2	11.00	150	1.00
3	19.00	305	4.00

Final Time: 26.83

10.1.3 The basic injection port parameters are as follows for the Gerstel Large Volume Injection System:

"Splitless" mode
Initial Temp: 46 °C
Initial Time: 0.30 min
Ramp Rate: 300°/second
Final Temp: 300°C
Final Time: 30.0

Cryo: ON
Cryo Use temp: 25 °C
Cryo Timeout: 30.0 min
Cryo Fault: ON
Purge Flow: 25 ml/min
Purge Time: 2.50 min
Gas Saver: off
Gas: Helium

Mode: Constant Pressure
Average Velocity: 30cm/sec
Initial flow: 1.3 ml/min

10.1.4 The basic GC parameters are as follows for the Agilent Split/Splitless injector System:

Oven Equib Time: 0.20 min
Oven Max: 325°C
Initial Temp.: 30°C

Level	Rate (°C/min)	Final Temp. (°C)	Final Time (min)
1	0	30	2.00
2	5	50	0.00
3	50	300	2.00

Final Time: 13.00

10.1.5 The basic injection port parameters are as follows for the Agilent Split/Splitless injector System:

:

Mode: Splitless
Temperatuire: 200 °C
Flow: 1.0 mL/min
Velocity: 36.074 cm/sec
Septum Purge: 3mL/min
Purge Flow to Split Vent: 60 mL/min at 0.3 min

10.1.6 MS Acquisition Information:

Tune file: dftpp.u
Acquisition Mode: SIM
Solvent Delay: 7.70 min
MS Source temp: 280°C

10.1.7 Tuning

10.1.7.1 Before the analytical standards are analyzed the mass spectrometer must be adjusted to meet the proper ion criteria for DFTPP. This is demonstrated by injecting into the GC/MS system 1uL of a 50ug/mL DFTPP solution. After the analysis of the DFTPP, evaluate the tune as follows:

- Enter into the “Environmental Data Analysis” (off-line) screen.

- Go to “File” and select the tune data file.
- Go into “Tuner” and select “Eval DFTPP”, then select “AutoFind DFTPP to Screen,” to evaluate the tune file, based on the pre-set SW-846 criteria**.** The software will evaluate the tune by selecting three scans of the DFTPP peak and will display the ion intensities on the screen. That is, one scan at the apex, one scan directly preceding the apex and one scan following the apex and averages them, then takes one background subtracted scan, 20 seconds before the beginning of the DFTPP peak. If the criteria below are met, repeat, select “AutoFind to Printer”, for a hardcopy of the tune evaluation for the record.

If the “AutoFind” tune evaluation does not meet the criteria below, manual evaluation of the tune can be performed by attempting either of the options below:

- Blow up the DFTPP peak on the screen and select either one single scan at the apex of the peak, or a scan immediately preceding or following the apex. Go into “Tuner” and select “Evaluate DFTPP to Screen,” or “Evaluate DFTPP to Printer,” as described above, OR,
- Take the average of the scans across the entire peak. Go into “Tuner” and select “Evaluate DFTPP to Screen,” or “Evaluate DFTPP to Printer,” as described above.

10.1.7.2 The following DFTPP mass intensity criteria should be used.

DFTPP KEY MASSES AND ABUNDANCE CRITERIA

Mass	m/z Abundance criteria
51	10-80 percent of mass 198.
68	Less than 2 percent of mass 69.
70	Less than 2 percent of mass 69.
127	10-80 percent of mass 198.
197	Less than 2 percent of mass 198.
198	Base peak, or >50 percent of Mass 442.
199	5-9 percent of mass 198.
275	10-60 percent of mass 198.
365	Greater than 1 percent of mass 198.
441	Present but less than 24 percent of mass 442.
442	Base Peak, or > 50 percent of mass 198.
443	15-24 percent of mass 442.

10.1.8 Tune acceptance should be verified at the beginning of every 12 hour analytical shift. The DFTPP may be combined with the calibration verification standard as long as both tuning and calibration acceptance criteria are met.

10.2 Initial Calibration

10.2.1 After the DFTPP passes criteria, a set of multi-level calibration standards listed in Section 8.7 are analyzed, from low concentration to high. A minimum of five calibration levels are analyzed. The calibration standards are stored in amber vials in the standards freezer. The labeling convention allows each standard to have a unique identifier which distinguishes it from field samples. The naming convention used throughout the laboratory identifies the standard as semivolatile, hydrocarbon, pesticide/PCB or volatile.

An example of this would be SW042407E, meaning it is a semivolatile (S) working (W) standard made on April 24, 2007 and that it was the fifth standard made that day. All certificates of analysis that are shipped with standards are filed with their receipt ID written on it to insure traceability.

10.2.2 Once the standards have been analyzed, they are reduced by the search software of the Enviroquant data system. Once all the components are identified, a linear curve is calculated for the components. The criteria for evaluation are as follows:

10.2.2.1 The average RF for each compound must be greater than 0.05.

10.2.2.2 The %RSD of each compound must not exceed 20%. If they do this may be an indication that the chromatographic system is too reactive for analysis to begin. This indicates the instrument may need maintenance.

10.2.2.3 Alternatively, a linear regression model may be employed, provided that the coefficient of determination (COD or r2) is $\geq$0.99. Otherwise, construct a nonlinear calibration of no more than a third order equation. Statistical considerations in developing a non-linear calibration model require more data than the more traditional linear approach. A quadratic (second order) model requires six standards, and a third order polynomial requires seven standards. In setting model parameters, do not force the line through the origin. The COD or r^2 must be greater than or equal to 0.99.

10.2.2.4 Once the calibration curve is reviewed, an Initial Calibration Checklist must be completed.

10.2.3 All samples and standards are spiked with Internal Standards (IS) before analysis. Refer to section 8.4 for specific internal standard spiking information. The IS is intended to be used for both quantitation and the establishment of relative retention times. Internal standard acceptance criteria can be found in Section 9.7.2.

10.2.4 Independent check standards (Section 8.8) from a separate source or different lot are analyzed after every initial calibration for evaluation against calibration standard solutions. The % Difference (%D) should not be greater than $\pm$30%.

10.3 Equipment Operation and Sample Processing

10.3.1 Tuning

A DFTPP standard must be analyzed and pass criteria before a continuing calibration verification standard or any samples are analyzed. The DFTPP may be combined with the calibration verification standard as long as both tuning and calibration acceptance criteria are met. A DFTPP tune standard must be analyzed before each 12 hour analytical shift. Please refer to Section 10.1.5 for tuning criteria and other information.

10.3.2 Daily Calibration

On a daily basis after the DFTPP has passed, a mid-level (usually 1000 ng/mL) continuing calibration standard which contains all of the analytes of interest is analyzed. The criteria for acceptance are:

10.3.2.1 All analytes must have response factors greater than 0.05.

10.3.2.2 The % D must be $\pm$20% D from the initial calibration.

10.3.3 Sample Analysis

10.3.3.1 The prep lab staff will transfer the samples to the instrument laboratory. The samples are generally brought to a 10 mL final volume for liquids or 4 ml for soil samples; 1 mL is transferred and the remaining sample volume is archived. One aliquot of each sample is then placed in the sample extract holding refrigerator located in the instrument laboratory.

10.3.3.2 All of the samples at 1 mL (including the batch QC samples) are spiked with 20 uL internal standard (see section 8.4 for specifics regarding the internal standard). The samples are shaken briefly after the internal standard is added to ensure mixing. A sample will need a dilution for target analytes that are over calibration.

10.3.3.3 After the daily tune and CCAL have passed criteria, the analyst places the samples onto the autosampler tray. (Generally, the samples will be analyzed in order of color (lightest to darkest.) with QC samples being analyzed first. The instrument sequence is typed into the HP Chemstation Software. Next run "Simulate Sequence" (also under the "Sequence" dropdown list) without clicking the "Overwrite Files" box. Compare the order of the vials on the instrument versus the sequence to confirm all the samples and standards are in the right places. Next click "Run Sequence" also under the HP Chemstation "Sequence" dropdown list.

10.3.3.4 After the samples have been analyzed, the data files from the MS are quantitated versus the proper quantitation method. The QCPRN1.MAC macro creates a form with which to easily check internal standard and surrogate criteria are met. The following should be reviewed initially:

10.3.3.4.1 Are all the surrogates within QC criteria? Please see Section 9.7.1 for surrogate information.

10.3.3.4.2 Is the internal standard- 1,4 Dichlorobenzene-d4, within 50-200% of the daily CCAL? If not, the samples should be checked for matrix interferences that may be causing these issues. The IS peaks should also be evaluated for peak splitting or incorrect integration by the software. A sample may not need to be reanalyzed if it can be determined (with guidance from a supervisor) that the QC is exceeded due to matrix interference.

10.3.3.4.3 Are all target analytes within calibration range? If not, the sample(s) should be diluted and re-analyzed. If a dilution is performed after the internal standard has already been added, it will be necessary to add additional IS in order to make up for the impact of the original IS added also being diluted. Conversely, if a sample has been over-diluted, it may need to be analyzed at less of a dilution to detect target analytes that may have been diluted out. Note: for this method, due to the isotope dilution, it is necessary to refer to area counts to determine if dilution is required. The area response for 1,4-Dioxane should be compared to the area of the highest level of the ICAL standard for that target. If the area of 1,4-Dioxane in the extract is greater than the area of 1,4-Dioxane in the Highest level of the Calibration Curve, then a dilution is necessary. The analyst **CAN NOT** use the concentration of 1,4-Dioxane found in the extract to determine if dilution is required because the concentration of 1,4-Dioxane in the sample is surrogate corrected in Chemstation based on the use of the extraction surrogate as an Internal Standard. Once it is determined based on the peak area that a sample

requires a dilution, the analyst must pay attention to the concentration of IS in the dilution. Although the analyst will adjust the 1,4-Dichlorobenzene-d4 concentration by adding the appropriate amount of additional IS to the dilution, there is no way for the analyst to make up for the dilution of the extraction surrogate 1,4 dioxane-d8 (also used as internal standard). To account for this the analyst must change the concentration of the 1,4-Dioxane-d8 within the calibration table to a value which accounts for the dilution. (Example: If the analyst performs a 10x dilution then the concentration of 1,4-Dioxane–d8 must be changed within the ICAL to 50.) The analyst must ensure that only the diluted sample is calculated against this value and that the method is returned to the proper concentration once the analysis of the dilution is complete.

10.3.3.4.4 Are all analyses within 12 hour tune time? If a sample is analyzed outside tune time, it will need to be re-analyzed in another tune clock.

10.3.3.4.5 The sequence should also be printed out from Chemstation, initialed and dated, and placed in the logbook.

10.3.3.4.5.1 If anything in the initial review of the data indicates that there should be a re-analysis, the reason for re-analysis should be noted on the sequence.

10.3.3.4.5.2 Also, note the time the tune standard was analyzed, the time of the last sample analysis. If there are samples analyzed outside tune time, the time the last sample within tune time ran should be noted, as well as the last sample analyzed.

10.3.3.4.5.3 If a re-extract is required, the “Request for Repreparation/Reclean” book should be filled out and a photocopy of the appropriate page should be given to the Preparation Group leader or the Organics Section Head.

10.4 Continuing Calibration

Continuing Calibration Criteria is outlined in section 10.3.2

10.5 Preventive Maintenance

If performing any maintenance on any piece of equipment it must be documented in the *Instrument Maintenance Logbook* located in the laboratory specific to each instrument.

Daily

Injection port maintenance: Maintenance should be done when the daily CCAL starts to demonstrate degradation either by %D outliers or area responses <50% as compared to the ICAL areas. Several tune clocks may be injected before maintenance is needed. The type of samples analyzed will have an effect on how soon maintenance should be performed. Injection port maintenance should be done as needed. General maintenance includes replacing the single baffle liner packed lightly with glass wool, cutting about 2-4 inches off the head of the column, and replacing the septa. Refer to maintenance log for more specific information.

The Gerstel Injection port should be handled with care. The liners are quite thin. Do not force the Gerstel weldmen into place as the threads are soft metal and will cross thread. Always make sure the weldmen goes on straight. The Gerstel injection port does not require significant

tightening of either the weldmen or column nut, tighten enough to seal but there is no need to crank down on it.

11. Data Evaluation, Calculations and Reporting

11.1 Qualitative Analysis

11.1.1 The qualitative identification of compounds determined by this method is based on retention time and on comparison of mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method. The characteristic ions from the reference mass spectrum are defined as the two ions of greatest relative intensity, and are over 30% relative intensity. Compounds are identified when the following criteria are met.

11.1.2 The intensities of the characteristic ions of a compound must maximize in the same scan or within one scan of each other. A peak selected by the data system, based on the presence of target specific ions at a target specific retention time will be accepted as meeting these criteria.

11.1.3 The relative retention time of the sample component is within $\pm$ 0.06 RRT units of the RRT of the standard component.

11.1.4 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%.) The relative intensities are monitored daily. The relative intensities will be updated when they exceed established values from the reference spectrum.

11.1.5 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

11.1.6 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e. a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important. Selective ion monitoring eliminates this potential.

11.2 Quantitative Analysis

11.2.1 Response factors and % RSD to evaluate Initial Calibration acceptability.

Calculate RF by:

$$RF = \frac{area_{cmp}}{area_{is}} \times \frac{conc_{is}}{conc_{cmp}}$$

where:

area cmp = Area of the characteristic ion for the compound being measured.

area is = Area of the characteristic ion for the specific internal standard.
conc is = Concentration of the specific internal standard.
conc cmp = Concentration of the compound being measured.

Calculate % RSD by:

$$\%RSD = \frac{SD}{\bar{x}} \times 100 \qquad SD = \sqrt{\sum_{i=1}^{N} \frac{(x_i - \bar{x})^2}{N-1}}$$

where:

% RSD = percent relative standard deviation
x = average of RF's
SD = standard deviation
xi = analytical results of each level in the final reporting units
N = number of results (levels)

11.2.2 Calculate % Difference (%D) by:

$$\%D = \frac{\overline{RF}_i - RF_c}{\overline{RF}_i} \times 100$$

where:

RFi - Initial Calibration average RF
RFc = Continuing Calibration RF

11.2.3 Results of Water Analysis - calculation as performed in report form:

$$\text{Concentration (ug/L)} = \frac{\text{(Conc) (Vf) (DF)}}{\text{(Vi)}} \times 1000$$

11.2.4 Results of Soil/Sediment Analysis – calculation as performed in report form:

$$\text{Concentration (ug/Kg)} = \frac{\text{(Conc) (Vf) (DF)}}{\text{(Vi) \%S}} \times 1000$$

where:
Conc = Raw on-column concentration obtained from the quantitation report using Initial Calibration results.
Vf = Final volume of extract (mL)
Vi = Volume of sample extracted (mL), or weight of sample extracted in grams (g)
DF = Dilution factor, for manually prepared dilutions, not instrumental "dilutions".
%S = percent solids, as a decimal

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Section 9.0 outlines sample batch QC acceptance criteria. If non-compliant organic compound results are to be reported, the Organic Section Head and/or the Laboratory Director, and the Operations Manager must approve the reporting of these results. The laboratory Project Manager shall be notified, and may chose to relay the non-compliance to the client, for approval, or other corrective action, such as re-sampling and re-analysis. The analyst, Data Reviewer, or Department Supervisor performing the secondary review initiates the project narrative, and the narrative must clearly document the non-compliance and provide a reason for acceptance of these results.

All results for the organic compounds of interest are reportable without qualification if extraction and analytical holding times are met, preservation requirements (including cooler temperatures) are met, all QC criteria defined in the table below are met, and matrix interference is not suspected during extraction or analysis of the samples. If any of the below QC parameters are not met, all associated samples must be evaluated for re-extraction and/or re-analysis.

QC Parameter	Acceptance Criteria
Method Blank	No analyte above the reporting limit The results are qualified with a "B" for any associated sample concentrations that are less than 10x the blank concentration for this analyte
Surrogate Recovery	15% - 110%
Laboratory Control Samples	40% - 140% and 30% RPD
Matrix Duplicate	30% RPD
Matrix Spike	40% - 140%
Matrix Spike Duplicate	40% - 140% and 30% RPD

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP 1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP 1732 Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ)
SOP 1739 Demonstration of Capability (DOC) Generation
SOP 1731 Manual Integration & Compound Rejection
SOP 1797 Hazardous Waste and Sample Disposal

SOP 1816 Reagent, Solvent and Standard Control

16. Attachments

None

Alkalinity Titration Method

Reference: Method 2320 B. Standard Methods for the Examination of Water and Wastewater. APHA-AWWA-WEF. Standard Methods Online

1. Scope and Application

Matrices: This method is applicable to water matrices.

Definitions: Refer to Alpha Analytical Quality Manual.

The alkalinity of a water is its acid-neutralizing capacity. It is the sum of all the titratable bases. The measured value may vary significantly with the end-point pH used. Alkalinity is a measure of an aggregate property of water and can be interpreted in terms of specific substances only when the chemical composition of the sample is known.

Alkalinity is significant in many uses and treatments of natural waters and wastewaters. Because the alkalinity of many surface waters is primarily a function of carbonate, bicarbonate, and hydroxide content, it is taken as an indication of the concentration of these constituents. The measured values also may include contributions from borates, phosphates, silicates, or other bases if these are present. Alkalinity in excess of alkaline earth metal concentrations is significant in determining the suitability of a water for irrigation. Alkalinity measurements are used in the interpretation and control of water and wastewater treatment processes. Raw domestic wastewater has an alkalinity less than, or only slightly greater than, that of the water supply. Properly operating anaerobic digesters typically have supernatant alkalinities in the range of 2000 to 4000mg calcium carbonate ($CaCO_3$)/L.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

Hydroxyl ions present in a sample as a result of dissociation or hydrolysis of solutes react with additions of standard acid. Alkalinity thus depends on the end-point pH used, and for total alkalinity a pH of 4.5 is used for the end-point.

When alkalinity is due entirely to carbonate or bicarbonate content, the pH at the equivalence point of the titration is determined by the concentration of carbon dioxide (CO_2) at that stage. Carbon dioxide concentration depends, in turn, on the total carbonate species originally present and any losses that may have occurred during titration. “Phenolphthalein alkalinity” is the term traditionally used for the quantity measured by titration to pH 8.3 irrespective of the colored indicator used in the determination.

The results obtained from the phenolphthalein and total alkalinity determinations offer a means for stoichiometric classification of the three principal forms of alkalinity present in many waters. The classification ascribes the entire alkalinity to bicarbonate, carbonate, and hydroxide, and assumes the absence of other (weak) inorganic or organic acids, such as silicic, phosphoric, and boric acids. It further presupposes the incompatibility of hydroxide and bicarbonate alkalinities. Because the calculations are made on a stoichiometric basis, ion concentrations in the strictest sense are not

represented in the results, which may differ significantly from actual concentrations, especially at a pH > 10.

2.1 Method Modifications from Reference

The reporting limit for this method is less than that in the Reference Method.

0.02N NaOH (Sodium Hydroxide) is used for LCS instead of 0.05N Na_2CO_3 (potassium carbonate) for LCS. All reagents are commercially prepped and have certificate of analysis.

3. Reporting Limits

The Reporting Limit is 2.0 mg/L

4. Interferences

Soaps, oily matter, suspended solids, or precipitates may coat the glass electrode and cause a sluggish response. Allow additional time between titrant additions to let the electrode come to equilibrium or clean the electrode between samples.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples for alkalinity analysis are collected in exclusive plastic or glass bottles, with alkalinity as the only analysis from the bottle. The bottles are filled completely, without any headspace, and capped tightly. The sample container is not opened until the time of analysis.

6.2 Sample Preservation

Store at 4 ± 2°C.

6.3 Sample Shipping

No specific requirement.

6.4 Sample Handling

The sample holding time is 14 days from collection. Initial sample pH measurement is documented during the beginning of alkalinity analysis. The pH results are included on the printout with the Alkalinity data.

7. Equipment and Supplies

7.1 T70 Mettler Toledo Titrator: With InMotion Flex Autosampler, terminal, and dosing units

7.2 Mettler Toledo Combination Electrode: Incorporates measuring and referenced functions; solid, gel-type filling material

7.3 Computer: with related accessories: LabX2014 software, version 6.1.0

7.4 Pipets: 1 mL, 5 mL glass volumetric

7.5 Plastic Cups: Mettler Toledo brand, 100 mL volume

7.6 KCl 3 mol/L electrolyte solution: Mettler Toledo brand for electrode or equivalent

7.7 50 mL conical centrifuge tubes

8. Reagents and Standards

8.1 Standard Sulfuric Acid, 0.1N: A commercially prepared standard solution which has been standardized against a NIST standard. A certificate of analysis is kept on file. Store at room temperature. Expires upon manufacturer's specified date.

8.2 Standard Sulfuric Acid, 0.02N: A commercially prepared standard solution which has been standardized against a NIST standard. A certificate of analysis is kept on file. Store at room temperature. Expires upon manufacturer's specified date.

8.3 Sodium Hydroxide Solution, 0.1N: A commercially prepared solution. A certificate of analysis is kept on file. Store at room temperature. Expires upon manufacturer's specified date.

8.4 Sodium Hydroxide Solution, 0.02N: A commercially prepared solution. A certificate of analysis is kept on file. Store at room temperature. Expires upon manufacturer's specified date.

8.5 pH Buffer Solutions: pH 4 and pH 10; two sources/Lot numbers of pH 7. Use to calibrate pH meter. Store at room temperature. Expires upon manufacturer's specified date.

8.6 Reagent Water: All reference to water in this method refer to Deionized Water (DI) from Alpha's water treatment system.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank

Analyze one method blank per batch of 20 samples or less. The blank consists of 50 mL of DI water (8.6). The blank results must be less than the reported detection limit. No samples may be analyzed until an acceptable blank is obtained.

9.2 Laboratory Control Sample (LCS)

Analyze one LCS per batch of 20 samples or less. A 100 ppm LCS is prepared by adding 5 mL of 0.02N sodium hydroxide solution (8.4) to 45 mL of DI water (8.6). The LCS must be recovered within control limits generated by QA. If the LCS falls outside of acceptance criteria,

it is reprepared and reanalyzed. If failure continues, the titrator is rinsed and the LCS analyzed again. No samples may be analyzed until an acceptable LCS recovery is obtained.

9.3 Initial Calibration Verification (ICV)

Same as the LCS, see section 9.2.

9.4 Continuing Calibration Verification (CCV)

A continuing calibration verification (CCV) (equivalent to the LCS) and a continuing calibration blank (CCB) (equivalent to method blank) pair is analyzed at the end of each analytical run to ensure that calibration is still valid.

Acceptance criteria for the CCV are 90-110% of the true value. If the CCV fails these criteria, all samples in the associated batch are reanalyzed.

The CCB must be less than the RL of 2.0 mg/L. If the CCB fails these criteria but the associated sample concentrations are non-detect, the sample results are reported with a narrative. If the CCB fails these criteria but the associated sample concentrations are greater than 10x the reporting limit, the sample results are reported with a narrative. Otherwise, if the CCB fails, all samples in the associated batch are reanalyzed.

9.5 Matrix Spike

Analyze one matrix spike per batch of 20 samples or less. A 100 mg/L spike is prepared by adding 1.0 mL of 0.1 N sodium hydroxide solution (8.3) to 50 mL of sample. The MS recovery must be within control limits. If the MS is outside of acceptance criteria, the sample and its spike are reanalyzed. If failure continues, report the data with a narrative to be included on the final report

9.6 Laboratory Duplicate

Analyze one sample in duplicate per batch of 20 samples or less. The %RPD between the sample and its duplicate must within control limits. If %RPD is outside of acceptance criteria the sample and its duplicate are reanalyzed. If failure continues, report the data with a narrative to be included on the final report.

9.7 Method-specific Quality Control Samples

None.

9.8 Method Sequence

- **Calibration** with pH 4, pH 7, and pH 10 buffers
- **Calibration verification** with second source pH 7 buffer
- **Rinse** burette with fresh acid
- **Analytical Run** Rinse, Blank, LCS, Samples, Duplicate, Matrix Spike, CCV, CCB
- **Titrate** to the pH end point on the auto titrator
- **Calculate** the results

10. Procedure

10.1 Equipment Set-up

Calibrate the pH meter of the Mettler Toledo titrator each day prior to use. Follow the manufacturer's instructions for calibration, using pH 4, pH 7, and pH 10, buffer solutions, using method 1 in LabX2014 software. Based on the manufacture specifications, 100% for slope will correspond to -59.16 mV/pH. Calibration is successful, if SLOPECal will fall between -56.20 and -62.12mV/pH. Calibration also will be validated by using a second source pH 7 buffer, using method 2 in LabX2014 software. The results must be within ± 0.05 pH units, otherwise recalibration is necessary.

10.2 Initial Calibration

Initial calibration is same as the pH calibration described in Section 10.1.

10.3 Equipment Operation and Sample Processing

10.3.1 Ensure rinse bottle is filled with DI water and the standard acid container is filled with sulfuric acid (8.2).

10.3.2 Open LabX2014 software and calibrate according to 10.1.

10.3.3 Record standards and reagents in log book and write down the analytical sequence.

10.3.4 After the calibration and pH check, purge any old acid and air bubbles from the burette using the "burette" tab by rinsing out the burette twice, using the LabX2014 software.

10.3.5 Using method 3 for total alkalinity or using method 4 for 2 step alkalinity, type in the sequence for analytical run. Right click on the appropriate method and click on create task. Name the sequence using the date and add the correct number of samples.

10.3.5.1 Always start analytical run with rinse, blank, and LCS and following with all samples, duplicate and matrix spike.

10.3.6 Label plastic cups (7.5) with marker in correct analytical sequence.

10.3.7 Shake samples and measure out 50 mL of sample into centrifuge tubes and then transfer to label cup and place them in the auto sampler.

10.3.8 Under tasks, click start to begin the analytical run.

10.3.9 After the analytical run is complete, print out results. The initial sample pH results are included on the data print out and are available upon request.

10.3.10 NOTE: Only 30 mL of titrant (8.2) may be used. If samples use more than 30 mL of titrant, sample dilution and reanalysis are required. Dilutions are prepared by using a known amount of standard acid (8.2) and then titration a smaller volume of sample.

10.3.11 Samples with low Alkalinity (less than 20mg/l): Use "Low Alkalinity Method". Record volume and pH at pH 4.5, then titrate to pH 4.2. Record volume titrated to pH 4.2. Use calculation for Low Alkalinity. Software will calculate based on two readings.

10.4 Continuing Calibration

See Section 9.4.

10.5 Preventive Maintenance

10.5.1 The pH electrode is filled with electrolyte (7.7) and is replaced as necessary.

10.5.2 Prior to each analytical run, the auto titrator burette is checked and purged of any air bubbles present.

10.5.3 Auto titrator burette is checked quarterly by the QA department to ensure accuracy.

11. Data Evaluation, Calculations and Reporting

11.1 Alkalinity

$$\text{mg } CaCO_3/L = \frac{\mathbf{A} \times \mathbf{N} \times 50{,}000}{\text{mL sample}}$$

Where:

A = mL of the standard acid titrated
N = normality of the standard acid

For samples with Low Alkalinity (less than 20 mg/l) use following calculation:

$$\text{mg } CaCO_3/L = \frac{\mathbf{(2B-C)xN} \times 50{,}000}{\text{mL sample}}$$

Where:

B=mls of titrant used to first recorded pH (4.5)
C= mls of titrant used to second recorded pH (4.2)
N= normality of the standard acid

11.2 Calculation of Alkalinity Relationships: The mathematical conversion of the results is shown in Table 1.

11.2.1 Carbonate (CO_3) Alkalinity is present when phenolphthalein alkalinity is not zero but is less than total alkalinity.

11.2.2 Hydroxide (OH) Alkalinity is present if phenolphthalein alkalinity is more than half the total alkalinity.

11.2.3 Bicarbonate (HCO_3) Alkalinity is present if phenolphthalein alkalinity is less than half the total alkalinity.

11.2.4 Carbon Dioxide (CO_2)

11.2.4.1 Free CO_2 is present if the total Alkalinity of a water is due almost entirely to hydroxides, carbonates or bicarbonates, and Total Dissolved Solids is not greater than 500mg/L.

$$\textbf{mg Free } CO_2\textbf{/L} = 2.0 \times B \times 10^{(6-pH)}$$

Where:

B = Bicarbonate alkalinity, mg $CaCO_3$/L

11.2.4.2 Total CO_2 is calculated from the Free CO_2, bicarbonate alkalinity and carbonate alkalinity

$$\textbf{mg Total } CO_2\textbf{/L} = [A + 0.44\,(2B + C)]$$

Where:

A = mg free CO_2/L (Section 11.2.4.1)

B = Bicarbonate alkalinity, mg $CaCO_3$/L

C = Carbonate alkalinity, mg $CaCO_3$/L

Table 1: Alkalinity Relationships

Result of Titration	Hydroxide Alkalinity as $CaCO_3$	Carbonate Alkalinity as $CaCO_3$	Bicarbonate Concentration as $CaCO_3$
P = 0	0	0	T
P < ½ T	0	2P	T – 2P
P = ½ T	0	2P	0
P > ½ T	2P – T	2 (T – P)	0
P = T	T	0	0

Key: P = Phenolphthalein Alkalinity

T = Total Alkalinity

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedance and improper preservation are noted on the nonconformance report form.

Review of LCSs, blanks, spikes and duplicates occur for each batch of samples. See section 9.1, 9.2, 9.5, and 9.6 for acceptance criteria. Record any trends or unusual performance on a nonconformance action form.

If the LCS recovery falls outside the designated acceptance range, the laboratory performance is judged to be out of control, and the problem must be immediately identified and corrected. Immediate corrective action includes reanalyzing all affected samples by using any retained sample before the expiration of the holding time. In case laboratory cannot reanalyze samples (due to lack of additional aliquot), samples will be reported with narrative; customer service will be notified and customers will be contacted.

13. Method Performance

13.1 Detection Limit Study (DL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the DL, LOD, and/or LOQ as outlined in Alpha SOP ID 1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP ID 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Hazardous Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

2121 Chemical Hygiene Plan

1732 Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ) SOP

1739 Demonstration of Capability (DOC) Generation SOP

1728 Hazardous Waste Management and Disposal SOP

16. Attachments

None.

Determination of Inorganic Anions by Ion Chromatography

Reference Methods: **EPA 300.0,** Methods for the Determination of Inorganic Substances in Environmental Samples, EPA/600/R-93/100, August, 1993.

Method 9056A, SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Revision IV, February 2007.

1. Scope and Application

Matrices: Drinking water, surface water, mixed domestic and industrial wastewaters, groundwater, reagent waters, solids (after extraction) leachates (when no acetic acid is used).

Definitions: See Alpha Laboratories Quality Manual Appendix A.

Regulatory Parameter List:

Parameter
Bromide
Chloride
Fluoride
Nitrate – N
Sulfate

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Laboratory Services Manager, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in use of ion chromatography and in the interpretation of ion chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

A small volume of sample is introduced into an ion chromatograph. The anions are separated and measured, using a system comprised of a guard column, analytical column, suppressor device, and conductivity detector.

2.1 Method Modifications from Reference

Use of other eluents that improve method performance are minor modifications of the method and are considered by the method to be acceptable.

3. Detection Limits

The laboratory follows the procedure found in 40CFR Part 136 to determine the MDL on a semi-annual basis. The method detection limits determined by the laboratory are on file for review.

4. Interferences

4.1 Interferences can be caused by substances with retention times that are similar to and overlap those of the anion of interest. Large amounts of an anion can interfere with the peak resolution of an adjacent anion. Sample dilution and/or fortification can be used to solve most interference problems associated with retention times.

4.2 Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baseline in ion chromatograms.

4.3 Samples that contain particles larger than 0.45 microns and reagent solutions that contain particles larger than 0.20 microns require filtration to prevent damage to instrument columns and flow systems.

4.4 Any anion that is not retained by the column or only slightly retained will elute in the area of fluoride and interfere. Known coelution is caused by carbonate and other small organic anions. At concentrations of fluoride above 1.5mg/L, this interference may not be significant, however, it is the responsibility of the user to generate precision and accuracy information in each sample matrix.

4.5 The acetate anion elutes early during the chromatographic run. The retention times of the anions also seem to differ when large amounts of acetate are present. Therefore, this method is not recommended for leachates of solid samples when acetic acid is used for pH adjustment.

4.6 The quantitation of unretained peaks should be avoided, such as low molecular weight organic acids (formate, acetate, propionate, etc.) which are conductive and coelute with or near fluoride and would bias the fluoride quantitation in some drinking and most waste waters.

4.7 Any residual chlorine dioxide present in the sample will result in the formation of additional chlorite prior to analysis. If any concentration of chlorine dioxide is suspected in the sample, purge the sample with an inert gas (argon or nitrogen) for about five minutes or until not chlorine dioxide remains.

5. Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

Sulfuric acid used in this method has the potential to be highly toxic or hazardous.

6. Sample Collection, Preservation, and Handling

6.1 Sample Collection

Samples are collected in glass or plastic bottles of sufficient volume to allow replicate analyses of the anions of interest.

6.2 Sample Preservation

Samples are refrigerated at 4°C.

6.3 Sample Handling

The sample holding time is 48 hours for the following anions: Nitrate –N.

The sample holding time is 28 days for the following anions: Bromide, Chloride, Fluoride, and Sulfate.

7. Equipment and Supplies

7.1 Balance: Analytical, capable of weighing to 0.0001g.

7.2 Ion Chromatograph: Analytical system (Dionex ICS-2000) complete with ion chromatograph and all required accessories including syringes, autosampler, analytical columns, compressed gasses and detectors.

7.2.1 Anion guard column: AG-18 (Dionex PN 060551) A protector of the separator column. If omitted from the system the retention times will be shorter. Usually packed with a substrate the same as that in the separator column.

7.2.2 Anion analytical column: AS-18 (Dionex PN 060549). This column produces the separation shown in Figure 1.

7.2.3 Anion supressor: ASRS Ultra II 4mm (PN 061561). The supressor column is packed with a high capacity cation exchange resin that is capable of converting the eluent and separated anions to their respective acid forms.

7.2.4 Detector: DS6 (PN 057985) Temperature controlled, heated conductivity cell

7.2.5 Eluent Generator: EG40 (Dionex PN 058900) Prepares the eluent electronically, controlled by the software; equipped with KOH cartridge.

7.3 Software: The Dionex IC Instrument uses Chromeleon Software.

7.4 0.45µm Membrane Filter Syringes.

7.5 Volumetric Flasks: Various volumes.

7.6 Volumetric Pipets: Various volumes.

7.7 0.5mL Vials with Caps: Dionex PN 038142.

8. Standards and Reagents

Note: All analytical standards used for calibration and calibration verification must be traceable to NIST. Each standard is recorded in a Logbook and unique ID is assigned to each standard. The unique IDs must also be included in all analytical sequences.

8.1 Reagent Water: Deionized water, free of the anions of interest. Water should contain particles no larger than 0.20 microns.

8.2 Eluent Solution: 32mM KOH, Prepared by the Eluent Generator.

8.3 Stock Calibration / ICV Standard Solutions, 1000mg/L (1mg/mL): Stock standards for all analytes are usually purchased as certified solutions. Certificates of analysis are kept on file.

However, if it is necessary, the Stock Solutions may be prepared from ACS reagent grade materials (dried at 105°C for 30 minutes) as listed below. The ICV Standards must be prepared from a different source than the calibration standards.

NOTE: Stock calibration/ ICV standards are stable for at least six months when stored at 4°C. Dilute working standards are prepared weekly.

8.3.1 Standard 1: Fluoride Stock Standard, 1000mg F^-/L

In a 250mL volumetric flask, dissolve 0.5526g of sodium fluoride (NaF, CASRN 7681-49-4) in about 200mL reagent water. Dilute to the mark with reagent water, and invert to mix.

8.3.2 Standard 2: Fluoride Stock Standard, 100mg F^-/L

In a 250mL volumetric flask, pipet 25mL of Standard 1, dilute to the mark with reagent water, and invert to mix.

8.3.3 Standard 3: Chloride Stock Standard, 1000mg Cl^-/L

In a 250mL volumetric flask, dissolve 0.4121g of sodium chloride (NaCl, CASRN 7647-14-5) in about 200mL reagent water. Dilute to the mark with reagent water, and invert to mix.

8.3.4 Standard 4: Bromide Stock Standard, 1000mg Br^-/L

In a 250mL volumetric flask, dissolve 0.3219g of sodium bromide (NaBr, CASRN 7647-15-6) in about 200mL reagent water. Dilute to the mark with reagent water, and invert to mix.

8.3.5 Standard 5: Bromide Stock Standard, 100mg Br^-/L

In a 250mL volumetric flask, pipet 25mL of Standard 4, dilute to the mark with reagent water, and invert to mix.

8.3.6 Standard 8: Nitrate Stock Standard, 1000mg NO_3^--N /L

In a 250mL volumetric flask, dissolve 1.5170 g of sodium nitrate ($NaNO_3$, CASRN 7631-99-4) in about 200mL reagent water. Dilute to the mark with reagent water, and invert to mix.

8.3.7 Standard 9: Nitrate Stock Standard, 100mg NO_3^--N /L

In a 250mL volumetric flask, pipet 25mL of Standard 8, dilute to the mark with reagent water, and invert to mix.

8.3.8 Standard 12: Sulfate Stock Standard, 1000mg SO_4^{2-} /L

In a 250mL volumetric flask, dissolve 0.4535 g of anhydrous dibasic, potassium sulfate (K_2SO_4, CASRN 7778-80-5) in about 200mL reagent water. Dilute to the mark with reagent water, and invert to mix.

8.3.9 Stock Bromide Standard, 1000ppm

Commercially available. Certificate of analysis required and kept on file. Use separate sources for the ICV and Calibration standards listed below.

8.3.9.1 ICV Bromide Standard, 100ppm

In a 100mL volumetric flask, add 1mL of Stock 1000ppm Bromide standard (Section 8.4.13). Bring to volume with reagent water.

8.4 Working Mixed Stock Standard A (Calibration Stock)

In a 200mL volumetric flask, transfer using volumetric pipets, 2mL each of Standards Bromide 1000ppm and Nitrate 1000ppm; 20mL of Chloride 1000ppm and Fluoride 100ppm (Section 8.3). Dilute to the mark with reagent water and invert to mix. Store at 4 ± 2°C for up to one month.

This makes Standard A containing F^-, Cl^-, NO_2^- -N, Br^-, NO_3^- -N and SO_4^{2-} at the concentrations of 10, 100, 10, 10 and 200ppm respectively.

8.4.1 Analyte Matrix Spike Solution

Volumetrically prepare the spike solution by bringing 1.0 mL of the calibration stock standard (Section 8.4) up to a 25mL final volume with the sample.

8.5 Working Mixed Standards B through G (Calibration Curve)

Working mixed standards B through F are prepared by diluting Standard A as summarized in the following Table. These are prepared fresh as needed for calibration.

Std.	Std. A (mL)	Final Vol. (mL)	F^-	Cl^-	Br	NO_3^- -N	SO_4^{2-}
B	5	10	5.0	50.0	5.0	5.0	100.0
C	5.0	25	2.0	20.0	2.0	2.0	40.0
D	1.25	25	0.5	5.0	0.5	0.5	10.0
E	0.5	25	0.2	2.0	0.2	0.2	4.0
F	1 of Std D	10	0.05	0.5	0.05	0.05	1.0
G	0.5 of Std D	10	0.025	0.25	0.025	0.025	0.5

Example: To make up Standard B, take 25mL of Standard A in a 10mL volumetric flask and dilute to the mark with reagent water.

Note: The dynamic range for the method is two orders of magnitude. The concentrations for the standards could be changed to bracket the concentrations of the samples to be analyzed.

8.6 ICV Stock Standard (Second Source Verification)

To a 200mL volumetric flask add 20mL of the following standards: Chloride 1000ppm and Sulfate 1000ppm, and 2mL of Fluoride 1000ppm, Bromide 1000ppm and Nitrate 1000ppm (Section 8.3). Dilute to volume with reagent water.

8.6.1 ICV Working Standard / LCS Solution

In a 25mL volumetric flask, add 2mL of the ICV Stock Standard. Bring to volume with reagent water. Store at 4 ± 2°C. Prepare weekly.

ICV working standard will have the following concentrations: 0.8 mg/L for Fluoride, Nitrate and Bromide; 8.0 mg/L for Chloride and Sulfate.

8.7 CCV Working Solution

The CCV Working Solution is made by diluting 2.5 ml of Mixed Stock Standard A up to 25 ml of DI Section 8.4. Final concentrations are: 1.0 mg/L for Fluoride, Nitrate and Bromide; 10.0 mg/L for Chloride and 20.0mg/l for Sulfate. Store at 4 ± 2°C. Prepare weekly.

9. Procedure

9.1 SET-UP

9.1.1 Determination of Linear Calibration Range (LCR)

The LCR must be determined initially and verified every six months or whenever a significant change in instrument response is observed or expected. The initial demonstration of linearity must use sufficient standards to ensure that the resulting curve is linear. The verification of linearity must use a minimum of a blank and three standards. If any verification data exceeds the initial values by ± 10%, linearity must be reestablished. If any portion of the range is shown to be nonlinear, sufficient standards must be used to clearly define the nonlinear portion.

9.1.2 **Prime Pump:** The pump must be primed prior to analysis, to ensure that there is no gas entering the column.

9.1.3 **Monitor Baseline:** From the main Panel screen, press the “Startup” Button. This will turn on, in order, the pump, the eluent generator, and the conductance cell. Allow the instrument to warm up for 10 – 20 minutes to ensure the baseline is stable and flat.

9.1.4 While the baseline stabilizes, the sample sequence can be written and the autosampler may be loaded.

9.1.5 When the baseline is stabilized, the sample sequence may be loaded into the analytical run, and started from the Chromeleon software.

9.1.6 **Operating Conditions: Dionex IC Instrument**

Eluent Concentration: 32mM KOH

Flow rate: 1.0mL / minute

Injection volume: 100µL

ASRS: ON

Conductivity Cell Temperature: 30 °C

9.1.7 **Monitor instrument stability:** Prior to QC sample and sample analysis, analyze a DI water blank to ensure the instrument is stable.

9.1.8 **Sample filtration:** Autosampler vials are equipped with a filter. If additional filtration is necessary, samples may be filtered through a 0.45µm membrane filter attached to

a syringe. **NOTE:** If samples require filtration, all associated batch QC samples must also be filtered.

9.1.9 **Extraction of solid materials :** Add a volume of reagent water equal to 10 times the weight of dry solid material taken as a sample. This slurry is mixed for 10 minutes using a magnetic stirring device. Filter the resulting slurry before injecting using a 0.45µ membrane filter attached to a syringe. Care should be taken to show that good recovery and identification of peaks is obtained with the user's matrix through the use of matrix spikes (Section 10.5).

9.2 Calibration Curve Generation

For each analyte of interest, prepare calibration standards at a minimum of three concentration levels and a blank by adding accurately measured volumes of one or more stock standards (Section 8.5) to a volumetric flask and diluting to volume with reagent water. If a sample analyte concentration exceeds the calibration range, the sample may be diluted to fall within the range.

Using injections of 100µL of each calibration standard, tabulate peak height or area responses against the concentration. The results are used to prepare a calibration curve for each analyte. During this procedure, retention times must be recorded.

The calibration curve for each analyte is prepared by plotting instrument response against the standard concentration. A correlation coefficient of 0.995 or greater is considered acceptable for all analytes.
All calibration points are back calculated by Dionex software and should be within 10% from true concentration, except 2 lowest points of calibration curve. %recoveries for low range will be wider, but shouldn't exceed 100% and correlation coefficient will not be worse then 0.995.

9.2.1 **Initial Calibration Verification (ICV/LCS):** The calibration curve must be verified on each working day, and after every 20 samples. The ICV/LCS sample is prepared from a different source than that used for the calibration standards (Section 8.6). If the response or retention time for any analyte varies from the expected values by more than ± 10%, the analysis must be repeated, using fresh calibration standards. If the results are still more than ± 10%, a new calibration curve must be prepared for that analyte.

9.3 Standardization (Continuing Calibration Verification)

This standard (Standard D: Section 8.5) is prepared weekly. The CCV is analyzed at the beginning of each run, after every tenth sample, and at the end of the sample run. The % Recovery of this standard must be within ± 10% of the calibration standard. Refer to Section 10.3 if % Recovery falls outside of the acceptance range.

9.4 Equipment Operation and Sample Analysis

9.4.1 An automated constant volume injection system is used. Load and inject a fixed amount of well-mixed sample. Flush injection loop thoroughly, using each new sample. Use the same size loop for standards and samples. Record the resulting peak size in area or peak height units.

9.4.2 The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time can be used to

calculate a suggested window size for each analyte. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

9.4.3 If the response for the peak exceeds the working range of the system, dilute the sample with an appropriate amount of reagent water and reanalyze.

9.4.4 If the resulting chromatogram fails to produce adequate resolution, or if identification of specific anions is questionable, fortify the sample with an appropriate amount of standard and reanalyze.

Note: Retention time is inversely proportional to concentration. Nitrate and sulfate exhibit the greatest amount of change, although all anions are affected to some degree. In some cases this peak migration may produce poor resolution or identification.

9.5 Preventative Maintenance

Follow the Preventative Maintenance Schedule as outlined on the Dionex ICS-2000 CDROM.

As Needed

- Check the eluent reservoir to see if it needs to be refilled.

Daily

- Check the ICS-2000 component mounting panel for leaks or spills. Wipe up spills. Isolate and repair leaks. Rinse off any dried eluent with reagent water.
- Check the waste container daily and empty when needed.

Weekly

- Once a week, check fluid lines for crimping or discoloration. Relocate any pinched lines. Replace damaged lines.
- Check the junctions between the pump heads and the pump casting for evidence of liquid leaks. If piston seal wash tubing is not connected, check the drain tubes at the rear of the pump heads for evidence of moisture. Normal friction and wear may gradually result in small liquid leaks around the piston seal. If unchecked, these leaks can gradually contaminate the piston housing, causing the pump to operate poorly. If leaks occur, replace the piston seals.

- Check the end-line filter (PN 045987) and change if needed. When new, end-lline filters are pure white. If the system is in continuous operation, change the end-line filter weekly, or whenever it becomes discolored. Replace the filter more often if bacterial buildup is visible or if the eluent does not contain solvent.

  NOTE: It is especially important to regularly replace end-line filters when using aqueous eluents, which may contaminate the filter with bacteria or algae. The bacterial buildup may not be visible.

Yearly (performed by Dionex technician)

- Calibrate the cell.
- Calibrate the vacuum degas assembly
- Replace the pump piston rinse seals and piston seals.

9.6 Calculations

9.6.1 Compute the sample concentration by comparing sample response with the standard curve. Multiply the result by the appropriate dilution factor.

9.6.2 Report only those values that fall between the lowest and the highest calibration standards. Samples exceeding the highest standard should be diluted and reanalyzed.

10. Quality Control and Data Assessment

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a calibration verification standard is used to confirm the measurements were performed in an in-control mode of operation.

10.1 Demonstration of Capability

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method. Each time a method modification is made, the analyst is required to repeat the procedure.

When one or more of the parameters tested fail at least one of the acceptance criteria, the analyst must locate and correct the source of the problem and repeat the test for failed parameters of the method.

Repeated failure confirms a general problem with the measurement system or analytical technique of the analyst. If the failure repeats, locate and correct the source of the problem and repeat the test for all parameters listed in the method.

10.2 Method Blank

One Method Blank consisting of an aliquot of reagent water is analyzed with each batch of 20 samples or less. Data produced are used to assess contamination from the laboratory environment. Method Blank results must be less than the Reporting Limit (RL) for the analyte.

Note: If samples have to be filtered prior to analysis, all associated batch QC must also be filtered.

10.3 Continuing Calibration Verification (CCV) Standard

The CCV Standard is the equivalent of Standard D in Section 8.5. Store at 4 ± 2°C. Prepare weekly. The CCV is analyzed at the beginning of each run, after every tenth sample, and at the end of the sample run. The % Recovery of this standard must be within ± 10% of the calibration standard.

10.4 Initial Calibration Verification (ICV) Standard / Laboratory Control Sample (LCS)

The ICV/LCS is analyzed at the beginning of each run, but after the CCV standard. This standard is prepared from a different source than that used to prepare the calibration standards (Section 8.6). The % Recovery of this standard must be within ± 10% of the calibration standard.

10.5 Matrix Spike

Prepare and analyze one spiked sample per batch of 10 samples or less (Section 8.4.1). Recovery of the Matrix Spike must be within +/-10% of the true value

10.6 Duplicates

Prepare and analyze one duplicate sample per batch of 20 samples or less. The RPD for the duplicate measurements must be within the Laboratory defined control limits (Section 10.7).

10.7 Control Limits

The laboratory maintains performance records to document the quality of data that is generated. Method accuracy for samples is assessed and records maintained. After the analysis of 20 spiked samples, and 20 laboratory control samples, calculate the average percent recovery (R) and the standard deviation of the percent recovery (S).

Control limits for the method parameters are generated by the QC staff and distributed to the analysts. The control limits are calculated based on in-house performance data. The limits are compared to the control limits found in the reference method.

10.8 Analytical Sequence

- Instrument calibration
- DI Blank
- CCV
- ICV
- Ten samples
- CCV
- Blank
- Shut-down

11. Method Performance

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDL concentrations were obtained using reagent water. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

MDL's must be established for all analytes, using reagent water (blank) fortified at a concentration of two to three times the estimated calculated detection limit.

Method performance data is on file in the laboratory QC department. Comparison of method performance data for the laboratory to the reference method criteria occurs when laboratory in-house acceptance limits are generated. In-house generated data must be within the specifications of the reference method or the analysis is not continued until corrective action is completed.

12. Corrective Actions

If the Method Blank result exceeds the Reporting Limit (RL) for the analyte, the Blank is reanalyzed. If the second result remains > RL, notify the Laboratory Manager to ensure maintenance is performed on the water filtration system and seek an alternate reagent water source within the laboratory. If the alternate reagent water source is acceptable, this source must be utilized for all blanks, standards and sample dilutions for the sample batch. If the second source reagent water also fails, the Laboratory Manager is notified.

If the Continuing Calibration cannot be verified within the specified limits, reanalyze the CCV solution. Record the reason for re-injection. If the second analysis of the CCV solution confirms calibration to be outside the limits, sample analysis must be discontinued, the cause determined and/or in the case of drift, the instrument recalibrated. All samples following the last acceptable CCV solution must be reanalyzed. The analysis data of the calibration blank and CCV solution must be kept on file with the sample analyses data.

If the ICV/LCS acceptance criterion cannot be met, reanalyze the standard. If failure continues, the instrument is recalibrated.

If the Matrix Spike acceptance criteria is not met, the spiked sample is reanalyzed (if possible). If failure continues and if all other QC performance criteria are met, the data is reported and a narrative is included with the final report.

If the RPD for the Duplicate measurements falls outside the Laboratory defined control limits (Section 10.7), the sample is reanalyzed (if possible). If failure continues, and if all other QC performance criteria are met, the data is reported and a narrative is included with the final report.

Holding time exceedence and improper preservation are noted on the nonconformance report form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

Review of standards, blanks and standard response for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form.

If the CCV or LCS recovery of any parameter falls outside the designated acceptance range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked samples is suspect and is only reported for regulatory compliance purposes with the appropriate nonconformance action form. Immediate corrective action includes reanalyzing all affected samples by using any retained sample before the expiration of the holding time.

13. Pollution Prevention

See Chemical Hygiene Plan for pollution prevention operations.

14. Waste Management

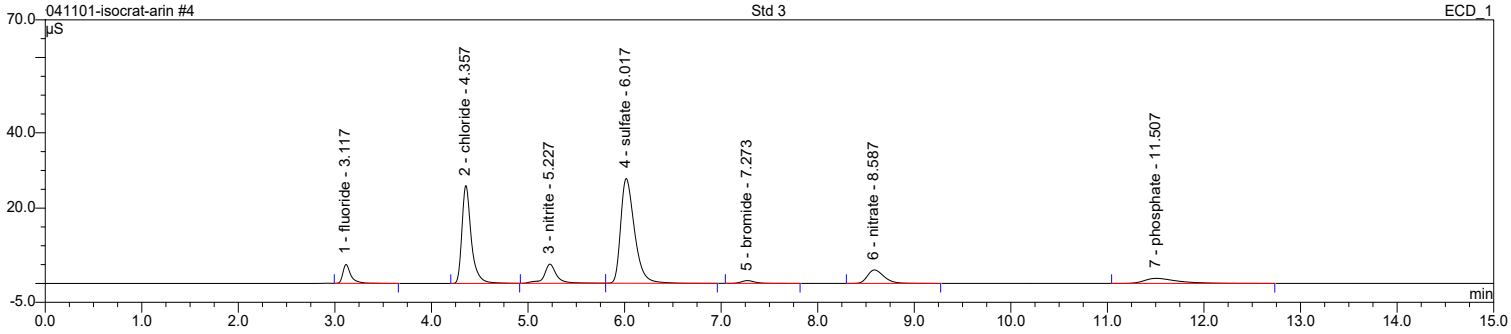
See Chemical Hygiene Plan for waste handling and disposal.

15. Attachments

Figure 1: Isocratic Anion Standard Separation

Figure 1

Isocratic Anion Standard Separation


041101-isocrat-arin #4
Std 3
ECD_1
uS
70.0
40.0
20.0
-5.0
1 - fluoride - 3.117
2 - chloride - 4.357
3 - nitrite - 5.227
4 - sulfate - 6.017
5 - bromide - 7.273
6 - nitrate - 8.587
7 - phosphate - 11.507
min
0.0
1.0
2.0
3.0
4.0
5.0
6.0
7.0
8.0
9.0
10.0
11.0
12.0
13.0
14.0
15.0

Determination of Perchlorate by IC/MS/MS

Reference Methods: **EPA Method 332.0**, Determination of Perchlorate in Drinking Water by Ion Chromatography with Suppressed Conductivity and Electrospray Ionization Mass Spectrometry, Rev. 1.0, March, 2005.

EPA METHOD 331.0 DETERMINATION OF PERCHLORATE IN DRINKING WATER BY LIQUID CHROMATOGRAPHY ELECTROSPRAY IONIZATION MASS SPECTROMETRY, Revision 1.0, January 2005.

EPA Method 6860, Perchlorate in Water, Soils and Solid Wastes using IC/ESI/MS or IC/ESI/MS/MS. January, 2007.

1. Scope and Application

Perchlorate (ClO_4^-) is an anion that originates as a contaminant in ground water and surface waters when the salts of ammonium, potassium, magnesium, or sodium dissolve in water. One major source of contamination is the manufacture or improper disposal of ammonium perchlorate that is used as the primary component in solid propellant for rockets, missiles, and fireworks.

This method covers the determination of Perchlorate using Ion Chromatography coupled with MS/MS detection.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Laboratory Services Manager, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation and the interpretation of IC/MS/MS data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

Matrices: Drinking Water, Ground Water, Surface Water, Soil

Definitions: See Alpha Laboratories Quality Manual Appendix A

Regulatory Parameter List:

Parameter	CAS
Perchlorate	14797-73-0

2. Summary of Method

A 100µL injection of sample or sample extract is introduced into the Ion Chromatograph (IC). Perchlorate is separated using a system comprised of an ion chromatographic pump, sample injection valve, guard column, analytical column, suppressor device, and conductivity detector. Perchlorate is subsequently detected by negative electrospray ionization mass spectrometry. A remote controlled valve is used to divert early eluting cations and anions to waste. Prior to the elution of perchlorate, the valve is switched sending the chromatographic eluent to the mass spectrometer. This diversion helps prevent unnecessary fouling of the electrospray source.

Chlorine has two naturally occurring isotopes (Cl^{35} and Cl^{37}) that appear in a scientifically established ratio of 3:1. Therefore, there are two isotopes of Perchlorate to monitor: $Cl^{35}O_4$ (MW 99) and $Cl^{37}O_4$ (MW 101). The loss of an oxygen (in the Collision cell (Q2)) produces two transitions that are monitored: 101/85 and 99/83. According to Method 331.0 (Section 10.2.3), since MRM analysis is employed, quantitation is performed on the 99/83 transition and confirmation is performed with the 101/85 transition, while the ratio between them verifies the presence of Perchlorate. Quantitation is also monitored with the use of an isotopically labeled Perchlorate standard ($^{18}O_4$-ClO_4) at MW 107. The isotopically labeled Perchlorate Internal Standard is added to all samples and standards prior to analysis.

2.1 Method Modifications from Reference

None

3. Detection Limits

The Minimum Reporting Limit (MRL) for liquid samples is 0.05µg Perchlorate/L. The Minimum Reporting Limit (MRL) for soil samples is 0.5µg Perchlorate/Kg.

The MRL is verified and confirmed by determining the Half Range for the prediction interval of results using the equation in Section 9.9.2.

4. Interferences

4.1 Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baselines in an ion chromatogram. These interferences can lead to false positive results for the target analyte as well as reduced detection limits as a consequence of elevated baseline noise. **Subtracting blank values from sample results is not permitted.**

4.2 All reagent solutions (eluents) used by the instrument must be filtered through no larger than a 0.45 µm nominal pore size membrane or frit to remove particulates and prevent damage to the instrument, columns and flow systems. Sample filtration must also be employed on every sample prior to analysis. This applies not only to field samples but also to the laboratory reagent blank (LRB) and laboratory fortified blank (LFB). The LRB and LFB samples function as controls and must be filtered to confirm no bias is attributable to the filtration. Filter the samples through a membrane or frit with a 0.45 µm nominal pore size. Syringe mounted filters work well. Filters specifically designed for IC applications should be used. In addition, samples are filtered using sterile sample filters to remove any bacteria from the sample that may cause Perchlorate to become converted to another state. QC samples that are

associated with an analytical batch are filtered with the same lot of sterile filters to matrix match the samples and remove any suspicion of contamination.

4.3 Other interferences may come from compounds with the same (or very similar) molecular weight as Perchlorate. The tracking of the Transition Ratio is imperative for Quantitation. This ratio aids in the proper identification of Perchlorate. When the Ratio is beyond acceptance criteria, the true identification of the sample should be questioned, and appropriate measures should be taken to ensure proper identification.

5. Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, and Handling

6.1 Sample Collection

Samples may be collected in plastic or glass bottles. All bottles must be thoroughly cleaned and rinsed with reagent water. The volume collected should be sufficient to ensure a representative sample (20mL minimum for liquid samples; 100g minimum for soil samples). Allow extra volume for replicate analysis and laboratory fortified matrix analysis, if required, and minimize waste disposal.

6.2 Sample Preservation

Samples must be chilled during shipment and must not exceed 10 °C during the first 48 hours after collection. Samples should be confirmed to be at or below 10 °C when they are received at the laboratory. Samples stored in the lab must be held at or below 6 °C until analysis. Samples should not be frozen.

6.3 Sample Handling

6.3.1 **Sample sterilization:** Perchlorate is known to be susceptible to microbiological degradation by anaerobic bacteria. Samples and extracts are filtered to remove microbes and stored with headspace to minimize the possibility that anaerobic conditions develop during storage.

6.3.2 **Liquids**

Once a representative liquid sample is obtained in the field, it must be sterile filtered immediately, prior to shipment to the Laboratory, to remove any native microorganisms. At a minimum, leave the top one third of the bottle empty to reduce the potential for degradation by any remaining anaerobic organisms.

6.3.2.1 Remove a sterile syringe (Section 7.14) from its package and draw up 20 mL of the sample. Remove a sterile syringe filter (Section 7.13) from its package without touching the exit Luer connection. Connect the filter to the syringe making sure that no water from the syringe drops on the exterior of the filter.

Open a sterile sample container (Section 7.12) without touching the interior. Using gentle pressure, pass the sample through the filter into the sample container. During this process do not let the syringe or filter make contact with the sample container. Following filtration, seal the sample container tightly, label and prepare the container for shipment. Syringes and filters are single use items and must be discarded after each sample.

6.3.3 Soil Extracts

Immediately following soil sample extraction (per Section 9.1.3), the extracts are filtered to remove any native microorganisms. Follow Section 6.3.1.1.1 above.

6.3.4 Holding Time: Properly sterilized liquid samples and soil sample extracts must be analyzed within 28 days of collection.

7. Equipment and Supplies

7.1 Ion chromatograph (IC) - Analytical system complete with eluent reservoirs, an ion chromatographic pump, injection valves, both guard and analytical separator columns, suppressor, conductivity detector, and computer based data acquisition system.

7.1.1 Anion guard column - Dionex AG16 2mm (P/N 055379), or equivalent. This column functions as a protector of the separator column. If omitted from the system, the retention times will be shorter.

7.1.2 Anion separator column - Dionex AS16, 2mm (P/N055378), or equivalent.

7.1.3 Anion suppressor device – Thermo scientific/Dionex Anion Electrolytically Regenerated Suppressor (Dionex AERS 500 2mm, P/N 082541). An equivalent suppressor device may be utilized provided comparable conductivity detection limits are achieved and adequate baseline stability is attained as measured by a combined baseline drift/noise of no more than 5 nS per minute over the background conductivity.

The ASRS is set to perform electrolytic suppression at a current setting of 45mA using the external water mode. External water is delivered to the suppressor directly from a pressurized source.

7.1.4 Detector - Conductivity cell (Dionex CD25, P/N 056961)

7.1.5 Eluent Generator - (Dionex EG50, P/N 060585) Capable of generating the specified molar concentration (45mM KOH) of eluent necessary. Equipped with a EluGen II KOH Cartridge (Dionex P/N 058900)

7.1.6 Autosampler - AS50 AutoSelect, PEEK, with internal injection valve (Dionex P/N 056836)

7.1.7 Eluent Pump- GS50 Gradient Pump (Dionex P/N 059481) Capable of a flow rate of 0.3mL/min.

7.1.8 Solvent Pump- AXP-MS (Dionex P/N 060684) Capable of delivering 0.2mL/min to the MS source.

7.1.9 **Data Acquisition System** - Dionex Chromeleon Chromatography Software, Version 6.5, copyright 1994 – 2003.

7.2 Triple Quadrapole MS/MS System- capable of providing electrospray ionization with negative ion detection.

7.2.1 **Detector**- API 2000 MS/MS system (Applied Biosystems P/N API2000)

7.2.2 **Source**- ESI source (Applied Biosystems P/N WLO19415)

7.2.3 **Syringe with Syringe Pump**- (Applied Biosystems P/N WCO13873)

7.2.4 **Data Acquisition System** – Analyst Version 1.4.1 (Applied Biosystems P/N 1014283)

7.3 Pipetter

7.3.1 Brinkmann Eppendorf Reference Series 2000. Brinkmann # 27-47-0302, adjustable from 100 – 1000µL.
7.3.2 Wheaton Acura 811 #09051275, fixed volume of 10µL.

7.4 Disposable Pipet Tips

7.4.1 1mL: Fisher PN 02-681-176.
7.4.2 10µL: Fisher PN 02-681-440.
7.4.3 200µL: Eppendorf PN 022491938.

7.5 Volumetric Flasks: Class A glass 250mL, 100mL, 50mL, 25mL, and 10mL volumes.

7.6 Volumetric Pipets: Class A glass 25mL, 10mL, 5mL, 2mL, 1mL and 0.5mL volumes.

7.7 2mL glass 11mm vials: Agilent, pn 5182-0714.

7.8 Blue screw cap, pre-slit PTFE/sil septa: Agilent, pn 5185-5865.

7.9 Hand Vial Crimper (11mm): Fisher P/N 06-406-20

7.10 Particulate Filters: 0.45 micron syringe filters. (National Scientific Company P/N 66030-853)

7.11 Disposable 10mL syringes: With Luer-Lok tip. Schleicher & Schuell, Reference Number 309604.

7.12 Sterile Sample Containers: 125-mL sterile high-density polyethylene (HDPE) bottles (IChem125-mL sterile HDPE bottle, Fisher Cat. No. N411-0125 or equivalent).

7.13 Sterile Sample Filters: Sterile sample filters (Corning 26-mm surfactant free cellulose acetate 0.2-µm filter, Fisher Cat. No. 09-754-13 or equivalent). If alternate filters are used they should be certified as having passed a bacterial challenge test. Additionally, if alternate

filters or different lots of the recommended filters are used, they must be tested using a LRB and a LFB fortified at the MRL as outlined in Section 9.2 to insure that they do not introduce interferences or retain perchlorate.

7.14 Sterile disposable syringes, 20mL: Fisher Catalog# 14-820-19, or equivalent).

7.15 Nitrogen: High purity compressed gas used for desolvation in the mass spectrometer. The gas purity and pressure requirements will depend on the instrument manufacturers' specifications.

7.16 Helium: High purity compressed gas at 60 psi to pressurize eluent, deliver water to the suppressor, and move the injection valve from LOAD to INJECT and back again.

7.17 Auxillary 6-Port Valve: Allows diversion of the flow to waste after conductivity detection, and directs flow to MS for Perchlorate detection (after conductivity detection).

7.18 15mL Vials: Glass, Teflon-lined caps. (VWR P/N 66009-560)

7.19 Ultrasonic Bath, with timer: Branson 2510 MTH

7.20 Vortex: VWR Vortexer 2

7.21 Analytical Balance: Accurate to 0.0001g.

8. Standards and Reagents

8.1 Reagent water: Distilled or deionized water 18.2 Mohm or better, free of the anions of interest. Water should contain particles no larger than 0.20 microns.

8.2 Eluent Solution, 45mM KOH: Prepared by the eluent generator installed as a component of the IC System equipped with KOH cartridges. The eluent generator (Section 7.1.5) prepares the eluent utilizing the KOH cartridge (Section 7.1.5) installed as a component of the Eluent Generator.

8.3 Perchlorate Stock Calibration Standard Solution, 1000 mg/L: May be purchased commercially prepared (Ultra Scientific ICC-013 or Accustandard IC-PER-10x-1) or prepared from ACS reagent grade, sodium salt as listed below. (NOTE: Sodium perchlorate represents a molar weight fraction of 81.2 % perchlorate anion).

To prepare using sodium salt: dissolve 0.1231g of sodium perchlorate ($NaClO_4$, CASRN [7601-89-0] in reagent water and dilute to 100 mL in a volumetric flask.

NOTE: Perchlorate stock standards, stored at room temperature, appear to be very stable and may be stable for an extended period of time. However, specified expiration dates should be marked on each prepared stock standard. Perchlorate stock standards purchased commercially, expire according to vendor specified date stated on the label. Expiration date of intermediate stock standards prepared in the lab from perchlorate salts or from purchased stock standards is no more than 12 months and is clearly specified on the label.

8.3.1 Intermediate Calibration Stock Solution, 100 000 µg/L (ppb)
Dilute 1.0mL of 1000 mg/L Perchlorate Stock Standard Solution (Section 8.3) brought to 10mL volume with reagent water.

8.3.2 Intermediate Calibration Stock Solution, 10 000 µg/L (ppb)
Dilute 100uL of 1000 mg/L Perchlorate Stock Standard Solution (Section 8.3) brought to 10mL volume with reagent water

8.3.3 Intermediate Calibration Stock Solution, 1 000 µg/L (ppb)
Dilute 100uL of 100 000 ug/L Intermediate Calibration Stock Solution (Section 8.3.1) brought to 10mL volume with reagent water

NOTE: Any of the following calibration standards (Section 8.3.2) may be used to perform the LFB, CCCS, ECCS, or LFM at required concentrations (sections 10.3, 10.4, 10.5)

8.3.4 Intermediate Calibration Stock Solution, 100 µg/L (ppb): *e.g.*, 100uL of Intermediate Calibration Stock Solution , 10 000 µg/L (Section 8.3.2) diluted to 10mL with reagent water. Note: All calibration stock solutions expire one year after preparation and should be stored at room temperature. Individual calibration point standards (0.02ug/L through 10ug/L) expire one month after preparation.

8.3.4.1 10µg/L Calibration Standard: 2.5mL of 100µg/L Calibration Standard (Section 8.3.3.) diluted to 25mL with reagent water.

8.3.4.2 5µg/L Calibration Standard: 1.25mL of 100µg/L Calibration Standard (Section 8.3.3.) diluted to 25mL with reagent water.

8.3.4.3 2µg/L Calibration Standard: 0.5mL of 100µg/L Calibration Standard (Section 8.3.3.) diluted to 25mL with reagent water.

8.3.4.4 1µg/L Calibration Standard: 2.5mL of 100µg/L Calibration Standard (Section 8.3.3) diluted to 250mL with reagent water.

8.3.4.5 0.5µg/L Calibration Standard: 1.25mL of 10µg/L Calibration Standard (Section 8.3.2.1) diluted to 25mL with reagent water.

8.3.4.6 0.2µg/L Calibration Standard: e.g., 400uL of 100µg/L intermediate Calibration stock solution (Section 8.3.3) diluted to 200mL with reagent water.

8.3.4.7 0.1µg/L Calibration Standard: 0.25mL of 10µg/L Calibration Standard (Section 8.3.2.1) diluted to 25mL with reagent water.

8.3.4.8 0.05µg/L Calibration Standard: 2.5mL of 5µg/L Calibration Standard (Section 8.3.2.2) diluted to 250mL with reagent water.

8.3.4.9 0.02µg/L Calibration Standard: 0.20mL of 1µg/L Calibration Standard (Section 8.3.2.4) diluted to 10mL with reagent water.

8.3.4.10 0.01µg/L Calibration Standard: 0.10mL of 1µg/L Calibration Standard (Section 8.3.2.4) diluted to 10mL with reagent water.

8.4 QCS, Perchlorate stock standard solution, 1000 mg/L: May be purchased commercially prepared (eg Accustandard IC-PER-10x-1 or Ultra Scientific ICC-013) or prepared from ACS reagent grade, sodium salt as listed below. (NOTE: Sodium perchlorate represents a molar weight fraction of 81.2 % perchlorate anion.

Perchlorate (ClO_4^-) 1000 mg/L: Dissolve 0.1231g sodium perchlorate ($NaClO_4$, CASRN [7601-89-0] in reagent water and dilute to 100 mL in a volumetric flask.

NOTE: Perchlorate stock standards, stored at room temperature, appear to be very stable and may be stable for an extended period of time. However, specified expiration dates should be marked on each prepared stock standard. Perchlorate stock standards purchased commercially, expire according to vendor specified date stated on the label. Expiration date of intermediate stock standards prepared in the lab from perchlorate salts or from purchased stock standards is no more than 12 months and is clearly specified on the label.

8.4.1 **Perchlorate QCS Intermediate Stock (2nd source) Solution #1, 10 000µg/L**. This standard must be prepared from a purchased source other than that used to prepare initial calibration standards (Section 8.3). e.g., 100uL of 1000 mg/L QCS Perchlorate Stock Standard Solution (Section 8.4) brought up to final volume of 10mL with reagent water.

8.4.2 **Perchlorate QCS Intermediate Stock (2nd source) Solution #2, 100µg/L**. This standard must be prepared from a purchased source other than that used to prepare initial calibration standards (Section 8.3). *E.g.*, 100uL of Perchlorate QCS Intermediate Stock (2nd source) Solution #1, 10 000µg/L (Section 8.4.1) brought up to final volume of 10mL with reagent water.

8.4.2.1 **5µg/L QCS Standard working solution:** e.g., 500uL of the 100µg/L Solution (Section 8.4.2) brought to 10mL final volume with reagent water.

8.4.2.2 **1.0µg/L QCS Standard working solution:** e.g., 100uL of the 100µg/L Solution (Section 8.4.2) brought to 10mL final volume with reagent water

8.5 Mixed Common Anion Stock Solution: Contains the anions bicarbonate, chloride, sulfate and carbonate each at 25 mg/mL anion concentration. This solution is used to prepare simulated common anion samples in the determination of a Conductivity Maximum. This solution is also used to prepare the Laboratory Simulated Sample Matrix Standard and Blank (Sections 10.1.7). Store at room temperature for no more than 12 months. Record the date of expiration on the storage container.
Dissolve the following salts in reagent water to a final volume of 25.0 mL: (Final concentration is 25,000mg/L of each anion.)

1.0 g sodium chloride (NaCl, CASRN [7647-14-5] Fisher ACS Reagent Grade*) = 0.61 g Cl*

0.93g sodium sulfate or 1.14g potassium sulfate, (Na_2SO_4 , CASRN [7757-82-6] Mallinckrodt ACS Reagent Grade) = *0.63 g SO4* ; (K_2SO_4 , emd px1595-1).

1.1g sodium carbonate (Na_2CO_3 , CASRN [497-19-8] Fisher HPLC Grade / ACS Grade) = *0.62 g CO3*

0.86g sodium bicarbonate ($NaHCO_3$, CASRN [144-55-8], fisher 5233-3) = *0.625g HCO3*.

8.5.1 **LSSM Blank**: Contains anions at 1000mg/L each to demonstrate low background at high levels of anions. Bring 1mL of the Mixed Common Anion Stock Solution (Section 8.5) to a final volume of 25mL. This blank is analyzed during the IDC and quarterly.

8.5.2 **LSSM LCS:** Contains anions at 1000mg/L each to demonstrate that high anion fortification does not interfere with the detection of Perchlorate. Bring 1mL of the Mixed Common Anion Stock Solution (Section 8.5) and 1.25mL of the 10ppb Cal Standard

(Section 8.3.2.2) to a final volume of 25mL with reagent water. The final concentration of the LSSM LCS is 0.5ppb. This standard is analyzed during the IDC and quarterly. The standard expires after one month of preparation.

8.6 **$Cl^{18}O_4^-$ Internal Standard Stock:** 1mg of $Cl^{18}O_4^-$ in 0.55g $H_2{}^{18}O$, purchased standard from ICON ISOTOPES/BERRY ASSOCIATES P/N IO6455 (or equivalent). Chemical purity 98%, isotopic enrichment 95%. As sodium perchlorate (MW 130.44). Store in the fridge or at room temperature for no more than 12 months after opening. Record the date of opening on the container.

8.6.1 **10,000µg/L Working IS stock:** 71.7uL of $Cl^{18}O_4^-$ Internal Standard Stock (Sec. 8.6) brought up to a volume of 10mL with reagent water. This standard expires 12 months after date of preparation.

8.6.2 **100µg/L Working IS:** 100uL of Working IS Stock (Sec. 8.6.1) brought up to a volume of 10mL with reagent water. This standard expires 12 months after date of preparation, or by the expiration date of Working IS stock, whichever is earlier.

*Add 10µL of this standard to 1mL of sample to obtain the correct Internal Standard final concentration of 1µg/L.

8.6.3 **100µg/mL $^{18}O_4$-ClO_4** Prepared from a solid source: To prepare this solution from a solid standard, weigh out 12.1 mg of $NaCl^{18}O_4$ into a 100-mL volumetric flask and dilute to 100 mL with reagent water.

8.7 **PPG 3000 (Neg) Standard:** Component of the "Mass Spectrometer Standards Kit" (PE SCIEX P/N 401936). Store at 4 ± 2°C. Standard expires one year from date of opening or by manufacturer specified date, whichever is earlier. Record opening date on the standard container.

Tuning/Calibration Masses:

59.050	175.133	616.464	906.673	1254.925	1545.134	1777.702

8.8 **Perchlorate mass calibration verification standard**.

This standard is a mixture of 100ug/L $Cl^{18}O_4^-$ and 100ug/L ClO_4^-. It is prepared by diluting 10 000ug/L stock solutions, e.*g.*, add 100uL of 10,000µg/L Working IS stock (Sec. 8.6.1) and 100uL of Intermediate Calibration Stock Solution, 10 000 µg/L (ppb) (Sec. 8.3.2) and bring up to final volume of 10mL with reagent water. This standard expires after 12 months from date of preparation or by expiration date of any of the stocks used in preparation, whichever is earlier.This standard is used to verify mass accuracy of target analytes after each new mass calibration according to procedure outlined in Section 9.2.4.

8.9 **HPLC grade Acetonitrile:** (Fisher P/N AC61001-0040)

8.10 **Ottawa Sand:** 20 – 30 mesh. eg. EMD, P/N SX0075-1.

9. Procedure

9.1 Set-Up

9.1.1 **MS/MS Instrument**:

Open Analyst Software from Icon on Desktop.

Under the “Configure” Tab, open Hardware Configuration
Click on “MassSpec Only” and Press “Activate Hardware”

Under the “Acquire” Tab
Press the “Ready” Icon and verify that the Syringe Icon and MassSpec icon are Green

Perchlorate Acquisition Parameters: (Negative Ion Mode) MRM Analysis. An example of method operational conditions is given below, actual conditions my vary slightly:

Ion Source:	TurboSpray (ESI)
Curtain Gas (CUR):	50.0 [psi]
Collision Gas (CAD):	6 [psi]
Ion Spray Voltage (IS):	-4000.0 [Volts]
Temperature (TEM):	450.0 [°C]
Ion Source Gas 1 (GS1):	60.0 [psi]
Ion Source Gas 2 (GS2):	70.0 [psi]
Interface Heater:	On
Declustering Potential (DP):	-30.0 [Volts]
Focusing Potential (FP):	-300.0 [Volts]
Entrance Potential (EP):	-10.0 [Volts]
Collision Energy (CE):	-35.0 [Volts]
Collision Cell Exit Potential (CXP):	-17.0 [Volts]
Ion Energy 1 (IE1):	-1.0 [Volts]
Ion Energy 3 (IE3):	-3.5 [Volts]
Q1 Resolution:	Unit
Q3 Resolution:	Unit
Deflector (DF):	255.0 [Volts]
CEM (CEM):	2100-2500[Volts]
Scans in Period:	1904
Scan Type:	MRM
Polarity:	Negative

Q1 Mass (amu)	Q3 Mass (amu)	Dwell time (msec)	
99.00	83.00 (loss of ^{16}O)	150.00	($^{35}ClO_4$ transition)
101.00	85.00 (loss of ^{16}O)	150.00	($^{37}ClO_4$ transition)
107.00	89.00 (loss of ^{18}O)	150.00	($^{35}Cl^{18}O_4$ Internal Std)

Synchronization Mode:	LC sync
Acquisition Duration:	9.997 min
Number of Cycles:	1904

9.1.2 IC Instrument:

Open Chromeleon Software from the Icon on the Desktop and enable the Server.
Open the IC/MS/MS Project Folder
Open "My Panels" and Select "Conductivity Setup" to open the Control Panel for the IC.

Perchlorate Method Parameters:

Pressure Lower Limit:	200psi
Pressure High Limit:	3000psi
Typical Back Pressure:	1700-1900psi
Autosampler Flush Volume:	500µL
Syringe Speed:	5
Mobile Phase:	60mM KOH
Suppressor Current:	45mA
Flow Rate:	0.30mL/min
Run Time:	10 min
Column Oven Temperature:	35 °C

9.1.3 Soil sample extraction

Prior to sample analysis, soil samples are extracted as follows, and sterilized as per Section 6.3.1.2.

- Using an analytical balance, weigh 1.0g of each sample and QC sample into separate 15mL glass vials. Weigh 1.0g Ottawa Sand for the Blank and LCS samples.
- Add 0.1ml of 100ppb IS Solution (Section 8.6.2) to each vial.

NOTE: Because the IS concentration must remain constant for quantitation, the extract may not be diluted prior to analysis. If upon analysis the sample requires dilution to quantitate the value to be within the calibration curve, there are two options:

Option 1: Prepare the soil sample again, using a smaller amount of sample, or

Option 2: Prepare the soil sample again using the same amount of sample, but adding the IS at the time of analysis, following extract dilution.

- Add the appropriate Spiking Solution to the batch LCS and MS samples (typically 0.1mL of 100ppb, Section 8.3.2).
- Add 10mL of Reagent Water (Section 8.1)
- Cap and vortex each vial for 30 seconds.
- Sonicate in the Ultrasonic Bath for 15 minutes.
- Remove vials from the Bath and vortex again for another 30 seconds.
- Allow the vials to stand for 15 minutes. If necessary, centrifuge the extracts before filtration.
- Filter the extracts through a 0.45µm filter as per Section 6.3.1.2.
- Samples are ready for analysis.

9.2 MS/MS Instrument Calibration

Instrument Calibration is achieved using the PPG 3000 Negative Standard (Section 8.7) as follows:

9.2.1 Tuning Q1

Open the Tune Tab Click on the "T" to reserve the Instrument for Tuning (ensure that the Icons in the lower portion of the screen turn from Yellow to Green). Double Click the Manual Tuning Tab, and ensure that the correct Project folder (Perchlorate) is open.

Select the Q1NegPPG data acquisition file.

Choose Scan Type Q1 MS and Polarity of Negative.
Check Center/Width and define the window as 6amu.
Click on the Advanced MS Tab and confirm that the Scan Mode is Profile and the Step Size is 0.1
Click on the Resolution Tab. The Q1 Resolution should be set to Unit and the Ion Energy should be 0.5. The Q3 Resolution should be set to Unit and the Ion Energy should be 2.0.
Uncheck the MCA box.
Fill the Syringe with the PPG solution and start the syringe pump.

Click Start to begin the scan.

Look for a constant TIC, and when it achieved, right click and Open File.
Right Click over one of the eight spectra windows and select List Data.
Click Calibration Peak List tab to display the mass list and their widths.
The masses must be between 0.6 and 0.8 amu.
To adjust widths, change the offset values in the Resolution Table by no more that 0.005 units at a time. Once the 0.6 to 0.8 windows are achieved, click the Apply button.

When optimization is achieved, return to the Manual Tune window and click the stop button.

Proceed to Calibration (Section 9.2.2).

9.2.2 Calibration of Q1

On the MS tab in the Manual Tune window, check the MCA box and enter a period of 10 cycles.

Click the Start button to begin the scan.
Once the TIC window opens, right mouse click over the spectra and click Open File, which will open the spectra for the 7 individually defined mass windows (masses defined in Section 8.7).

Once the 10 cycles have been collected, and unit resolution (of 0.6 to 0.8 amu) is confirmed, click one of the spectra and click the Calibrate from Spectra button in the Toolbar.
A Mass Calibration Option dialog opens and shows the settings for calibration.

Click the Start button to begin the calibration procedure.

A Mass Calibration Report appears containing three graphs:
Mass Shift: difference between expected and found masses
Peak Width: width for each mass
Intensity Difference: differences in intensity between this data and previous calibration

Minimize the Mass Calibration Report to display the Mass Calibration Results text page. The slope variation should be equal to 1 +/- 0.002.

Return to the Mass Calibration Report and press the Replace Calibration button for a full mass range calibration replacement.

9.2.3 **Tuning and Calibration for Q3**: Follow the Tuning and Calibration procedure for Q1, replacing the Q1NegPPG data acquisition method with Q3NegPPG data acquisition method.

9.2.4 **Verification of new mass calibration**. Mass calibration must be verified prior to the analysis of field samples by infusing Perchlorate mass calibration verification standard (Sec. 8.8) and acquiring a full scan continuum mass spectrum that covers the *m/z* range of the target analyte (i.e., 95-115amu). To perform the test open the instrument in Manual Tuning mode and use *PercMassVerification.dam* method to scan the defined mass range while infusing Perchlorate mass verification standard (Sec.8.8). The peaks should be symmetric around the target mass and perchlorate ion ($^{35}ClO_4$) should have a *m/z* of 99 ±0.3, the ($^{35}ClO_4$) isotope should have a *m/z* of 101 ± 0.3 and the internal standard ($^{35}Cl^{18}O_4$) should have a *m/z* of 107 ± 0.3. If these parameters are not achieved new mass calibration and/or instrument maintenance need to be performed. (Sec. 10.2 of Method 332.0 and Sec. 10.2.1 of Method 331.0).

9.3 Analyte Calibration

Demonstration and documentation of acceptable initial calibration is required prior to the IDC and before any samples are analyzed. It is also required intermittently throughout sample analysis to meet required QC performance criteria outlined in this method and summarized in Table 2. Initial calibration verification is performed using a QCS as well as with each analysis batch using initial, continuing (when more than 10 field samples are analyzed), and ending calibration check standards. It must also be demonstrated that the highest calibration point does not carryover into the next sample. To demonstrate this, a blank must be analyzed directly following the highest calibration point.

Establish Ion Chromatographic and MS/MS operating parameters equivalent to those indicated in Section 9.1.

Estimate the Linear Calibration Range (LCR): The LCR should cover the expected concentration range of the field samples and should not extend over more than two orders of magnitude in concentration. The restriction of two orders of magnitude is prescribed since beyond this it is difficult to maintain linearity throughout the entire calibration range.

A minimum of five calibration standards are required for a curve that extends over a single order of magnitude and a minimum of seven calibration standards are required if the curve covers two orders of magnitude. Since the anticipated concentration range for perchlorate in actual field samples is expected to cover two orders of magnitude, the use of at least seven calibration standards in the range 0.05 – 10µg/L is recommended. Typically, Alpha utilizes 8 calibration standards.

Prepare the calibration standards (as detailed in Sections 8.3.2) at concentrations of 0.01µg/L, 0.02µg/L, 0.05µg/L, 0.10µg/L, 0.20µg/L, 0.50µg/L, 1.0µg/L, 2.0ug/L, 5.0µg/L, and 10µg/L. Add 10µL of the IS Working Standard (Section 8.6.2) to 1mL of each standard. Cap the vial and Vortex for 5 seconds to ensure proper mixing.

***Note:** Calibration Solutions are NOT processed with sample collection devices.

Prepare an Acquisition Batch for the MS/MS and a Sequence for the IC (as outline in Section 9.5), and Load the Autosampler accordingly (as outlined in Section 9.6).
Inject 100µL of each calibration standard. Tabulate peak area responses against the Perchlorate concentration. Calculate the Transition Ratio (Section 10.8) and the Internal Standard Peak Area response (Section 10.9). If all parameters are within acceptable limits, the results can be used to calculate a calibration curve. Acceptable calibration is confirmed after reviewing the curve for linearity (Linear and 1/x regressions acceptable) and passing the criteria for the QCS as outlined in section 10.1.5. A correlation coefficient (r^2) of 0.995 or better is acceptable. The calculated value for each standard equal to or less than the MRL should be within 50-150% of true value. All other calibration points should have a calculated value 80-120% within true value. If these criteria are not met, the curve is considered invalid, and should be prepared and analyzed again.

Peak areas must be used as a measure of response since they have been found to be more consistent, in terms of quantitation, than peak heights. Using peak areas, it is the analyst's responsibility to review all chromatograms to ensure accurate baseline integration of target analyte peaks, since poorly drawn baselines will significantly influence peak areas.

INITIAL CALIBRATION CURVE VERIFICATION: After establishing or reestablishing calibration curves, the accuracy of this calibration must be verified through the analysis of a QCS or externally prepared second source (Section 8.4.1). The QCS should be prepared at a concentration near the middle of the calibration curve. As specified in Section 10.1.5, determined concentrations must fall within ± 20% of the stated values. It must be established that there is no carryover present by analyzing a blank immediately following the highest calibration point. The blank must adhere to the specifications noted in Section 10.2.

9.4 Standardization (Continuing Calibration Verification)

The calibration curve must be verified (each analysis day) as the first standard of the day, at the midrange of the curve. In addition, a Reporting Limit Check Standard (Section 10.7) must be within acceptable limits.

Upon calibration verification, samples may be analyzed, and a continuing calibration verification standard must be analyzed every 10 samples. The concentration of this standard should alternate, spanning the range of the curve.

9.5 Sample Analysis

Ensure the samples have come to room temperature prior to conducting sample analysis.

9.5.1 Build an MS/MS Acquisition Batch:

Batches of samples to be analyzed must have an MS/MS "Acquisition Batch" and an IC Sequence".

Open "Build Acquisition Batch" by double clicking it in the menu on the Left.

Press “Add Set” then “Add Samples” and enter the amount of samples intended for analysis.

Under the Sample Tab, Type in the Sample Name and the Date File name. Also pick the Quantitation Method (Perchlorate) as well as the Acquisition Method (Perchlorate) from the drop down menus. Add a column for Workgroup and Analyst ID, and insert proper information.

Under the Quantitation Tab, give each sample a “sample type”

“Standard” for Calibration points

“Double Blank” for DI H_2O blanks

“Blank” for blanks with Internal Standard added

“QC” for quality control standards

“Unknown” for samples

Fill in the appropriate concentrations for all Standards and QC samples.

Save the Acquisition Batch, the name of the file should indicate the date.

Under the Submit Tab, Press submit to send the acquisition batch to queue.

Press the Acquire Tab. The acquisition batch that was just submitted should be sitting “in queue”.

Press the Start Sample Button. The sample collection will begin when the sync from the LC is activated.

9.5.2 Build an IC Sequence:

Open the Browser in the Chromeleon Software and Open a new Sequence under the IC/MS/MS Folder.

Name the Sequence with the same file name as the Acquisition Batch for the MS/MS

Add the same amount of lines as the Acquisition Batch

Each sample type will remain “Unknown”

Type in the Autosampler position for each Sample

Type in the Injection Volume (100µL)

Choose the “Perchlorate” Program from a drop down menu for each sample

Choose the “Perchlorate” Method from a drop down menu for each sample

Type in Dilution Factors for samples that will be analyzed as a dilution.

Save the file by pressing the disk icon

Under the “Batch” tab, Press Edit, and Add the Sequence for analysis.

When samples are loaded into the Autosampler, pressing “Start” will send the LC sync, allowing analysis and data acquisition to begin.

Record the Batch Sequence in the Run Log Notebook.

9.6 Autosampler Loading

9.6.1 Liquid Sample Extracts

- Label a 2mL vial for each sample in the Sequence.
- Add 1mL of each Instrument Standard, QC sample, and Liquid sample extract to its appropriately labeled vial.
- Add 10μL of the 100μg/L Internal Standard (Section 8.6.2) to each vial.
- Place a cap on each vial and Crimp it securely.
- Vortex each vial at high speed for approximately 5 seconds per vial.

9.6.2 Soil Sample Extracts

- Label a 2mL vial for each sample in the Sequence.
- Add 1mL of each Instrument Standard, QC sample, and Soil sample extract to its appropriately labeled vial.
- Add 10μL of the 100μg/L Internal Standard (Section 8.6.2) to each Instrument Standard vial only.
- Place a cap on each vial and Crimp it securely.
- Vortex each vial at high speed for approximately 5 seconds per vial.

To Open the Autosampler door: On the front panel of the AS50, Press Menu, #5,#7, then scroll to YES and enter. Open the door and remove the Autosampler tray. Place each vial in the appropriate position, corresponding to its labeled position in the Sequence.
Replace the tray and close the door.

Samples are now ready for analysis, and the batch may be started.

9.7 Quantitation

Perchlorate is quantitated on the 99/83 transition. The 101/85 transition is used for confirmation of the transition area ratio.

Open the Quantitation Wizard in the Analyst (MS/MS) Software

Select the samples from the Data files that were entered into the Acquisition Batch.

Hit Next, then Next, Choose the quantitation method (Perchlorate) and press Finish.

Check the chromatograms individually by double clicking on the first sample and scrolling through the entire batch.

- Ensure that all peaks (the 101/85 transition, the 99/83 transition, and the 107 Internal Standard) are integrated properly.
- Make adjustments with the parameters button when necessary.
- Verify that minimum QC requirements for the batch have been analyzed, that all QC standards are within acceptable limits, and that all samples have been analyzed within the range of calibration.
- If any quantitated value is greater than the calibration range, proceed as follows:

1) **For Liquid sample extracts**, dilute the sample extract with reagent water (Section 8.1) to be within the calibration range. Proceed to Section 9.6.1.

2) **For Soil sample extracts,** re-prep the sample according to Section 9.1.3, without adding Internal Standard. Prior to reanalysis, dilute the extract with reagent water (Section 8.1) to be within the calibration range. Proceed to Section 9.6.1 for Liquid sample extracts.

When the batch is properly integrated and quantitated, save the Quantitation batch under a file name that indicates the date, and print the file.

The Quantitation batch may now be exported to the LIMS system.

When a dilution is performed, the MDL and MRL values are proportionally increased to reflect the dilution factor.

9.8 Preventative Maintenance

The suggested Preventative Maintenance Schedule is outlined below:

As Needed

Check the eluent and solvent reservoirs to see if they need to be refilled.

Check Regenerant DI water reservoir and refill if needed.

Lubricate autosampler arm rails.

Daily

Check the ICS-2500 component mounting panel and API2000 for leaks or spills. Wipe up spills. Isolate and repair leaks. Rinse off any dried eluent with reagent water.

Check the waste container daily and empty as needed.

Weekly

Once a week, check fluid lines for crimping or discoloration. Relocate any pinched lines. Replace damaged lines.

Check the junctions between the pump heads and the pump casting for evidence of liquid leaks. If piston seal wash tubing is not connected, check the drain tubes at the rear of the pump heads for evidence of moisture. Normal friction and wear may gradually result in small liquid leaks around the piston seal. If unchecked, these leaks can gradually contaminate the piston housing, causing the pump to operate poorly. If leaks occur, replace the piston seals.

Check the end-line filter (PN 045987) and change if needed. When new, end-line filters are pure white. Any discoloration of the filter might indicate bacterial/algae(green) buildup .The filter should be replaced immediately and reservoir cleaned to remove contamination (using mild caustic solution, NaOH, works well; rinse thoroughly with DI water prior to use)

NOTE: It is especially important to regularly replace end-line filters when using aqueous eluents, which may contaminate the filter with bacteria or algae. The bacterial buildup may not be visible.

Perform Flush and Prime cycles using autosampler control panel.

Monitor the top of inject port for any "pooling" liquid. Excessive "pooling" can cause carryover and poor reproducibility of injections and must be addressed immediately. Check needle for signs of wear, inspect tubing/valve for blockage and perform alignment of the sampling needle in the inject port (Sec. 5.7 of AS50 Autosampler Manual).

Every six to twelve months (performed using Preventive Maintenance kits):

Replace HS602 vacuum pump oil using HE200 oil or equivalent. (API2000)

Thoroughly rinse eluent reservoirs using methanol followed by DI water (use mild caustic solution, NaOH, if bacterial/algae contamination is suspected).

Replace end-line filters. (IC)

Clean orifice, curtain and skimmer plates and Q0 region as described in API2000 coursebook manual.

Visually inspect the dark teflon coating on the sampling needle and replace the needle assembly (P/N 054271) if the coating is worn (refer to Section 5.3 of AS50 autosampler operator's manual for instructions). (IC)

Perform the AS50 preventive maintenance procedure. An AS50 Preventive Maintenance Kit is available for this purpose (P/N 055644, with PEEK parts, or P/N 055643, with stainless steel parts). The kit provides parts and instructions for performing the procedure. Rebuild the injection valve.

9.9 Calculations

9.9.1 Calculate the MDL as follows:

$$MDL = (t) \times (S_{n-1})$$

Where:

t = student's value for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom [t = 3.14 for seven replicates]

S_{n-1} = sample standard deviation (n-1) of the seven replicate analyses.

9.9.2 Determine the Half Range for the prediction interval of results (HR_{PIR}) using the equation below

$$HR_{PIR} = 3.963S$$

Where: S is the standard deviation, and 3.963 is a constant value for seven replicates.

9.9.2.1 Confirm that the upper and lower limits for the Prediction Interval of Result (PIR =Mean + HRPIR) meet the upper and lower recovery limits as shown below

The Upper PIR Limit must be <150 percent recovery.

$$\frac{\text{Mean} + HR_{PIR}}{\text{Fortified Concentration}} \times 100$$

The Lower PIR Limit must be >50 percent recovery.

$$\frac{\text{Mean - } HR_{PIR}}{\text{Fortified Concentration}} \quad x100$$

9.9.3 Calculate the IDA (Section 10.1.3) by calculating the mean measured concentration (Cx) of the replicate values as follows:

$$C_x = \frac{(C_1 + C_2 + C_3 + ...Cn)}{N}$$

Where:

C_x = Mean recovered concentration of the replicate analysis

C_1, C_2, ...Cn = Recovered concentrations of the replicate 1, 2...n.

N = 7

9.9.4 Calculate the IDP (as outlined in Section 10.1.4) by determining the %RSD of the replicates used for determining the IDA:

$$\%RSD = \frac{(S_{n-1})}{(C_x)} \times 100$$

Where:

S_{n-1} = sample standard deviation (n-1) of the replicate analyses.

C_x = mean recovered concentration of the replicate analysis

9.9.5 Calculate the percent recovery for each LFM, corrected for concentrations measured in the unfortified sample. Percent recovery should be calculated using the following equation:

$$\%REC = \frac{(Cs - C)}{s} \times 100$$

Where:
%REC = percent recovery,
Cs = measured perchlorate in the fortified sample,
C = measured native perchlorate sample concentration, and
s = concentration equivalent of analyte added to sample.

9.9.6 Calculate the relative percent difference (RPD) for each Duplicate or MS/MSD of the initial quantitated concentration (IC) and duplicate quantitated concentration (Dc) using the following formula:

$$RPD = \frac{|(IC - Dc)|}{([IC + Dc]/2)} \times 100$$

9.9.7 Reporting Calculations:

9.9.7.1 Liquid Samples:

Perchlorate concentrations are calculated in µg/L with the Date Acquisition Software and multiplied by any applicable dilution factor.

9.9.7.2 Soil Samples:

Perchlorate, µg/Kg =

$$\text{Perchlorate µg/L} \times \frac{\text{Final volume of sample extract (L)}}{\text{Weight of soil sample (Kg)}}$$

9.9.8 Calculation of the Transition Ratio (Expected Value = 3)

$$\frac{\text{Peak Area of the 99/83 Transition}}{\text{Peak Area of the 101/85 Transition}} = \text{Ratio of 99:101}$$

9.9.9 Calculation of the RRT

$$\frac{\text{Retention Time of Perchlorate Ion}}{\text{Retention Time of Internal Standard Ion}} = \text{RRT}$$

10. Quality Control and Data Assessment

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a calibration verification standard is used to confirm the measurements were performed in an in-control mode of operation.

Each laboratory using this method is required to operate a formal quality control (QC) program. The requirements of this program consist of an initial demonstration of laboratory capability, and subsequent analysis in each analysis batch of an Laboratory Reagent Blank (LRB), Initial Calibration Check Standard (ICV), RL Check Standard, Laboratory Fortified Blank (LFB), Continuing and End Calibration Check Standards (CCCS/ECCS), Laboratory Fortified Sample Matrix (LFM) and either a Field, Laboratory or LFM duplicate sample analysis.

This section details the specific requirements for each of these QC parameters. The QC criteria discussed in the following sections are summarized in Table 2.

10.1 Demonstration of Capability

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method. Each time a method modification is made, the analyst is required to repeat the procedure.

When the parameter tested fails at least one of the acceptance criteria, the analyst must locate and correct the source of the problem and repeat the test.

Repeated failure confirms a general problem with the measurement system or analytical technique of the analyst. If the failure repeats, locate and correct the source of the problem and repeat the test.

10.1.1 The Initial Demonstration of Capability (IDC): This is used to characterize instrument and laboratory performance prior to performing analyses by this method.

The laboratory must closely monitor the perchlorate retention time for this analysis. If sudden shifts of more than 5% are observed in the perchlorate retention time, some type of instrument problem may be present. Potential problems include improperly prepared eluent, erroneous method parameters programmed such as flow rate or some other system problem. The observed retention time for perchlorate should closely replicate the times established when the column was originally installed. As a column ages, it is normal to see a gradual shift and shortening of retention times, but if after several years of use, extensive use over less than a year, or use with harsh samples, this retention time has noticeably shifted to any less than 80% of the original recorded value, the column requires cleaning (according to manufacturer's instructions) or replacement. A laboratory should retain a historic record of retention times for perchlorate to provide evidence of an analytical column's continued performance.

10.1.2 Initial demonstration of low system background: A blank (LRB) must be analyzed and determined to have less than 1/3 the amount of perchlorate than the MRL. An LRB must also be analyzed after the highest calibration point in the curve to ensure that there is no carryover. This must be performed for during the IDC and in all subsequent calibration curves. The LRB must have less than 1/3 the amount of perchlorate than the MRL.

10.1.3 Initial Demonstration of Accuracy (IDA): Prepare and analyze 7 replicate LFBs fortified at the Reporting Limit (0.025 µg/L). Calculate the mean measured concentration (Cx) of the replicate values as outlined in Section 9.9.3. To pass the IDA, the value derived for Cx must be within ± 20% of the true value.

10.1.4 Initial Demonstration of Precision (IDP): Using the data generated for Section 10.1.3, calculate the percent relative standard deviation (%RSD) of the replicate analysis, as indicated in Section 9.9.4. To pass the IDP, the %RSD must be less than 20%.

10.1.5 Quality Control Sample (QCS): After calibration curves have initially been established or have been re-established, or as required to meet data quality needs, verify both the calibration and acceptable instrument performance with the preparation and analyses of an external/second source QCS, analyzed at the midpoint of the calibration curve. If the determined concentrations are not within ± 20% of the stated values, performance of the determinative step of the method is unacceptable. The source of the problem must be identified and corrected before either proceeding with the IDC or continuing with on-going analyses.

10.1.6 Minimum Reporting Level (MRL): The MRL is the threshold concentration of an analyte that a laboratory can expect to accurately quantitate in an unknown sample. The MRL should be established at an analyte concentration either greater than three times the MDL or at a concentration which would yield a response greater than a signal to noise ratio of five. Setting the MRL too low may cause repeated QC failure upon analysis of the RL Standard. **Although the lowest calibration standard may be below the MRL, the MRL must never be established at a concentration lower than the lowest calibration standard.**

10.1.7 Laboratory Simulated Sample Matrix (LSSM) An LSSM Blank and LSSM LCS are analyzed during the IDC and again quarterly to demonstrate that high anionic content does not interfere with the background levels or the detection of Perchlorate. The LSSM Blank (Section 8.5.1) must not have a calculated value higher than 1/3 of the MRL. The LSSM LCS (Section 8.5.2) must fall within an 80-120% recovery for perchlorate.

10.2 Blank

An LRB must be prepared and treated exactly as a typical field sample including exposure to all glassware, equipment, solvents, filtration and reagents that are used with field samples. Data produced are used to assess instrument performance of a blank sample and evaluate contamination from the laboratory environment. Values that exceed ½ the MRL indicate a laboratory or reagent contamination is present. The source of the contamination must be determined prior to conducting any sample analysis. Any sample included in an automated analysis batch which has an invalid LRB, indicated by a quantitated perchlorate that exceeds ½ the MRL, must be reanalyzed in a subsequent analysis batch after the contamination problem is resolved.

Two Blanks should be analyzed with each batch, a blank that contains Internal Standard (fortified at a level of 1μg/L) that will be labeled as “Blank IS” in the Acquisition Batch and a blank with no fortification that should be labeled as “Blank” in the Acquisition Batch.

NOTE: Blanks must be processed in the same manner as field samples, as described in Section 6.3.

10.3 Calibration Verification

Initial calibrations may be stable for extended periods of time. Once the calibration curve has been established it MUST be verified for each analysis batch, prior to conducting any field sample analysis using an Initial Calibration Verification Standard and an RL Check Standard. Continuing Calibration Check Standards and End Calibration Check Standards are also required as described in the sections below.

10.3.1 INITIAL CALIBRATION CHECK VERIFICATION (ICV): For each analysis batch the calibration must initially be verified prior to analyzing any samples. A point midrange in the calibration curve must be used. Percent recovery for the ICV must be in the range or 80-120% before continuing the analysis batch and conducting any sample analyses.

10.3.2 CONTINUING CALIBRATION CHECK/END CALIBRATION CHECK STANDARDS (CCCS/ECCS): Continuing calibration check standards MUST be analyzed after every tenth field sample analysis and at the end of the analysis batch as an end calibration check standard. If more than 10 field samples are included in an analysis batch, the analyst must alternate between the middle and high continuing calibration check standard levels. The percent recovery for perchlorate in the CCCS/ECCS must be between 80-120% (However, if the fortification is at or below the MRL, the recovery limits are 50-150%).

10.3.2.1 If during the analysis batch, the measured concentration for perchlorate in the CCCS or ECCS differs by more than the calibration verification criteria shown above, or if the perchlorate peak retention time shifts outside the retention time window (as defined in Section 10.1.1), all samples analyzed after the last acceptable check standard are considered invalid and must be reanalyzed. The source of the problem must be identified and resolved before reanalyzing the samples or continuing analyses.

10.3.2.2 In the case where the end calibration fails to meet performance criteria, but the initial and middle calibration checks are acceptable, the samples

bracketed by the acceptable calibrations may be reported. However, all field samples between the middle and end calibration checks MUST be reanalyzed.

10.4 Laboratory Control Samples (LFB)

Prepare a secondary dilution stock (Section 8.3.1), and use this to fortify the LFB and the LFMs (Section 10.5). An external source stock or QCS (Section 8.4), which is used to verify the accuracy of the calibration curve when it was initially prepared (Section 10.1.5), should not be used to prepare this secondary dilution stock. Laboratories are required to analyze a LFB (filtered as if it were a field sample) with each analysis batch. The LFB must be prepared with the same solution used to prepare the LFM and should be prepared at a concentration near the midpoint of the calibration curve. By analyzing the LFB initially, a control check is performed on the concentrated solution used to prepare the LFM. If any deviations in the perchlorate concentration are present, it will be reflected in the LFB and not exclusively attributed to a matrix upon analysis of the LFM. Calculate accuracy as percent recovery. The recovery for perchlorate must fall in the range of 80 - 120% prior to analyzing samples. If the LFB recovery for an analysis batch does not meet these recovery criteria the data are considered invalid, and the source of the problem should be identified and resolved before continuing analyses. The level of fortification must vary between low, mid and high levels (within the calibration curve).

NOTE: LFB's must be processed in the same manner as field samples, as described in Section 6.3.

10.5 Matrix Spike (LFM)

The laboratory must add a known amount of each target analyte to a minimum of 5% of the collected field samples or at least one with every analysis batch, whichever is greater. For a LFM to be valid, the target analyte concentrations must be greater than the native level. It is recommended that the solutions used to fortify the LFM be prepared from the same stocks used to prepare the calibration standards and not from external source stocks. This will remove the bias contributed by an externally prepared stock and focus on any potential bias introduced by the field sample matrix.

10.5.1 The fortified concentration must be equal to or greater than the native sample concentration. Fortified samples that exceed the calibration range must be diluted to be within the linear range.

10.5.2 Upon calculation of the native amount of perchlorate in the sample, the spike should be prepared near the same concentration and added to the analytical sequence. If no native perchlorate is detected, the LFM must be performed at the RL.

10.5.3 Calculate the percent recovery for each target analyte, corrected for concentrations measured in the unfortified sample. Percent recovery should be calculated using the equation outlined in Section 9.9.5.

10.5.4 Recoveries may exhibit a matrix dependence. If the recovery for perchlorate falls outside 80 - 120% for Liquid samples (50-150% if fortified at the RL), or 70 - 130% for Soil samples, and the laboratory's performance for all other QC performance criteria is acceptable, the accuracy problem encountered with the fortified sample is judged to be matrix related, not system related. The result for that analyte in the unfortified sample and the LFM must be labeled suspect/matrix to inform the data user that the result is suspect due to matrix effects with a narrative on the data report. Repeated failure to meet

suggested recovery criteria indicates potential problems with the procedure and should be investigated.

10.5.5 Analysts may replace the use of a Duplicate sample (Section 10.6) in a batch with the analysis of a Matrix Spike Duplicate (LFM-D). The acceptable percent recovery range is 80-120% with an RPD of 30.

10.6 Duplicates

FIELD, LABORATORY DUPLICATES OR DUPLICATE LFM: The laboratory must analyze either a field duplicate, a laboratory duplicate, or a duplicate LFM for a minimum of 5% of the collected field samples or at least one with every analysis batch, whichever is greater. The sample matrix selected for this duplicate analysis must contain measurable concentrations of the target anions in order to establish the precision of the analysis set and ensure the quality of the data. Without prior knowledge or strong suspicion that an unknown sample has measurable perchlorate concentrations, the best alternative is to analyze a duplicate LFM.

Calculate the relative percent difference (RPD) of the initial quantitated concentration (IC) and duplicate quantitated concentration (Dc) using the formula outlined in Section 9.9.6.

Duplicate analysis may exhibit a matrix dependence. If the RPD for the duplicate measurements of perchlorate falls outside ± 20% (+/- 50% for those concentrations less than 2xMRL) and if all other QC performance criteria are met, laboratory precision is out of control for the sample and perhaps the analytical batch. The result for the sample and duplicate should be labeled as suspect/matrix to inform the data user that the result is suspect due to a potential matrix effect, which led to poor precision. This should not be a chronic problem and if it frequently recurs (>20% of duplicate analyses), it indicates a problem with the instrument or individual technique that must be corrected.

10.7 RL Check Standard

A Reporting Limit (RL) check standard is analyzed at the beginning of each batch, following the ICV. Preparation instructions for this standard are in Section 8.3.1. This standard recovery must be within 50 – 150%. Analysis cannot continue until acceptable recovery is achieved.

If the RL check standard fails the acceptance criteria, refer to Section 12.8 for corrective action procedure.

10.8 Transition Ratio

The ratio between the 99/83 and 101/85 transitions must be monitored for all Calibration standards, QC standards, and samples. The expected ratio is 3.08.
A ratio less than 2.31 or greater than 3.85 should never be used for quantitation samples. A ratio beyond these requirements indicates that Perchlorate is not present, or that the instrument may be out of tune or calibration. Reanalyze the sample to verify the ratio, or re-tune and re-calibration is mandated (Sections 9.2 and 12.9).

10.9 Internal Standard Peak Area

The Peak Area for the Internal Standard must be monitored for all Calibration standards, QC standards, and samples. The criterion for this parameter is +/- 30% of the IS response in opening ICV of the day. Refer to Sec. 12.10 of this SOP for further explanation on how to proceed in case of deviation greater than 30%.

10.10 Relative Retention Time Acceptance Criteria (RRT)

All calibration, QC, and field samples must meet the Relative Retention Time acceptance criteria. The RRT is calculated according to section 9.9.9, and must be 1.0 +/-2% (0.98-

1.02) for a peak to be identified as perchlorate. Refer to Section 12.11 for corrective action procedure.

10.11 Control Limits

The laboratory maintains performance records to document the quality of data that is generated. Method accuracy for samples is assessed and records maintained.

10.12 Analytical Sequence

The analytical sequence is found in Table 1.

11. Method Performance

The MDL should be established using reagent water (blank) fortified at a concentration of 0.05µg/L. To determine MDL values, take seven replicate aliquots of the fortified reagent water and process through the entire analytical method. Perform all calculations defined in the method and report the concentration values in the appropriate units. Calculate the MDL as outlined in Section 9.9.1. Verify the Half Range for the prediction interval of results as outlined in Section 9.9.2. MDLs should be periodically verified, but MUST be initially determined when a new operator begins work or whenever there is a significant change in the background, or instrument response.

NOTE: Do not subtract blank values when performing MDL calculations.

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDL concentrations were obtained using reagent water. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

Method performance data is on file in the laboratory QC department. Comparison of method performance data for the laboratory to the reference method criteria occurs when laboratory in-house acceptance limits are generated. In-house generated data must be within the specifications of the reference method or the analysis is not continued until corrective action is completed.

12. Corrective Actions

Holding time exceedence, improper preservation, improper sterilization and observed insufficient sample headspace are noted on the nonconformance report form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

Review of calibration standards, blanks, and QC standards response for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form.

12.1 Failure of the QCS: If the determined concentration is not within ± 20% of the stated value, performance of the determinative step of the method is unacceptable. The source of the problem must be identified and corrected before either proceeding with the IDC or continuing with on-going analyses.

12.2 Failure of the LRB: Any sample included in an analysis batch which has an invalid LRB, indicated by a quantitated perchlorate value that exceeds 1/3 the MRL, must be reanalyzed in a subsequent analysis batch after the contamination problem is resolved.

12.3 Failure of the ICV: Percent recovery for the ICV must be in the range or 80 - 120% before continuing the analysis batch and conducting any sample analyses. The source of the problem must be identified and resolved before reanalyzing the samples or continuing analyses if the recovery is not within acceptable limits.

12.4 Failure of the CCCS/ECCS: The percent recovery for perchlorate in the CCCS/ECCS must be between 80-120% (However, if the CCCS is fortified at or below the MRL, the recover limits are 50-150%). If during the analysis batch, the measured concentration for perchlorate in the CCCS or ECCS differs by more than the calibration verification criteria stated, or if the perchlorate peak retention time shifts outside the retention time window (as defined in Section 10.1.1), all samples analyzed after the last acceptable check standard are considered invalid and must be reanalyzed. The source of the problem must be identified and resolved before reanalyzing the samples or continuing analyses. In the case where the end calibration fails to meet performance criteria, but the initial and middle calibration checks are acceptable, the samples bracketed by the acceptable calibrations may be reported. However, all field samples between the middle and end calibration checks MUST be reanalyzed.

12.5 Failure of the LFB: The recovery for perchlorate must fall in the range of 80 - 120% prior to analyzing samples. If the LFB recovery for an analysis batch does not meet these recovery criteria the data are considered invalid, and the source of the problem should be identified and resolved before continuing analyses.

12.6 Failure of the LFM: If the recovery for perchlorate in Liquid samples falls outside 80 - 120%, or outside 70 – 130% for Soil samples, and the laboratory's performance for all other QC performance criteria is acceptable, the accuracy problem encountered with the fortified sample is judged to be matrix related, not system related. The result for that analyte in the unfortified sample and the LFM must be labeled suspect/matrix to inform the data user that the result is suspect due to matrix effects.

12.7 Failure of the Duplicate: If the RPD for the duplicate measurements of perchlorate falls outside ± 20% (or +/- 30% for an LFM-Dup) (or +/-50% if the native Perchlorate level is less than 2xMRL) and if all other QC performance criteria are met, laboratory precision is out of control for the sample and perhaps the analytical batch. The result for the sample and duplicate should be labeled as suspect/matrix to inform the data user that the result is suspect due to a potential matrix effect, which led to poor precision. This should not be a chronic problem and if it frequently recurs (>20% of duplicate analyses), it indicates a problem with the instrument or individual technique that must be corrected.

12.8 Failure of the RL Check Standard: If the RL check standard is not recovered within 50 – 150%, then analysis is deemed unacceptable. The source of the problem must be identified and resolved before sample analysis can begin. If failure continues, the instrument is recalibrated.

12.9 Failure of the Transition Ratio:

If the ratio for a given sample is beyond acceptance criteria, the sample should be reanalyzed with and without a spike to verify the presence and concentration of Perchlorate.

If an entire batch of data has transition ratios that are non-compliant, the instrument should be tuned and calibrated according to Section 9.2.1 and 9.2.2.

A ratio less than 2.31 or greater than 3.85 should never be used when quantitating samples. If unacceptable ratios continue to be found, re-tune and re-calibration is mandated (Section 9.2.1 and 9.2.2).

12.10 Failure of the Internal Standard Peak Area: If the Peak Area of the Internal Standard for any Calibration Standard, QC standard, or sample deviates by more than ± 30% of the initial ICV for the day, a second aliquot of the sample is injected as a part of the same or new analytical batch within the holding time of the sample. If re-injected aliquot produces an acceptable IS response, results from that aliquot are reported. If the IS area counts of the re-injected aliquot still do not meet the IS criterion and the IS area counts of the most recent CCV is within criteria, sample results should be reported as “suspect matrix”. If the IS area counts criterion is not met for both the sample and the most recent CCV, corrective action, such as instrument maintenance and/or calibration, might be necessary. Once proper operating conditions are re-established all affected samples are reanalyzed.

12.11 Failure of the Relative Retention Time: If the relative retention time deviates by more than ± 2% from 1.0, the Mass Spec may be out of tune or calibration. Failure upon reanalysis mandates recalibration of the instrument (Section 9.2).

13. Definitions

13.1 ANALYSIS BATCH: A sequence of samples which includes no more than 20 field samples. An Analysis Batch must also include all required QC samples, which do not contribute to the maximum field sample total of 20. The required QC samples include:

- Laboratory Reagent Blank (LRB) (Blank IS)
- Laboratory Fortified Blank (LFB)
- Continuing Calibration Check Standard (CCCS), when the batch contains more than 10 field samples
- End Calibration Check Standard (ECCS)
- Laboratory Fortified Matrix (LFM)
- Either a Field Duplicate, a Laboratory Duplicate or a duplicate of the LFM

NOTE: Every field sample analysis, excluding any LFM or Duplicate field sample analysis which qualify as QC samples, must be applied to the maximum of 20 total field samples permitted in an analysis batch.

13.2 CALIBRATION STANDARD (CAL): A solution prepared from the primary dilution standard solution(s) or stock standard solutions. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

13.3 INITIAL CALIBRATION STANDARDS: A series of CAL solutions used to initially establish instrument calibration and develop calibration curves for individual target anions (Section 9.3).

13.3.1 INITIAL CALIBRATION CHECK STANDARD (ICV): A CAL solution, which is analyzed initially, prior to any field sample analyses, which verifies the previously established calibration curve. The concentration for the initial calibration check standard MUST be at about the midpoint of the calibration curve. The ICV is discussed in section 10.3.1.

13.4 CONTINUING CALIBRATION CHECK STANDARDS (CCCS): A CAL solution which is analyzed after every tenth field sample analyses, not including QC samples, which verifies the previously established calibration curve and confirms accurate analyte quantitation for the previous ten field samples analyzed. The concentration for the continuing calibration check standards should alternate levels (Section 10.3.2)

13.5 END CALIBRATION CHECK STANDARD (ECCS): A CAL solution which is analyzed after the last field sample analyses which verifies the previously established calibration curve and confirms accurate analyte quantitation for all field samples analyzed since the last continuing calibration check. The end calibration check standard should vary in concentration levels (Section 10.3.2).

13.6 FIELD DUPLICATES (FD): Two separate samples collected at the same time and placed under identical circumstances and treated exactly the same throughout field and laboratory procedures. Analyses of field duplicates indicate the precision associated with sample collection, preservation and storage, as well as with laboratory procedures (Section 10.6).

13.7 LABORATORY DUPLICATE (LD): Two sample aliquots (LD1 and LD2), taken in the laboratory from a single sample bottle, and analyzed separately with identical procedures. Analyses of LD1 and LD2 indicate precision associated specifically with the laboratory procedures by removing variation contributed from sample collection, preservation and storage procedures (Section 10.6).

13.8 LABORATORY FORTIFIED BLANK (LFB): An aliquot of reagent water, or other blank matrix, to which a known quantity of perchlorate is added in the laboratory. The LFB is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements (Section 10.4).

13.9 LABORATORY FORTIFIED SAMPLE MATRIX (LFM): An aliquot of an environmental field sample to which a known quantity of perchlorate is added in the laboratory. The LFM is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical result (when compared to the result for the LFB). The background concentrations of perchlorate, in the sample matrix, must be initially determined in a separate aliquot and the measured value in the LFM corrected for this background concentration (Section 10.5).

13.10 LABORATORY REAGENT BLANK (LRB): An aliquot of reagent water or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, filtration and reagents that are used with other samples. The LRB is used to determine if perchlorate or other interferences are present in the laboratory environment, the reagents, or the apparatus (Section 10.2).

13.11 LINEAR CALIBRATION RANGE (LCR): The concentration range over which the instrument response is linear.

13.12 MATRIX CONDUCTIVITY MAXIMUM (MCM): Demonstrates that the system can handle large levels of conductivity without affecting the quantitation of Perchlorate. The conductance of a sample matrix is proportional to the common anions present in the matrix (which contribute to the level of total dissolved solids [TDS]). The value for this threshold is dependant on the conditions, hardware, and state of the hardware employed. Consequently, this threshold is not method defined and must be determined by the

individual analytical laboratory during the Initial Demonstration of Capability (IDC). The MCM is verified quarterly with a Blank and LCS fortified at the MCM to ensure that all instrumentation is performing consistently. Matrix conductivity is measured in microsiemens/cm (µS/cm).

13.13 METHOD DETECTION LIMIT (MDL): The minimum concentration of an analyte that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero.

13.14 MINIMUM REPORTING LEVEL (MRL): The minimum concentration that can be reported as a quantitated value for a target analyte in a sample following analysis. This defined concentration can be no lower than the concentration of the lowest calibration standard and can only be used if acceptable quality control criteria for this standard are met (Section 10.1.6).

13.15 PROFICIENCY TESTING (PT) or PERFORMANCE EVALUATION (PE) SAMPLE:
A certified solution of method analytes whose concentration is unknown to the analyst. Often, an aliquot of this solution is added to a known volume of reagent water and analyzed with procedures used for samples. Results of these analyses are typically used as part of a laboratory certification program to objectively determine the capabilities of a laboratory to achieve high quality results.

13.16 QUALITY CONTROL SAMPLE (QCS): A solution of method analytes of known concentrations that is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check laboratory performance with externally prepared test materials (Section 10.1.5).

13.17 STOCK STANDARD SOLUTION (SSS): A concentrated solution containing perchlorate which is either prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source.

13.18 RL CHECK STANDARD: A calibration solution that is analyzed with each analytical batch to verify the Reporting Limit. (Section 10.7)

13.19 Transition Ratio: Chlorine naturally exists in two isotopic states: ^{35}Cl and ^{37}Cl. Since the formula for Perchlorate is ClO_4^- the natural abundance ratio of ^{35}Cl to ^{37}Cl apply. The naturally occurring ratio of ^{35}Cl to ^{37}Cl is 3.08. It is imperative that this ratio remain within acceptance criteria to accurately identify and quantitate Perchlorate. (Section 10.8)

13.20 Internal Standard: An internal standard will be detected in the same manner as the analyte of interest under the same chromatographic and mass spec conditions. In this case, the Oxygen has been labeled as ^{18}O instead of the naturally occurring ^{16}O. This allows the analyte of interest (Perchlorate) to be monitored without affecting quantitation.

13.21 MRM Monitoring: A mass spectrometric technique where a parent ion is first isolated then subsequently fragmented into a product ion(s). Quantitation is accomplished by monitoring a specific product ion.

14. Pollution Prevention

See Chemical Hygiene Plan for pollution prevention operations.

15. Waste Management

See Chemical Hygiene Plan for waste handling and disposal.

16. Attachments

Table 1: Analytical Sequence
Table 2: IDC AND QC REQUIREMENTS

TABLE 1

EXAMPLE SAMPLE ANALYSIS BATCH WITH QUALITY CONTROL REQUIREMENTS

Injection #	Sample Description	Acceptance Criteria
1	Blank	-
1	Blank IS	Less than 1/3 RL
2	ICV at midrange of calibration curve	80-120%
3	RL Check Standard (0.05µg/L)	50-150%
4	Batch Blank (LRB)	Less than 1/3 RL
5	Batch LCS (LFB) at midrange of calibration curve	80-120%
6	Sample 1	normal analysis
7	Sample 1 - Laboratory Duplicate (LD)	± 20% RPD (+/- 50% if Perchlorate is less than 2xMRL)
8	Sample 2 (sample with highest conductivity)	normal analysis
9	Sample 2 - Laboratory Fortified Matrix (LFM)	Recovery of 80 - 120% (Recovery of 50-150% when fortified at or below MRL)
10	Sample 2- Laboratory Fortified Matrix-Duplicate	+/-20%RPD (+/-50% when Perchlorate level is at or below 2x the MRL
11	Sample 3	normal analysis
12	Sample 4	normal analysis
13	Sample 5	normal analysis
14	Sample 6	normal analysis
15	Sample 7	normal analysis
16	Sample 8	normal analysis
17	Sample 9	normal analysis
18	CCCS (rotate level)	80-120%
19	Sample 10	Normal analysis
20	Sample 11	normal analysis
21	Sample 12	
22	Sample 13	normal analysis
23	Sample 14	normal analysis
24	Sample 15 (sample over calibration range)	normal analysis
25	Sample 15 (Analyzed at a dilution)	dilution analysis; RL is elevated to reflect the dilution
26	Sample 16	normal analysis
27	Sample 17	normal analysis
28	ECCS (rotate level)	80-120%

TABLE 2

INITIAL DEMONSTRATION OF CAPABILITY AND QC REQUIREMENTS
Requirements prior to beginning any analysis batch

Requirement	Specification and Frequency	Acceptance Criteria
Initial Demonstration of Low System Background	Analyze a method blank (LRB) and an LSSM Blank to determine that all target analytes are below 1/3 of the proposed MRL prior to performing the IDC	The LRB and LSSM Blank concentration must be less than 1/3 of the proposed MRL
Initial Demonstration of Accuracy (IDA)	Analyze 7 replicate LFBs fortified with perchlorate at RL. Calculate the mean recovered concentration	The mean recovery must be +/- 20% of the true value
Initial Demonstration of Precision (IDP)	Calculate the percent relative standard deviation (%RSD) of IDA replicates.	%RSD must be less than 20%
Quality Control Sample (QCS)	Initially, upon reestablishing calibration or at least quarterly, analyze a QCS from a second source	QCS must be +/- 20% of true value
Method Detection Limit (MDL) Determination	Select a fortifying level at 0.05µg/L. Analyze 7 replicate LFBs and calculate the MDL	
Minimum Reporting Level (MRL)	An MRL should be established for perchlorate during the IDC	The low CAL standard can be lower than the MRL, but the MRL must be no lower than the low CAL standard.
Matrix Conductivity Maximum (MCM)	Prepare a series of LFB samples, each containing a suggested perchlorate concentration of 0.5µg/L, at sequentially increasing fortified levels of common anions.	MCM, based upon Recovery at RL in fortified matrix.
MRL verification	Verify the MRL in a solution prepared at the MCM	Prepared within +/- 10% of the MCM. Perchlorate recovery must be 50-150% of the MRL
Sample Holding time/ Preservation/ Storage	Perchlorate holding time is 28 days. Samples must be filtered using sterile technique during collection and kept at a maximum of 10 degrees Celsius.	Holding time must not be exceeded, sterilization must be performed during collection.
Initial Calibration	Generate calibration curve. At least 5 calibration standards are recommended, using IS technique.	The lowest level standard should recover 50-150%. All others 80-120%
Test for System Carryover	An LRB is analyzed immediately after the Highest Calibration Standard during the IDC and after each calibration.	The calculated concentration must be less than 1/3 of the MRL.

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TABLE 2 (Continued)

INITIAL DEMONSTRATION OF CAPABILITY QC REQUIREMENTS
Requirements during an analytical batch

Requirement	Specification and Frequency	Acceptance Criteria
Initial Calibration Check (ICV)	With each analysis batch, initially verify calibration at the midpoint of the Calibration curve.	Recovery must be 80-120% of the true value
Continuing Calibration Check (CCCS) and End Calibration Check (ECCS)	Alternately analyze separate mid and high level CCCS/ECCS after every 10 samples and after the last sample in an analysis batch.	Recoveries must fall between 80-120%
Laboratory Reagent Blank (LRB)	Include LRB with every analysis batch (up to 20 samples) Analyze prior to analyzing field samples.	Perchlorate must be less than 1/3 of the MRL
RL verification	Daily, at the beginning of analysis.	Recovery must be 50-150%
Laboratory Fortified Blank (LFB)	Daily, or with each Analysis batch of up to 20 field samples.	Recovery for the LFB must be 80-120% (or 50-150%when fortified at the MRL)
MRL Confirmation	Calculate the Mean and Half Range (HR). Confirm the Upper PIR and Lower PIR meet recovery criteria.	Upper PIR <150% Lower PIR >50%
Laboratory Fortified Sample Matrix (LFM)	Must add known amount of perchlorate to a minimum of 5% of field samples or at least one within each analysis batch. LFM should be fortified near or above the native.	Recovery must be 80-120% for Liquid samples. (Recovery of 50-150% when Perchlorate level is less than 2xMRL.) Recovery must be 70-130% for Soil samples. If fortified sample fails the recovery criteria, label both as suspect matrix.
Field or Laboratory Duplicates or LFM Duplicate	Analyze either a field, laboratory, or LFM duplicate for a minimum of 5% of field samples or at least one within each analysis batch.	RPD must be +/-20% (+/-50%when Perchlorate is less than 2x MRL) And +/- 30% for an LFM duplicate.

Standard Operating Procedure:

Validation of Volatile Organics in Air Samples by Methods TO-3, TO-15, and TO-17

1.0 SCOPE AND APPLICATION

Data Validation determines the technical usability of analytical data. It is a process by which a body of data is assessed for validity and defensibility, compared to a set of acceptance criteria, and flagged with qualifiers when acceptance criteria are not met. A report is prepared that documents the degree of compliance to the intended purpose that is unbiased and technically sound.

2.0 SUMMARY

This SOP details the process of evaluating and verifying Volatile Organics in air (Methods TO-3, TO-15, and TO-17), comparing to it stated Data Quality Objectives (DQOs) and acceptance criteria and preparing a technically sound validation report that provides the data user with a reliable basis for decision making. Extensive laboratory knowledge, knowledge of published methods, and the ability to apply consistent professional judgment are essential. This SOP should be used in conjunction with ddms SOP ECS-SOP-012-Rev.0– General DV: 'Validation and Review of Analytical Data', the site-specific QAPPs and SAPs, regional data review guidelines, and analytical methods. This SOP provides ddms' professional judgment.

All data have the potential to go to litigation. Part of the validation process is to identify any data and/or documentation issues that could be problematic if the data are used in litigation. All documents are discoverable during litigation, including data validation/review reports. Any issues identified during the data review process must be clearly documented in the report.

When only a reduced review of the data is requested, the procedure outlined in this SOP is followed, but calculations are not performed. Only a review of the laboratory summary forms is performed unless that review reveals major quality issues. If at any time a situation arises that is not addressed in this SOP, contact with the other ddms chemists is required. Where PJ is referenced in the guidelines and no PJ in this SOP covers the situation, communication with other ddms chemists is required. Always document in the report when and why ddms PJ is applied.

When verifying calculations, not all regions specify an acceptable %D between laboratory-reported and validator-calculated values. It is ddms' PJ to contact the laboratory when discrepancies greater than 10% occur. The error term of 10% is considered to be allowable experimental error in general laboratory practice.

Each of the analytical methods has method-specific sample collection, preparation, and analysis elements. Because many of these elements are not well-defined in the method, validation must rely on professional judgment in assessing many of the quality elements. Discussion with other ECS chemists is essential. Decisions and data or guidance relied upon to make decisions must be clearly documented in the validation report with supporting data as necessary and appropriate. Documentation of the decisions in the project file may be required.

ACRONYMS

COC – Chain of Custody
DQO – Data Quality Objective
QAPP – Quality Assurance Project Plan
GC/MS – Gas Chromatography/Mass Spectrometry
LIMS – Laboratory Information Management System
QA – Quality Assurance
QC – Quality Control
SAP – Sampling and Analysis Plan
SOP – Standard Operating Procedure
VOC – Volatile Organic Compound
TCL – Target Compound List
RL – Reporting Limit
QL – Quantitation Limit
MDL – Method Detection Limit
QL – Quantitation Limit
RPD – Relative Percent Difference
IC – Initial Calibration
ICV – Initial Calibration Verification
CC – Continuing Calibration
BFB – Bromofluorobenzene
RRF – Relative Response Factor
%D – Percent Difference
LCS – Laboratory Control Sample
RRT – Relative Retention Time
RT – Retention Time
RSD – Relative Standard Deviation
TIC – Tentatively Identified Compound
PJ – Professional Judgment
BAL – Blank Action Level
ISTD – Internal Standard

3.0 PROCEDURE

3.1 Identify Project Specific Requirements

3.1.1 Refer to SOP ECS-SOP-012-Rev.0– General DV: 'Validation and Review of Analytical Data'.

3.1.2 Contact the client for approval to contact the laboratory directly should any questions arise during validation process.

3.1.3 Project Specific Documents: before beginning the validation process, it is important to identify and understand the project specific documents. These documents may include, but are not limited to:

- QAPPs
- SAPs
- Laboratory SOPs
- Published analytical methods
- Field notes

QAPPs and SAPs will include pertinent information such as required sample collection techniques, required parameters and compound lists, and required quantitation limits. Laboratory SOPs can be used to determine whether laboratory procedures comply with project requirements as well as the method used to analyze the samples. The extent to which these documents apply or exist will vary from project to project.

The requirements of the QAPPs and SAPs take precedence during the data review process. The criteria set forth in the applicable EPA or other agency data review guidelines may then be applied. The analytical method and agency guidelines are consulted; however, ddms professional judgment is used when determining the need for data qualification. When a data quality situation occurs that has not been addressed in this SOP, it warrants discussion with the other ddms chemists for a uniform decision. Professional judgment should always be applied as necessary and appropriate.

NOTE: Any incomplete, missing, or unclear information discovered during the validation process warrants contacting the laboratory for clarification and/or correction. If calculations do not agree within 10% of the reported value, resolution with the laboratory is warranted. This must be documented in the validation report along with the laboratory's corrective action. If corrective action or resolution was not required or not possible, this must also be documented in the validation report.

3.1.4 Chain of Custody/Sample Integrity

3.1.4.1 Qualify sample results as estimated (J, UJ) for samples analyzed outside the 30 day hold time but less than 60 days. Reject (R) non-detect and estimate (J) detected results for samples analyzed greater than 60 days from date of collection.

3.1.5 Canister certification information (TO-15)

3.1.5.1 Verify the canister blank(s) analyzed after the canister cleaning does not contain any target analytes.

3.1.5.1.1 If any target analyte is detected and the sample concentration is >5x the blank concentration, no qualification of sample results is made.

3.1.5.1.2 If any target analyte is detected and the sample concentration is ≤ 5x the blank concentration, qualify the sample as not-detected (U) at the reported value or RL, whichever is greater.

3.1.6 Residual Vacuum Evaluation

3.1.6.1 Verify there is residual vacuum in the canister upon receipt at the laboratory.

3.1.6.1.1 If no residual vacuum was present a leak is suspected. Estimate (J, UJ) sample results and document in the validation report.

3.2 Review, Verification, and Evaluation of Analytical Results

3.2.1 GC/MS Instrument Performance Check (BFB tuning standard – for GC/MS methods TO-15 and -17)

3.2.1.1 All results for samples analyzed under a BFB that does not meet acceptance criteria are rejected (R). If samples are analyzed outside of the analysis clock, the validator should request all tunes and CC standards for the next analytical sequence on that instrument and assume no systemic changes have been made to the instrument between analytical sequences. If the tune and CC standards are acceptable, no qualification is necessary, but rationale must be documented in the report. If the tunes or CC standards exceed acceptance criteria, discussion with other ddms chemists is necessary for resolution.

3.2.1.2 Proper instrument performance must be determined through review of the reference standards data for all methods. Good chromatography and resolution must be demonstrated. Professional judgment is used and discussion with the ECS team is recommended when questions arise.

3.2.2 Initial Calibration

3.2.2.1 Although these methods have no minimum RF (or CF) requirements, use PJ to determine if the instrument sensitivity is adequate to ensure target compounds will be detected. Consultation with other ddms chemists is necessary to determine any action necessary.

3.2.2.2 If the %RSD in the IC is >30% but less than 50%, qualify sample results as estimated (J, UJ). If the %RSD is >50% consultation with ddms chemists is necessary to determine course of action.

3.2.2.3 If the %RSD is greater than 30% in the IC, determine the cause of non-linearity by eliminating either the high or low point of the curve and recalculating the %RSD. If the %RSD is less than 30% after eliminating the high point, accept positive detects in the linear portion of the curve, estimate (J) positive detects at the high, non-linear end of the curve, and accept non-detects. If the %RSD is less than 30% after eliminating the low point on the curve, accept positive detects in the linear portion of the curve, estimate (J) positive detects in the low, non-linear portion of the curve and estimate (UJ) non-detects.

3.2.2.4 GC/MS methods TO-15 and TO-17 allow only use of average RF for establishing calibration. If the laboratory uses an alternate calibration model, qualify the data as estimated (J, UJ).

3.2.2.5 For GC method TO-3, the validator must use professional judgment to determine whether or not the calibration established is valid and unbiased. Discussion with other ECS chemists may be necessary.

NOTE: Action warranted based on IC issues affect all sample results calculated using the IC. Qualifiers applied based on unacceptable calibration are applied to all samples calculated on the IC.

3.2.2.6 A second-source initial calibration verification (ICV) should be analyzed following the IC and prior to samples. The objective of the ICV is to verify the concentration of the primary standards used to establish the calibration. If the percent difference of any target exceeds 30%, affected results must be qualified as estimated (J, UJ). In case of extreme variance, it may be necessary to reject (R) results.

3.2.2.7 If a second source ICV has not been analyzed, other supporting data may be available from the laboratory to support the calibration accuracy. This would require contact with the laboratory and the use of PJ for a determination.

NOTE: Action warranted based on ICV issues affects all sample results calculated using the IC. Qualifiers applied based on unacceptable calibration are applied to all samples calculated on the IC.

3.2.3 Continuing Calibration

3.2.3.1 If the percent difference is >30%, estimate (J) detects. If there is an increase in sensitivity, non-detects are not qualified. If there is a decrease in sensitivity (30-50%), estimate non-detects (UJ).

3.2.3.2 If any target compound has a %D greater than 50% exhibiting a decrease in sensitivity, qualify detects as estimated (J) and non-detects as rejected (R).

3.2.4 Blanks

3.2.4.1 Method Blank

3.2.4.1.1 If any target analytes were detected in the laboratory method or clean canister blank and the associated samples are not-detected, no qualifications will be made.

3.2.4.1.2 If any target analytes were detected in either the laboratory method or clean canister blank, the blank action level (BAL) is equal to the concentration of the contamination in the blank for all compounds excluding common lab contaminants (methylene chloride, acetone and 2-butanone(MEK). The BAL for common lab contaminants is determined by multiplying the highest contamination concentration by two.

3.2.4.1.3 If the concentration of the sample result is less than the BAL, then the analyte is qualified as not-detected (U), at the sample quantitation limit (QL). If the concentration of the sample is $\geq$ the BAL, professional judgement should be applied.

3.2.4.1.4 If the concentration of the sample result for an analyte is greater than or equal to the QL, but less than the BAL, raise the QL to the sample concentration and qualify the result not-detected (U).

3.2.4.1.5 If the concentration of the sample result for an analyte is greater than or equal to the QL and > Blank result or BAL, professional judgement should be applied.

3.2.5 Laboratory Control Sample (LCS)

3.2.5.1 Verify that the LCS recoveries for all analytes are within 70-130%.

3.2.5.1.1 If the recovery of an analyte in either the LCS or LCSD is ≥130%, estimate (J) detects for that compound in the samples associated with that LCS, indicating potential high bias. Non-detects are not qualified.

3.2.5.1.2 If the recovery of an analyte in either the LCS or LCSD is less than 70% but ≥10%, estimate detects (J) and all non-detects (UJ) in all samples associated with the LCS, indicating potential low bias.

3.2.5.1.3 If the recovery of an analyte in either the LCS or LCSD is less than 10%, estimate (J) detects and reject (R) all non-detects in samples associated with that LCS.

3.2.5.1.4 If more than 30% of the analytes are outside QC limits, contact ddms chemists for discussion and resolution.

3.2.5.2 When the laboratory has analyzed an LCS/LCSD pair, the RPD between the paired results should be less than 15%. Qualify results as estimated (J) for all samples in the associated analytical batch when the RPD exceeds 15%. When the RPD exceeds 30%, discussion with other ddms chemists is necessary for qualification of data.

3.2.6 Surrogates

3.2.6.1 ddms acceptance criteria are 70-130 %R for aqueous and non-aqueous samples.

3.2.6.2 If the recovery of any one surrogate is less that 70% but greater than or equal to 10%, estimate (J) all detects and non-detects (UJ) in that sample.

3.2.6.3 If the recovery of any one surrogate is less than 10%, estimate (J) all detects and reject (R) all non-detects in that sample.

3.2.6.4 If the recovery is >130% estimate (J) all detects in that sample.

3.2.7 Internal Standards (ISTD) for Methods TO-15 and TO-17

3.2.7.1 If any sample ISTD area count is > 140% when compared against the most current continuing calibration standard, estimate (J) all positive detects for compounds quantitated using that ISTD. Accept all non-detects. Professional judgement (PJ) may be applied when the validator feels necessary.

3.2.7.2 If any ISTD area count is < 40% but > 10%, ddms professional judgment is to estimate (J) all positive detects and estimate (UJ) all non-detects for compounds quantitated using that ISTD. Professional judgement may be applied when the validator feels necessary.

3.2.7.3 If the area of ISTD is less than 10% ddms professional judgment is to reject (R) non-detects.

3.2.7.4 If the RT of any ISTD is outside the acceptance limit (± 20 seconds), closely examine the affected sample chromatogram to determine the potential for false positives or false negatives.

3.2.7.5 If the RT of the ISTD is outside the acceptance limit, consultation with ddms chemist is necessary

3.2.8 Field Duplicates (FDs)

3.2.8.1 The RPD for FDs should be less than 20% for all analytes that are detected at concentrations greater than or equal to 5x the sample QL. If the RPD is greater than 20%, estimate (J) all positive detects and estimate (UJ) all non-detects for that analyte in all samples from the same location and day.

3.2.8.2 The absolute difference for all analytes detected at concentrations less than 5x the sample QL (including where one duplicate sample is not-detected) should be less than or equal to 2x the sample QL. If the absolute difference is greater than 2x the sample QL, estimate (J) all positive detects and estimate (UJ) all non-detects for that analyte in all samples of the same matrix.

3.2.8.3 If any analyte is detected at estimated (J) concentrations less than the sample QL in both field duplicate samples, no additional qualification is performed.

3.2.8.4 If an analyte in a field duplicate pair has a non-detect and a duplicate positive detect that is greater than ten times the sample RL, contact other ddms chemists for qualification of samples in the sample delivery group.

3.2.9 Target Compound Identification

NOTE: Professional judgment is often required to determine if a correct compound identification was made. Often, co-eluting compounds may mask or skew the mass spectrum for an individual compound. If it is determined that the identification is incorrect, contact with the laboratory may be necessary to have the data corrected and resubmitted. If a discrepancy remains, use professional judgment to qualify or reject and document in the validation report.

3.2.9.1 Refer to ddms SOP ECS-012-Rev.0 – General DV: 'Validation and Review of Analytical Data'

3.2.9.2 When a sample contains an on-column concentration that exceeds the highest concentration standard of the calibration curve, careful examination of the sample analyzed immediately following the high concentration sample is warranted. Consult with other ddms chemists to discuss carryover possibility and resolution.

3.2.9.3 GC/MS provide identification and confirmation of target analytes based on responses and ratios of characteristic ions. Where method criteria are not provided, follow MS convention for S/N and ion ratio criteria.

3.2.9.4 For GC methods, analyte identification is dependent upon matching retention times with the reference standards. The laboratory establishes RT windows for search and ID of the target analytes. In the absence of

method criteria, PJ must be used to make determination of accurate identification.

3.2.10 Tentatively Identified Compounds (TICs) – GC/MS methods only

3.2.10.1 Refer to ddms SOP ECS-SOP-012-Rev.0 – General DV: 'Validation and Review of Analytical Data'.

3.2.11 Compound Quantitation

3.2.11.1 Refer to ddms SOP ECS-SOP-012-Rev.0 – General DV: 'Validation and Review of Analytical Data'.

3.2.11.2 Recalculate, from the raw data, the concentration of at least one positive detect in every field sample. The calculated value should agree within 10% of the reported value. Verify conversion from ppbv to ug/m3 is correct.

3.2.12 Reported Quantitation Limits (QLs)

3.2.12.1 Refer to SOP ECS-SOP-012-Rev.0 – General DV: 'Validation and Review of Analytical Data'.

3.2.12.2 Any sample concentration reported below the QL (and above the laboratory determined Method Detection Limit [MDL]) must be qualified as estimated (J) by the laboratory.

3.2.13 Documentation

3.2.13.1 Refer to ddms SOP ECS-SOP-012-Rev.0 – General DV: 'Validation and Review of Analytical Data'.

4.0 Preparation of Reports and Other Deliverables

4.1.1 Refer to ddms SOP ECS-SOP-012-Rev.0 – General DV: 'Validation and Review of Analytical Data'.

Standard Operating Procedure:

Validation and Review of Semivolatile Organic Data

1.0 Scope and Application

Data validation determines the technical usability of analytical data. It is a process by which a body of data is compared to a set of acceptance criteria and flagged with qualifiers when acceptance criteria are not met. A report is prepared that documents the degree of compliance to the intended purpose that is unbiased and technically sound. There may be criteria in this standard operating procedure (SOP) that are more stringent than those outlined in regional guidelines. The focus of validation is always on the technical usability and defensibility of the data.

2.0 Summary

This SOP details the process of evaluating and verifying Semivolatile Organic data, comparing it to stated Data Quality Objectives (DQOs) and acceptance criteria, and preparing a technically sound validation report that provides the data user with a reliable basis for decision making. Extensive laboratory knowledge, knowledge of published methods, and the ability to apply consistent professional judgment are essential. This SOP should be used in conjunction with ddms SOP ECS-SOP-012-Rev.0– General Data Validation: "Validation and Review of Analytical Data," the site-specific quality assurance project plans (QAPPs) and sampling and analysis plans (SAPs), regional data review guidelines, if applicable, and analytical methods. This SOP provides ddms professional judgment. **When 1,4-dioxane is a contaminant of concern, and analyzed by SIM (Select Ion Monitoring), please refer to ECS SOP 004-SIM – Validation and Review of Organic Analyses Using Selective Ion Monitoring (SIM).**

All data have the potential to go to litigation. Part of the validation process is to identify any data and/or documentation issues that could be problematic if the data are used in litigation. All documents are discoverable during litigation, including validation/review reports. Any issues identified during the data review process must be clearly stated in the report.

When only a review of the data is requested, the procedure outlined in this SOP is followed, but calculations are not performed. Only a review of the data summary forms is performed unless that review reveals major quality issues. If at any time a situation arises that is not addressed in

this SOP, contact with the other ddms chemists is required. Where professional judgment (PJ) is referenced in the guidelines and no PJ in this SOP covers the situation, communication with the other ddms chemists is required. Always document in the report when ddms PJ is applied and the rationale for decisions made.

When verifying calculations, not all regions specify an acceptable %D between laboratory-reported and validator-calculated values. It is ddms' PJ to contact the laboratory when discrepancies greater than 10% occur. The error term of 10% is considered to be an allowable experimental error in general laboratory practice.

3.0 Acronyms

%Diff – Percent Difference
%R – Percent Recovery
BAL – Blank Action Level
BN – Base Neutral
CC – Continuing Calibration
COC – Chain of Custody
DFTPP – Decafluorotriphenylphosphine
DQO – Data Quality Objective
DU – Decision Unit
DV – Data ValidationFD – Field Duplicate
GC – Gas Chromatography
GC/MS – Gas Chromatography/Mass Spectroscopy
IC – Initial Calibration
ICV – Initial Calibration Verification
IS – Internal Standard
LCS/LCSD – Laboratory Control Sample/Laboratory Control Sample Duplicate
LIMS – Laboratory Information Management System
MB – Method Blank
MDL – Method Detection Limit
MS/MSD – Matrix Spike/Matrix Spike Duplicate
ND – Not Detected
QA – Quality Assurance
PAH – Polynuclear Aromatic Hydrocarbon
PAL – Project Action Limit
PJ – Professional Judgment
PSC – Project Specific Criteria
QAPP – Quality Assurance Project Plan
QC – Quality Control
QL – Quantitation Limit

RL – Reporting Limit
RPD – Relative Percent Difference
RRF – Relative Response Factor
RRT – Relative Retention Time
RSD – Relative Standard Deviation
RT – Retention Time
SAP – Sampling and Analysis Plan
SOP – Standard Operating Procedure
SS – Surrogate Standard
SVOC – Semivolatile Organic Compound
TCL – Target Compound List
TI – Tentative Identification
TIC – Tentatively Identified Compound
TQL – Target Quantiation Limit

4.0 Procedure

4.1 Identify Project Specific Requirements- Refer to SOP ECS-SOP-012-Rev.0– General DV: "Validation and Review of Analytical Data."

4.2 Sample Preservation and Holding Time

4.2.1 Verify that the temperature of the samples in the cooler(s) upon receipt at the laboratory was documented and was 4 ° C ± 2°. If the temperature was outside the lower range, no qualification is made, but it is documented in the validation report. If the temperature of the cooler was outside the upper range, qualify based on appropriate data validation guidelines. In the absence of regional validation guidelines regarding temperature excursions, apply ddms professional judgment. If the receipt temperature of the samples exceeds the acceptance criteria, but it is noted that the samples were received "on ice", review the documentation to verify that there was insufficient time (nominally 4 hours) from sample collection to laboratory receipt for the samples to have cooled. No qualification of data is necessary, but it is noted in the DV report. If the elapsed time from sample collection to laboratory receipt exceeds 4 hours, estimate (J, UJ) the results.

4.2.2 In the absence of regional validation guidelines regarding holding time excursions, apply ddms professional judgment. Qualify sample results as estimated (J, UJ) for samples prepared and/or analyzed outside the hold time. Reject (R) non-detect sample results and estimate (J) detected results for samples prepared outside of two times the hold time. Reject (R) non-detect sample

results and estimate (J) detected results for samples analyzed more than 60 days after preparation.

4.3 Review, Verification and Evaluation of Analytical Results

4.3.1 Decafluorotriphenylphosphine Performance Check (DFTPP Tune)

Although agency validation guidelines state that all results for samples analyzed under a DFTPP that does not meet acceptance criteria are rejected (R), prior to qualifying data, contact with the laboratory is necessary to determine if an incorrect tune was provided in the data package. If samples are analyzed outside of the analysis clock, the validator should request all tunes and CC standards for the next analytical sequence on that instrument. If the tune and CC standards are acceptable, no qualification is necessary, but it should be documented in the report. If the tunes or CC standards exceed acceptance criteria, discussion with other ddms chemists is necessary for resolution.

Revision 8270E: Tuning requirements for this revision of the SVOCs method specify MS tuning only prior to the IC and allow use of DFTPP or other vendor-recommended tuning compound. DFTPP criteria can be used, or vendor-specified criteria may be used.

4.3.2 Initial Calibration

4.3.2.1 The average RRF for all target analytes in the IC should be greater than or equal to 0.05. If any target analyte has an average RRF less than 0.05, the chemist will review the instrument response for the IS and the target compound and in conjunction with other ddms chemists make a uniform decision on data qualification. NOTE: data qualification will not be based on RRF alone. (If 8270D Rev 5 or later is used for analysis, the Table 4 compounds listed below may have RRFs as low as 0.010.)

Compound	RRF
Benzaldehyde	0.010
2,2—Oxybis-(1-chloropropane)	0.010
Acetophenone	0.010
4-Chloroaniline	0.010
Hexachlorobutadiene	0.010
Caprolactam	0.010
1,1’-Biphenyl	0.010
2-Nitroaniline	0.010
Dimethyl phthalate	0.010
3-Nitroaniline	0.010
2,4-Dinitrophenol	0.010

4-Nitrophenol	0.010
Diethyl phthalate	0.010
1,2,4,5-Tetrachlorobenzene	0.010
4-Nitroaniline	0.010
4,6-Dinitro-2-methylphenol	0.010
N-nitroso-diphenylamine	0.010
Atrazine	0.010
Carbazole	0.010
Di-n-butyl phthalate	0.010
Butyl benzyl phthalate	0.010
3,3'-Dichlorobenzidine	0.010
Bis(2-ethylhexyl)phthalate	0.010
Di-n-octyl phthalate	0.010
2,3,4,6-Tetrachlorophenol	0.010

4.3.2.2 If the %RSD is greater than 20% in the IC, determine the cause of non-linearity. If the curve is acceptable after eliminating the high point of the curve and recalculating %RSD accept positive detects in the linear portion of the curve, estimate (J) positive detects at the high end of the curve and accept non-detects. If the %RSD is less than 20% after eliminating the low point on the curve, accept positive detects in the linear portion of the curve, estimate (J) positive detects in the low portion of the curve. In the absence of regional qualification guidelines, ddms practice will be to raise the RL to the concentration at which linearity was demonstrated.

4.3.2.3 In situations where use of the average RRF was not acceptable, the laboratory may have used an alternate calibration model, such as a linear regression or quadratic equation. Follow the procedure outlined in Section 4.2.2.2. When the outlier is not at the low or high end of the calibration range, contact other ddms chemists for qualification of other samples in the sample delivery group.

4.3.2.4 If a linear regression was used to establish calibration, the low point of the calibration must be re-calculated against the curve and produce a concentration within 30% of the true value. If this criterion is not met, results at the low end of the curve will be qualified as estimated (J).

4.3.2.5 If a quadratic equation was used to establish calibration, verify that six points were used. Determine whether or not the curve will apply a high or low bias and qualify sample results accordingly. [Note: A

quadratic model should only be used as a last resort for any but the poorest performers under the SVOC method]

4.3.3 Initial Calibration Verification (ICV)

4.3.3.1 Verify that the ICV was a second source standard. If a second source verification was not analyzed after the IC, the IC has not been verified as valid and all sample results reported under this IC must be qualified as estimated.

4.3.3.2 ddms ICV acceptance criteria are ± 20%. If the recovery of any compound falls outside of 80-120%, qualify all sample results analyzed under the affected IC. Because the source of the error in concentration (primary standard or secondary source) generally cannot be determined, no direction of bias can be assigned. If recovery is less than 80%, qualify detects (J) and non-detects (UJ). If the recovery is greater than 120%, qualify detects (J) and accept non-detects.

4.3.4 Continuing Calibration

4.3.4.1 Verify that the continuing calibration (CC) standard was analyzed on each instrument used to analyze samples at the beginning of the 12 hour (or 24 hours for 500 and 600 series methods) following the analysis of a tune standard that meets acceptance criteria and before any blanks and QC or field samples were analyzed.

4.3.4.2 Verify that the CC was compared against the most recent IC. If the CC was not compared against most recent IC, contact the laboratory to correct the CC and subsequent sample results.

4.3.4.3 If any target compound has an RRF less than the method defined criterion or less than 0.05 for those with no method criterion, the chemist will review the instrument response for the IS and the target compound and in conjunction with other ddms chemists make a uniform decision on data qualification. NOTE: data qualification will not be based on RRF alone.

EXCEPTION: 1,4-dioxane. See ECS SOP 004-Validation and Review of Organic Analyses Using Selective Ion Monitoring (SIM)

4.3.4.4 When the percent difference (drift) exceeds 20%, qualify detects as estimated (J). If the shift represents an increase in sensitivity, non-detects are not qualified. If the shift represents a decrease in sensitivity, qualify non-detects as estimated (UJ). If any target

compound has a %D greater than 100.0%, exhibiting an increase in sensitivity, consult ddms chemists for further action.

4.3.5 Blanks

4.3.5.1 Be aware that some Regions do not qualify data based on FB results. ddms' SOP is **to** qualify sample results based on FB results. It is not ddms' policy to qualify soil data based on associated FB sample results without consultation with other ddms chemists.

4.3.5.2 If any target analytes were detected in either the laboratory method blank, TCLP/SPLP LEB, EB, or FB, that blank action level (BAL) is equal to the concentration of the contamination in the highest blank. If the concentration of the sample result for an analyte is greater than or equal to the QL, but less than the BAL, raise the QL to the sample concentration and qualify the results as not-detected (U). If the concentration of the sample results for an analyte is greater than or equal to the QL and greater than the blank result/BAL, professional judgment should be applied.

4.3.5.3 Scenario 1: The BAL in the MB is less than the BAL of the FB. The sample result is greater than the MB BAL, but less than the FB BAL. Even though the governing agency specifically states that data not be qualified based on FB results, based on ddms professional judgment, results would be qualified as not detected (U) at the sample-specific RL or reported value, whichever is greater.

4.3.5.4 Scenario 2: Samples are analyzed on two different instruments on the same day. On one of the instruments, a common laboratory contaminant is reported in the MB and on the other instrument the same analyte is ND. The sample results on both instruments were less than the BAL on the instrument with the contaminant. ddms professional judgment is to qualify the results from the analytical runs on both instruments as not detected (U) at the sample specific RL or reported value, whichever is greater.

4.3.5.4.1 Ideally, a method blank should be analyzed on each instrument on which samples from the same preparation batch were analyzed, in order to assess any contamination present in the individual analytical system as well as artifacts from sample preparation.

4.3.6 Matrix Spike/Matrix Spike Duplicates (MS/MSD) - Refer to SOP ECS-SOP-012-Rev.0– General DV: "Validation and Review of Analytical Data."

Note: When applying qualifiers based on MS/MSD recoveries, qualification is made to all samples of the same matrix. Determination of decision units (DUs) or other site-specific associations should be made and documented before project initiation. See the Project Kick-Off Form in Appendix F.

4.3.7 Field Duplicate Samples- Refer to SOP ECS-SOP-012-Rev.0– General DV: "Validation and Review of Analytical Data."

4.3.8 Laboratory Control Sample (LCS)/ Laboratory Control Sample Duplicate (LCSD)

4.3.8.1 Verify an LCS was extracted as appropriate. ddms PJ (70-130%R) is as outlined below:

4.3.8.1.1 If the recovery of an analyte is ≥130%, estimate (J) detects for that compound in the samples associated with that LCS, indicating potential high bias. Accept non-detects without qualification.

4.3.8.1.2 If the recovery of an analyte is less than 70% but ≥10%, estimate (J) detects in all samples associated with the LCS, and estimate (UJ) all non-detects in all samples associated with the LCS, indicating potential low bias.

4.3.8.1.3 If the recovery of analyte is less than 10%, estimate (J) detects in samples associated with that LCS and reject (R) all non-detects in samples associated with that LCS.

4.3.8.1.4 When the laboratory has analyzed an LCS/LCSD pair, the RPD between the paired results should be less than 20%. Qualify results as estimated (J) for all samples in the associated analytical batch when the RPD exceeds 20%. When the RPD exceeds 40%, discussion with other ddms chemists is necessary for qualification of data.

Note: The LCS (and LCSD if applicable) should include all reported target analytes. If any reported analytes have not been included, use PJ and discussion with other ddms chemists to determine the impact on the reported results, which will depend on other information available in the

data package to support that laboratory's ability to extract and detect the(se) compound(s).

4.3.9 Surrogates

4.3.9.1 ddms' acceptance criteria are 70-130%R for all surrogate compounds.

4.3.9.1.1 For full-target-list SVOCs, If the recovery of any one BN surrogate and/or one acid surrogate is (are) less than 70% but greater than 10%, estimate all detects (J) and non-detects (UJ) in that sample as indicated in the table below:

Surrogate	**Compounds**
Nitrobenzene-d_5	Aniline Benzaldehyde 1,4-Dioxane n-Nitroso-di-n-propylamine Bis (2-chloroethyl) ether Bis(2-chloro-1-methylethyl)ether 2, 2'-Oxybis (1-chloropropane) 1,2-Dichlorobenzene 1,3-Dichlorobenzene 1,4-Dichlorobenzene Acetophenone Hexachloroethane Nitrobenzene Isophorone Bis(2-chloroethoxy)methane Naphthalene 4-Chloroaniline n-Nitrosodimethyl amine Pyridine Hexachlorobenzene 2-Methylnaphthalene 1-Methylnaphthalene 1,2,4-Trichlorobenzene Ethyl methanosulfonate Methyl methanosulfonate 2-Picoline Caprolactam α,α-Dimethylphenethylamine n-Nitrosodi-n-butylamine

Surrogate	Compounds
	n-Nitrosopiperidine
Fluorobiphenyl	Hexachlorocyclopentadiene 1,1'-Biphenyl 4-Aminobiphenyl Atrazine 1-Chloronaphthalene 2-Chloronaphthalene 2-Nitroaniline Diethyl phthalate 2,6-Dinitrotoluene Acenaphthylene 3-Nitroaniline Acenaphthene Dibenzofuran 2,4-Dinitrotoluene Dimethyl phthalate Fluorene 4-Chlorophenylphenyl ether 4-Nitroaniline 1,2,4,5-Tetrachlorbenzene n-Nitrosodiphenylamine 4-Bromophenyl-phenyl ether Hexachlorobenzene Phenanthrene Anthracene Carbazole Di-n-butylphthalate Fluoranthene Pentachloronitro-benzene 1-Naphthylamine 2-Naphthylamine Pentachlorobenzene 4-Nitroquinoline-oxide Phenacetin Pronamide
Terphenyl-d_{14}	Benzidine Pyrene Butyl benzyl phthalate

Surrogate	Compounds
	Chrysene 3,3'-Dichlorobenzidine Benzo(a)anthracene Bis(2-ethylhexyl)phthalate Di-n-octyl phthalate Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene Benzo(e)pyrene Indeno(1,2,3-c,d)pyrene Dibenz(a,h)anthracene Benzo(g,h,i)perylene Dimethyl aminobenzene Dimethylbenz(a)anthracene Dibenz(a,j)acridine 3-Methylcholanthrene Perylene
2-Fluorophenol or Phenol-d_5 (If both compounds are out of acceptance criteria, all acid compounds will be qualified accordingly.)*	Phenol Benzyl alcohol Benzoic acid 2-Chlorophenol 2-Methylphenol 3-/4-Methylphenol 2-Nitrophenol 2,4-Dimethylphenol 2,4-Dinitrophenol* 2,4-Dichlorophenol 2,6-Dichlorophenol 4-Nitrophenol* 4-Chloro-3-methylphenol 2,4,5-Trichlorophenol* 2,4,6-Trichlorophenol*
2,4,6-Tribromophenol*	Pentachlorophenol 4,6-Dinitro-2-methylphenol 2,3,4,6-Tetrachlorophenol*

*Determine which IS is referenced for the surrogate compound and qualify all targets referenced to the same IS accordingly.

4.3.9.1.2 If the recovery of any one surrogate is less than 10%, contact another ddms chemist for resolution. Non-detects associated with surrogate recoveries <10 % may be rejected as unusable. Positive results may be estimated with a potential low bias.

4.3.9.1.3 If two or more surrogates are outside of acceptance criteria for a single fraction, all analytes for that fraction are qualified accordingly.

4.3.9.1.4 If only selected compounds are reported from the SVOC target list (i.e., PAHs, or other subgroups), the number of surrogates with potential impact on the sample results may be different than those mentioned above. PJ and consultation with other ddms chemists will be used to determine how recoveries will apply.

4.3.10 Internal Standards (IS)

4.3.10.1 If the area of the IS- is between 25% and 50% of the IS area of the midpoint standard IC or of the most recent CC standard analyzed, qualify detects and non-detects associated with that ISTD as estimated (J, UJ). If the area of the IS is between 200 % and 250% of the IS area of the most recent calibration standard qualify detects associated with that ISTD as estimated (J) and accept non-detects.

4.3.10.2 If the area of the IS is less than 25% or greater than 250%, contact the ddms chemists for discussion and resolution. Low IS responses may result in estimation of positive results and estimation or rejection of non-detects. Based on PJ, the RLs for the affected target compounds may be raised.

4.3.10.3 IS responses less than 10% will require rejection (R) of associated non-detects and estimation of positive results with the potential for high bias.

4.3.10.4 If the RT of any IS is outside the acceptance limit, closely examine the affected sample chromatogram to determine if target compounds have been identified correctly and whether any false positive or false negatives exist. Contact the ddms chemists for discussion and resolution.

4.3.10.5 The laboratory should re-analyze or re-prep and analyze any samples with unacceptable IS response. If any sample was not re-analyzed due to unacceptable IS- area and/or RT, this must be noted in the validation report and results may require qualification.

4.3.11 Target Compound Identification

NOTE: Professional judgment is often required to determine if a correct compound identification was made. Examination of RTs and spectra compared with the reference standards is required.

If it is determined that the laboratory's identification is incorrect or cannot be confirmed, contact with the laboratory may be necessary to have the data corrected and resubmitted. If a discrepancy remains, use professional judgment to qualify or reject the result(s), and document in validation report.

4.3.11.1 If a blank has not been analyzed following a sample containing an on-column concentration which exceeds the highest concentration standard of the calibration curve, inspect the sample(s) analyzed following the high concentration sample to verify no carryover occurred and the analyte was not reported as a false positive in the following sample(s). ddms procedure is to contact the laboratory for explanation and resolution. If the resolution is not achieved with the laboratory's response, contact other ddms chemists for discussion and decisions on qualification of data.

4.3.12 Tentatively Identified Compounds (TICs)

4.3.12.1 Confirm with the client that an evaluation of the TICs is a requirement of the data validation effort. If the data do not support the tentative identification (TI) provided by the laboratory or in the validator's PJ, the TI is incorrect, revise the identification on the reporting form (Form 1) and document the correction in the validation report.

4.3.12.2 Verify that the TICs reported by the laboratory are not surrogates, ISs or missed target analytes (even if not project-specific targets) If compounds are included in the reference standards analyzed by the laboratory, confirmation is supported and TI can be revised to confirmed ID.

4.3.12.3 Any TICs identified that are common laboratory contaminants, laboratory or field sampling artifacts and are not true sample components should be discussed in the validation report.

4.3.13 Compound Quantitation

4.3.13.1 If the concentration for an analyte was outside the calibration range in the undiluted analysis but was reported as a non-detect in the diluted analysis, evaluate the raw data or refer to historical data, if available, to determine if the non-detect in the re-analysis was a result of over-dilution or if the analyte was a false positive in the initial, undiluted analysis. If the non-detect in the re-analysis was a

result of over-dilution, it is ddms' procedure to accept the result from the initial, undiluted analysis as the reportable concentration and qualify this result as estimated (J), because it was above the calibration range. If the result in the initial, undiluted analysis is determined to be a false positive, reject (R) the result in the initial analysis.

4.3.14 Reported Quantitation/Reporting Limits (QLs or RLs)

4.3.14.1 Verify that sample RLs are supported by the data provided. The RLs must be equal to or greater than the lowest concentration used to establish the calibration. If low points were dropped for any of the target analytes in the IC, verify that the RLs have been adjusted for the samples to take this into account.

4.3.14.2 Any RLs not supported by the initial calibration need to be recalculated, corrected and documented in the validation report for all associated samples.

4.3.14.3 Verify that the RLs meet the project specific required RLs, project action limits (PALs), project specific criteria (PSC) or target quantitation limits (TQLs). Any samples and/or analytes that do not meet the project specific RLs should be documented in the validation report.

4.3.14.4 Correct reported non-detects at the MDL to non-detected at the RL. Because the RLs (or lowest concentration used to establish calibration) are the lowest values supported in the data package, MDLs should not be used to report non-detects. Results reported between the RL and the MDL should be qualified by the laboratory as estimated (J) because their accuracy is not supported by the calibration.

4.3.15 Documentation - Refer to SOP ECS-SOP-012-Rev.0– General DV: "Validation and Review of Analytical Data."

5.0 Preparation of Reports - Refer to SOP ECS-SOP-012-Rev.0– General DV: "Validation and Review of Analytical Data."

Standard Operating Procedure:

Validation and Review of Volatile Organic Data

1.0 SCOPE AND APPLICATION

Data validation determines the technical usability of analytical data. It is a process by which a body of data is compared to a set of acceptance criteria and flagged with qualifiers when acceptance criteria are not met. A report is prepared that documents the degree of compliance to the intended purpose that is unbiased and technically sound. The focus of validation is always on the technical usability of the data rather than method compliance.

2.0 SUMMARY

This SOP details the process of evaluating and verifying Volatile Organic data, comparing it to stated Data Quality Objectives (DQOs) and acceptance criteria and preparing a technically sound validation report that provides the data user with a reliable basis for decision making. Extensive laboratory knowledge, knowledge of published methods, and the ability to apply consistent professional judgment are essential. This SOP should be used in conjunction with ddms SOP ECS-SOP-012-Rev.0– General DV: 'Validation and Review of Analytical Data', the site-specific QAPPs and SAPs, regional data review guidelines, and analytical methods. This SOP provides ddms professional judgment.

All data have the potential to go to litigation. Part of the validation process is to identify any data and/or documentation issues that could be problematic if the data are used in litigation. All documents are discoverable during litigation, including data validation/review reports. Any issues identified during the data review process must be clearly identified in the report.

When only a review of the data is requested, the procedure outlined in this SOP is followed, but calculations are not performed. Only a review of the data summary forms is performed unless that review reveals major quality issues. If at any time a situation arises that is not addressed in this SOP, contact with the other ddms chemists is required. Where PJ is referenced in the guidelines and no PJ in this SOP covers the situation, communication with the other ddms chemists is required. ddms professional judgment must be documented in the report.

When verifying calculations, not all regions specify an acceptable %D between the laboratory's reported results and the validator's calculations. It is ddms' professional judgment to contact the laboratory when discrepancies greater than 10% occur.

3.0 ACRONYMS

COC – Chain of Custody
DQO – Data Quality Objective
QAPP – Quality Assurance Project Plan
GC – Gas Chromatography
GC/MS – Gas Chromatography/Mass Spectrometry
LIMS – Laboratory Information Management System
QA – Quality Assurance
QC – Quality Control
SAP – Sampling and Analysis Plan
SOP – Standard Operating Procedure
VOC – Volatile Organic Compound
TCL – Target Compound List
RL – Reporting Limit
MDL – Method Detection Limit
RL – Reporting Limit
RPD – Relative Percent Difference
IC – Initial Calibration
CC – Continuing Calibration
BFB – Bromofluorobenzene
ISTD – Internal Standard
RRF – Relative Response Factor
%D – Percent Difference
%R – Percent Recovery
RRT – Relative Retention Time
RT – Retention Time
RSD – Relative Standard Deviation
SURR – Surrogate Standard
TIC – Tentatively Identified Compound
BAL – Blank Action Level

4.0 PROCEDURE

4.1 Identify Project Specific Requirements

4.1.1 Refer to SOP ECS-SOP-012-Rev.0– General DV: 'Validation and Review of Analytical Data'.

4.2 Sample Preservation and Holding Times

Verify that the temperature of the samples in the cooler(s) upon receipt at the laboratory was documented and was 4° C ± 2°. If the temperature was below the lower limit, no qualification is made, but it is documented in the validation report. If the temperature of the cooler was >6 °C but <10°, professional judgement is

applied. If the temperature is >10 °C, qualify based on appropriate data validation guidelines, as estimated (J, UJ). Caution must be used when determining directional bias. In the absence of regional validation guidelines regarding temperature excursions, apply ddms professional judgment. If the receipt temperature of the samples exceeds the acceptance criteria, but it is noted that the samples were received "on ice", review the documentation to determine whether there was sufficient time (nominally 4 hours) from sample collection to laboratory receipt for the samples to have cooled. If not, no qualification of data is necessary, but it is noted in the DV report. If the elapsed time from sample collection to laboratory receipt exceeds 4 hours, estimate (J, UJ) the results.

Aqueous samples preserved with HCl to a pH of $\leq$2 for compounds excluding acrolein, acrylonitrile, and 2-chloro vinyl ether have a holding time of 14 days. Aqueous samples should be unpreserved when reported analytes include acrolein, acrylonitrile, and 2-chloro vinyl ether and have a holding time of 7 days.

NOTE: For non-aqueous samples placed in methanol at sample collection, no qualification of data is necessary. Non-aqueous samples collected via encore samples should be lab preserved within 48hrs of collection with methanol for high concentrations and water for low concentrations. Low concentration water preserved samples should be immediately frozen once they have been prepared. Methanol samples should be kept between 2° and 6° C to prevent evaporation of methanol.

Holding Times

In the absence of regional validation guidelines regarding holding time excursions, apply ddms professional judgment. Qualify sample results as estimated (J, UJ) for samples analyzed outside the hold time. Reject (R) non-detect sample results and estimate (J) detected results for samples analyzed outside of two times the hold time.

Estimate (J) reported results for any samples received with air bubbles <6mm. Air bubbles exceeding 6mm should be discussed with other ddms chemists regarding potential for further qualification.

4.3 Review, Verification and Evaluation of Analytical Results

4.3.1 Bromofluorobenzene Performance Check (BFB Tune)

Although agency validation guidelines state that all results for samples analyzed under a BFB that does not meet acceptance criteria are rendered unusable and rejected (R), contact with the laboratory is necessary to determine if the documentation provided is accurate. If an incorrect BFB was provided in the data package, the laboratory must provide the correct information. If samples are

analyzed outside of the analysis clock, the validator should request all tunes and CC standards for the next analytical sequence on that instrument and assume no systemic changes have been made to the instrument between analytical sequences. If the tune and CC standards are acceptable, no qualification is necessary, but rationale for accepting results as reported must be documented in the report. If the tunes or CC standards exceed acceptance criteria, discussion with other ddms chemists is necessary for resolution.

NOTE: For 8260D Tune checks are only required prior to IC. If BFB is used as a surrogate, the surrogate solution may be used for the tune. BFB criteria differs and should be referenced from the method.

4.3.2 Initial Calibration

4.3.2.1 The average RRF for all target analytes in the IC should be greater than or equal to 0.05. If any target analyte has an average RRF less than 0.05, the chemist will review the instrument response for the ISTD and the target compound and in conjunction with other ddms chemists make a uniform decision on data qualification. NOTE: Data qualification will not be based on RRF alone.

EXCEPTION: 1,4-Dioxane by SIM. See ECS SOP 004 – 1,4-dioxane by SIM

4.3.2.2 If the %RSD across the calibration range is greater than 20% in the IC, determine the cause of non-linearity by eliminating either the high or low point of the curve and recalculating %RSD. If the %RSD is less than 20% after eliminating the high point, accept positive detects in the linear portion of the curve, estimate (J) positive detects at the high end of the curve, and accept non-detects. If the %RSD is less than 20% after eliminating the low point on the curve, accept positive detects in the linear portion of the curve, estimate (J) positive detects in the low portion of the curve and estimate (UJ) non-detects.

4.3.2.3 In situations where the instrument response was not linear the laboratory may have used an alternate calibration model, such as a linear regression or quadratic equation. Evaluate the linearity and qualify data as described in SOP ECS-SOP-012-Rev.0– General DV: 'Validation and Review of Analytical Data'.

4.3.3 Initial Calibration Verification (ICV)

4.3.3.1 Verify that the ICV was a second source standard. If a second source verification was not analyzed after the IC, the IC has not been verified

as valid and all sample results reported under this IC must be qualified as estimated.

4.3.3.2 ddms acceptance criteria are ± 20%. If the recovery of any compound falls outside of 80-120%, qualify all sample results analyzed under the affected IC. Because the source of the error in concentration (primary standard or secondary source) generally cannot be determined, no direction of bias can be assigned. If recovery is less than 80%, qualify detects (J) and non-detects (UJ). If the recovery is greater than 120%, qualify detects (J) and accept non-detects.

4.3.4 Continuing Calibration

4.3.4.1 If any target compound has an RRF less than 0.05, evaluate the data as described for the IC.

EXCEPTION: 1,4-dioxane by SIM. See ECS SOP 004 Validation and Review of Organic Analyses Using Selective Ion Monitoring (SIM)

4.3.4.2 When the percent difference (drift) exceeds 20%, qualify detects as estimated (J). If the shift represents an increase in sensitivity, non-detects are not qualified. If the shift represents a decrease in sensitivity, qualify non-detects as estimated (UJ). If any target compound has a %D greater than 100%, exhibiting an increase in sensitivity, consult ddms chemists for further action.

4.3.5 Blanks

4.3.5.1 Be aware that some EPA Regions do not qualify data based on trip blank (TB) and field blank (FB) results. ddms' professional judgment is **to** qualify sample results based on TB and FB results. It is not ddms' policy to qualify soil data based on associated aqueous TB and FB sample results without consultation with other ddms chemists.

4.3.5.2 If any target analytes were detected in either the laboratory method blank, FB, or EB, the blank action level (BAL) is equal to the concentration of the contamination in the blank for all compounds excluding common lab contaminants (methylene chloride, acetone and 2-butanone(MEK). The BAL for common lab contaminants is determined by multiplying the highest contamination concentration by two.

4.3.5.3 If the concentration of the sample result is less than the BAL then the analyte is qualified as not-detected (U), at the sample quanititation

limit (QL). If the concentration of the sample is ≥ the BAL, professional judgment should be applied.

4.3.5.4 If the concentration of the sample result for an analytes is greater than or equal to the QL, but less than the BAL, raise the QL to the sample concentration and qualify the result (non-detected (U).

4.3.5.5 If the concentration of the sample result for an analyte is greater than or equal to the QL and > BAL results or BAL, professional judgment should be applied.

4.3.5.6 Scenario: The Blank Action Level (BAL) in the method blank (MB) is less than that of the TB and/or FB BAL. The sample result is greater than the MB BAL, but less than the TB and/or FB BAL. Even though the governing agency specifically states that data not be qualified based on TB and/or FB results, based on ddms professional judgment, results would be qualified as not detected (U) at the sample-specific RL or reported value, whichever is greater.

4.3.5.7 If a target compound is detected in more than one type of blank (e.g., MB, TB, FB, RB, etc.) the BAL is calculated from the highest concentration blank.

4.3.5.7.1 Only sample results are qualified based on blank contamination. FB, TB, EB, etc. are not qualified but a statement should be included in the data validation report clarifying lab or field contamination.

4.3.5.8 A method blank must be analyzed once during every 12 hour period for method 8260 or 24 hour for methods 524.2 and 624. However, if the 12-hour or 24-hour clock has not been met, then the blank from the start of the next analytical sequence is applied to all samples in that batch and assume no systemic changes have been made to the instrument between analytical sequences. Discussion with other ddms chemists may be necessary to qualify sample results when on-column concentrations of common laboratory contaminants are consistent from sample to sample but not present in the MB.

4.3.5.9 Instrument Blanks – Ideally, these should be analyzed following a sample that contained high concentrations of a particular analyte(s) and will identify if instrument contamination is present. Calculate a BAL for any analyte detected in an instrument blank and qualify data in the following samples using standard blank guidelines. Use professional judgment to qualify sample results when on-column

concentrations for the preceding samples exceeded the highest standard of the calibration curve and no instrument blank followed the sample analysis.

4.3.6 Matrix Spike/Matrix Spike Duplicates (MS/MSD)

4.3.6.1 Some regions specify qualification only if both the MS and MSD fall outside of criteria. It is ddms' professional judgment to qualify data if either the MS or MSD fail to meet acceptance criteria.

4.3.6.2 Document in the report if a blank or non-site specific sample was used for the MS/MSD analyses. No data are qualified when a non-site specific sample is used for the MS/MSD analyses.

4.3.6.3 The recovery of the MS/MSD analytes should be within ddms' professional judgment acceptance criteria of **70-130%**. Data are qualified when the %Rs fall outside of these limits to alert the data user of potential basis.

4.3.6.3.1 If the recovery of any analyte is less than 70% but greater than or equal to 10% recovery, estimate (J) all positive detects and estimate (UJ) all non-detects, indicating potential low bias of sample results.

4.3.6.3.2 If the recovery of any analyte is less than 10%, estimate (J) all positive detects and reject (R) all non-detects.

4.3.6.3.3 If the recovery of any analyte is greater than 130% recovery, estimate (J) all positive detects, indicating potential high bias of sample results. Non-detect results do not require qualification.

4.3.6.3.4 If the RPD is outside the QC acceptance limit, (30% RPD for waters and 50% RPD for soils), estimate (J) positive detects and estimate (UJ) non-detects.

NOTE: When applying qualifiers based on MS/MSD recoveries, qualification is made to all samples of the same matrix except discrete sampling locations or types (i.e., different buildings, different soil types).

4.3.7 Field Duplicate Samples

4.3.7.1 Refer to SOP ECS-SOP-012-Rev.0– General DV: 'Validation and Review of Analytical Data'.

4.3.8 Laboratory Control Sample (LCS) or Laboratory Control Sample Duplicate (LCSD)

4.3.8.1 Verify that an LCS was analyzed every 12 hours for method 8260 (or 24 hours for methods 524.2 and 624) and for every 20 samples of the

same matrix on the same instrument. However, if the 12-hour or 24-hour clock has not been met, then the LCS from the start of the next analytical sequence assuming no systemic changes have been made to the instrument between analytical sequences is applied to all samples in that batch. ddms applies qualifiers based on acceptance limits of 70 to 130%R:

4.3.8.1.1 If the recovery of an analyte in either the LCS or LCSD is >130%, estimate (J) detects for that compound in the samples associated with that LCS, indicating potential high bias.

4.3.8.1.2 If the recovery of an analyte in either the LCS or LCSD is less than 70% but ≥10%, estimate (J) detects, and estimate (UJ) all non-detects in all samples associated with the LCS, indicating potential low bias.

4.3.8.1.3 If the recovery of an analyte in either the LCS or LCSD is less than 10%, estimate (J) detects and reject (R) all non-detects in samples associated with that LCS.

4.3.8.1.4 If more than 30% of the analytes are outside QC limits, contact ddms chemists for discussion and resolution.

4.3.8.2 When the laboratory has analyzed an LCS/LCSD pair, the RPD between the paired results should be less than 20%, regardless of matrix. Results for all samples in the associated analytical batch when the RPD exceeds 20% will be qualified as estimated (J, UJ). When the RPD exceeds 40%, discussion with other ddms chemists is necessary for qualification of data.

4.3.9 Surrogates

4.3.9.1 ddms acceptance criteria are 70-130% R for aqueous and non-aqueous samples.

4.3.9.2 If the recovery of any one surrogate is >130%, estimate (J) all detects in that sample.

4.3.9.3 If the recovery of any one surrogate is less than 70% but greater than or equal to 10%, estimate (J) all detects and non-detects (UJ) in that sample.

4.3.9.4 If the recovery of any one surrogate is less than 10%, estimate (J) all detects and reject (R) all non-detects in that sample.

4.3.10 Internal Standards (ISTD)

4.3.10.1 If the area of an ISTD is between 25% and 50% of the ISTD area of the midpoint standard or of the most recent CC standard, qualify detects and non-detects associated with that ISTD as estimated (J, UJ). If the area of ISTD is >200% of the ISTD area of the most recent calibration standard qualify detects associated with that ISTD as estimated (J) and accept non-detects.

4.3.10.2 If the area of ISTD is less than 25% or greater than 250%, contact the ddms chemists for discussion and resolution. Low IS responses may result in estimation of positive results and estimation or rejection of non-detects. Based on PJ, the RLs for the affected target compounds may be raised.

4.3.10.3 IS responses less than 10% will require rejection (R) of associated non-detects and estimation of positive results with the potential for high bias.

4.3.10.4 If the RT of any ISTD is outside the acceptance limit, closely examine the affected sample chromatogram in comparison to the opening CCV or mid-point standard for target analytes to determine if any false positive or false negatives exist and contact the ddms chemists for discussion and resolution.

4.3.10.5 If any sample was not reanalyzed due to failing ISTD area and/or RT outside acceptance criteria, this must be noted in the validation report.

4.3.11 Target Compound Identification

NOTE: Professional judgment is often required to determine if a correct compound identification was made based on comparison of the mass spectrum and relative retention time (RRT) of the analyte to that of the opening CCV or the mid-point of the calibration curve.

If it is determined that the identification is incorrect, contact with the laboratory may be necessary to have the data corrected and resubmitted. If a discrepancy remains, use professional judgment to qualify as presumptively present (NJ) or reject (R) and document in the validation report.

A blank should be analyzed following a sample containing an on-column concentration which exceeds the highest concentration standard of the

calibration curve. If a blank was not analyzed, inspect the sample(s) analyzed following the high- concentration sample to verify no carryover occurred and was not incorrectly reported in the following sample(s). ddms' professional judgment is to contact the laboratory for explanation and resolution. If the validator's opinion differs from the laboratory's response, contact other ddms chemists for verification of opinion and qualification of data.

4.3.12 Tentatively Identified Compounds (TICs)

Confirm with the client whether or not an evaluation of the TICs is a requirement of the data validation effort before proceeding with this portion of the validation effort. If the validator disagrees with the tentative identification provided by the laboratory, revise the identification on the Form 1 and document the correction in the validation report.

Verify that the TICs reported are not surrogates, ISTDs or missed target analytes.

Any TICs identified that are common laboratory contaminants or artifacts and are not true sample components should be discussed in the validation report.

4.3.13 Compound Quantitation

Recalculate, from the raw data, the concentration of at least one positive detect in every field sample. The calculated value should agree within 10% of the reported value. If the validator calculated concentration differs by more than 10%, contact the laboratory for clarification. If there is no resolution, contact other ddms chemists for resolution.

If the concentration for an analyte was above the calibration range in the undiluted analysis, but was reported as a non-detect in the diluted analysis, evaluate the raw data and/or refer to historical data, if available, to determine if the non-detect in the reanalysis was a result of over-dilution, or if the analyte was a false positive in the initial, undiluted analysis due to carryover from a previous sample. If the non-detect in the reanalysis was a result of over dilution, it is ddms' professional judgment to accept the result from the initial, undiluted analysis as the reportable concentration and qualify the result as estimated (J) because it was detected outside of the calibration range. If the result in the initial, undiluted analysis is determined to be a false positive, reject (R) the result in the initial analysis.

4.3.14 Reporting Limits (RLs)

4.3.14.1 Verify that the sample RLs meet the project specific required RLs. Any samples that do not meet the project specific RLs should be documented in the validation report. Any RLs not supported by the initial calibration need to be recalculated and corrected for all associated samples. Correct reported non-detects at the MDL to non-detected at the RL. Because the RLs (or lowest concentration used to establish calibration) are the lowest values supported in the data package, MDLs should not be used to report non-detects. Results reported between the RL and the MDL should be qualified by the laboratory as estimated (J) because their accuracy is not supported by the calibration.

4.3.15 Documentation

If documentation issues arose during the validation process, discuss the action taken and the impact to the reported results, addressing technical usability and potential issues if the data are used for litigation. The data user should be instructed to have the missing information inserted into all copies of the data package to ensure complete and accurate information is available for future distributions of the data.

5.0 PREPARATION OF REPORTS

5.1 Refer to SOP ECS-SOP-012-Rev.0– General DV: 'Validation and Review of Analytical Data'.

Standard Operating Procedure:

Validation and Review of Metals Data

1.0 Scope and Application

Data Validation determines the technical usability of analytical data. It is a process by which a body of data is compared to a set of acceptance criteria and flagged with qualifiers when acceptance criteria are not met. A report is prepared that documents the degree of compliance to the intended purpose that is unbiased and technically sound. The focus of validation is always on the technical usability of the data rather than method compliance.

2.0 Summary

This SOP details the process of evaluating and verifying Metals data, comparing it to stated Data Quality Objectives (DQOs) and acceptance criteria and preparing a technically sound validation report that provides the data user with a reliable basis for decision making. Extensive laboratory knowledge, knowledge of published methods, and the ability to apply consistent professional judgment are essential. This SOP should be used in conjunction with ddms SOP ECS-SOP-012-Rev.0– General DV: 'Validation and Review of Analytical Data', the site-specific QAPPs and SAPs, regional data review guidelines, and analytical methods. This SOP provides ddms' professional judgment.

All data have the potential to go to litigation. Part of the validation process is to identify any data and/or documentation issues that could be problematic if the data are used in litigation. All documents are discoverable during litigation, including validation/review reports. Any issues identified during the data review process must be clearly identified in the report.

When only a review of the data is requested, the procedure outlined in this SOP is followed, but calculations are not performed. Only a review of the data summary forms is performed unless that review reveals major quality issues. If at any time a situation arises that is not addressed in this SOP, contact with the other ddms chemists is required. Where professional judgement (PJ) is referenced in the guidelines and no PJ in this SOP covers the situation, communication with the other ddms chemists is required. Always document in the report when ddms PJ is applied.

When verifying calculations, not all regions specify an acceptable %D. It is ddms' PJ to contact the laboratory when discrepancies greater than 10% occur.

3.0 Acronyms

ICP-AES – Inductively Coupled Plasma-Atomic Emission Spectrometry
ICPMS - Inductively Coupled Plasma Mass Spectrometry
ICS - Interference Check Sample
SD – Serial Dilution
PS – Post Spike
ISTD – Internal Standard
%D – Percent Difference
RSD – Relative Standard Deviation
BAL - Blank Action Level

4.0 Procedure

4.1 Identify Project Specific Requirements

4.1.1 Refer to SOP ECS-SOP-012-Rev.0– General DV: 'Validation and Review of Analytical Data'.

4.2 Holding Time

Samples to be analyzed for dissolved metals must be filtered and preserved in the field as soon as possible after collection. Physical and chemical changes can occur in the unpreserved samples after collection, due to temperature, redox and pH changes. In the event that samples are filtered after receipt at the laboratory, results for dissolved metals are qualified as estimated (J, UJ).

4.3 Review, Verification and Evaluation of Analytical Results

4.3.1 Tune (ICP-MS only)

Although agency validation guidelines state that all results for samples analyzed under a sequence where a tune was not performed, are rejected (R), prior to qualifying data, contact with the laboratory is necessary to determine if the tune was erroneously omitted from the data package. If samples are analyzed outside of the analysis clock, the validator should request all tunes for the next analytical sequence on that instrument. If the tune and CC standards are acceptable, no qualification is necessary, but rationale must be documented in the report. If the tunes or CC standards exceed acceptance criteria, discussion with other ddms chemists is necessary for resolution.

The tune solution should be analyzed at least 5 times consecutively, with a %RSD of <5%. If the %RSD is >5%, samples should be qualified as estimated (J, UJ). If the tune is

not analyzed at least 5 times, discussion with other ddms chemists is necessary for resolution.

The resolution of mass calibration should be within 0.1 amu. If the resolution is not with 0.1 amu, associated analytes should be qualified in the samples as estimated (J,UJ).

4.3.2 Initial Calibration

If any correlation coefficient is less than 0.995, estimate (J) all detects and estimate (UJ) all non-detects in all samples associated with that IC.

4.3.3 Initial and Continuing Calibration Verification Standards (ICV, CCV)

For ICP(6010) and ICPMS(6020), if the recovery is excessively high (>125%) indicating the potential for high bias and false positives or low (<75%) indicating potential for low bias and false negatives, or more than 10% of the analytes are outside acceptance limits, use professional judgment and estimate detects when recoveries >125% and estimate detects (J) and reject (R) non-detects when recoveries <75% with the associated ICV or CCV. This may indicate instrument instability. Take into consideration the magnitude of the results compared to the QL to determine the significance of the potential bias.

For Mercury (7470/7471), if the recovery is excessively high (>130%) indicating the potential for high bias and false positives or excessively low (<70%) indicating potential for low bias or false negatives, use professional judgement to estimate detects when recoveries >130% (J) and reject (R) non-detects with recoveries <70% with the associated ICV or CCV. This may indicate instrument instability. Take into consideration the magnitude of the results compared to the QL to determine the significance of the potential bias.

4.3.4 Quantitation Limit Check Standard.

Verify a QL check standard was analyzed prior to sample analysis. For method 6020, a lower limit of quantitation check sample (LLQC) and mid-level QL standard must be analyzed. NOTE: Method 6010 and Method 200.8 do not require the analysis of a LLQC.

If a QL check standard was not analyzed, use professional judgment to assess analytical sensitivity and qualify sample results as warranted. If the recovery of either standard, in the case of 6020 analysis, is excessively low (<50%), there is the potential for low bias and

false negatives, so use professional judgment to estimate (J) detects less than 10x the QL and reject (R) non-detects.

If the recovery is between 50% and 90% qualify results less than 10x the QL as estimated (J, UJ). If the recovery is >150%, there is a potential for high bias and/or false positives at low concentrations, so qualify positive results less than 10x the QL as estimated.

4.3.5 When using professional judgement, consider also any parallel support for analyte recovery that may be present in the data package, i.e., laboratory fortified blanks or LCSs that may lend support to the laboratory's ability to recovery the element during the analytical process. If possible, determine the source of the QL check standard (same source as IC or different).

4.3.6 Blanks

If a contaminant(s) is(/are) found, determine the Blank Action Level (BAL) for each analyte present by multiplying the concentration of the analyte by 5 to determine the BAL.

More than one BAL may exist for a given analyte, therefore, the ddms professional judgment is to use the highest BAL for qualification. Be aware that some agencies qualify data based on the blanks that bracket the analytical run, however ddms' professional judgment should be applied.

If the concentration in the associated sample is > the BAL but < the sample QL, no further qualification is performed, as the sample result should have been reported as estimated by the laboratory. If the result has not been reported by laboratory as estimated, qualify the result, define the reason for qualification as less than the QL and not due to blank contamination.

4.3.6.1 Review the ICB and CCB raw data and determine if a value ≤ the negative MDL for any target analyte is present. The Blank Action Level (BAL) is the absolute value of 5-times then lowest negative value reported in all the blanks.

4.3.6.2 If the concentration in the associated sample is > the negative BAL and ≥ the sample QL, no qualification of sample results is performed.

4.3.6.3 If the concentration in the associated sample is > the negative BAL but < the sample QL, even though the sample result should have been reported as

estimated by the laboratory, estimate the sample result (J, UJ) as the negative blank result adds another level of uncertainty to the reported result.

4.3.6.4 If the concentration in the associated sample is ≤ the negative BAL but is ≥ the sample QL, estimate (J) sample results and note the potential low bias.

4.3.6.5 If the concentration in the associated sample is ≤ the negative BAL and is < the sample QL, no further qualification is performed, as the sample result should have been reported as estimated by the laboratory.

4.3.6.6 Estimate (UJ) non-detects in all samples associated with the negative blank to indicate potential low bias.

4.3.6.7 For samples with associated positive and negative blank values and positive sample results, qualify data as follows:

-if the sample concentration is greater than the positive and negative BAL, no qualification is needed.

-if the sample concentration is less than either the positive or negative BAL, qualify the sample result as not detected (U) at the RL or the reported value, whichever is greater, and estimated (J).

4.3.7 Interference Check Samples (ICSA and ICSAB)

4.3.7.1 If the recovery of any analyte is excessively low (<50%), use professional judgment and estimate (J) detects and reject (R) non-detects for that analyte in all samples associated with the ICSAB.

4.3.7.2 If the recovery of any analyte is excessively high (>150%), use professional judgment and estimate (J) detects for that analyte in all samples associated with the ICSAB.

4.3.7.3 Using professional judgment, any analyte that does not meet acceptance criteria in the ICSA but meets ICSAB criteria to qualify sample results as estimated (J).

4.3.8 Matrix Spike/Matrix Spike Duplicates (MS/MSD)

It is ddms' professional judgement to qualify data as summarized below:

Methods 6010/6020/7470 only

Criteria	Action	
	Detect	Non-detect
Matrix Spike %R < 10% regardless of Post-digestion spike %R	J-	R
Matrix Spike %R < 75%, but ≥ 10% Post-digestion spike %R < 75%, but ≥ 10%	J-	UJ
Matrix Spike %R < 75%, but ≥ 10% Post-digestion spike %R ≥ 75%	J	UJ
Matrix Spike %R > 125% Post-digestion spike %R > 125%	J+	No qualification
Matrix Spike %R > 125% Post-digestion spike %R ≤ 125%, but ≥ 75%	J	No qualification
Matrix Spike %R > 125% Post-digestion spike %R <75%, but ≥ 10%	J	UJ
Matrix Spike %R < 30% No post-digestion spike performed (e.g., not required for Ag)	J-	R
Matrix Spike %R 30-74% No post-digestion spike performed (e.g., not required for Ag)	J-	UJ
Matrix Spike %R 75-125% Post-digestion spike not required	No qualification	No qualification
Matrix Spike %R > 125% No post-digestion spike performed (e.g., not required for Ag)	J+	No qualification

Methods 200.7/200.8/245.1
(Post digestion spike not required for Method 200.8)

Criteria	Action	
	Detect	Non-detect
Matrix Spike %R < 10% regardless of Post-digestion spike %R	J-	R
Matrix Spike %R < 70%, but ≥ 10% Post-digestion spike %R < 85%, but ≥ 10%	J-	UJ
Matrix Spike %R < 70%, but ≥ 10% Post-digestion spike %R ≥ 85%	J	UJ
Matrix Spike %R > 130% Post-digestion spike %R > 115%	J+	No qualification
Matrix Spike %R > 130% Post-digestion spike %R ≤ 115%, but ≥ 85%	J	No qualification
Matrix Spike %R > 130% Post-digestion spike %R <85%, but ≥ 10%.	J	UJ
Matrix Spike %R < 30% No post-digestion spike performed (e.g., not required for Ag)	J-	R
Matrix Spike %R 30-74% No post-digestion spike performed (e.g., not required for Ag)	J-	UJ
Matrix Spike %R 75-125% Post-digestion spike not required	No qualification	No qualification
Matrix Spike %R > 125% No post-digestion spike performed (e.g., not required for Ag)	J+	No qualification

Method 7471 (Post Digest Spike recommended by the method, but not required)

Criteria	Action	
	Detect	Non-detect
Matrix Spike %R < 10% regardless of Post-digestion spike %R	J-	R
Matrix Spike %R < 80%, but ≥ 10% Post-digestion spike %R < 85%, but ≥ 10%	J-	UJ
Matrix Spike %R < 80%, but ≥ 10% Post-digestion spike %R ≥ 85%	J	UJ
Matrix Spike %R > 120% Post-digestion spike %R > 115%	J+	No qualification
Matrix Spike %R > 120% Post-digestion spike %R ≤ 115%, but ≥ 85%	J	No qualification
Matrix Spike %R > 120% Post-digestion spike %R <85%, but ≥ 10%.	J	UJ
Matrix Spike %R < 30% No post-digestion spike performed	J-	R
Matrix Spike %R 30-79% No post-digestion spike performed	J-	UJ
Matrix Spike %R 80-120% Post-digestion spike not required	No qualification	No qualification
Matrix Spike %R > 120% No post-digestion spike performed	J+	No qualification

4.3.9 Serial Dilution (SD)

If percent difference (%D) for any analyte in the SD is greater than 10% and less than 50% and the SD sample result is > undiluted sample, estimate (J) detects and estimate (UJ) non-detects in all samples of the same matrix prepared and analyzed by the same method, indicating potential low bias. If the %D is greater than 50%, contact the ddms chemists for discussion and resolution.

4.3.10 Field Duplicate Samples

Refer to SOP ECS-SOP-012-Rev.0– General DV: 'Validation and Review of Analytical Data'.

4.3.11 Laboratory Control Sample (LCS)

Refer to SOP ECS-SOP-012-Rev.0– General DV: 'Validation and Review of Analytical Data'. Actions taken due to unacceptable LCS or LCS/LCSD results are as outlined below:

Methods 6010/6020/7470/7471

Criteria	**Action**	
	Detect	**Non-detect**
LCS %R < 10%	J-	R
LCS %R < 80%, but ≥ 10%	J-	UJ
LCS %R > 120%	J+	No qualification
Matrix Spike %R 80-120% Post-digestion spike not required	No qualification	No qualification

Method 200.7/200.8/245.1

Criteria	Action	
	Detect	Non-detect
LCS %R < 10%	J-	R
LCS %R < 85%, but ≥ 10%	J-	UJ
LCS %R > 115%	J+	No qualification
Matrix Spike %R 85-115% Post-digestion spike not required	No qualification	No qualification

4.3.12 Internal Standards (ISTDs)

ISTD intensities are method 200.8 and 6020 defined.

For data analyzed by EPA Method 200.8, the absolute response of any one internal standard must not deviate more than 60-125% of the original response in the calibration blank.

For data analyzed by EPA Method 6020B, the acceptance limit for ISTD response is 30% of the IS in the IC. If response falls below 30%, corrective action must be taken.

If any ISTD intensity is outside the acceptance limits, estimate (J) all detects and estimate (UJ) all non-detects for all analytes associated with that ISTD. Contact other ddms chemists and use professional judgment to reject (R) sample non-detects.

4.3.13 Target Analyte Quantitation

If the concentration for a positive detect is outside the linear range and a diluted analysis was not performed, estimate (J) those analytes that exceed the calibration range.

4.3.14 Reported Quantitation Limits (QLs)

Verify the sample QLs meet the project specific required QLs. Any samples that do not meet the project specific QLs should be documented in the validation report.

4.4 Total vs. Dissolved Metals (Aqueous samples)

If both total and dissolved metals were reported, verify the total concentration for each analyte is greater than the corresponding dissolved analyte concentration.

4.4.1 If the dissolved concentration of any analyte is greater than the total concentration, calculate the percent difference (%D).

4.4.2 If the %D is greater than 10%, estimate (J) in both the total and dissolved fractions, the concentration of that analyte.

4.4.3 If the %D is less than 10%, no qualification of sample results is performed, however this should be identified in the validation report and attributed to experimental error.

4.4.4 If the dissolved concentration of any analyte is greater than the total concentration, where the dissolved concentration is detected, and the total concentration is not-detected, qualify both samples as estimated (J, UJ).

5.0 PREPARATION OF REPORTS

5.1.1.1 Refer to SOP ECS-SOP-012-Rev.0– General DV: 'Validation and Review of Analytical Data'.

Standard Operating Procedure:
Validation and Review of Per- and Polyfluorinated Alkyl Substances (PFAS) Analysis Data

1.0 Scope and Application

Data validation determines the technical usability of analytical data. It is a process by which a body of data is compared to a set of acceptance criteria and flagged with qualifiers when acceptance criteria are not met and in response to various other impacts to the data quality. An unbiased and technically sound report is prepared that documents the degree of compliance to the intended purpose. There may be criteria in this SOP that are more stringent than those outlined in regional guidelines. The focus of validation is always on the technical usability and defensibility of the data. This SOP is applicable to data produced under the following methods:

EPA Method 537	EPA Method 537 Modified
ASTM D7968 – 17a	EPA SW-846 Method 8327 (draft)
ASTM D7979 - 17	EPA SW-846 Method 8328 (draft)
	EPA SW-846 Method 8329 (draft)

2.0 Summary

This SOP details the process of evaluating and verifying PFAS data, comparing it to stated Data Quality Objectives (DQOs) and acceptance criteria, and preparing a technically sound validation report that provides the data user with a reliable basis for decision making. Extensive laboratory knowledge, knowledge of published methods, and the ability to apply consistent professional judgment are essential. This SOP should be used in conjunction with ddms SOP ECS-SOP-012-Rev.0– General Data Validation: "Validation and Review of Analytical Data," the site-specific Quality Assurance Project Plans and Sampling and Analysis Plans, regional, national, or other agency data review guidelines, if applicable, and analytical methods. This SOP provides ddms' professional judgment.

All data have the potential to go to litigation. Part of the validation process is to identify any data and/or documentation issues that could be problematic if the data are used in litigation. All documents are discoverable during litigation, including validation/review reports. Any documentation issues that potentially affect the usability of the data identified during the review process must be clearly stated in the report.

When only a review (in lieu of a validation) of the data is requested, the procedure outlined in this SOP is followed, but calculations are not performed. Only a review of the data summary forms (raw data are not reviewed) is performed, unless that review reveals major quality issues. If at any time a situation arises that is not addressed in this SOP, contact with the other ddms chemists is required. Where professional judgement (PJ) is referenced in the guidelines and no PJ in this SOP covers the situation, communication with the other ddms chemists is required. Always

document in the report when ddms professional judgment is applied and the rationale for decisions made.

When verifying calculations, not all regions specify an acceptable %D between laboratory-reported and validator-calculated values. It is ddms' professional judgment to contact the laboratory when discrepancies greater than 10% occur. The error term of 10% is considered to be allowable experimental error in general laboratory practice.

3.0 Acronyms

%D – Percent Difference
%R – Percent Recovery
BAL – Blank Action Level
BS/BSD – Blank Spike/BS Duplicate
CC – Continuing Calibration
COC – Chain of Custody
DQO – Data Quality Objective
DV – Data Validation
FB – Field Blank
FRB – Field Rinsate Blank
IC – Initial Calibration
ICV – Initial Calibration Verification
ID - Identification
IS – Internal Standard
LC – Liquid Chromatography
LC/MS/MS – Liquid Chromatography/Tandem Mass Spectroscopy
LCS/LCSD – Laboratory Control Sample/LCS Duplicate
LIMS – Laboratory Information Management System
LRB – Laboratory Reagent Blank
MB – Method Blank
MDL – Method Detection Limit
MRL – Method Reporting Limit
PDS – Primary Dilution Standard
PFAS – Per- and Polyfluorinated Alkyl Substances
PJ – Professional Judgement
RF – Response Factor
QA – Quality Assurance
QAPP – Quality Assurance Project Plan
QC – Quality Control
QCS – Quality Control Sample
RL – Reporting Limit
RPD – Relative Percent Difference
RF – Response Factor
RRF – Relative Response Factor
RRT – Relative Retention Time
RT – Retention Time
RSD – Relative Standard Deviation

SAP – Sampling and Analysis Plan
S/N – Signal to Noise
SOP – Standard Operating Procedure
SS – Surrogate Standard
TCL – Target Compound List
TV – True Value

A list of acronyms for the PFAS and labeled analogs is provided in Attachment A.

4.0 Definitions

4.1 Due to inconsistent naming of the various QC elements among the high resolution methods, the following definitions specific to the use of isotope dilution are provided to avoid confusion:

4.1.1 Internal Standards / Injection Standards – These labeled compounds are added to each sample after extraction and prior to injection. They provide a means to assess successful injection into the measurement system.

4.1.2 Labeled Analogs / Surrogate Standards / Recovery Standards – These labeled compounds are added to all samples before preparation. Recoveries for these labeled compounds are used to assess the efficiency of the extraction procedure. The responses for these labeled compounds are used to calculate the concentrations of the native target compounds in the sample extract.

5.0 Procedure

5.1 Identify Project Specific Requirements - Refer to SOP ECS-SOP-012-Rev.0– General DV: "Validation and Review of Analytical Data."

5.2 Review, Verification and Evaluation of Analytical Results

5.2.1 Sample Integrity, Preservation, and Holding Time

5.2.1.1 Custody

5.2.1.1.1 Verify that complete, consistent, unbroken COC documentation is provided for all samples.

5.2.1.1.2 Ensure that a cross-reference between field IDs and lab IDs is provided.

5.2.1.2 Preservation

5.2.1.2.1 Verify that the temperature of the samples in the cooler(s) upon receipt at the laboratory was documented and was ≤10°C. If the temperature of the cooler was above the upper limit, qualify based on appropriate data validation guidelines. In the absence of regional validation guidelines regarding temperature excursions, apply ddms professional judgment. If the receipt temperature of the samples exceeds the acceptance criteria, but it is noted that the samples were received "on ice," review the documentation to verify that there was sufficient time (nominally 4 hours) from sample collection to laboratory receipt for the samples

to have cooled to the appropriate temperature. No qualification of data is necessary, but it is noted in the data validation report. If the elapsed time from sample collection to laboratory receipt exceeds 4 hours, use professional judgment to determine whether or not to estimate (J, UJ) the results.

5.2.1.2.2 Determine if Trizma was added to all samples, if appropriate, to remove chlorine and microbes and to maintain the pH at 7.0. If not and it should have been, use PJ to determine whether or not to qualify results.

5.2.1.2.3 Upon lab receipt, and based on documentation provided by the lab, samples should be maintained at ≤ 6°C and protected from light.

5.2.1.2.4 If samples were not preserved or stored properly, use PJ to determine action.

5.2.1.3 Holding Time

5.2.1.3.1 The methods stipulate a 14-day holding time from collection to extraction for water samples and a 28-day holding time from collection to extraction for solid samples, if samples are properly preserved, and 28 days from extraction to analysis.

5.2.1.3.2 In the absence of regional validation guidelines regarding holding time excursions, ddms professional judgment is to qualify all results as estimated (J, UJ) if samples were extracted or analyzed beyond the method holding time. If the holding time is grossly exceeded (>28 days for water sample extraction, >56 days for solid sample extraction, >60 days to analysis), qualify detects estimated (J) and reject (R) non-detects.

5.2.2 Extraction

5.2.2.1 If extraction procedures were not followed properly, use PJ to determine potential effects on results, and qualify data as estimated (J, UJ) or, if appropriate, rejected (R). The following situations may be encountered and should be documented by the lab:

5.2.2.1.1 Full sample volume or mass was not available or was not used. Preparation for aqueous samples requires use of full sample volume. Ensure that sample containers used are appropriate for full-volume extraction (nominally 250 mL sample bottle used). Use PJ to determine if there are any adverse effects from use of sample aliquot instead of entire volume.

5.2.2.1.2 Samples were transferred from original sampling container for measurement before preparation based on documentation provided from the lab.

5.2.2.1.3 Samples contained color, particulates, precipitate, etc., and/or special preparation steps were performed.

5.2.2.1.4 The appropriate solvents (96:4% MeoH:water) and the correct solid phase extraction (SPE) cartridges were not used.

5.2.2.1.5 Cartridges were not properly conditioned before use.

5.2.2.1.6 All equipment used was not constructed of polypropylene or polyethylene and was not verified to be PFAS free. PFAS will adsorb to some materials.

5.2.2.1.7 It cannot be determined if all equipment and supplies used met QC criteria. (If applicable, testing records for each lot of consumables may be requested from the laboratory.)

5.2.3 System Performance

5.2.3.1 The validator must verify that acceptable mass calibration and resolution was achieved before analyses were initiated. Because there is no prescribed tuning compound for PFAS analysis (manufacturer's instructions are referenced for use), verify appropriate tuning and resolution based on data provided for the instrument tune, if available. In the absence of mass tuning data, evaluate the mass resolution and sensitivity in the calibration standards. Documentation for mass calibration and resolution may be requested from the laboratory if needed. Polypropylene glycol (PPG) is commonly used for calibration, annually at a minimum. If any of the mass calibration/resolution or chromatographic criteria are not met or were not verified, use PJ to determine the effect on sample results and qualify accordingly.

5.2.3.2 Because much of the data generation for this instrumentation is blind to the data reviewer, it is reasonable to request clarification from the laboratory on instrument conditions, mass data acquisition parameters, etc. Determine (and/or request from the laboratory for verification) that all analyses have been performed under the same conditions and that all integrations of the reference and unlabeled target analytes have been performed the same throughout all of the applicable analyses (for all initial and continuing calibration standards and all field and QC samples). If the same conditions, including the acquisition and integration parameters, have not been used for all analyses, use PJ to determine the potential effect on data and qualify accordingly. It may be appropriate to qualify results as estimated or to reject results.

5.2.3.3 Chromatographic performance for PFAS analysis is evaluated based on the peak shapes, retention time stability, sensitivity, and resolution in the calibration standards.

5.2.3.4 Verify a minimum of 10 scans across the chromatographic peak for a mid-level calibration standard.

5.2.3.5 Verify that the peak asymmetry ratio for each peak is 0.8-1.5.

5.2.3.6 Signal to noise (S/N) should be at least 10:1 for defensible identification of target compounds.

5.2.3.7 Verify that any manual integrations were performed correctly, documented appropriately, and done consistently throughout the data set.

5.2.4 Calibration

5.2.4.1 Initial Calibration

5.2.4.1.1 A minimum of five calibration points must be used.

5.2.4.1.2 The calibration range must span a 20-fold concentration range.

5.2.4.1.3 Quadratic curves must have six points and must be forced through zero (may be weighted).

5.2.4.1.4 If calculated against the curve, all standard recoveries must be 70-130% of the true concentration, except the lowest, which must be 50-150% of the true value.

5.2.4.1.5 IS areas must be within ±50% of the average area calculated from the IC and within 70-140% of the most recent CC.

5.2.4.1.6 The RPD between high and low areas for each IS must be ≤20%. If exceeded, qualify associated detect results as estimated. Use PJ for non-detects.

5.2.4.1.7 Verify that all peaks (if multiple peaks are present) observed for PFHxS, PFOS, NMeFOSAA, and NEtFOSSA were integrated and the total area used for calculation. Branched isomers must be included in the integration for all analyses.

5.2.4.1.8 If the isotope dilution method is used for quantitation, evaluate RFs used to establish the calibration for the labeled analogs and the native compounds.

5.2.4.1.9 If internal standards are used, evaluate the peak shape and response for the IS (single or multiple) used.

5.2.4.1.10 If multiple IS are used, verify that the appropriate relationship between labeled analog of target analytes to the IS has been established.

5.2.4.1.11 Verify that the IS are added to all standards and samples (QC and field) to achieve the same concentration.

5.2.4.1.12 Evaluate peak shapes and responses/recoveries of the labeled analogs for each of the compounds analyzed.

5.2.4.1.13 Where there are target compounds present without a labeled analog, verify that assignment to a labeled analog is appropriate for each of those compounds.

5.2.4.1.14 If the %RSD is less than 20% across the IC standards, the average response factor should be used. If the %RSD is greater than 20% in the IC, the laboratory should use a linear model for calibration. The validator should examine the data to determine any causes for non-linearity. If by eliminating either the high or low point of the curve and recalculating %RSD an acceptable RSD is achieved, this approach is appropriate. If the high point is eliminated,

accept positive detects in the linear portion of the curve; estimate (J) positive detects at the higher end of the curve, and accept non-detects. If the low point on the curve is removed, accept positive detects in the linear portion of the curve, estimate (J, UJ) positive detects and non-detects in the lower portion of the curve. In the absence of regional qualification guidelines, ddms practice will be to raise the RL to the concentration at which linearity was demonstrated.

5.2.4.1.15 In situations where the instrument response was not linear and the laboratory may have used an alternate calibration model, such as a quadratic equation, follow the procedure outlined in Section 5.2.4.1.14. If a quadratic equation was used to establish calibration, it indicates that there are problems with the measurement system. Careful assessment should enable the validator to determine which portion of the calibration is reliable, if any. If only a portion of the calibration is reliable, qualify the data accordingly. Results within the range may be acceptable as reported, while results outside of this usable range should be either qualified as estimated or rejected, based on the potential error. PJ will be necessary and other ddms validators should be consulted.

5.2.4.1.16 If internal standards were not used, determine if and how the laboratory assessed valid injection and what criteria were used.

5.2.4.1.17 If the labeled analog recoveries are used as the acceptance criteria for acceptable injections, determine how those are calculated.

5.2.4.1.18 If single-point calibrations are used for the labeled analogs, ddms applies limits of 50-150% for recoveries since calibration has only been established and verified at the concentration spiked.

5.2.4.1.19 If external calibrations are used for the target compounds, verify that the calibration models used are valid. If quadratic equations are used, verify that a minimum of six points is included.

5.2.4.2 Retention Time Window

5.2.4.2.1 If internal standards are used, assess relative retention times for each mass peak selected for identification, confirmation, and quantification.

5.2.4.2.2 Relative retention times for target analytes, internal standards, and labeled analogs should be within 0.2 retention time units. Retention times outside this window may be acceptable. PJ will be necessary and other ddms validators should be consulted.

5.2.4.2.3 One chromatogram of the five (minimum) concentrations must show peaks at 25-100% of the full scale.

5.2.4.3 An initial calibration verification (ICV) standard (second-source quality control sample) must be analyzed with each new lot of primary dilution standard (PDS) or new initial calibration, and at a minimum, quarterly.

5.2.4.3.1 Verify that the ICV was a second-source standard and that ICV results are calculated on the correct IC. ddms acceptance criteria are ±30%. If the recovery of any compound falls outside of 70-130%, contact the laboratory to determine if the ICV and CC standards are from the same working lot. If the standards are from the same working lot and recovery is less than 70%, qualify detects (J) and non-detects (UJ). If the recovery is greater than 130%, qualify detects (J) and accept non-detects.

5.2.4.3.2 If analysis of the ICV is not included in the data package, request from the laboratory. If an ICV was not analyzed at the appropriate frequency, qualify results. Without acceptable performance of the ICV, the true concentrations of the of the IC standards have not been verified and the accuracy of results calculated on the IC are not supported, unless the CC was from a second source from the IC.

5.2.4.4 Continuing Calibration

5.2.4.4.1 Verify that continuing calibration (CC) standards are analyzed on each instrument used to analyze samples at the beginning of each sequence before any blanks, samples or QC samples are analyzed and at the end of the sequence. The opening CC must be at the MRL. A CC must be analyzed after every tenth sample (not including batch QC samples) and must alternate throughout the analytical sequence, in concentrations to cover the IC range.

5.2.4.4.2 Verify the CC was compared against the most recent IC. If CC was not compared against most recent IC, contact the laboratory to correct CCs and subsequent sample results.

5.2.4.4.3 Recoveries must be 70-130% for all but the lowest concentration and 50-150% for the lowest concentration.

5.2.4.4.4 If recoveries are outside acceptance limits and the shift represents an increase in sensitivity, positive results are qualified as estimated (J), but non-detects are not qualified. If the shift represents a decrease in sensitivity, qualify positive results and non-detects estimated (J, UJ). If any target compound has a %D greater than 100.0% exhibiting an increase in sensitivity, consult ddms chemists for further action.

5.2.4.4.5 If the closing CC is unacceptable for a compound and the opening CC was acceptable, review all of the samples included in the bracket and consult with other ddms chemists. If spiked samples (LCS/MS) are present to support continued performance, the results may be acceptable. Labeled compound responses in the samples may provide support for continued performance. PJ must be used on a case by case basis to determine the effect on the sample data impacted by the unacceptable CC.

5.2.4.4.6 Verify that CCs concentrations were varied. A low-level standard must be analyzed with each analytical sequence to verify the laboratory's continued ability to meet the reporting limits

5.2.5 Blanks

5.2.5.1 Laboratory Reagent Blank (LRB)

5.2.5.1.1 A method Blank should be analyzed on each instrument on which samples from the same preparation batch were analyzed.

5.2.5.1.2 All target analytes must be <1/3 of the MRL.

5.2.5.2 Field-generated Blanks - For PFAS, special sampling procedures are critical because the potential for contamination from sampling containers, equipment, and personal clothing and care products is very high. Field-submitted blanks are critical due to the ubiquitous nature of the compounds. If a field-submitted blank was not collected and provided, assess the impact on the samples and determine appropriate action. Consultation with other ddms chemists is recommended.

5.2.5.2.1 Some Regions do not qualify data based on field-submitted blank results. ddms' SOP is **to** qualify sample results based on field-submitted blank results. It is not ddms' policy to qualify soil data based on associated field-submitted sample results without consultation with other ddms chemists.

5.2.5.2.2 An FRB (at a minimum) is required with each collection event for PFAS to assess contamination; however, the presence and the type(s) of field-submitted blank(s) collected may be subject to individual project requirements.

5.2.5.3 Blank Scenarios

5.2.5.3.1 Scenario 1: The blank action level (BAL) in the MB is less than that of the FB BAL. The sample result is greater than the MB BAL, but less than the FB BAL. Even though the governing agency specifically states that data not be qualified based on FB results, based on ddms professional judgment, results would be qualified as not detected (U) at

the sample-specific RL or the reported value, whichever is greater.

5.2.5.3.2 Scenario 2: Samples are analyzed on two different instruments on the same day. On one of the instruments, a common laboratory contaminant is reported in the MB and on the other instrument the same analyte is ND. The sample results on both instruments were less than the BAL on the instrument with the contaminant. ddms professional judgment is to qualify the results from the analytical runs on both instruments as not detected (U) at the sample-specific RL or the reported value, whichever is greater.

5.2.5.3.3 If any target analytes were detected in any of the blanks, the blank action level (BAL) is calculated as ten times the maximum contamination level of the blank for the associated samples.

5.2.6 Labeled Analogs - ddms acceptance limits for recovery are 40-140%. A list of target analyte associations with the labeled analogs is provided in Attachment B. Not all target PFAS have labeled analogs; be aware that associated labeled analogs for these analytes may be different from laboratory to laboratory.

5.2.6.1 If the recovery of a labeled analog is less than 40% but greater than or equal to 10%, estimate detects (J) for the native target compound(s) and non-detects (UJ).

5.2.6.1 If the recovery of a labeled analog is less than 10%, estimate (J) detects and reject (R) non-detects.

5.2.6.2 If the recovery of the labeled analog is greater than 140%, positive results are qualified as estimated (J) and non-detects are not qualified.

5.2.6.3 If internal standards are not used, then the labeled analogs must also be evaluated in place of the IS. Responses for these compounds must be within 50-150%. These compounds must be assessed to determine effective injection as well as recovery through the extraction procedure.

5.2.6.4 If the labeled analogs were calibrated with only a single concentration in the calibration, only that concentration has been demonstrated to be accurate. This generates the need to evaluate the responses as both IS and as surrogates.

5.2.7 Internal Standards (IS)

5.2.7.1 If internal standards are used, IS areas must be within 70-140% of the most recent CC and within 50-150% of the associated IC.

5.2.7.2 If the IS area is outside the specified limits, qualify detects and non-detects associated with that IS as estimated (J, UJ).

5.2.7.3 If the IS area is less than 25% or greater than 200%, contact other ddms chemists for discussion and resolution. Excessively low or

high IS responses indicate the potential for high variability in the analysis and will result in qualification of results.

5.2.7.4 If any sample was not reanalyzed due to an IS area outside acceptance criteria, this must be noted in the validation report.

5.2.8 Laboratory Control Sample (LCS) / Laboratory Control Sample Duplicate (LCSD) - Refer to SOP ECS-SOP-012-Rev.0– General DV: "Validation and Review of Analytical Data."

5.2.9 Matrix Spike/Matrix Spike Duplicates (MS/MSD) - Refer to SOP ECS-SOP-012-Rev.0– General DV: "Validation and Review of Analytical Data."

5.2.10 Field Duplicate Samples - Refer to SOP ECS-SOP-012-Rev.0– General DV: "Validation and Review of Analytical Data."

5.2.11 Target Compound Identification

5.2.11.1 Mass peak RTs, S/N ratio, and the ratio between primary and secondary masses are assessed against the reference standards for identification of the individual target compounds. Professional judgment is often required to determine if a correct identification was made.

5.2.11.2 If it is determined that the identification is incorrect, contact with the laboratory may be necessary to have the data corrected and resubmitted. If a discrepancy remains, use professional judgment to qualify or reject and document the reason in the validation report.

5.2.11.3 Inspect any sample(s) analyzed following a high concentration sample to verify that no carryover occurred and was not incorrectly reported in the following sample(s). ddms PJ is to contact the laboratory for explanation and resolution. If the validator's opinion differs from the laboratory's response, contact other ddms chemists for verification of opinion and qualification of data.

5.2.12 Compound Quantitation

5.2.12.1 Examine the method of calculation for the target analytes. Ensure that these are calculated in consistency with the established calibration.

5.2.12.2 Verify calculations of some selected labeled analog recoveries and unlabeled target analyte concentrations.

5.2.12.3 If a dilution was performed because the concentration of an analyte was outside of the calibration range in the undiluted analysis, but that analyte was reported as a non-detect in the diluted analysis, evaluate the raw data or refer to historical data, if available, to determine if the non-detect in the reanalysis was a result of over dilution or if the analyte was a false positive in the initial, undiluted analysis. If the non-detect in the reanalysis was a result of over dilution, it is ddms PJ to accept the result from the initial, undiluted analysis as the reportable concentration and qualify this result as estimated (J) because it was detected outside of the calibration range. If the result

in the initial, undiluted analysis is determined to be a false positive, reject (R) the result in the initial analysis.

5.2.12.4 If multiple dilutions have been performed, assess all of the available data to determine the most usable results.

5.2.12.5 If the laboratory has supplemented the internal standards and/or surrogate standards after dilution of the sample extract, determine the method used for assessing successful analysis and for calculating the target analyte concentrations. Use PJ to determine whether or not results require qualification based on the laboratory's procedure.

5.2.13 Reported Quantitation/Reporting Limits (QLs or RLs)

5.2.13.1 Verify the sample QLs or RLs meet the project specific required QLs or RLs. Any samples or target analytes that do not meet the project specific RLs should be documented in the validation report.

5.2.13.2 Any RLs not supported by the initial calibration need to be recalculated and corrected for all associated samples.

5.2.13.3 Correct reported non-detects at the MDL to non-detected at the RL.

5.2.13.4 Where the Analytical Report lists "ND" for sample results and lists both the MDL and RL, the "ND" should be understood to be ND at the RL. This should be narrated in the validation report.

5.2.14 Documentation - Refer to SOP ECS-SOP-012-Rev.0– General DV: "Validation and Review of Analytical Data."

6.0 Preparation of Reports - Refer to SOP ECS-SOP-012-Rev.0– General DV: "Validation and Review of Analytical Data."

ATTACHMENT A

LIST OF ACRONYMS FOR PFAS AND LABELED ANALOGS

Per- and Polyfluorinated Target Compounds

Compound Name	Acronym	CAS number
Perfluoroalkylcarboxylates		
Perfluorobutanoic acid	PFBA	375-22-4
Perfluoropentanoic acid	PFPeA	2706-90-3
Perfluorohexanoic acid	PFHxA	307-24-4
Perfluoroheptanoic acid	PFHpA	375-85-9
Perfluorooctanoic acid	PFOA	335-67-1
Perfluorononanoic acid	PFNA	375-95-1
Perfluorodecanoic acid	PFDA	335-76-2
Perfluoroundecanoic acid	PFUnDA	2058-94-8
Perfluorododecanoic acid	PFDoDA	307-55-1
Perfluorotridecanoic acid	PFTrDA	72629-94-8
Perfluorotetradecanoic acid	PFTeDA	376-06-7
Perfluorohexadecanoic acid	PFHxDA	67905-19-5
Perfluorooctadecanoic acid	PFODA	16517-11-6
Perfluoroalkylsulfonates		
Perfluorobutanesulfonic acid	PFBS	375-73-5
Perfluoropentanesulfonic acid	PFPeS	2706-91-4
Perfluorohexanesulfonic acid	PFHxS	355-46-4
Perfluoroheptanesulfonic acid	PFHpS	375-92-8
Perfluorooctanesulfonic acid	PFOS	1763-23-1
Perfluorononanesulfonic acid	PFNS	68259-12-1
Perfluorodecanesulfonic acid	PFDS	335-77-3
Perfluorododecanesulfonic acid	PFDoDS	79780-39-5
Perfluorooctanesulfonamides		
Perfluorooctanesulfonamide	PFOSA	754-91-6
N-methyl Perfluoro-1-octanefulfonamide	NMePFOSA	31506-32-8
N-ethyl Perfluoro-1-octanesulfonamide	NEtPFOSA	4151-50-2

Compound Name	Acronym	CAS number
Alkyl Perfluorooctanesulfonamidoacetic Acids		
Methylperfluorooctanesulfonamidoacetic acid	NMeFOSAA	2355-31-9
Ethylperfluorooctanesulfonamidoacetic acid	NEtFOSAA	2991-50-6
Alkyl Perfluroroalkane Sulfonamidoethanols		
2-(N-methylperfluoro-1-octanesulfonamido)-ethanol	NMePFOSAE	2444809-7
2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol	NEtPFOSAE	1691-99-2
Fluorotelomersulfonates		
4:2-Fluorotelomersulfonate	4:2-FTS	757124-72-4
6:2-Fluorotelomersulfonate	6:2-FTS	27619-97-2
8:2-Fluorotelomersulfonate	8:2-FTS	39108-34-4
10:2-Fluorotelomersulfonate	10:2-FTS	120226-60-0
Perfluoroalkyl Ether Carboxylates		
2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)-propanoic acid	HFPODA	13252-13-6
Sodium dodecafluoro-3H-4,8-dioxanonanoate	NaDONA	958445-44-8
4,8-dioxa-3H-Perfluorononanoic acid	DONA	919005-14-4
Labeled Perfluoroalkylcarboxylic Acids		
$^{13}C_4$-Perfluorobutanoic acid	13C4-PFBA	
$^{13}C_5$-Perfluoropentanoic acid	13C5-PFPeA	
$^{13}C_5$-Perfluorohexanoic acid	13C5-PFHxA	
$^{13}C_4$-Perfluoroheptanoic acid	13C4-PFHpA	
$^{13}C_8$-Perfluorooctanoic acid	13C8-PFOA	
$^{13}C_9$-Perfluorononanoic acid	13C9-PFNA	
$^{13}C_6$-Perfluorodecanoic acid	13C6-PFDA	
$^{13}C_7$-Perfluoroundecanoic acid	13C7-PFUnDA	
$^{13}C_2$-Perfluorododecanoic acid	13C2-PFDoDA	
$^{13}C_2$-Perfluorotetradecanoic acid	13C2-PFTeDA	

Compound Name	Acronym
Labeled Perfluoroalkylsulfonates	
$^{13}C_3$-Perfluorobutanesulfonic acid	13C3-PFBS
$^{13}C_3$-Perfluorohexanesulfonic acid	13C3-PFHxS
$^{13}C_8$-Perfluorooctanesulfonic acid	13C8-PFOS
Perfluorooctanesulfonic Acids	
$^{13}C_8$-Perfluorooctanesulfonamide	13C8-PFOSA
d_3-N-methyl Perfluoro-1-octanefulfonamide	d3-NMePFOSA
d_5-N-ethyl Perfluoro-1-octanesulfonamide	d5-NEtPFOSA
Labeled Perfluorooctanesulfonamidoacetic Acids	
d_3-Methylperfluorooctanesulfonamidoacetic acid	d3-NMeFOSAA
d_5-Ethylperfluorooctanesulfonamidoacetic acid	d5-NEtFOSAA
d_7-2-(N-methylperfluoro-1-octanesulfonamido)-ethanol	d7-NMePFOSAE
d_9-2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol	d9-NEtPFOSAE
Labeled Fluorotelomersulfonates	
$^{13}C_2$-4:2-Fluorotelomersulfonate	13C2-4:2-FTS
$^{13}C_2$-6:2-Fluorotelomersulfonate	13C2-6:2-FTS
$^{13}C_2$-8:2-Fluorotelomersulfonate	13C2-8:2-FTS
Labeled Perfluoroalkyl Ether Carboxylic Acids	
$^{13}C_3$-2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)-propanoic acid	13C3-HFPODA

ATTACHMENT B

TARGET ANALYTE ASSOCIATIONS WITH LABELED ANALOGS

Target Analyte Associations with Labeled Analogs		
Associated Target Analyte	**Labeled Analog**	**Alternate Labeled Analog**
PFBA	13C4-PFBA	
PFPeA	13C5-PFPeA	
PFBS	13C3-PFBS	
PFPeS	13C3-PFBS	13C3-PFHxS
4:2-FTS	13C2-4:2-FTS	
PFHxA	13C5-PFHxA	
HFPODA	13C3-HFPODA	
PFHpA	13C4-PFHpA	
DONA	13C4-PFHpA	
PFHxS	13C3-PFHxS	
PFHpS	13C3-PFHxS	13C8-PFOS
6:2-FTS	13C2-6:2-FTS	
PFOA	13C8-PFOA	
PFOS	13C8-PFOS	
PFNS	13C8-PFOS	
PFDS	13C8-PFOS	
PFDoDS	13C8-PFOS	
PFNA	13C9-PFNA	
PFDA	13C6-PFDA	
8:2-FTS	13C2-8:2-FTS	
10:2-FTS	13C2-8:2-FTS	
PFOSA	13C8-PFOSA	
NMeFOSAA	d3-NMeFOSAA	
PFUnDA	13C7-PFUnDA	
NEtFOSAA	d5-NEtFOSAA	
PFDoDA	13C2-PFDoDA	
PFTrDA	13C2-PFDoDA	
NMePFOSAE	d7-NMePFOSAE	
NMePFOSA	d3-NMePFOSA	
NEtPFOSAE	d9-NEtPFOSAE	
NEtPFOSA	d5-NEtPFOSA	
PFTeDA	13C2-PFTeDA	
PFHxDA	13C2-PFHxDA	13C2-PFTeDA
PFODA	13C2-PFHxDA	13C2-PFTeDA

Standard Operating Procedure:

Validation and Review of Gas Chromatography (GC) Data

1.0 Scope and Application

Data validation determines the technical usability of analytical data. It is a process by which a body of data is compared to a set of acceptance criteria and flagged with qualifiers when acceptance criteria are not met. An unbiased and technically sound report is prepared that documents the degree of compliance with the intended purpose. There may be criteria in this standard operating procedure (SOP) that are more stringent than those outlined in regional guidelines. The focus of validation is always on the technical usability and defensibility of the data. This SOP is applicable to data produced under GC methods for analysis. Target analytes may include organochlorine pesticides, polychlorinated biphenyls (PCBs) as Aroclors, chlorinated herbicides, dissolved gases, nonhalogenated organics (excluding petroleum hydrocarbons, phenols, alcohols, explosives, phthalate esters, nitrosamines, haloethers, and other analytes or chemical classes amenable to GC analysis.

2.0 Summary

This SOP details the process of evaluating and verifying GC data, comparing it to stated data quality objectives (DQOs) and acceptance criteria, and preparing a technically sound validation report that provides the data user with a reliable basis for decision making. Extensive laboratory knowledge, knowledge of published methods, and the ability to apply consistent professional judgment are essential. This SOP should be used in conjunction with ddms SOP ECS-SOP-012-Rev.0– General Data Validation: "Validation and Review of Analytical Data," the site-specific quality assurance project plans (QAPPs) and sampling and analysis plans (SAPs), regional data review guidelines, if applicable, and analytical methods. This SOP provides ddms' professional judgment.

All data have the potential to go to litigation. Part of the validation process is to identify any data and/or documentation issues that could be problematic if the data are used in litigation. All documents are discoverable during litigation, including validation/review reports. Any issues identified during the data review process must be clearly identified in the report.

When only a review of the data is requested, the procedure outlined in this SOP is followed, but calculations are not performed. Only a review of the data summary forms is performed, unless that review reveals major quality issues. If at any time a situation arises that is not addressed in this SOP, contact with the other ddms chemists is required. Where professional judgment (PJ) is referenced in the guidelines and no PJ in this SOP covers the situation, communication with the other ddms chemists is required. Always document in the report when ddms PJ is applied and the rationale for decisions made.

When verifying calculations, not all regions specify an acceptable %D between laboratory-reported and validator-calculated value. It is ddms' PJ to contact the laboratory when discrepancies greater than 10% occur. The error term of 10% is considered to be allowable experimental error in general laboratory practice.

3.0 Acronyms

%D – Percent Difference
%R – Percent Recovery
BAL – Blank Action Level
CC – Continuing Calibration
COC – Chain of Custody
DQO – Data Quality Objective
DV – Data Validation
FB – Field Blank
FD – Field Duplicate
GC – Gas Chromatography
GC/MS – Gas Chromatography/Mass Spectroscopy
IC – Initial Calibration
ICV – Initial Calibration Verification
IS – Internal Standard
LCS/LCSD – Laboratory Control Sample/Laboratory Control Sample Duplicate
LIMS – Laboratory Information Management System
MB – Method Blank
MDL – Method Detection Limit
MS/MSD – Matrix Spike/Matrix Spike Duplicate
ND – Not Detected
PCB – Polychlorinated Biphenyl
QA – Quality Assurance
QAPP – Quality Assurance Project Plan
QC – Quality Control
QL – Quantitation Limit

RL – Reporting Limit
RPD – Relative Percent Difference
RRF – Relative Response Factor
RRT – Relative Retention Time
RSD – Relative Standard Deviation
RT – Retention Time
SAP – Sampling and Analysis Plan
SOP – Standard Operating Procedure
SS – Surrogate Standard
TCL – Target Compound List

4.0 Procedure

4.1 Identify Project Specific Requirements- Refer to SOP ECS-SOP-012-Rev.0– General DV: "Validation and Review of Analytical Data."

4.2 Sample Preservation and Holding Time

4.2.1 Verify that sample conditions between collection and receipt at the laboratory protected the integrity of the samples, including temperature, chemical preservation if applicable, proper containers for the parameters requested, and condition of samples and containers (intact, no headspace for dissolved gases, leaking containers, etc.) If conditions were not met, apply qualifiers as recommended by the reference method, validation guidance, or project-specific requirements. Use PJ as appropriate, where necessary.

4.2.2 Verify that the temperature of the samples in the cooler(s) upon receipt at the laboratory was documented and was ≤ 6°C (or 4°C ± 2°C). If the temperature was outside the lower range, no qualification is made, but it is documented in the validation report. If the temperature of the cooler was outside the upper range, qualify based on appropriate data validation guidelines. In the absence of regional validation guidelines regarding temperature excursions, apply ddms professional judgment. If the receipt temperature of the samples exceeds the acceptance criteria, but it is noted that the samples were received "on ice," review the documentation to verify that there was sufficient time (nominally 4 hours) from sample collection to laboratory receipt for the samples to have cooled to the appropriate temperature. No qualification of data is necessary, but it is noted in the DV report. If the elapsed time from sample collection to laboratory receipt exceeds 4 hours, estimate (J, UJ) the results.

4.2.3 In the absence of regional validation guidelines regarding holding time excursions, apply ddms professional judgment. Qualify sample results as estimated (J, UJ) for samples prepared and/or analyzed outside the method-specified holding time. Reject (R) non-detect sample results and estimate (J)

detects for samples prepared outside of two times the holding time. It may be appropriate to reject (R) non-detect sample results and estimate (J) detected results for samples analyzed more than 60 days after preparation. Note, however, that PJ should be applied in the case of missed holding times.

4.2.4 Chapter 4 of SW-846 does not specify a holding time for extraction or analysis for PCBs, although it is recommended that extracts be analyzed within 40 days of extraction. Note that since Aroclors are stable in the environment, it may not be necessary to qualify results when analysis is performed more than 40 days after extraction. Use PJ to estimate or accept the results.

4.3 Review, Verification, and Evaluation of Analytical Results

4.3.1 Initial Calibration

4.3.1.1 Chromatographic performance for GC analyses is evaluated based on the retention time stability, sensitivity, peak shape, and resolution in the calibration standards. For multicomponent analytes such as Aroclors, performance is also evaluated based on a characteristic chromatographic pattern. Comparison between columns should be assessed if the analysis was performed with dual columns.

4.3.1.2 For pesticides, a resolution check mixture and performance evaluation mixture (PEM) may be analyzed prior to initial calibration.

4.3.1.2.1 For the resolution check mixture, resolution between adjacent peaks must be ≥80% on the primary column and must be ≥50% on the confirmation column if a single standard mixture is used for initial calibration. If two individual standard mixtures are used, resolution between adjacent peaks on both columns must be ≥60%.

4.3.1.2.2 For the PEM, %Ds for all target analytes and surrogates must be ±25%, absolute RTs must be within the established RT windows, and all target analytes and surrogate compounds must be 90% resolved on each column.

4.3.1.3 With the exception of Aroclors, the initial calibration must include at least 5 points for all target analytes. Aroclor methods specify that at least five points are required only for Aroclors 1016/1260. Although many laboratories perform multi-point initial calibrations for Aroclors other than 1016 and 1260, single points may be run (for pattern recognition). At least five peaks are selected for identification and quantitation of Aroclors 1016 and 1260, none of which overlap. For the remaining Aroclors, a minimum of 3 peaks (preferably 5) should be selected. Each peak must be characteristic of the Aroclor and must

be at least 25% of the height of the largest Aroclor peak. At least one selected peak for each Aroclor should be unique to it.

4.3.1.4 For an external standard calibration, calibration factors (CFs) are calculated and used to evaluate linearity.

4.3.1.5 For an internal standard calibration, response factors (RFs) are calculated and used to evaluate linearity.

4.3.1.6 If the %RSD is greater than 20% in the IC (15% for dissolved gases), determine the cause of non-linearity by eliminating either the high or low point of the curve and recalculating the %RSD. If the %RSD is less than 20% after eliminating the high point, accept positive detects in the linear portion of the curve, estimate (J) positive detects at the higher end of the curve, and accept non-detects. If the %RSD is less than 20% after eliminating the low point on the curve, accept positive detects in the linear portion of the curve, estimate (J) positive detects in the lower portion of the curve. In the absence of regional qualification guidelines, ddms practice will be to raise the RL to the concentration at which linearity was demonstrated.

4.3.1.7 In situations where the instrument response was not linear, the laboratory may have used an alternate calibration model, such as a linear regression or quadratic equation. At least 6 points must be used for a quadratic equation. Follow the procedure outlined in Section 4.3.1.5 to determine if all or some portion of the model can be used to produce usable results. Use PJ to determine which, if any, results should be qualified (i.e., positive results may warrant qualification as estimated [J] and non-detects as estimated, rejected, or not detected at an elevated RL depending on the position of the outlier[s] in the model). Discussion with other ddms chemists is recommended for these instances.

4.3.1.8 Many methods require a read-back, or re-calculation, of analyzed standards against the calibration models used. Relative standard error (RSE) or percent error is determined and must meet method or laboratory-established requirements to support results calculated against the model. If no guidance is available, ddms default criteria are ±50% for the lowest level of the calibration and ±30% for all other levels.

4.3.1.9 A DDT standard may be included in the Aroclors analysis to determine if 4,4’-DDT, 4,4’-DDE, or 4,4’-DDD elute at the same RTs as any of the target Aroclor peaks.

4.3.2 Retention Time Windows

4.3.2.1 If internal standards are used, assess relative retention times. If external calibration was used, evaluate absolute retention times for each target analyte or Aroclor peak selected for identification and quantification.

4.3.2.2 RT windows should be ±0.07 minutes for the major Aroclor peaks, ±.05 for TCX, and ±0.10 for DCB. Procedures for determining RT windows for other analysis parameters are as specified in the applicable method.

4.3.2.3 One chromatogram of the five (minimum) concentrations must show peaks at 25-100% of the full scale.

4.3.3 Initial Calibration Verification (ICV)

4.3.3.1 Verify that the ICV was a second-source standard. ddms acceptance criteria are ± 20% (±15% for dissolved gases). If the recovery of any analyte falls outside of the acceptance limits, contact the laboratory to determine if the ICV and CC standards are from the same working lot. If the standards are from the same working lot and recovery is less than 80% (or 85% for dissolved gases), qualify detects (J) and non-detects (UJ). If the recovery is greater than 120% (or 115% for dissolved gases), qualify detects (J) and accept non-detects. All sample results calculated using the associated IC are qualified.

Note: For RSK-175, a second source standard is analyzed at the end of the sample set.

NOTE: If the IC and ICV standards are from the same working lot, data qualification as above is necessary even if the samples were not analyzed within the analysis clock following the IC.

4.3.3.2 For pesticides analysis, endrin/DDT breakdown is evaluated at the beginning of each 12-hour shift. The % breakdown for each must be <15%.

4.3.4 Continuing Calibration

4.3.4.1 Verify that the continuing calibration (CC) standard was analyzed on each instrument used to analyze samples at the beginning of each sequence before any blanks, samples or QC are analyzed and, if required, at the end of the sequence.

4.3.4.2 The CC standard for pesticides should be alternating high and low concentration mixtures of single- and multi-component analytes. Other methods reference use of the mid-level IC concentration for the CC standard.

4.3.4.3 Verify that the CC was compared against the most recent IC. ddms' acceptance limit for the CC standard is ±20%. Some GC methods specify a more stringent acceptance limit (e.g., ±15%). Use PJ to determine if the more stringent limit should be applied.

4.3.4.3.1 If the CC was not compared against most recent IC, contact the laboratory to correct the CC summary form and, if warranted, any subsequent sample results.

4.3.4.3.2 When the percent difference (drift) exceeds 20%, qualify detects as estimated (J). If the shift represents an increase in sensitivity, non-detects are not qualified. If the shift represents a decrease in sensitivity, qualify non-detects as estimated (UJ). If any target compound has a %D greater than 100% exhibiting an increase in sensitivity, consult ddms chemists for further action.

4.3.4.3.3 All selected peaks for multi-component analytes must be reported by the laboratory and evaluated for the CC standard.

4.3.4.3.4 CC standard acceptance limits are 85-115% for the RSK-175 method dissolved gases analyses.

4.3.5 Blanks

4.3.5.1 Laboratory Method Blank (MB) – A laboratory method blank must be prepared and/or analyzed with each preparation batch.

4.3.5.2 Field-submitted Blanks – Field-submitted blanks can include trip blanks, equipment blanks, or rinse blanks.

4.3.5.2.1 If a field-generated blank was not collected and provided, assess the impact on the samples and determine appropriate action. Consultation with other ddms chemists is recommended.

4.3.5.2.2 Some Regions do not qualify data based on FB results. ddms' SOP is **to** qualify sample results based on FB results. It is not ddms' policy to qualify soil data based on associated FB sample results without consultation with other ddms chemists.

4.3.5.3 Blank Scenarios

4.3.5.3.1 Scenario 1: The analyte concentration in the MB is less than that of that in the FB. The sample result is greater than the MB, but less than the FB. Even though the governing agency specifically states that

data are not qualified due to FB results, based on ddms professional judgment, results would be qualified as not detected (U) at the sample-specific RL or reported value, whichever is greater.

4.3.5.3.2 Scenario 2: Samples are analyzed on two different instruments on the same day. On one of the instruments, a target compound is reported in the MB, and on the other instrument the same analyte is ND. The sample results on both instruments were less than the concentration detected on the instrument with the contaminant. ddms professional judgment is to qualify the results from the analytical runs on both instruments as not detected (U) at the sample-specific RL or reported value, whichever is greater.

4.3.6 Matrix Spike/Matrix Spike Duplicates (MS/MSD) - Refer to SOP ECS-SOP-012-Rev.0– General DV: "Validation and Review of Analytical Data."

4.3.7 Field Duplicate Samples - Refer to SOP ECS-SOP-012-Rev.0– General DV: "Validation and Review of Analytical Data."

4.3.8 Laboratory Control Sample (LCS)/ Laboratory Control Sample Duplicate (LCSD)

4.3.8.1 Verify that an LCS was prepared and/or analyzed as appropriate. ddms PJ (70-130%R) is as outlined below: Some GC methods may specify more stringent acceptance limits. Use PJ to determine if the more stringent limits should be applied.

4.3.8.1.1 If the recovery of an analyte is ≥130%, estimate (J) detects for that compound in the samples associated with that LCS, indicating potential high bias. Non-detects are not qualified.

4.3.8.1.2 If the recovery of an analyte is less than 70% but ≥10%, estimate (J) detects in all samples associated with the LCS, and estimate (UJ) all non-detects, indicating potential low bias.

4.3.8.1.3 If the recovery of analyte is less than 10%, estimate (J) detects in samples associated with that LCS and reject (R) all non-detects in samples associated with that LCS.

4.3.8.1.4 When the laboratory has analyzed an LCS/LCSD pair, the RPD between the paired results should be less than 20%. Qualify results as estimated (J) for all

samples in the associated analytical batch or sequence when the RPD exceeds 20%. When the RPD exceeds 40%, discussion with other ddms chemists is necessary for qualification of data.

4.3.8.1.5 All selected peaks for multi-component analytes must be reported by the laboratory and evaluated for the LCS.

4.3.8.1.6 Note: The LCS (and LCSD if applicable) should include all reported target analytes. If any reported analytes have not been included, use PJ and discussion with other ddms chemists to determine the impact on the reported results, which will depend on other information available in the data package to support that laboratory's ability to extract and/or detect the(se) compound(s).

4.3.9 Surrogates

4.3.9.1 Verify that the RT of the surrogate compound, if surrogates are being used, is within the established retention time window.

4.3.9.2 ddms acceptance criteria for surrogate recoveries are 70-130%.

4.3.9.2.1 If the recovery of any one surrogate is less than 70% but greater than 10%, estimate (J) all detects and non-detects (UJ) in that sample as indicated in the table below:

Surrogate	**Analytes**
Tetrachloro-m-xylene (TCX)	Pesticides, PCBs as Aroclors
Decachlorobiphenyl (DCB)	
2,4-Dichlorophenylacetic acid (DCAA)	Herbicides
None specified	Nonhalogenated Organics
None required	Dissolved Gases
None required	Alcohols
2,4-Dibromophenol	Phenols
2,5-Dinitrotoluene (2,5-DNT)	Explosives
3,4-Dinitrotoluene (3,4-DNT)	
Diphenyl phthalate	Phthalate Esters
Diphenyl isophthalate	
Dibenzyl phthalate	
None specified	Nitrosamines
2,4-Dichlorophenyl ether	Haloethers
2,3,4-Trichlorodiphenyl ether	

4.3.9.2.2 If the recovery of any one surrogate is less than 10%, associated positive results are estimated (J) and non-detect results are rejected (R). Use PJ to determine analyte associations with the affected surrogate since it may or may not be appropriate to qualify all target analytes based on an unacceptable surrogate recovery.

4.3.9.2.3 If two or more surrogates are outside of acceptance criteria, all analytes are qualified accordingly.

4.3.9.2.4 Where dual column analyses are performed, positive sample results are qualified based on surrogate recoveries only if the affected analyte was reported from the column on which the unacceptable recovery was obtained. In the case of non-detects, if surrogate recoveries are unacceptable on one column but acceptable on the second column, no qualifiers are required.

4.3.10 Internal Standards (IS)

4.3.10.1 If any IS RT is outside the acceptance limit, closely examine the affected sample chromatogram to determine if any false positive or false negatives exist and contact other ddms chemists for discussion and resolution.

4.3.10.2 When internal standards are used, IS areas must be within 50-150% of the average area from the IC, and retention times must be within ±30 seconds of the average RT from the IC.

4.3.10.3 If the IS area is between 25% and 50% or between 150% and 200% of the IS area of the most recent calibration standard, qualify detects and non-detects associated with that IS as estimated (J, UJ).

4.3.10.4 If the IS area is less than 25% or greater than 200%, contact other ddms chemists for discussion and resolution.

4.3.10.5 If any sample was not reanalyzed due to IS area and/or RT outside acceptance criteria, this must be noted in the validation report and results may require qualification.

4.3.11 Target Compound Identification

4.3.11.1 For single-component target analytes, the peak retention time must be within the established retention time window on both columns. For multi-component target analytes, retention times for the selected peaks must be within the established retention time window on both columns, and characteristic peak patterns are assessed

against the reference standards. Note that professional judgment may be required to determine if a correct identification was made.

4.3.11.2 Identification of detected compounds for GC analyses is typically confirmed through the use of a second, dissimilar column.

4.3.11.3 GC/MS confirmation can be performed if the concentration of the analyte is high enough to be detected by the mass spectrometer.

4.3.11.4 If it is determined that the laboratory's identification is incorrect or cannot be confirmed, contact with the laboratory may be necessary to have the data corrected and resubmitted or additional supporting data supplied. If a discrepancy remains, use professional judgment to qualify (tentatively identified: N) or reject and/or document the issue in the validation report.

4.3.11.5 Inspect samples analyzed following a high concentration sample to verify that no carryover occurred and the analyte was not reported as a false positive in the following sample(s). If carryover is suspected, ddms procedure is to contact the laboratory for explanation and resolution. If resolution is not achieved from the laboratory's response, contact other ddms chemists for discussion and decisions on qualification of data.

4.3.12 Compound Quantitation

4.3.12.1 If the concentration for an analyte was outside the calibration range in the undiluted analysis but was reported as a non-detect in the diluted analysis, evaluate the raw data or refer to historical data, if available, to determine if the non-detect in the reanalysis was a result of over dilution or if the analyte was a false positive or high-biased in the initial, undiluted analysis. If the non-detect in the reanalysis was a result of over dilution, it is ddms procedure to accept the result from the initial, undiluted analysis as the reportable concentration and qualify this result as estimated (J) because it was detected above the calibration range. If the result in the initial, undiluted analysis is determined to be a false positive, reject (R) the result in the initial analysis.

4.3.12.2 Toxaphene may be quantified using the total area between RTs of the first and last eluting toxaphene components in the standard or using the areas 4 to 6 major peaks. If the total area is used, the concentration is calculated with the CF for the total area in the associated IC. If using individual peaks, the concentration of each selected peak is calculated with the CF for that peak from the associated IC, and the average concentration of all selected peaks is

reported. Calculations performed must be consistent with those used for the calibration standards.

4.3.12.3 Chlordane may be quantified using the total area or the areas of 3 to 5 major peaks. If the heptachlor epoxide peak in the sample is relatively small, it is included as part of the total area. If large peaks are present for heptachlor and/or heptachlor epoxide, their concentrations are calculated separately, and the areas are subtracted from the total area. A third option for chlordane is to quantitate the cis-chlordane, trans-chlordane, and heptachlor peaks separately and report individual results for these analytes.

4.3.12.4 For Aroclors, the area of each selected peak is quantified using the CF for that peak from the associated IC, and the average concentration of all selected peaks is reported. Concentrations of all selected peaks are expected to be approximately the same for each selected Aroclor. Note that in some cases, not all selected peaks for an Aroclor in a field sample are quantified by the laboratory. Inspect the chromatogram to determine if an interference is present at the applicable retention time or if the peak is not detected. Since each Aroclor includes numerous chromatographic peaks, having a selected peak not detected or not quantified due to an interference is not typically an issue, as long as at least three of the selected peaks are used for quantitation. Use PJ to determine the appropriateness of the laboratory's approach in this situation.

4.3.12.5 If dual column analyses were performed, verify that the appropriate concentration was reported. The Contract Laboratory Program Statement of Work specifies that the lower of the two concentrations be reported, while SW-846 specifies that the higher of the two concentrations be reported. In other cases, the analyst and/or laboratory QC reviewer may use PJ to determine that the method-specified concentration should not be reported. Use PJ to determine if laboratory's approach should be accepted.

4.3.13 Column Agreement in Dual Column Analysis

4.3.13.1 The concentrations determined for the two analytical columns are compared and evaluated against the applicable acceptance criterion. Where column agreement is unacceptable, determine whether or not interferences are present which affected the quantitation. If the acceptance criterion is exceeded, the result is qualified as estimated (J). Where interferences are observed, professional judgment should be applied to determine if qualification is warranted. In the absence of an acceptance criterion, ddms' acceptance criterion is ≤25%D.

4.3.14 Reported Quantitation/Reporting Limits (QLs or RLs)

4.3.14.1 Verify that sample QLs or RLs are supported by the data provided. The RLs must be equal to or greater than the lowest concentration used to establish the calibration. If low points were dropped for any of the target analytes in the IC, verify that the RLs have been adjusted for the samples to take this into account. If dilutions have been performed, or non-nominal sample volumes/masses have been used, ensure that the QLs or RLs have been adjusted accordingly. When results are reported from multiple extractions or injections, QLs/RLs should reflect the appropriate value for each analyte.

4.3.14.2 Any RLs not supported by the initial calibration must be recalculated and corrected by the validator for all associated samples.

4.3.14.3 Correct non-detects reported at the MDL to non-detected at the RL. Because the RLs (or lowest concentration used to establish calibration) are the lowest values supported in the data package, MDLs should not be used to report non-detects. Results reported between the RL and the MDL should be qualified by the laboratory as estimated (J) because their accuracy is not supported by the calibration. If the MDL has been used to support non-detects at a project action limit that was not attainable with the laboratory's calibration, this should be noted in the validation report.

5.0 **Documentation** - Refer to SOP ECS-SOP-012-Rev.0– General DV: "Validation and Review of Analytical Data."

6.0 **Preparation of Reports** - Refer to SOP ECS-SOP-012-Rev.0– General DV: "Validation and Review of Analytical Data."

Standard Operating Procedure:

Validation and Review of Analytical Data

1.0 Scope and Application

Data validation determines the technical usability of analytical data. It is a process by which a body of data is evaluated to determine validity and usability. The process will include comparison to a set of acceptance criteria, evaluation of method adherence, suitability of methodology for the intended use, sample integrity, data completeness, and other parameters which affect the technical veracity of the reported results. An objective, unbiased report is prepared to document the degree of compliance to the intended purpose, the usability of the data, and any issues observed that may impact the reliability of the results. Criteria used by ddms may be more stringent than those outlined in data review guidelines and/or reference methods. Criteria used may represent those stipulated in a project-specific or program-specific quality assurance project plan (QAPP) or other documented requirements. The focus of validation is always on the technical usability and defensibility of the data. Available validation guidance may be consulted, and the validator's professional judgment and experience are used, when applicable, to make decisions on the data usability.

2.0 Summary

This standard operating procedure (SOP) details the process of evaluating and verifying analytical data, comparing it to stated Data Quality Objectives (DQOs) and acceptance criteria, and preparing a technically sound report that provides the data user with a reliable basis for decision making. Extensive laboratory knowledge, knowledge of published methods, and the ability to apply consistent professional judgment are essential. This SOP should be used in conjunction with the site-specific QAPPs and sampling and analysis plans (SAPs), data review guidelines, if applicable, parameter-specific ddms SOPs, and analytical methods. This SOP includes reliance on the professional judgment of ddms validators. Qualification of data is based on the most conservative criteria in order to be protective of human health and the environment.

This SOP provides a framework for the validation process that follows the process order and structure of Contract Laboratory Program (CLP)-like validation described in the EPA National Functional Guidelines (National Functional Guidelines are applicable to CLP only) and provides non-parameter-specific guidance only. Individual ECS SOPs, reference methods, and other applicable references must be consulted for detailed guidance and requirements for specific analytical parameters. The validator is encouraged to refer to other references as needed, especially when use of professional judgment is required for decisions on qualification of results. When these supplemental resources are used to form the basis for qualification of results, the

validator will include such references in the final validation report to clarify and support decisions made.

All data have the potential to go to litigation. Part of the validation process is to identify any data and/or documentation issues that could be problematic if the data are used in litigation. All documents are discoverable during litigation, including validation/review reports and validator notes and mark-ups. Any issues identified during the data review process must be clearly identified in the report.

When only an evaluation (e.g., Stage 2A or Stage 2B review) of the data is requested, the procedure outlined in this SOP is followed, but calculations are not performed. Only a review of the data summary forms is performed, unless that review reveals critical quality issues.

When verifying calculation of sample results, not all regions specify an acceptable %D between laboratory-reported and validator-calculated values. It is ddms' professional judgment to contact the laboratory when discrepancies greater than 10% occur. The error term of 10% is considered to be allowable experimental error in general laboratory practice.

3.0 Acronyms

BAL – Blank Action Level
BFB – Bromofluorobenzene
BS/BSD – Blank Spike/Blank Spike Duplicate
CC – Continuing Calibration
CCB – Continuing Calibration Blank
CCV – Continuing Calibration Verification
CF – Calibration Factor
CLP – Contract Laboratory Program
COC – Chain of Custody
DFTPP – Decafluorotriphenylphosphine
DOD – Department of Defense
DQA – Data Quality Assessment
DQO – Data Quality Objective
DUE – Data Usability Evaluation
DUSR – Data Usability Summary Report
DV – Data Validation
EB – Equipment Blank
EDD – Electronic Data Deliverable
EPA – Environmental Protection Agency
FB – Field Blank
FD – Field Duplicate
GC – Gas Chromatography
GC/MS – Gas Chromatography/Mass Spectroscopy

IB – Instrument Blank
IC – Initial Calibration
ICB – Initial Calibration Blank
ICV – Initial Calibration Verification
IS or ISTD – Internal Standard
LC – Liquid Chromatography
LCS/LCSD – Laboratory Control Sample/Laboratory Control Sample Duplicate
LIMS – Laboratory Information Management System
MB – Method Blank
MDL – Method Detection Limit
MRL – Method Reporting Limit
MS – Mass Spectrometry
MS/MSD – Matrix Spike/Matrix Spike Duplicate
NFG – National Functional Guidelines
PB – Preparation Blank
PE – Performance Evaluation
PJ – Professional Judgment
PT – Proficiency Test
RF – Response Factor
RL – Reporting Limit
RPD – Relative Percent Difference
RRF – Relative Response Factor
RRT – Relative Retention Time
RSD – Relative Standard Deviation
RSE – Relative Standard Error
RT – Retention Time
QA – Quality Assurance
QAPP – Quality Assurance Project Plan
QC – Quality Control
SAP – Sampling and Analysis Plan
SDG – Sample Delivery Group
SOP – Standard Operating Procedure
SURR – Surrogate Standard
TB – Trip Blank
TIC – Tentatively Identified Compound
TCL – Target Compound List
%D – Percent Difference or Percent Drift
%R – Percent Recovery

4.0 Project Specific Requirements

4.1 Project Specific Documents: Before beginning the validation process, it is important to identify and understand the project-specific documents. These documents may include, but are not limited to:

- QAPPs
- SAPs
- Laboratory SOPs
- Applicable EPA regional and state data validation guidelines
- Other agency guidelines (i.e., EPA National Functional Guidelines, DOD Quality Systems Manual, etc.)
- Published analytical reference methods
- Field notes (well logs, boring logs, sampler field logs, etc.)

QAPPs and SAPs should include pertinent information, such as required sample collection techniques, required parameters and analyte lists, and required quantitation limits. Laboratory SOPs can be used to determine whether laboratory procedures used to analyze the samples comply with project requirements, as well as with the reference method. The individual documents which are available and the extent to which these documents apply will vary from project to project. However, a thorough understanding of the applicable data validation guidelines or other governing agency requirements is essential before beginning the data evaluation process.

The requirements of the QAPPs and SAPs take precedence during the data review process. The criteria set forth in the applicable EPA or other agency data review guidelines are then applied. The analytical method and agency guidelines are consulted; however, ddms professional judgment is used when determining the need for data qualification. When a data quality situation occurs that has not been addressed in this SOP or the parameter-specific ddms SOP, it warrants discussion with the other ddms chemists for a uniform decision. Professional judgment should always be applied as necessary and appropriate. There may be instances where ddms PJ is in conflict with guidelines or QAPP requirements. These must be discussed internally and documented in the report, whether they result in qualification of results or not, as they may potentially affect the usability or defensibility of the data.

NOTE: Any incomplete, missing, or unclear information discovered during the validation process warrants contacting the laboratory for clarification and/or correction. This must be documented in the validation report, along with the laboratory's corrective action. If corrective action was not required or not possible, this must also be documented in the validation report. If the deadline to complete the validation is approaching and a rapid response is needed, communicate that information to the laboratory. In any event, it is reasonable to expect a response from the laboratory within 48 hours. Contact the laboratory again if no response has been received after 48 hours. It may be appropriate to contact the client to determine whether or not to complete the report without the requested information. If revisions or requested

data/documentation are not received by the time the report is released, this is also noted in the validation report.

4.2 Identify the level of review required. Typical validation/evaluation/review levels are shown in the table below.

TABLE 1 – Levels of Data Evaluation and Validation with Parameters Assessed

QC Component Reviewed and/or Verified	Stage 4	Stage 3	Stage 2B	Stage 2A
Chain of Custody	X	X	X	X
Requested Method Performed	X	X	X	X
Project-Specific Analyte Lists	X	X	X	X
Reporting Limits Meet Project-Specific Limits	X	X	X	X
Sample Related QC				
Holding Times	X	X	X	X
Sample Receipt Conditions	X	X	X	X
Preservation	X	X	X	X
Surrogates	X	X	X	X
Method Blanks	X	X	X	X
Field-Related Blanks	X	X	X	X
Laboratory Control Samples/Laboratory Control Sample Duplicate (LCS/LCSD)	X	X	X	X
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	X	X	X	X
Field and/or Laboratory Duplicates	X	X	X	X
Analyte Identification Verified using Provided Mass Spectra	X	---	---	---
Chromatograms	X	---	---	---
Total vs. Dissolved Concentrations	X	X	X	X
Recalculate Reported Concentrations	X	X	---	---
Instrument Related QC				
Instrument Tunes	X	X	X	---
Internal Standards	X	X	X	---

TABLE 1 – Levels of Data Evaluation and Validation with Parameters Assessed				
QC Component Reviewed and/or Verified	**Stage 4**	**Stage 3**	**Stage 2B**	**Stage 2A**
Initial and Continuing Calibrations - Response Factors - %RSD or %D, as appropriate - Fit and Appropriateness of IC Curve Used	X	X	X	---
Calibration Blanks	X	X	X	---
Recalculate Instrument Related Reported Values	X	---	---	---

4.3 Identify the validation guidelines to be used. Links to current validation guidance are provided in Appendix A. Guidance documents are also available at: R:\Company Resources\Group Admin\ECS\Validation Guidance Documents.

4.4 Identify the validation qualifiers to be applied. These are typically defined in the applicable validation guidance document. EPA qualifier definitions are provided in Appendix B.

4.5 Identify the report format to be used. ddms reports are discussed in Section 6.0 of this SOP, and links to several example report formats are provided in Appendix C.

4.6 Identify the laboratory deliverables that are required. Types of laboratory deliverables are described in Appendix D. These requirements may be specified by the regulatory agency or site-specific QAPP.

4.7 For new projects, the Project Kick-Off Form may be completed and included in the project folder. A link to this form is provided in Appendix E.

5.0 Procedure for Review, Verification and Evaluation of Analytical Data

5.1 Sample Preservation and Holding Time

5.1.1 Condition and Custody

Verify that sample conditions between collection and receipt at the laboratory protected the integrity of the samples, including temperature, chemical preservation, proper containers for the parameters requested, and condition of samples and containers (intact, no or limited headspace for volatile components or those affected by exposure to oxygen, protection from light for photosensitive components, leaking containers, frozen samples for biological components, etc.). If conditions were not met, apply qualifiers as recommended by the reference method, validation guidance, or project-specific requirements. Use PJ as appropriate, where necessary.

Ensure that the COC documentation supports secure transport, handling, and storage of the samples from time of collection, throughout any intermediate

transfers and/or storage, to receipt at the laboratory. If samples require internal laboratory COC records, ensure that these are included in the data package or can be produced by the laboratory in a fashion that provides legally defensible custody records from receipt at the laboratory to the production of the analytical result. It may be necessary to obtain field records to support that stage of sample custody, if COC records and laboratory login documentation are incomplete or unclear.

Verify that the temperature of the samples in the cooler(s) upon receipt at the laboratory was documented and was acceptable. (Programs may stipulate criteria such as 4 °C ± 2°C, <6°C but not frozen, or <10°C, but not frozen). Generally, if the temperature was between 0°C and the lower limit of the acceptable range, no qualification is made, but it is documented in the validation report. If the temperature of the cooler was outside the upper range, qualify based on appropriate data validation guidelines, or apply PJ to determine whether qualification is warranted.

Note: Freezing is appropriate for some analytical parameters and/or sample matrices but may have adverse effects for others. Follow method and/or program guidance when samples are received at temperatures below freezing.

Most air samples, metals and radiological analytes, and some other analytical parameters and sample types do not require refrigeration or have other, very specific, handling and storage requirements; consult reference methods and program guidance for proper handling.

In the absence of regional validation guidelines regarding temperature excursions, apply ddms professional judgment. If the receipt temperature of the samples exceeds the acceptance criteria, but it is noted that the samples were received “on ice”, review the documentation to verify whether or not there was sufficient time (nominally four hours) from sample collection to laboratory receipt for the samples to have cooled. No qualification of data is necessary, but it is noted in the DV report. If the elapsed time from sample collection to laboratory receipt exceeds four hours, use professional judgment to determine if results should be estimated (J, UJ).

5.1.2 Holding Times

In the absence of method-defined actions or validation guidelines regarding holding time excursions, apply ddms professional judgment:

- Qualify sample results as estimated (J, UJ) for samples prepared and/or analyzed outside the holding time.

- Reject (R) non-detect sample results and estimate (J) detected results for samples prepared outside of two times the holding time.
- Reject (R) non-detect sample results and estimate (J) detected results for samples analyzed more than 60 days after preparation.

Note: The above is general guidance only and may not be applicable to all situations.

For analyze-immediately parameters (i.e., pH, dissolved oxygen, oxygen reduction potential, ferrous iron, etc.), if the analysis was not performed within 15 minutes of sample collection, results are qualified as estimated (J, UJ).

If both total metals and dissolved metals are to be analyzed, a separate aliquot of sample should be filtered and acid preserved in the field as soon as possible after collection. If samples were filtered and preserved or preserved and filtered after receipt at the laboratory, results are qualified as estimated (J, UJ) as there is no certainty that metals concentrations remained unchanged (due to dissolution, precipitation, adsorption, etc.) between the time of sampling and time of preparation and analysis.

If samples for parameters requiring preservation are received by the laboratory insufficiently preserved or unpreserved, the laboratory must add preservatives on receipt and follow method-specific requirements (e.g., adding nitric acid to metals samples and waiting 18-24 hours before preparing for analysis). Ensure that such requirements were followed. If not, qualify based on guidance or PJ, as appropriate. This is general guidance only and may not be applicable to all situations.

5.2 Measurement system performance – Method requirements and/or program requirements must be consulted for applicable criteria. Where criteria are not available, PJ must be used to determine whether reported results represent good laboratory practice and sound analytical performance. Qualifiers are applied based on published requirements, or PJ, as applicable.

5.2.1 Selectivity – It must be demonstrated that the measurement system can accurately identify the target analyte(s) and that potential interferences are recognized and will be noted in the documentation, if encountered. Acceptable calibration documentation and other documentation of reference materials analyses (blank and matrix spikes, interference check samples, GC and MS performance evaluation mixtures, and isomer and/or homolog resolution/window mixes, etc.) are examples of supporting data for instrument selectivity.

5.2.2 Sensitivity – It must be demonstrated that the measurement system can detect and/or quantitate the target analyte at demonstrated concentration levels used for reporting. (Project requirements may also be evaluated with respect to the laboratory's ability to meet them.) Low-level standards or spikes, RL verification

standards, documentation of analyte MDLs, etc., are data that can be used to assess instrument or measurement system sensitivity.

5.2.3 Verification of contamination-free measurement system – This is demonstrated by analysis of a variety of laboratory-generated blanks (Instrument blank [IB], initial calibration blank/continuing calibration blank [ICB/CCB], method blank [MB], preparation blank [PB], etc.) which are dependent on the parameter, instrumentation, and methodology.

5.2.4 Performance Checks – This quality element demonstrates, with each day/sequence/analytical event, that the measurement system continues to allow accurate identification and quantitation of the target analytes. It is an assessment of stability of analytical conditions and may assess chromatographic resolution (gas chromatography [GC], liquid chromatography [LC], ion chromatography [IC], etc.) taking the form of peak shape and retention time, and peak resolution (valley), mass calibration and resolution (mass spectrometry [MS]), and/or background noise. Continuing calibration standards and reference material spikes can also be used in many situations to determine continued system performance.

5.2.5 Batch / Sequence / Analysis Time Period – Many methods specify a required frequency for calibration and/or system performance verification. Samples must be analyzed within this time period in order for the results to be supported by the laboratory's documented performance. Use published criteria or PJ to determine qualification of results, if applicable.

5.3 Calibration

5.3.1 Initial Calibration

5.3.1.1 Reference materials used for calibration and performance checks must be traceable to certified sources of authentic target analytes for proper identification. Where second-source standards are required, verify that those have been used appropriately. When traceability of reference materials is not included, the validator may request these from the laboratory, if appropriate.

5.3.1.2 When validating non-standard methods, modified methods, or new methods that have not become well-established, the traceability of the standards may be essential for support of the results. Use PJ to determine whether or not supporting data is adequate to provide defensibility of analyte identification.

5.3.1.3 Determine the relation of standard preparation to the sample preparation (i.e., same reagents used, extracted/not extracted, derivatization, etc.). These will be method and matrix dependent.

5.3.1.4 Determine the model of calibration established. This model may be method-specified, there may be multiple options, and there may be

a hierarchy recommended. The initial calibration is the model established, using known concentrations of the analytes, to be used for the determination/calculation of unknown concentrations in samples. Consult method or program criteria for compliance.

5.3.1.5 Models most commonly used are average response factors (RFs) or calibration factors (CFs), and for these percent relative standard deviation (%RSD) is used to determine linearity. Consult the applicable parameter-specific ddms SOP for acceptance limits. If the %RSD in the IC is greater than the maximum RSD specified, determine the cause of non-linearity. If the curve is acceptable after eliminating the high point of the curve and recalculating the %RSD, accept positive detects in the linear portion of the curve, estimate (J) positive detects at the high end of the curve, and accept non-detects. If the %RSD is less than the maximum RSD specified in the method after eliminating the low point on the curve, accept positive detects in the linear portion of the curve and estimate (J) positive detects in the low portion of the curve. In the absence of regional qualification guidelines, ddms practice will be to raise the RL to the lowest concentration at which linearity was demonstrated.

5.3.1.5.1 Ensure that the laboratory has not used the "grand mean" approach to determine compliance with calibration criteria.

5.3.1.5.2 The use of marginal exceedance requires careful evaluation to determine if the individual project analytes exhibit acceptable calibration.

5.3.1.6 Linear regression is the second most commonly used model and uses correlation coefficient or R-squared to determine acceptable linearity. Follow method requirements and criteria. Use PJ where appropriate. If a linear regression was used to establish calibration, the low point of the calibration must be re-calculated against the curve and must produce an analyte concentration within 30% of the true value. If this criterion is not met, sample results at the low end of the curve will be qualified as estimated (J).

5.3.1.7 In situations where the average CF or RF was not used and the laboratory used an alternate calibration model, such as a linear regression or quadratic equation, follow the procedure outlined in Section 5.3.1.5. to determine if all or some portion of the model can be used to produce usable results. Use PJ to determine which, if any, results should be qualified (i.e., positive results may warrant qualification as estimated (J) and non-detects as estimated, rejected, or not detected at an elevated RL depending on the position of the

outlier[s] in the model). Discussion with other ddms chemists is recommended for these instances. A calibration solver worksheet is available at R:\Company Resources\SOPs\In Progress\ECS.

5.3.1.8 In situations where the instrument response was not linear, the laboratory may have used an alternate calibration model, such as a quadratic equation. Quadratic equations require a minimum of six unique concentration points. Follow method requirements and criteria. A quadratic equation should be used only as a last resort, since the curve can result in a very high bias at the low or high ends of the calibration range. If a quadratic equation was used for an analyte that typically does not require one, review the data carefully to ensure that the curve for that analyte is acceptable.

5.3.1.9 Many methods require a read-back, or re-calculation, of analyzed standards against the calibration models used. Relative standard error (RSE) or percent error is determined and must meet method or laboratory-established requirements to support results calculated against the model. If no guidance is available, ddms default criteria are ±50% for the lowest level of the calibration and ±30% for all other levels.

NOTE: The IC validity affects ALL analyses performed in association with the IC.

5.3.2 Initial Calibration Verification (ICV) – This quality element is an independent, second-source reference standard intended to verify the concentrations of the prepared IC standards. If the second source does not match the IC, the laboratory must investigate the discrepancy and resolve the issue before analyzing samples. Where possible (if the data package includes reference standard traceability documentation), verify that the ICV was a second-source standard. ddms acceptance criteria for the ICV standard are provided in the parameter-specific ddms SOPs. If the recovery of any analyte falls outside of acceptance criteria, estimate detects (J) and non-detects (UJ).

NOTE: The IC/ICV validity affects ALL analyses performed in association with the IC. In the absence of documentation of the sources of the IC and ICV standards, assume that a second source was used. If required to support data for a regulatory or legal application, the laboratory may be able to provide documentation to support the IC, if they participate in a PE program or use internal vendor-supplied PTs, etc. If such documentation is needed, request, review, and determine validity, using PJ.

5.3.3 Continuing Calibration

5.3.3.1 Verify that the continuing calibration (CC) standard(s) was/were analyzed at the appropriate frequency (examples are 12- or 24-hour sequences, every 10 injections of samples, etc.) and concentration(s). Most CCs are analyzed at a mid-point concentration. Some methods and/or projects require analysis of a low-level standard or standard at the quantitation limit to verify continued sensitivity of the measurement system for all sample analyses. Some methods require alternating concentrations of the CC.

5.3.3.2 Verify that the CC was compared against the most recent IC. If this is not the case, contact the laboratory to request an explanation and correction for the CC and associated sample results. If such correction cannot be provided by the laboratory or is not provided in time for completion of the project deadlines, results must be qualified as appropriate. Use PJ and assessment of all other available data to determine appropriate course of action. Results may be rejected as unusable in some cases.

5.3.3.3 When the percent difference (drift) or percent recovery is outside the applicable limit, qualify detects as estimated (J). If the shift represents an increase in sensitivity, non-detects are not qualified. If the shift represents a decrease in sensitivity, qualify non-detects as estimated (UJ). For excessively high or low responses (e.g., %D >100%), consult other ddms chemists for further action.

5.3.3.4 Other continuing calibration verifications - Some methods have specific requirements for additional verifications. Refer to the reference method, lab SOP, and program- or project-specific requirements for evaluation and qualification of data.

5.4 Blanks - Blanks are intended to demonstrate contaminant-free conditions for sample collection, handling, and storage. General guidance is provided below; refer to the parameter-specific ddms SOPs for detailed guidance.

5.4.1 Laboratory Blanks - Laboratory blanks include, but are not limited to, instrument blanks, method blanks, preparation blanks, and storage blanks.

5.4.1.1 A method or preparation blank must be prepared and/or analyzed as specified in the analytical method.

5.4.1.2 Discussion with other ddms chemists may be necessary to make decisions on qualification of sample results when on-column concentrations of common laboratory contaminants are consistent from sample to sample, but the contaminant is not present in the laboratory blank(s).

5.4.1.3 A potential scenario: Samples are analyzed for volatile organics on two different instruments on the same day. On one of the instruments, a common laboratory contaminant (such as acetone or methylene chloride) is reported in the MB, and on the other instrument the same analyte in the MB is not detected. The sample results on both instruments were less than the blank action level (BAL) on the instrument with the contaminant. ddms' professional judgment is to qualify the results from the analytical runs on both instruments as not detected (U) at the sample-specific RL or reported value, whichever is greater.

5.4.1.4 Instrument blanks may be analyzed as a part of the analytical calibration or start-up sequence. Instrument blanks should also be analyzed following a sample that contained high concentrations of a particular analyte(s) to identify if instrument carryover is present. However, since most samples are analyzed on unattended autosamplers, instrument blanks may not have been run in these situations. If a blank was not analyzed, inspect the sample(s) analyzed following the high concentration sample to verify that no carryover occurred and that analyte(s) were not incorrectly reported in the subsequently-analyzed sample(s). ddms' PJ is to contact the laboratory for explanation and resolution. If the validator's opinion differs from the laboratory's response, contact other ddms chemists to discuss interpretation of the data and any recommended qualification of data.

5.4.2 Field-submitted Blanks - Field-submitted blanks include field blanks, rinse blanks, equipment blanks, trip blanks, and others where applicable to the methods/matrix sampled.

5.4.2.1 The model for application of field-submitted blanks to field samples should be determined before the project initiation and documented on the Project Kick-Off Form (Appendix F). Where this information is not available, it may be necessary to contact the project manager or other personnel for specifics of field-submitted blank collection to determine how contamination may impact field samples.

5.4.2.2 Be aware that some Regions do not qualify data based on field blank results. ddms' SOP is to apply such qualification for samples of the same matrix where no indication contrary to this practice is present. It is not ddms' policy to qualify soil data based on associated field blank results, without consultation with other ddms chemists.

5.4.2.3 In another scenario, the BAL in the MB is less than that of the FB BAL. The sample result is greater than the MB BAL, but less than the FB BAL. Even if the governing agency specifically states that data not be

qualified due to FB results, based on ddms professional judgment, results would be qualified as not detected (U) at the sample-specific RL or reported value, whichever is greater, based on the high FB concentration.

5.4.3 If a target compound is detected in more than one type of blank (i.e. MB, TB, FB, RB, etc.) the BAL is calculated from the highest concentration blank.

5.4.4 Typically, only sample results are qualified based on blank contamination and FB, TB, EB, etc., results are not qualified. However, if it is apparent that, for example, analytes present in field-submitted blanks resulted from laboratory contamination, field-submitted results may be qualified based on professional judgment, or it may be noted in the validation report that analytes present in the field-submitted blanks are also present in the laboratory blank(s).

Note: Current EPA National Functional Guidelines specify that a BAL of one to two times the associated blank concentration be used to quality associated sample results greater than the contract required quantitation limit. Per these guidelines, professional judgment is applied when associated sample concentrations are greater than that BAL. ddms' professional judgment typically is to use a multiplier of five times the associated blank concentration as the BAL. Refer to the parameter-specific ddms SOPs for guidance.

5.5 Laboratory Control Sample/Duplicate - A Laboratory Control Sample (LCS) and Laboratory Control Sample Duplicate (LCSD) pair is a blank spike and spiked duplicate pair analyzed to demonstrate the laboratory's recovery and precision in an ideal situation, without any potential interference from the site sample matrix/matrices. The LCS or LCS/LCSD pair applies to all samples in the preparation batch or analysis batch, where applicable (i.e., where calibration is matrix-specific, or where there is no preparation for the analysis). Verify that an LCS was prepared and analyzed as appropriate to the method. Upper and lower acceptance limits are provided in the parameter-specific ddms SOPs. Actions taken due to unacceptable LCS or LCS/LCSD results are as outlined below:

5.5.1 If the recovery of an analyte is greater than the upper limit, estimate (J) detects for that analyte in the samples associated with that LCS, indicating potential high bias. Non-detects are not qualified.

5.5.2 If the recovery of an analyte is less than the lower limit but ≥10%, estimate (J) detects and estimate (UJ) all non-detects in all samples associated with the LCS, indicating potential low bias.

5.5.3 If the recovery of an analyte is less than 10%, estimate (J) detects and reject (R) all non-detects in samples associated with that LCS.

5.5.4 When the laboratory has analyzed an LCS/LCSD pair, the RPD between the paired results should be less than 20%. Qualify results as estimated (J) for all samples in the associated analytical batch when the RPD exceeds 20%. When the RPD

exceeds 40%, discussion with other ddms chemists is necessary for qualification of data.

5.6 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

5.6.1 Some regions specify qualification only if both the MS and MSD fall outside of criteria. It is ddms PJ to qualify data if either the MS or MSD fail to meet acceptance criteria.

5.6.2 Document in the report if a blank or non-site-specific sample was used for the MS/MSD analyses. No data are qualified when a non-site-specific sample is used for the MS/MSD analyses.

5.6.3 The recovery of the MS/MSD analytes should be within ddms professional judgment acceptance criteria of 70-130% (or method criteria, where more stringent). Data are qualified when the %Rs fall outside of these limits to alert the data user of potential bias.

5.6.4 If the recovery of any analyte is less than 70%, but ≥10% recovery, estimate (J) all positive detects and estimate (UJ) all non-detects, indicating potential low bias of sample results.

5.6.5 If the recovery of any analyte is less than 10%, estimate (J) all positive detect and reject (R) all non-detects.

5.6.6 If the RPD is greater than 30% for aqueous samples or greater than 50% for soil samples, estimate (J) positive detects and estimate (UJ) non-detects.

5.6.7 Where the reported concentration in the unspiked (parent) sample is greater than four times the spike concentration, data are not qualified based on the MS/MSD percent recoveries. A comparison of the three determinations (parent, MS, and MSD recoveries) may be performed, if appropriate. Use professional judgment to evaluate precision and/or contact other ddms chemists for input.

NOTE: When applying qualifiers based on MS/MSD recoveries, qualification is made to all samples of the same matrix. Determination of decision units (DUs) or other site-specific associations should be made and documented before project initiation. See the Project Kick-Off Form in Appendix F.

5.7 Laboratory Duplicate Analysis

5.7.1 Some methods require analysis of laboratory duplicates. In some cases, the laboratory will perform laboratory duplicate analyses not required by the method if required by their own SOP or by project or program requirements.

5.7.2 Refer to the method or program requirements for laboratory duplicate precision criteria. In the absence of method- or program-specified limits, ddms' PJ is to qualify associated aqueous sample results if the RPD is greater than 30% and to qualify associated non-aqueous sample results if the RPD is greater than 50%.

5.8 Field Duplicate Samples - Where there is one set of field duplicates (FDs) per day, only the samples collected on the same day will be qualified based on any FD excursions. Where one FD is associated with an entire sampling event, all samples for that event will be qualified based on the FD exceedances. In the case of a multi-day sampling event where FDs are not collected on each day of sampling, consult other ddms chemists for discussion of how to associate FD results to sample results. Qualification of sample results due to FD duplicate excursions is based on like matrices (e.g., groundwater, storm water, soil, sediment, etc.).

5.8.1 If both results of the FD pair are greater than five times the RL, field duplicate precision is evaluated using the calculated RPD.

5.8.1.1 Aqueous – ddms PJ is to qualify results when the RPD exceeds 30%.

5.8.1.2 Non-aqueous – ddms PJ is to qualify results when the RPD exceeds 50%.

5.8.1.3 Qualify as estimated (J) if the result in the associated sample is greater than five times the RL.

5.8.1.4 Qualify as estimated (J) if the result in the associated sample is less than five times the RL but is a positive result.

5.8.2 If both results of the FD pair are less than five times the RL, qualify results estimated (J) if the difference between the two results is greater than two times the RL.

5.8.2.1 This applies to aqueous and non-aqueous samples and assumes the same sampling methodology has been employed for each matrix.

5.8.2.2 If an associated sample result is less than five times the RL, qualify the result as estimated (J). If the associated sample result is greater than five times the RL, use professional judgment.

5.8.3 If one FD result is positive but less than two times the RL and the other FD result is not detected, estimate both results (J, UJ). Qualify all associated sample results less than two times the RL, including non-detects, as estimated (J, UJ).

5.8.4 If one FD result is positive and greater than two times the RL and the other FD result is not detected, consult with other ddms chemists for guidance. Consider rejection of data.

5.8.5 If the concentration in each sample of the FD pair is greater than five times the RL and the RPD is greater than 100%, consult other ddms chemists for guidance.

5.8.6 If both FD results are low (i.e., both less than two times the RL or one not detected) and the analyte concentration in the associated sample is high, no qualification of the associated sample result is necessary.

5.8.7 If multiple FD pairs are reported with conflicting RPDs (some acceptable, some not), qualify all associated sample results following the criteria defined above except in the paired FD samples where the RPD is acceptable.

5.8.8 Where appropriate due to dilution factor impact, assess on-column concentrations.

5.8.9 Where an FD result is qualified as not detected due to blank contamination and the paired sample is unqualified, it may be appropriate to use presumptively present (N) when qualifying sample results.

5.9 Surrogates / Labeled Analogs / Clean-up Recovery Spikes – These are non-project target analytes (may be isotopically labeled) added to the field and QC samples before preparation, carried through the preparation, and analyzed to demonstrate the laboratory's ability to recover the target analytes. Refer to the parameter-specific ddms SOP for acceptance limits.

5.9.1 If the recovery of a surrogate is greater than the upper limit, estimate (J) detects for the analyte(s) associated with that surrogate, in the sample, to indicate the potential for high bias. Non-detects are not qualified.

5.9.2 If the recovery of a surrogate is less than the lower limit but ≥10%, estimate (J) detects for all associated analytes in the sample and estimate (UJ) all associated non-detects, indicating potential low bias.

5.9.3 If the recovery of any one surrogate is less than 10%, associated positive results are estimated (J) and non-detect results are rejected (R).

5.9.4 If two or more surrogates are outside of acceptance criteria for a single fraction, all analytes are qualified accordingly.

5.9.5 Some methods require additional and/or specific recovery spikes for individual steps of the methodology (i.e., clean up standard for dioxins/furans analyses). These must be evaluated for the applicable samples. Use ddms parameter-specific recovery criteria and PJ to evaluate.

NOTE: If there is a greater than 10 times dilution factor or if the dilution factor causes surrogate concentrations to be below the RL, results are not qualified based on %Rs of surrogates.

5.10 Internal Standards (ISTD) / Recovery Standards / Injection Standards – These are non-target analytes spiked just before analysis to provide a common reference point across all of the standards, field samples, and QC samples. The criteria for ISTD response are method, SOP, and program specific. Refer to the applicable guidance and parameter-specific ddms SOPs for criteria.

5.11 Target Analyte Identification - Assess whether or not method or program criteria were met for target analyte identification. Additional information is provided in the parameter-specific ddms SOPs. In the absence of published criteria, use PJ, as appropriate.

5.11.1 Professional judgment is often required to determine if a correct analyte identification was made. If it is determined that the identification is incorrect, contact with the laboratory may be necessary to have the data corrected and

resubmitted. If a discrepancy remains, use professional judgment to qualify or reject and document in validation report.

5.11.2 Determine whether the supporting data indicate the potential for false negatives or positives and qualify if warranted. If indicated, false positives should be qualified as tentatively identified (N) or not detected (U) or U at an estimated value (UJ). If false negatives are likely, results are qualified as estimated (UJ). In cases where support for identification is absent or inadequate, results may require rejection (R) as unusable. It should be noted that some methods (microbiological methods, for example) include only positive and negative controls to demonstrate the potential for false positives or negatives.

5.12 Tentatively Identified Compounds (TICs)

5.12.1 If not provided in the project documents, confirm with the client that an evaluation of the TICs is a requirement of the data validation effort. If the validator disagrees with the tentative identification provided by the laboratory, revise the identification on the Form 1 and document the correction in the validation report. Where a specific compound or isomer has been identified by the software, it is often advisable to revise the identification to “unknown”, or unknown class (e.g., “unknown alkane”, etc.) to improve the defensibility of the data.

5.12.2 Verify that the TICs reported are not surrogates, ISTDs or missed target analytes.

5.12.3 Any TICs identified that are common laboratory contaminants or artifacts and are not true sample components should be discussed in the validation report. These should be removed from the TIC summary form and any electronic data deliverable if applicable.

5.13 Analyte Quantitation

5.13.1 Verify that detected analytes were properly quantified as specified in the analytical method.

5.13.2 If the concentration for an analyte was out of the calibration range in the undiluted analysis, but was reported as a non-detect in the diluted analysis, evaluate the raw data or refer to historical data, if available, to determine if the non-detect in the reanalysis was a result of over dilution or if the analyte was a false positive in the initial, undiluted analysis. If the non-detect in the reanalysis was a result of over-dilution, it is ddms’ PJ to accept the result from the initial, undiluted analysis as the reportable concentration and qualify this result as estimated (J) because it was detected at a concentration outside of the calibration range. If the result in the initial, undiluted analysis is determined to be a false positive, reject (R) the result in the initial analysis.

5.14 Reported Quantitation/Reporting Limits (QLs or RLs)

5.14.1 Verify the sample QLs or RLs meet the project-specific required QLs or RLs. Any samples or analytes that do not meet the project-specific QLs or RLs should be documented in the validation report.

5.14.2 Any QLs or RLs not supported by the initial calibration need to be recalculated and corrected for all associated samples.

5.14.3 Correct reported non-detects at the MDL to not detected at the QL or RL, unless project-specific criteria dictate otherwise. If non-detects are reported at the MDL, it is necessary to verify that the MDLs are supported.

5.14.4 For most analytical reports, both RLs/QLs and MDLs are reported. In these cases, no corrections are necessary if non-detects have been reported at the QL or RL. It can be noted in the validation report that the data user should consider the "less than values" to be the RLs/QLs rather than the MDLs.

5.14.5 If documentation for reported MDLs is not provided in the data package, include a statement in the validation report stating this and advising that the reported MDLs may or may not be defensible.

5.14.6 Percent solids determinations are made for most solid samples. Verify that these calculations were correctly performed and that analyte concentrations and QLs or RLs for non-detects in the solid sample have been properly adjusted for percent solids. If the percent solids is less than 30%, it is ddms PJ to estimate (J, UJ) all results for the sample.

5.15 Documentation

If documentation issues arose during the validation process, discuss the action(s) taken and the impact to the reported results, addressing technical usability and potential issues if the data are used for litigation. The data user should be instructed to have the missing information inserted into all copies of the data package to ensure complete and accurate information is available for future distributions of the data. Ensure that appropriate corrections or revisions have been made to the case narrative included in the revised data package.

Documentation issues frequently encountered include the following:

- Issues with chain of custody records, such as improperly made corrections or missing information;
- Incomplete or incorrect laboratory case narratives;
- Missing or incomplete data or summary forms; and
- Inclusion of data in the data package that are not applicable to the site sample analyses.

When a discrepancy exists between analyte naming in the laboratory data package and the EDD, use the terminology presented in the data package, note that the EDD is different, and list the synonym used.

6.0 Preparation of Reports and Other Deliverables

6.1 Preparation of Validation Report

The data validation report should provide the data user with an overall evaluation of the data, identifying any potential usability issues determined during the review of the data package and, when professional judgment was applied, including the rationale for such actions. Only QAPP- or agency-defined qualifiers are applied to the data and documented in validation report. Laboratory qualifiers may be removed or replaced through the data validation effort. All ECS data validation and data review reports receive internal review prior to being submitted to the client. The following sections are included in the data validation report:

6.1.1 Executive summary that identifies the project site name, number of samples, sample identifications, matrix (or matrices) and parameter(s). Also included is a summary of the qualifiers applied to the data as a result of the validation effort with reasons therefor, a discussion of any anomalies encountered (e.g., deviations from the sample nomenclature, etc.), and a discussion of all potential data usability issues including both sampling and analytical error associated with the sample results.

6.1.2 Introduction that provides statements that identify the method under which the samples were analyzed, validation guidelines under which the data were validated, and a summary of the available qualifiers and their definitions.

6.1.3 Holding Times, Preservation, and Sample Integrity

6.1.4 Documentation - Discuss any documentation issues observed during the data review and the impact to the data.

6.1.5 Data Review Elements Table – identifying the elements reviewed for a given analytical parameter, and whether or not they were acceptable. These may include the following:

- 6.1.5.1 Tuning or Instrument Performance
- 6.1.5.2 Initial and Continuing Calibration, ICV Standards
- 6.1.5.3 Blanks (Laboratory, Field, and Equipment, or Other)
- 6.1.5.4 Surrogate or Labeled Analog Responses or Recoveries
- 6.1.5.5 Interference Check Samples
- 6.1.5.6 LCS (LCS/LCSD)
- 6.1.5.7 MS/MSD. If no MS/MSD results were reported, note as n/a in the table.
- 6.1.5.8 Unspiked Laboratory Duplicate. If no laboratory duplicate results were reported, note as n/a in the table.
- 6.1.5.9 Field Duplicates. If no field duplicate was collected, note as n/a in the table.
- 6.1.5.10 Internal Standards
- 6.1.5.11 Serial Dilution Analysis
- 6.1.5.12 Target Analyte Identification
- 6.1.5.13 Analyte Quantitation and Reported Quantitation Limits
- 6.1.5.14 Intra-Parameter Relationships (e.g., dissolved v. total concentrations for aqueous metals, speciated metals evaluation, etc.)

6.1.6 Summary of Quality Control Issues Resulting in Qualifications - Those issues that affected reported results are discussed in subsequent sections. When explanation of the use of PJ or extenuating circumstances is required, additional narration may be included and additional (even if acceptable) results or performance information may be included in tables for illustration. Where a data quality element was found to be satisfactory, no further discussion is included in the validation report.

6.1.7 Attachments – Attachments are project specific and may include data summary tables and/or annotated analysis reports.

6.2 Preparation of Data Review Report

The data review report is a letter report format and, similar to the validation report, should provide the data user with an overall evaluation of the data, identifying any potential usability issues determined during the review of the data package and when professional judgment was applied including the rationale for such actions. Only EPA qualifiers are applied to the data and documented in the validation report. Laboratory qualifiers may be removed and/or replaced through the data validation effort. The data review report is formatted as follows:

6.2.1 Opening paragraphs which provide the sample identifications, matrix, parameters and identify the data review criteria applied.

6.2.2 A summary of the qualifiers applied to the data and the deficiencies which resulted in the data qualification.

6.2.3 The Data Review Elements Table, as described in Section 6.1.5.

6.2.4 Issues that affected reported results are discussed in subsequent sections. Where a data quality element was found to be satisfactory, no further discussion is included in the validation report.

6.2.5 Attachments – Attachments are project specific and may include data summary tables and/or annotated analysis reports.

6.3 Other Report Formats - Validation reports using formats other than those described above may be required for an individual project.

6.4 Other Deliverables – For many projects, electronic data deliverables or EDDs (electronic files generated by the laboratory for reporting of analytical data) are provided, after application of qualifiers as a part of the validation effort. Other information added to these by the validator may include reasons for qualification, date of validation, level of validation, and identity of the validator. Brief descriptions of the reasons for qualified results are included in the EDD in many cases; however, specific qualifier reason codes may be employed in certain cases. These reason codes are defined in the accompanying validation report.

APPENDIX A

CURRENT VALIDATION GUIDANCE

EPA National Functional Guidelines:

www.epa.gov/clp/superfund-clp-national-functional-guidelines-data-review.com

EPA Region 1

www.epa.gov/quality/managing-quality-evironmental-data-epa-region-1.com

EPA Region 2

www.epa.gov/quality/managing-quality-evironmental-data-epa-region-2.com

EPA Region 3

www.epa.gov/quality/epa-region-3-data-validation.com

EPA Region 4

www.epa.gov/quality/managing-quality-environmental-data-epa-region-4.com

EPA Region 5

www.epa.gov/sites/production/files/2019-03/documents/g5-final.pdf

Guidance for Quality Assurance Project Plans (EPA QA/G-5), which references Guidance on Environmental Data Verification and Data Validation (EPA QA/G-8)

EPA Region 6

www.epa.gov/quality/agency-wide-quality-system-documents#guidance.com

Guidance on Environmental Data Verification and Data Validation (EPA QA/G-8)

EPA Region 7

www.epa.gov/quality/agency-wide-quality-quality-system-documents#spec.com

Guidance on Environmental Data Verification and Data Validation (EPA QA/G-8)

EPA Region 8

www.epa.gov/quality/region-8-quality-management-plan.com

QA/G-8 and NFG mentioned in EPA Region 8 Quality Management Plan

EPA Region 9

www.epa.gov/quality/data-validation-laboratory-quality-assurance-region-9.com

EPA Region 10

www.epa.gov/quality/managing-quality-evironmental-data-epa-region-10.com

No documents specified

Non-EPA Validation Guidance:

Radiological:

- US Army Corps of Engineers Louisville District "Louisville Radiological Guideline (LRG), Data Analysis and Validation Guidelines," Version 4, September 2004:

  https://www.lrl.usace.army.mil/Portals/64/docs/Engineering/ENV/LRG.pdf

- Department of Defense, Department of Energy Consolidated "Quality Systems Manual (QSM) for Environmental Laboratories," Version 5.3, 2019:

  https://www.denix.osd.mil/edqw/documents/manuals/qsm-version-5-3-final/

DQA/DUE:

- New Jersey Department of Environmental Protection Site Remediation Program "Data Quality Assessment and Data Usability Evaluation Technical Guidance," Version 1.0, April 2014

  https://www.nj.gov/dep/srp/guidance/srra/data_qual_assess_guidance.pdf

- New Jersey Department of Environmental Protection Site Remediation Program "Data of Known Quality Protocols Technical Guidance," Version 1.0, April 2014:

  https://www.nj.gov/dep/srp/guidance/srra/known_quality_protocols_guidance.pdf

- CTDEP "Laboratory Quality Assurance and Quality Control Data Quality Assessment and Data Usability Evaluation Guidance Document," May 2009, revised December 2010

  https://portal.ct.gov/-/media/DEEP/site_clean_up/guidance/QAQC/DQADUEguidancepdf.pdf?la=en

DUSR:

- NYSDEC DER-10/Technical Guidance for Site Investigation and Remediation, 5/3/10 (Appendix 2B = Guidance for Data Deliverables and Development of Data Usability Summary Reports):

  https://www.dec.ny.gov/docs/remediation_hudson_pdf/der10.pdf

Validation guidance documents may also be found in the following folder:

R:\Company Resources\Group Admin\ECS\Validation Guidance Documents

ATTACHMENT B

VALIDATION QUALIFIERS

National Functional Guidelines – Inorganics and High Resolution

U The analyte was analyzed for but was not detected above the level of the reported sample quantitation limit.

J The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.

J+ The result is an estimated quantity, but the result may be biased high.

J- The result is an estimated quantity, but the result may be biased low.

UJ The analyte was analyzed for but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.

R The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria. The analyte may or may not be present in the sample.

National Functional Guidelines – Organics

U The analyte was analyzed for but was not detected above the level of the reported sample quantitation limit.

J The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.

J+ The result is an estimated quantity, but the result may be biased high.

J- The result is an estimated quantity, but the result may be biased low.

NJ The analyte has been "tentatively identified" or "presumptively" as present and the associated numerical value is the estimated concentration in the sample.

UJ The analyte was analyzed for but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.

R The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria. The analyte may or may not be present in the sample.

C The target pesticide or Aroclor analyte identification has been confirmed by Gas Chromatography/Mass Spectrometry (GC/MS).

X The target pesticide or Aroclor identification was not confirmed when GC/MS analysis was performed.

EPA Region 1

U The analyte was analyzed for but was not detected. The associated numerical value is the sample quantitation limit (SQL) or Contract Required Quantitation Limit (CRQL). The SQL/CRQL accounts for sample specific dilution factors, percent solids results, or sample sizes that deviate from those required by the method. SQLs are also referred to as Sample Reporting Limits (SRLs) and Contract Required Quantitation Limits (CRQLs) for Superfund CLP generated data.

J The associated numerical value is an estimated quantity. The analyte was positively identified, and the associated numerical value is the approximate concentration (due either to the quality of the data generated because quality control criteria were not met, or the concentration of the analyte was below the SQL).

J+ The associated numerical value is estimated; associated QC data indicate a positive bias.

J- The associated numerical value is estimated; associated QC data indicate a negative bias.

Note: J+ and J- qualifiers should only be used when QC results indicate an unambiguous direction of bias. For example, when all QC results are acceptable except for low spiked compound recoveries, then the data reviewer may quality the associated data using "J-." Also, there can be cumulative bias information, for example, when a PE compound has a low recovery and the compound is recovered low in the matrix spike. The data reviewer may apply "J-" to all the associated data and describe the cumulative low recoveries on the data summary table (DST) with a footnote.

UJ The analyte was analyzed for but was not detected at a level greater than or equal to the SQL. The sample quantitation limit is an estimated quantity.

NJ The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents an estimated quantity.

R The data are unusable (analyte may or may not be present). Re-sampling and analysis are necessary for verification. The R replaces the numerical value or SQL.

C This qualifier applies to pesticide and Aroclor results when the identification has been confirmed by Gas Chromatography/Mass Spectrometry (GC/MS).

X This qualifier applies to pesticide and Aroclor results when GC/MS analysis was attempted but was unsuccessful.

EB, TB, BB An analyte that was detected in an aqueous equipment blank (EB), trip blank (TB), or bottle blank (BB) that was used to assess field contamination associated with soil/sediment samples. **These qualifiers should only be applied to soil/sediment results.**

EPA Region 2 - Inorganics

U The analyte was analyzed for but was not detected above the level of the reported sample quantitation limit.

J The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.

J+ The result is an estimated quantity, but the result may be biased high.

J- The result is an estimated quantity, but the result may be biased low.

UJ The analyte was analyzed for but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.

R The data are unusable. The sample results are rejected due to serious deficiencies in meeting quality control (QC) criteria. The analyte may or may not be present in the sample.

EPA Region 2 – Organics – Volatile Organics and Semivolatile Organics

U The analyte was analyzed for but was not detected above the level of the reported sample quantitation limit.

J The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.

J+ The result is an estimated quantity, but the result may be biased high.

J- The result is an estimated quantity, but the result may be biased low.

NJ The analysis indicates the presence of an analyte the has been “tentatively identified” and the associated numerical value represents its approximate concentration.

UJ The analyte was analyzed for but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.

R The data are unusable. The sample results are rejected due to serious deficiencies in meeting quality control (QC) criteria. The analyte may or may not be present in the sample.

EPA Region 2 – Organics – Pesticides and Aroclors

U The analyte was analyzed for but was not detected above the level of the reported sample quantitation limit.

J The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.

J+ The result is an estimated quantity, but the result may be biased high.

J- The result is an estimated quantity, but the result may be biased low.

NJ The analysis indicates the presence of an analyte the has been "tentatively identified" and the associated numerical value represents its approximate concentration.

UJ The analyte was analyzed for but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.

R The data are unusable. The sample results are rejected due to serious deficiencies in meeting quality control (QC) criteria. The analyte may or may not be present in the sample.

C This qualifier applies to results when the identification has been confirmed by gas chromatography/mass spectrometry GC/MS)

X This qualifier applies to results when GC/MS analysis was attempted but unsuccessful.

Region 2 – SOPs HW-16 (Method 8330A) HW-22 (Method 8270D), HW-29 (Method 524.2), and HW-44 (Method 8081B)

U The analyte was analyzed for but was not detected above the reported sample quantitation limit.

J The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.

N The analysis indicates the presence of an analyte for which there is presumptive evidence to make a "tentative identification."

NJ The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.

UJ The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.

R The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

EPA Region 3 – Inorganics (Uses NFG Plus B Qualifier)

U The analyte was analyzed for but was not detected above the level of the reported sample quantitation limit.

J The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.

J+ The result is an estimated quantity, but the result may be biased high.

J- The result is an estimated quantity, but the result may be biased low.

UJ The analyte was analyzed for but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.

R The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria. The analyte may or may not be present in the sample.

B Not detected substantially above the level reported in laboratory or field blanks.

EPA Region 3 – Organics (Uses NFG Plus B Qualifier)

U The analyte was analyzed for but was not detected above the level of the reported sample quantitation limit.

J The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.

J+ The result is an estimated quantity, but the result may be biased high.

J- The result is an estimated quantity, but the result may be biased low.

NJ The analyte has been “tentatively identified” or “presumptively” as present and the associated numerical value is the estimated concentration in the sample.

UJ The analyte was analyzed for but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.

R The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria. The analyte may or may not be present in the sample.

B Not detected substantially above the level reported in laboratory or field blanks.

EPA Region 4 – Inorganics (SOP-12)

D-6 The sample results are confirmed by ICP-MS or other analytical technique including analysis of a reference standard (the reference standard is not a PES – a reference standard is usually a certified reference material independent of standards available through the CLP program).

J The analyte was positively identified in the sample, but the associated numerical value is an estimated concentration based on the associated quality control data/technical criteria.

R The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

U The analyte was included in the analysis but was not detected above the method detection limit as defined in ISM01.2.

EPA Region 4 – Inorganics (SOPs 13 and 14)

J The analyte was positively identified in the sample, but the associated numerical value is an estimated concentration based on the associated quality control data/technical criteria.

R The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

U The analyte was included in the analysis but was not detected above the method detection limit as defined in ISM01.2.

EPA Region 4 – PCDD/PCDF (SOP-16)

U The analyte was analyzed for but was not detected above the Estimated Detection Limit (EDL) as defined in DLM02.0, Exhibit D, Section 11.2.5.

J The analyte was positively identified, but the associated numerical value is an estimated concentration of the analyte in the sample based on its associated quality measures.

N The analysis indicates the presence of an analyte for which there is presumptive evidence to make a “tentative identification.”

R The sample results are rejected due to serious deficiencies in the ability to analyze sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

EPA Region 4 – Organics (SOP-25)

C The sample results are confirmed by other analytical techniques including analysis of a reference standard.

J The analyte was positively identified, but the associated numerical value is an estimated concentration of the analyte in the sample based on its associated quality measures.

N The analysis indicates the presence of an analyte for which there is presumptive evidence to make a “tentative identification.”

R The sample results are rejected due to serious deficiencies in the ability to analyze sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

U The analyte was analyzed for but was not detected above the method detection limit as defined in the SOWs.

Region 5 (from EPA QA/G-8)

U The analyte was analyzed for but was not detected above the reported sample quantitation limit.

UJ The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.

J The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.

R The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet QC criteria. The presence or absence of the analyte cannot be verified.

Region 6 (from EPA QA/G-8)

U The analyte was analyzed for but was not detected above the reported sample quantitation limit.

UJ The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.

J The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.

R The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet QC criteria. The presence or absence of the analyte cannot be verified.

Region 7 (from EPA QA/G-8)

U The analyte was analyzed for but was not detected above the reported sample quantitation limit.

UJ The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.

J The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.

R The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet QC criteria. The presence or absence of the analyte cannot be verified.

Region 8

From NFG:

National Functional Guidelines – Inorganics and High Resolution

U The analyte was analyzed for but was not detected above the level of the reported sample quantitation limit.

J The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.

J+ The result is an estimated quantity, but the result may be biased high.

J- The result is an estimated quantity, but the result may be biased low.

UJ The analyte was analyzed for but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.

R The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria. The analyte may or may not be present in the sample.

National Functional Guidelines – Organics

U The analyte was analyzed for but was not detected above the level of the reported sample quantitation limit.

J The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.

J+ The result is an estimated quantity, but the result may be biased high.

J- The result is an estimated quantity, but the result may be biased low.

NJ The analyte has been “tentatively identified” or “presumptively” as present and the associated numerical value is the estimated concentration in the sample.

UJ The analyte was analyzed for but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.

R The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria. The analyte may or may not be present in the sample.

C The target pesticide or Aroclor analyte identification has been confirmed by Gas Chromatography/Mass Spectrometry (GC/MS).

X The target pesticide or Aroclor identification was not confirmed when GC/MS analysis was performed.

From EPA QA/G-8

U The analyte was analyzed for but was not detected above the reported sample quantitation limit.

UJ The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.

J The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.

R The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet QC criteria. The presence or absence of the analyte cannot be verified.

Region 9

Qualifiers not defined.

Region 10

Qualifiers not defined.

Appendix C

ddms Example Reports

Validation Memorandum – Stage 2A and 2B (Industriplex)

R:\Projects\DEF\demax-1547\3204-Industri-PlexOU2\DataAnalysis\Data Review\2018-2019\Final Validation Memos\Iplex 2019 Final Validation Memos

DQA Table – Stage 2A and 2B (Omega)

R:\Projects\DEF\demax-1547\3139C-Omega\DataAnalysis\Validation\2019-Q4\Working\Groundwaters\Other

Full validation report – Stage 2B with some Stage 4 (Omega)

R:\Projects\DEF\demax-1547\3139C-Omega\DataAnalysis\Validation\2020-Q1

Letter report - Stage 2B or Stage 4 (NMI 2019 Comprehensive Groundwater Sampling Event):

R:\Projects\DEF\demax-1547\3243-NMI GW NTCRA\DataAnalysis\Validation\2019\Comprehensive Sampling Event

NJDEP DQA/DUE:

R:\Projects\MNOP\On-Site Environmental - 2121\0001-Olt Brothers\DataAnalysis\Validation

R:\Projects\GHI\H2M-2072\0028 - Franklin Twp\DataAnalysis\Validation

NYSDEC DUSR:

R:\Projects\JKL\JM Sorge-2074\0002-New Rochelle\DataAnalysis\Validation\2019

R:\Projects\ABC\Brickhouse Environmental-2114\0001-Binghamton Plaza\DataAnalysis\Validation

Appendix D

Laboratory Deliverables

Level 1

Narrative
Sample results and qualifiers
Chain of custody documents
No supporting QC

Level 2

Narrative
Sample results and qualifiers
Chain of custody documents
Surrogate results
Batch QC results (MS/MSD, LCS, MB, etc.)

Level 3

Narrative
Sample results and qualifiers
Chain of custody documents
Surrogate results
Batch QC results (MS/MSD, LCS, MB, etc.)
CLP-like summary forms:

- Organics:
  - Form 1 – Samples and QC Samples
  - Form 2 – Surrogates
  - Form 3 – LCS/LCSD, MS/MSD
  - Form 4 – Method Blank summary
  - Form 5 – Tune for mass spectrometry
  - Form 6 – initial calibration
  - Form 7 – continuing calibration
  - Form 8 – IS & RT (mass spectrometry)
  - Form 8 – Analytical Sequence (GC)
  - Form 10 – Identification (GC)
- Metals:
  - Form 1 – Samples
  - Form 2A – Calibration verification
  - Form 2B – CRQL Check
  - Form 3 – Instrument/method blanks
  - Form 4A –Interference check samples
  - Form 5 – MS/MSD/PDS

Form 6 –Laboratory duplicate
Form 7A – laboratory control sample
Form 7D – laboratory control sample duplicate
Form 8 –Serial Dilution
Form 9 –Detection limits
Form 10 –Correction factors
Form 12 – Preparation log
Form 13 – Run log
Form 14 – Tune (ICP/MS)
Form 15 – Internal standard relative intensity

General Chemistry:

Form 1 – Samples
Form 2 – Calibration
Form 3 –Method blanks
Form 5 – MS/MSD
Form 6 –Laboratory duplicate
Form 7A – laboratory control sample/LCSD
Form 9 –Detection limits
Form 12 – Preparation log
Form 13 – Run log

Level 4 (CLP or CLP like)

Narrative
Sample results and qualifiers
Chain of custody documents
Surrogate results
Batch QC results (MS/MSD, LCS, MB, etc.)
CLP-like summary forms:

Organics:

Form 1 – Samples and QC Samples
Form 2 – Surrogates
Form 3 – LCS/LCSD, MS/MSD
Form 4 – Method Blank summary
Form 5 – Tune for mass spectrometry
Form 6 – initial calibration
Form 7 – continuing calibration
Form 8 – IS & RT (mass spectrometry)
Form 8 – Analytical Sequence (GC)
Form 10 – Identification (GC)

Metals:

- Form 1 – Samples
- Form 2A – Calibration verification
- Form 2B – CRQL Check
- Form 3 – Instrument/method blanks
- Form 4A –Interference check samples
- Form 5 – MS/MSD/PDS
- Form 6 –Laboratory duplicate
- Form 7A – laboratory control sample
- Form 7D – laboratory control sample duplicate
- Form 8 –Serial Dilution
- Form 9 –Detection limits
- Form 10 –Correction factors
- Form 12 – Preparation log
- Form 13 – Run log
- Form 14 – Tune (ICP/MS)
- Form 15 – Internal standard relative intensity

General Chemistry:

- Form 1 – Samples
- Form 2 – Calibration
- Form 3 –Method blanks
- Form 5 – MS/MSD
- Form 6 –Laboratory duplicate
- Form 7A – laboratory control sample/LCSD
- Form 9 –Detection limits
- Form 12 – Preparation log
- Form 13 – Run log

Instrument raw data

Appendix E

Project Kick-Off Form

The Project Kick-Off Form (a fillable pdf form) is found in the following folder:

R:\Company Resources\Group Admin\ECS

Standard Operating Procedure:
Validation and Review of Perchlorate Analysis Data

1.0 Scope and Application

Data validation determines the technical usability of analytical data. It is a process by which a body of data is compared to a set of acceptance criteria and flagged with qualifiers when acceptance criteria are not met and in response to various other impacts to the data quality. An unbiased, objective report is prepared, that documents the degree of compliance to the intended purpose and that details the findings of the validation effort and impacts to the usability and defensibility of the reported results. There may be criteria in this SOP that are more stringent than those outlined in regional guidelines. The focus of validation is always on the technical usability and defensibility of the data. This SOP is applicable to data produced under the following methods:

EPA Method 331
EPA Method 332

2.0 Summary

This SOP details the process of evaluating and verifying perchlorate data, comparing it to stated Data Quality Objectives (DQOs) and acceptance criteria, and preparing a technically sound validation report that provides the data user with a reliable basis for decision making. Extensive laboratory knowledge, knowledge of published methods, and the ability to apply consistent professional judgment are essential. This SOP should be used in conjunction with ddms SOP ECS-SOP-012-Rev.0– General Data Validation: "Validation and Review of Analytical Data," the site-specific Quality Assurance Project Plans and Sampling and Analysis Plans, regional, national, or other agency data review guidelines, if applicable, and analytical methods. This SOP provides ddms' professional judgment.

All data have the potential to go to litigation. Part of the validation process is to identify any data and/or documentation issues that could be problematic if the data are used in litigation. All documents are discoverable during litigation, including validation/review reports. Any documentation issues that potentially affect the usability of the data identified during the review process must be clearly stated in the report.

When only a review (in lieu of a validation) of the data is requested, the procedure outlined in this SOP is followed, but calculations are not performed. Only a review of the data summary forms (raw data are not reviewed) is performed, unless that review reveals major quality issues. If at any time a situation arises that is not addressed in this SOP, contact with the other ddms chemists is required. Where professional judgment (PJ) is referenced in the guidelines and no PJ in this SOP covers the situation, communication with the other ddms chemists is required. Always document in the report when ddms professional judgment is applied and the rationale for decisions made.

When verifying calculations, not all regions specify an acceptable %D between laboratory-reported and validator-calculated values. It is ddms' professional judgment to contact the laboratory when discrepancies greater than 10% occur. The error term of 10% is considered to be allowable experimental error in general laboratory practice.

3.0 Acronyms

%D – Percent Difference
%R – Percent Recovery
CC – Continuing Calibration
COC – Chain of Custody
DQO – Data Quality Objective
DV – Data Validation
FB – Field Blank
IC – Initial Calibration
ICV – Initial Calibration Verification
IS – Internal Standard
LC – Liquid Chromatography
LCS/LCSD – Laboratory Control Sample/LCS Duplicate
LFB – Laboratory Fortified Blank
LFSSM - Laboratory Fortified Synthetic Sample Matrix
LFSSMB – Laboratory Fortified Synthetic Sample Matrix Blank
LRB – Laboratory Reagent Blank
LSSM – Laboratory Synthetic Sample Matrix
MB – Method Blank
MDL – Method Detection Limit
MRL – Method Reporting Limit
MRM – Multiple Reaction Mode
ND – Not Detected
PJ – Professional Judgment
QA – Quality Assurance
QC – Quality Control
QCS – Quality Control Sample
QL – Quantitation Limit
RL – Reporting Limit
RPD – Relative Percent Difference
RT – Retention Time
SIM – Selected Ion Monitoring
S/N – Signal to Noise
SOP – Standard Operating Procedure

4.0 Definitions

4.1 Refer to the analytical method referenced for definitions of method-specific terms.

4.2 Refer to SOP ECS-SOP-012-Rev.0– General DV: "Validation and Review of Analytical Data" for terms specific to the validation of analytical data.

5.0 Procedure

5.1 Identify Project Specific Requirements - Refer to SOP ECS-SOP-012-Rev.0– General DV: "Validation and Review of Analytical Data."

5.2 Review, Verification and Evaluation of Analytical Results

5.2.1 Sample Integrity, Preservation, and Holding Time

5.2.1.1 Custody record

5.2.1.1.1 Verify that complete, consistent, unbroken COC documentation is provided for all samples.

5.2.1.1.2 Ensure that a cross-reference between field IDs and lab IDs is provided.

5.2.1.2 Preservation

5.2.1.2.1 Verify that the temperature of the samples in the cooler(s) upon receipt at the laboratory was documented and was ≤10°C. If the temperature of the cooler was above the upper limit, qualify based on appropriate data validation guidelines. In the absence of regional validation guidelines regarding temperature excursions, apply ddms professional judgment. If the receipt temperature of the samples exceeds the acceptance criteria, but it is noted that the samples were received "on ice," review the documentation to verify that there was sufficient time (nominally 4 hours) from sample collection to laboratory receipt for the samples to have cooled to the appropriate temperature. No qualification of data is necessary, but it is noted in the data validation report. If the elapsed time from sample collection to laboratory receipt exceeds 4 hours, use professional judgment to determine whether or not to estimate (J, UJ) the results.

5.2.1.2.2 Samples must be filtered, using sterile syringes and filters after collection to preserve against biological activity, which will alter the sample. Determine if the samples were filtered. If not and they should have been, use PJ to determine whether or not to qualify results.

5.2.1.2.3 Sample containers should be filled to minimize headspace to prevent aerobic biodegradation.

5.2.1.2.4 Upon lab receipt, samples should be maintained at ≤ 6°C.

5.2.1.2.5 If samples were not collected or stored properly, use PJ to determine action.

5.2.1.3 Holding Time

5.2.1.3.1 The methods stipulate a 28-day holding time from collection to analysis of samples that are properly preserved.

5.2.1.3.2 In the absence of regional validation guidelines regarding holding time excursions, ddms professional judgment is to qualify all results as estimated (J, UJ) when analyzed beyond the method holding time. If the holding time is grossly

exceeded, >56 days, qualify detects estimated (J) and reject (R) non-detects.

5.2.2 System Performance

5.2.2.1 The validator must verify that acceptable mass calibration and resolution was achieved before analyses were initiated. Because there is no prescribed tuning compound for this analysis (manufacturer's instructions are referenced for use), verify appropriate tuning and resolution based on data provided for the instrument tune, if available. In the absence of mass tuning data, evaluate the mass resolution and sensitivity in the calibration standards. Documentation for mass calibration and resolution may be requested from the laboratory if needed. The tuning mixture must be appropriate to the mass range that will be monitored (including 80 - 120 m/z), especially since the instrumentation is typically used for high molecular weight materials and manufacturer's recommendations may not focus on low range masses. The method mentions use of a sodium iodide/rubidium iodide mixture. If any of the mass calibration/resolution or chromatographic criteria are not met or were not verified, use PJ to determine the effect on sample results and qualify accordingly.

5.2.2.2 Because much of the data generation for this instrumentation is blind to the data reviewer, it is reasonable to request clarification from the laboratory on instrument conditions, mass data acquisition parameters, etc. Determine (and/or request from the laboratory for verification) that all analyses have been performed under the same conditions and that all integrations of the reference and target analyte have been performed the same throughout all of the applicable analyses (for all initial and continuing calibration standards and all field and QC samples). If the same conditions, including the acquisition and integration parameters, have not been used for all analyses, use PJ to determine the potential effect on data and qualify accordingly. It may be appropriate to qualify results as estimated or to reject results.

5.2.2.3 Chromatographic performance for the analysis is evaluated based on the peak shape, retention time stability, sensitivity, and resolution in the calibration standards.

5.2.2.4 Verify that any manual integrations were performed correctly, documented appropriately, and done consistently throughout the data set.

5.2.3 Calibration

5.2.3.1 Initial Calibration

5.2.3.1.1 A minimum of five calibration points must be used.

5.2.3.1.2 Quadratic curves must have six points.

5.2.3.1.3 When calculated against the curve, all standard recoveries must be 80-120% of the true concentration, except the lowest, which must be 50-150% of the true value.

5.2.3.1.4 If any standard does not meet criteria when calculated against the curve, the curve is not valid.

5.2.3.1.5 The validator should examine the data to determine any causes for non-linearity. If it is the lowest or highest standard that is unacceptable, eliminate that standard and adjust the calibration range accordingly. If the high point is eliminated, accept positive detects in the linear portion of the curve; estimate (J) positive detects at the higher end of the curve, and accept non-detects. If the low point on the curve is removed, accept positive detects in the linear portion of the curve, estimate (J, UJ) positive detects and non-detects in the lower portion of the curve. In the absence of regional qualification guidelines, ddms practice will be to raise the RL to the concentration at which successful calibration was demonstrated

5.2.3.1.6 IS areas must be within ±30% of the first CC of the sequence and the CC area must be ≥50% of the average area calculated from the IC.

5.2.3.1.7 Verify that the IS are added to all standards and samples (QC and field) to achieve the same concentration.

5.2.3.1.8 In situations where the instrument response was not linear and the laboratory may have used an alternate calibration model, such as a quadratic equation, careful assessment should enable the validator to determine if the calibration is reliable. If only a portion of the calibration is reliable, qualify the data accordingly. Results within the range may be acceptable as reported, while results outside of this usable range should be either qualified as estimated or rejected, based on the potential error. PJ will be necessary and other ddms validators should be consulted.

5.2.3.1.9 The isotope ratio for perchlorate must be within ±25% of theoretical (for m/z 99/101 SIM or 83/85 MRM: theoretical 3.086; QC limits 2.31 – 3.85).

Analyte	**Precursor ion (m/z)**	**Product ion (m/z)**
$^{35}ClO_3$	99	83
$^{37}ClO_3$	101	85
$^{35}Cl^{18}O_3$ (IS)	107	89

5.2.3.1.10 Retention Time Window

5.2.3.1.10.1 Assess relative the relative retention time for each mass peak selected for identification, confirmation, and quantification (criteria 0.98-1.02).

5.2.3.1.10.2 Relative retention times for the target analyte and internal standard should be within 0.02 retention time units. Retention times outside this window may be acceptable. PJ will be

necessary and other ddms validators should be consulted.

5.2.3.1.11 Data from analysis of a laboratory synthetic sample matrix (LSSM) and laboratory fortified sample matrix (LFSSM) may be present (required quarterly at a minimum) to verify the laboratory's ability to separate the perchlorate peak from sulfate accurately. If the validator has concerns about the identification of the peak, these data should be requested from the laboratory.

5.2.3.1.12 A quality control sample (QSC) or initial calibration verification (ICV) standard (second-source quality control sample) must be analyzed with each new initial calibration, and at a minimum, quarterly.

5.2.3.1.12.1 Verify that the QCS/ICV was a second-source standard and that QCS/ICV results are calculated on the correct IC. ddms acceptance criteria are ±30%. If the recovery falls outside of 70-130%, contact the laboratory to determine if the ICV and CC standards are from the same working lot. If the standards are from the same working lot and recovery is less than 70%, qualify detects (J) and non-detects (UJ). If the recovery is greater than 130%, qualify detects (J) and accept non-detects.

5.2.3.2 Continuing Calibration

5.2.3.2.1 Verify that continuing calibration (CC) standards are analyzed on each instrument used to analyze samples at the beginning of each sequence before any blanks, samples or QC samples are analyzed and at the end of the sequence. A low level and mid-level CC must be analyzed at the beginning of each analysis batch. A CC must be analyzed after every tenth sample (not including batch QC samples).

5.2.3.2.2 Verify that the CC was compared against the most recent IC. If the CC was not compared against most recent IC, contact the laboratory to correct the CC and any affected subsequent sample results.

5.2.3.2.3 Recoveries must be 80-120% for all but the lowest concentration and 50-150% for the lowest concentration.

5.2.3.2.4 If recoveries are outside acceptance limits and the shift represents an increase in sensitivity, positive results are qualified as estimated (J), but non-detects are not qualified. If the shift represents a decrease in sensitivity, qualify positive results and non-detects estimated (J, UJ). If perchlorate exhibits a %D greater than 100.0%

exhibiting an increase in sensitivity, consult ddms chemists for further action.

5.2.3.2.5 If the closing CC is unacceptable and the opening CC was acceptable, review all of the samples included in the bracket and consult with other ddms chemists. If spiked samples (LCS, MS) are present to support continued performance, the results may be acceptable. Labeled compound response in the samples may provide support for continued performance. PJ must be used on a case by case basis to determine the effect on the sample data impacted by the unacceptable CC.

5.2.3.2.6 Verify that CC concentrations included a low-level at the beginning of the sequence. A low-level standard must be analyzed with each analytical sequence to verify the laboratory's continued ability to meet the reporting limits.

5.2.3.2.7 An analytical batch must be analyzed within 30 hours and includes:

- 20 field samples or fewer
- 1 LRB (minimum)
- 1 LFB (minimum)
- 1 LFSM (minimum)
- 1 LFSMD or 1 LD (minimum)

5.2.3.2.8 CC standards every 10 samples and at the beginning (low level and mid-level) and end of the sequence

5.2.4 Blanks

5.2.4.1 Laboratory Reagent Blank (LRB)

5.2.4.1.1 A laboratory reagent blank (LRB) must be prepared and analyzed with each preparation batch.

5.2.4.1.2 A laboratory synthetic sample matrix blank (LSSMB) must also be analyzed to verify that the system is free of contamination.

5.2.4.1.3 Both LRBs and LSSMBs must be filtered using the same sample collection devices used for the field samples.

5.2.4.1.4 Perchlorate detections in the blanks must be ND or ≤ 1/3 the MRL.

5.2.4.1.5 If perchlorate is detected in a blank at >1/3 MRL, all data must be considered invalid for that analysis batch. (PJ may be used to make decisions if sample concentrations are significantly greater than detections in the blank).

5.2.4.2 Field-generated Blanks

5.2.4.2.1 If a field-submitted blank was not collected and provided, assess the impact on the samples and determine appropriate action. Consultation with other ddms chemists is recommended.

5.2.4.2.2 Some Regions do not qualify data based on field-submitted blank results. ddms' SOP is **to** qualify sample results based on field-submitted blank results. It is not ddms' policy to qualify soil data based on associated field-submitted sample results without consultation with other ddms chemists.

5.2.4.3 Blank Scenarios

5.2.4.3.1 Scenario 1: The analyte concentration in the MB is less than that in the FB. The sample result is greater than the MB, but less than the FB. Even though the governing agency specifically states that data not be qualified based on FB results, based on ddms professional judgment, results would be qualified as not detected (U) at the sample-specific RL or the reported value, whichever is greater.

5.2.4.3.2 Scenario 2: Samples are analyzed on two different instruments on the same day. On one of the instruments, perchlorate is reported in the MB and on the other instrument this analyte is ND. The sample results on both instruments were less than the MB on the instrument with the contaminant. ddms professional judgment is to qualify the results from the analytical runs on both instruments as not detected (U) at the sample-specific RL or the reported value, whichever is greater.

5.2.5 Internal Standards (IS)

5.2.5.1 IS areas must be within 70-130% of the first CC of the sequence.

5.2.5.1.1 Note: the area of the IS in the first CC of the sequence should be ≥ 50% of the average of the IS area counts in the IC standards.

5.2.5.2 If the IS area is outside the specified limits, qualify detects and non-detects associated with that IS as estimated (J, UJ).

5.2.5.3 If the IS area is ≥10% and <25%, or greater than 200%, contact other ddms chemists for discussion and resolution. Excessively low or high IS responses indicate the potential for high variability in the analysis and will result in qualification of results.

5.2.5.4 If the IS area is less 10%, reject (R) non-detects, and estimate (J) detects with the potential for low bias.

5.2.5.5 If any sample was not reanalyzed due to an IS area outside acceptance criteria, this must be noted in the validation report.

5.2.6 Laboratory Fortified Blank (LFB)

5.2.6.1 Verify an LFB (or LFB/LFBD pair) was analyzed as appropriate.

5.2.6.2 LFBs must be processed and analyzed in the same manner as the field samples, using the same filtration equipment.

5.2.6.3 It is ddms PJ to qualify data if either the LFB or LFBD fail to meet acceptance criteria.

5.2.6.4 The method requires that the LFB be rotated between low, mid, and high concentrations batch to batch. If more than one batch has been analyzed in association with the data set, verify that altering concentrations have been used.

5.2.6.5 The recovery of perchlorate in the LFB/LFBD should be within ddms professional judgment acceptance criteria of 50-150% at concentration at or below the MRL and 80-120% for other concentrations. Data are qualified when the %Rs fall outside of these limits to alert the data user of potential bias.

5.2.6.5.1 If the recovery is >120%, estimate (J) detects for that compound in the samples associated with that LFB, indicating potential high bias. Non-detect results do not require qualification.

5.2.6.5.2 If the recovery of perchlorate is less than 80% but ≥10%, estimate (J) detects in all samples associated with the LFB, and estimate (UJ) all non-detects in all samples associated with the LFB, indicating potential low bias.

5.2.6.5.3 If the recovery of perchlorate is less than 10%, estimate (J) detects in samples associated with that LFB and reject all non-detects in samples associated with that LFB.

5.2.6.5.4 When the laboratory has analyzed an LFB/LFBD pair, the RPD between the paired results should be less than 20%. Qualify results as estimated (J) for all samples in the associated analytical batch when the RPD exceeds 20%. When the RPD exceeds 40%, discussion with other ddms chemists is necessary before making decisions on qualification of data.

5.2.7 Laboratory Fortified Sample Matrix /Laboratory Fortified Sample Matrix Duplicates (LFSM/LFSM–) - **Refer to SOP ECS-SOP-012-Rev.0– General DV: "Validation and Review of Analytical Data."**

5.2.8 Field Duplicate Samples – **Refer to SOP ECS-SOP-012 – General DV: "Validation and Review of Analytical Data."**

5.2.9 Target Compound Identification

5.2.9.1 Mass peak RTs, S/N ratio, and the ratio between characteristic SIM or MSM masses are assessed against the reference standards for identification of perchlorate. Professional judgment is often required to determine if a correct identification was made.

5.2.9.2 If it is determined that the identification is incorrect, contact with the laboratory may be necessary to have the data corrected and resubmitted. If a discrepancy remains, use professional judgment to qualify or reject and document the reason in the validation report.

5.2.9.3 Inspect any sample(s) analyzed following a high concentration sample to verify that reported positives are not due to carryover from the high concentration sample. ddms PJ is to contact the laboratory for explanation and resolution. If the validator's opinion differs from the

laboratory's response, contact other ddms chemists for verification of opinion and qualification of data.

5.2.10 Compound Quantitation

5.2.10.1 Examine the method of calculation for perchlorate. Ensure that these are calculated in consistency with the established calibration.

5.2.10.2 Verify calculations of some selected perchlorate concentrations.

5.2.10.3 If a dilution was performed because the concentration of perchlorate was outside of the calibration range in the undiluted analysis, but it was reported as a non-detect in the diluted analysis, evaluate the raw data or refer to historical data, if available, to determine if the non-detect in the re-analysis was a result of over-dilution or if the analyte was a false positive in the initial, undiluted analysis. If the non-detect in the reanalysis was a result of over dilution, it is ddms PJ to accept the result from the initial, undiluted analysis as the reportable concentration and qualify this result as estimated (J) because it was detected outside of the calibration range. If the result in the initial, undiluted analysis is determined to be a false positive, reject (R) the result in the initial analysis.

5.2.10.4 If multiple dilutions have been performed, assess all of the available data to determine the most usable result.

5.2.11 Reported Quantitation/Reporting Limits (QLs or RLs)

5.2.11.1 Verify the sample QLs or RLs meet the project specific required QLs or RLs. Any samples that do not meet the project specific RL should be documented in the validation report.

5.2.11.2 An RL not supported by the initial calibration needs to be recalculated and corrected for all associated samples.

5.2.11.3 Correct reported non-detects at the MDL to non-detected at the RL.

5.2.11.4 Where the Analytical Report lists "ND" for sample results and lists both the MDL and RL, the "ND" should be understood to be ND at the RL. This should be narrated in the validation report.

5.2.12 Documentation - **Refer to SOP ECS-SOP-012-Rev.0– General DV: "Validation and Review of Analytical Data."**

6.0 Preparation of Reports **- Refer to SOP ECS-SOP-012-Rev.0– General DV: "Validation and Review of Analytical Data."**

7.0 References

7.1 EPA Method 331.0, Rev. 1.0, Determination of Perchlorate in Drinking Water by Liquid Chromatography Electrospray Ionization Mass Spectrometry, 815-R-05-007, January 2005

7.2 EPA Method 332.0, Rev. 1.0, Determination of Perchlorate in Drinking Water by Ion Chromatography with Suppressed Conductivity and Electrospray Ionization Mass Spectrometry, EPA/600/R-05/049, March 2005

VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE FOR TOTAL, TOTAL INORGANIC, AND TOTAL ORGANIC CARBON (TOC)

(GL-GC-E-093 REVISION 16)

APPLICABLE TO METHODS:
EPA Method 415.1
EPA SW-846 Methods 9060/9060A
Standard Methods 22nd Edition, SM 5310 B-2011

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1.0 STANDARD OPERATING PROCEDURE FOR TOTAL, TOTAL INORGANIC, AND TOTAL ORGANIC CARBON (TOC)

2.0 METHOD CODE

2.1 EPA Method 415.1

2.2 EPA SW-846 Method 9060/9060A

2.3 Standard Methods 22nd Edition, SM 5310 B-2011

3.0 METHOD OBJECTIVE/PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to describe the procedure used to run samples for total, total inorganic, and total organic carbon using the OI Analytical Model 1030W Aurora TOC analyzer.

4.0 METHOD SUMMARY

4.1 Total organic carbon is converted to carbon dioxide by chemical oxidation of the organic carbon in the sample. The carbon dioxide is measured using a non-dispersive infrared detector.

4.2 Synonym: Non-purgeable organic carbon

5.0 APPLICABLE MATRICES

5.1 Groundwater

5.2 Drinking water

5.3 Domestic and industrial wastewater

NOTE: Clients may request that this analysis be performed on miscellaneous liquid or solid samples. If the sample is very viscous or heavily particulated, the sample is treated as a solid and SOP GL-GC-E-062 is used.

NOTE: SC DHEC requires analysis by SW-846 9060/9060A if samples are not drinking water or wastewater and the data are to be used for regulatory purposes.

6.0 HOLDING TIME

Holding time is 28 days from the time and date of collection until the start of analysis unless otherwise specified.

7.0 SAMPLE CONTAINER/PRESERVATION/COLLECTION/STORAGE REQUIREMENTS

7.1 Storage of samples in amber glass bottles is preferred. Polyethylene bottles may be used if blanks are collected to show that the containers do not contaminate the samples.

7.2 Unless samples are to be analyzed within 15 minutes of collection, they should be acidified to a pH of less than 2 with sulfuric acid, phosphoric acid or hydrochloric acid.

7.3 Samples should be stored at $0° \leq 6°$ C.

7.4 If the concentration of dissolved organic carbon is to be determined, samples should be filtered through a 0.45 µm filter at the time of collection.

8.0 INTERFERENCES

Carbonates and bicarbonates must be removed before analysis for TOC. The sparging of liquid samples removes carbonate and bicarbonate interferants.

9.0 PERFORMANCE CHARACTERISTICS

9.1 Method concentration range: 0.00 to 20 mg/L with the sample loop set at 5 mL loop size.

9.2 Calibration range: 1 to 20 mg/L.

9.3 Method detection limit (MDL): Refer to current MDL study.

9.4 Method precision: < or equal to 20% RPD.

9.5 Method accuracy: 85%-115%.

10.0 DEFINITIONS

10.1 AlphaLIMS: The Laboratory Information Management System used at GEL.

10.2 Laboratory Control Sample (LCS): A standard, usually of the same matrix as the sample batch being organized, taken through the same prep process as the samples then analyzed with the batch.

10.3 Total Organic Carbon: All carbon in a sample besides carbonates and bicarbonates.

10.4 Non-purgeable Organic Carbon: All organic carbon that is not removed by sparging.

10.5 Total Carbon: Total amount of carbon in a sample.

10.6 Total Inorganic Carbon: All inorganic carbon in a sample that is separated from organic carbon by acidification.

10.7 Dissolved Organic Carbon: All organic carbon in a sample that has been filtered through a 0.45 micron filter.

10.8 Refer to GL-QS-B-001 the Quality Assurance Plan for additional lab-wide used definitions.

11.0 ANALYST VERIFICATION

Technicians and analysts do not analyze client samples without supervision until trained by qualified personnel and upon successful analysis of a proficiency sample. Training records are maintained as quality records.

12.0 DOCUMENTATION OF DATA

As data are obtained, computer printouts of the data are generated. These dated hard copies of the data are stored in the TOC 1030W binder. Results are uploaded into AlphaLIMS.

13.0 SAFETY PRECAUTIONS AND HAZARD WARNINGS

13.1 Wear eye protection with side shields while performing procedures in the lab.

13.2 Treat all chemicals and samples as potential health hazards, and limit exposure to these chemicals to the lowest level possible. GEL maintains a current awareness file of OSHA regulations regarding the safe handling of the chemicals in the laboratory as well as a reference file of Material Safety Data Sheets (MSDS.) These documents and client sample MSDS forms are maintained in the laboratory.

14.0 SAMPLE RECEIPT FOR ANALYSIS

14.1 The analyst/technician gives the list of samples needed to the sample custodian. The sample custodian removes the appropriate samples from cooler and either delivers them to the analyst/technician or places them on the "pick-up" shelf in the main cooler.

14.2 Analysts and technicians are responsible for retrieving their own samples when the sample custodian is not available.

15.0 INSTRUMENTATION/EQUIPMENT/GLASSWARE

15.1 IO Analytical Model 1030W Aurora TOC Analyzer including:

15.1.1 Reaction module

15.1.2 Detector/electronics module

15.1.3 Printer module

15.1.4 Autosampler module

15.2 Compressed nitrogen (zero grade) and two stage regulator

15.3 Flow meter

15.4 Personal computer (for entering data)

16.0 REAGENTS

16.1 Raw materials: (Chemicals should be at least ACS grade or equivalent)

16.1.1 Potassium acid phthalate, $KHC_8H_4O_4$

16.1.2 Concentrated phosphoric acid, H_3PO_4 (85%)

16.1.3 5% (by volume) Phosphoric acid reagent: Carefully add 59 mL concentrated phosphoric acid to 500 mL DI water, and dilute to 1 L.

16.1.4 ASTM Type I deionized water (see GL-LB-E-016)

16.1.5 Sodium bicarbonate, $NaHCO_3$

16.2 Persulfate reagent:

16.2.1 Dissolve 100 g sodium persulfate in a 1 liter volumetric flask using deionized (DI) water.

16.2.2 Bring to volume with DI water.

17.0 PREPARATION OF SAMPLES

Not applicable.

18.0 PREPARATION OF STANDARDS

Potassium Phthalate (also known as potassium hydrogen phthalate) is used for the preparation of standards. The standard is valid for one year from opened date or manufacturer's expiration date, whichever is shortest.

18.1 TIC Standard Solution (2000mg/L)

18.1.1 Weigh 14 g of Sodium Bicarbonate and dilute to 500 mL of DI water in a volumetric flask. This yields a 2000 mg/L solution which expires in 6

months. This solution should be purchased from a certified vendor, if available.

18.2 Primary TOC Standard Solution (2000 mg/L)

18.2.1 Weigh 0.850 g of potassium acid phthalate and dilute to 200 mL volume with DI water in a 200 mL volumetric flask. This yields a 2000 mg/L solution. Standard expires six months from preparation date. This solution should be purchased from a certified vendor, if available.

18.3 TIC/TOC Working Standard (20 mg/L)

18.3.1 Transfer 2.5 mL of the primary TIC/TOC stock standard solution to the 250 mL volumetric flask and dilute to 250 mL with DI water.

18.4 TIC/TOC Working Standard (10 mg/L)

18.4.1 Transfer 2.5 mL of the primary TIC/TOC stock standard solution using 500 mL volumetric flask and dilute to 500 mL with DI water.

18.5 TIC/TOC Working Standard (1 mg/L)

18.5.1 Transfer 0.125 mL of the primary TIC/TOC standard solution to the 250 mL volumetric flask and dilute to 250 mL with DI water.

18.6 TOC Working Standard (5 mg/L)

18.6.1 Transfer 0.625 mL of the TIC/TOC to the partially filled flask. Dilute to 250 mL with DI water.

NOTE: Working standards expire 1 week after preparation.

18.7 Secondary TIC/TIC Stock Standard

18.7.1 Using a different lot number of Sodium Bicarbonate, weigh 14 g of Sodium Bicarbonate a dilute to 500 mL with DI water in a volumetric flask. This yields a 2000 mg/L solution which expire after 6 months. This solution should be purchased from a certified vendor, if available.

18.8 Secondary TOC Stock Standard

18.8.1 Using a different lot of potassium phthalate, weigh 0.850 g of potassium acid phthalate and dilute to 200 mL volume with DI water in a 200 mL volumetric flask. This yields a 2000 mg/L solution. Standard expires six months from preparation date. This solution should be purchased from a certified vendor, if available.

18.9 10 mg/L Working LCS/ICV/CCV

18.9.1 Transfer 2.5 mL of the secondary TIC/TOC stock standard to the 500 mL volumetric flask and dilute to 500 mL with DI water. This solution expires one week from preparation.

19.0 PREPARATION OF STANDARDS AND QUALITY CONTROL SAMPLES

19.1 Documentation of standards and their preparation are maintained in AlphaLIMS in accordance with GL-LB-E-007 for Laboratory Standards Documentation.

19.2 Laboratory Control Sample (LCS): (See 18.9)

19.3 Calibration standards: The concentrations used are listed below:

19.3.1 0.0 mg/L

19.3.2 1 mg/L

19.3.3 5 mg/L

19.3.4 10 mg/L

19.3.5 20 mg/L

19.3.6 Calibration standards are made up using primary source stock standard.

19.4 Initial Calibration Verification (ICV): The ICV is a 10.0 mg/L standard made from the second source stock standard. The ICV must be analyzed immediately after the calibration standards and analyzed at the start of each analytical week. The ICV standards are made up on a weekly basis. (See 18.9)

19.5 Continuing Calibration Verification (CCV): The CCV is a 10.0 mg/L standard made from the second source stock standard. A CCV must be run after every 10 samples and after the last sample in the run. The CCV standards are made up on a weekly basis. (See 18.9)

20.0 INSTRUMENT/EQUIPMENT START-UP PROCEDURE

20.1 Refer to Chapter 4: Operation of the OI Model 1030W Aurora TOC Analyzer Operator's Manual.

20.2 Before a run is started, make sure there is an adequate amount of sodium persulfate reagent to complete the analysis. Also make sure the 5% phosphoric acid solution and DI H_2O containers are full.

20.3 Prior to the start of analysis, ensure that the nitrogen flow is between 40 and 60 psi.

21.0 QUALITY CONTROL (QC) REQUIREMENTS

21.1 Instrument QC

21.1.1 An initial calibration verification (ICV) is analyzed immediately after the calibration standards and at the start of each analytical week. This standard must be made from a different source than the calibration standards.

21.1.2 An initial calibration blank (ICB) is analyzed following the ICV.

21.1.3 A continuing calibration verification (CCV) is analyzed after every 10 analytical samples and after the last analytical sample in the run.

21.1.4 A continuing calibration blank (CCB) is analyzed after every CCV.

21.2 Batch QC

21.2.1 A matrix spike and a matrix duplicate are analyzed for every batch of $\leq$ 10 samples and for each set of ten samples in batches with > 10 samples (unless otherwise required by client contract).

21.2.2 A method blank and laboratory control sample (LCS) are analyzed at least once for every batch of 20 samples or less.

21.2.3 For liquid samples, the LCS is normally a 10 mg/L standard taken through the same process as the samples.

21.2.4 LCS duplicates are analyzed if required by client contract.

21.3 Acceptance limits:

21.3.1 Correlation coefficient must be 0.995 or greater.

21.3.2 ICV recovery must be 90-110% for all batches (unless otherwise specified by client contract). If not, the ICV should be remade and reanalyzed. If the ICV continues to be out of range, a re-calibration is then required.

21.3.3 CCV recovery must be 90-110% for all batches (unless otherwise specified by client contract). Any samples bracketed by a failing CCV must be reanalyzed with passing bracketing CCVs. If the CCV continues to be out of range, the instrument must be recalibrated.

21.3.4 Matrix relative percent difference (RPD): < or equal to 20% of RPD.

21.3.5 Matrix spike recovery: 85% -115%.

21.3.6 Method blank: Must be less than the CRDL.

21.3.7 LCS: refer to current SPC limits which are static for Drinking Water samples.

21.3.8 LCS RPD: refer to current SPC limits which are static for Drinking Water samples.

21.3.9 If analysis by EPA Method 415.1 is requested, the samples are analyzed in duplicate. The relative percent difference (RPD) between the two values must be ≤ 20% when the values are greater than 5 mg/L.

21.3.10 If analysis by SW-846 9060/9060A is requested, samples are analyzed in quadruplicate. The relative percent difference (RPD) between the values must be ≤ 20% when the values are greater than 5 mg/L.

21.4 Handling out-of-control situations:

21.4.1 If a sample result exceeds the concentration of the highest calibration standard, the sample must be diluted appropriately with DI water and reanalyzed.

21.4.2 The correlation coefficient must be at least 0.995. If it is less than 0.995 the calibration standards must be reanalyzed. Analysis of samples cannot begin until a correlation coefficient of 0.995 is obtained.

22.0 RUN SEQUENCE

22.1 Calibration standards, including DI water blank

22.2 ICV

22.3 ICB

22.4 Up to 10 analytical samples including LCS, method blank, and sample QC (refer to Section 20)

22.5 CCV (Continuing Calibration Verification)

22.6 CCB

22.7 Repeat steps 21.5 through 21.7 for remaining samples in the run ending with CCV and CCB.

23.0 PROCEDURE

23.1 Analysis: Refer to Chapter 4 (pages. 62 through 71), Operation of the Model 1030W Aurora Analyzer Operator's Manual.

23.2 Calculation/reporting of results:

23.2.1 As the data are obtained, they are uploaded into AlphaLIMS.

23.2.2 If a sample is analyzed by EPA 415.1, the average of the two replicates is reported for the TOC or Total Carbon Concentration.

23.2.3 For samples analyzed by SW 846-9060/9060A, all four replicates plus the average are reported for the TOC concentration.

24.0 INSTRUMENT/EQUIPMENT SHUT-DOWN PROCEDURES

Refer to OI Analytical Model 1030W Aurora TOC analyzer.

25.0 DATA REVIEW, VALIDATION, AND APPROVAL PROCEDURE

Refer to GL-GC-E-092 for General Chemistry Data Review and Packaging.

26.0 DATA TRANSMITTAL

When a batch is issued "DONE" status, it is made available to reporting personnel.

27.0 RECORDS MANAGEMENT

All logbooks and data generated as a result of this procedure are maintained as quality records in accordance with GL-QS-E-008 for Quality Records Management and Disposition.

28.0 ROUTINE INSTRUMENT/EQUIPMENT MAINTENANCE

Refer to Chapter 4, page 74, Operation of the Model 1010 Wet Oxidation Total Organic Carbon Analyzer Operator's Manual.

29.0 LABORATORY WASTE HANDLING AND DISPOSAL

For the proper disposal of sample and reagent wastes from this procedure, refer to the Laboratory Waste Management Plan, GL-LB-G-001.

30.0 METHOD VERIFICATION

30.1 To ensure accuracy, % error is calculated for the duplicate values of all concentrations greater than 1 mg/L.

30.2 The % error must be 10% or less for samples logged according to Method 415.1. If not, the samples must be reanalyzed.

31.0 REFERENCES

31.1 Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, "Organic Carbon, Total," Method 415.1 (Combustion or Oxidation), March 1979.

31.2 Test Methods for Evaluating Solid Waste: Laboratory Manual Physical/ Chemical Methods, Volume 1C, SW-846, Third Edition, November 1986. Method 9060, "Total Organic Carbon," Revision 0, September 1996. USEPA Office of Solid Waste and Emergency Response, Washington, DC 20460.

31.3 Test Methods for Evaluating Solid Waste: Laboratory Manual Physical/ Chemical Methods, Volume 1C, SW-846, Third Edition, November 1986. Method 9060A,

"Total Organic Carbon," Revision 1, August 2002. USEPA Office of Solid Waste and Emergency Response, Washington, DC 20460.

31.4 OI Analytical Model 1030W Aurora Analyzer Operator's Manual.

31.5 Standard Methods 22nd Edition, SM 5310 B-2011. High-Temperature Combustion Method.

31.6 Dept. of Defense (DOD), Dept. of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories DOD QSM Version 5.3, July, 2019.

32.0 HISTORY

Revision 12: Preparation of Standards section added.

Revision 13: Updated procedure to include new instrument information and remove reference to old instrument. Updated the process of working standards.

Revision 14: Further method and procedure clarifications and updated to new instrument.

Revision 15: Updated the method concentration range from 0.2 to 0.00. Added reference to QAP in definitions. Added current DOD/DOE QSM version 5.1 and version 3.1, January 2017.

Revision 16: Updated sample preservation. Updated DoD QSM reference to Version 5.3, July 2019. Clarify acceptance limits for Drinking Water.

VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE

FOR

TOTAL HARDNESS BY TITRATION

(GL-GC-E-100 REVISION 8)

APPLICABLE TO METHODS:
Standard Methods, 22nd Edition, Method 2340C-97

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1.0 STANDARD OPERATING PROCEDURE FOR TOTAL HARDNESS BY TITRATION

2.0 METHOD CODE

Standard Methods, 22nd Edition, Method 2340C-97

3.0 METHOD OBJECTIVE / PURPOSE

This procedure is used to measure the Total Hardness of samples in mg/L of $CaCO_3$.

4.0 METHOD SUMMARY

4.1 Summary: Calcium and magnesium ions in the sample are sequestered upon the addition of disodium ethylenediamine tetraacetate (Na_2EDTA). The end point to the reaction is detected by means of an indicator, which has a red color in the presence of calcium and magnesium and a blue color when the cations are sequestered.

5.0 APPLICABLE MATRICES

5.1 Groundwater

5.2 Drinking water

5.3 Domestic and industrial wastewater

5.4 Saline water

5.5 Surface waters

6.0 HOLDING TIME

Samples must be analyzed within 180 days of collection.

7.0 SAMPLE CONTAINER, PRESERVATION, COLLECTION, STORAGE REQUIREMENTS

7.1 Samples may be stored in glass or plastic containers.

7.2 Preservation:

7.2.1 Samples are preserved with HNO_3 to a pH of < 2.

7.2.2 Documentation of temperature and pH are documented at Login. Refer to GL-SR-E-001 for Sample Receipt, Login, and Storage.

7.3 Refrigerate sample at $0 \leq 6°$ C until analysis.

8.0 INTERFERENCES

8.1 Excessive amounts of heavy metals can interfere. This is usually overcome by complexing the metals with cyanide.

9.0 PERFORMANCE CHARACTERISTICS

9.1 Method range: The method is suitable for all concentration ranges of hardness; however, in order to avoid large titration volumes, it is recommended that a sample aliquot containing no more than 25 mg $CaCO_3$ be used.

9.2 Method detection limit (MDL): Refer to current MDL study

9.3 Method precision

9.3.1 Sample Duplicate: Refer to current SPC limits.

9.3.2 Matrix Spike: Refer to current SPC limits.

9.4 Method accuracy: Refer to current SPC limits.

10.0 DEFINITIONS

10.1 AlphaLIMS - The data system used at GEL Laboratories, LLC.

10.2 Independent Calibration Verification (ICV) - A solution of method analytes of known concentrations that is used to fortify an aliquot of Blank or sample matrix. The ICV is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check laboratory performance with externally prepared test materials.

10.3 Laboratory Control Standard (LCS) - An aliquot of reagent water or other blank matrix to which known quantities of the method analytes are added in the laboratory. The LCS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

10.4 Method Blank (MB) - An aliquot of reagent water or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The MB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

10.5 Method Detection Limit (MDL) - The minimum concentration of an analyte that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero.

10.6 Spike (Matrix Spike or Post Spike) - An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS or PS is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS or PS corrected for background concentrations.

10.7 Statistical Process Control (SPC) Limits - Statistically derived limits which establish Acceptable Ranges for recoveries of analytes of interest, including LCS, MS, MSD, PS, PSD and internal standards.

10.8 Stock Standard Solution - A concentrated solution containing one or more method analytes prepared in the laboratory using certified reference materials or purchased from a reputable commercial source.

10.9 Refer to GL-QS-B-001 the Quality Assurance Plan for additional lab-wide used definitions.

11.0 ANALYST VERIFICATION

Technicians and analysts do not analyze client samples without supervision until trained by qualified personnel and upon successful analysis of a proficiency sample. Training records are maintained as quality records.

12.0 DOCUMENTATION OF DATA

As data is obtained, it is entered directly into AlphaLIMS.

13.0 SAFETY PRECAUTIONS AND HAZARD WARNINGS

13.1 Wear eye protection with side shields while performing procedures in the lab.

13.2 All chemicals and samples should be treated as a potential health hazard and exposure to these chemicals must be reduced to the lowest level possible. GEL maintains a current awareness file of OSHA regulations regarding the safe handling of the chemicals in the laboratory as well as a reference file of Material Safety Data Sheets (MSDS.) These documents are maintained in the laboratory. Individual sample MSDS forms provided by the clients are kept in login.

14.0 SAMPLE RECEIPT FOR ANALYSIS

14.1 The analyst/technician gives the list of samples needed to the sample custodian. The sample custodian removes the appropriate samples from cooler and either delivers them to the analyst/technician or places them on the "pick-up" shelf in the main cooler.

14.2 Analysts and technicians are responsible for retrieving their own samples when the sample custodian is not available.

15.0 INSTRUMENTATION/EQUIPMENT/GLASSWARE

15.1 Beakers: Capacity of 100 mL minimum required

15.2 Graduated cylinders: Minimum capacity of 100 mL is needed

15.3 Magnetic stir plate

15.4 Stir bar

15.5 Magnetic stir bar retriever

15.6 Squeeze bottle for deionized water

15.7 Hot plate

15.8 Buret with 0.05 mL increments: Recommended capacity, 10 mL to 20 mL

15.9 Watch glass

15.10 pH meter and probe

15.11 Oven capable of maintaining 250 ± 50° C

NOTE: The temperature of the oven is monitored in accordance with GL-LB-E-004.

15.12 Volumetric flask, 1 L

15.13 Aluminum weigh boat

15.14 Desiccator and desiccant

15.15 Analytical balance: capable of weighing to 0.0001g

16.0 REAGENTS

16.1 Buffer Solution

16.1.1 A commercially available "odorless buffer" is purchased (example: HACH Buffer Solution Hardness 1). The buffer solution expires 1 year from the date opened.

16.2 Indicator Solution

16.2.1 A commercially available indicator such as Calmagite indicator is used.

16.3 Standard EDTA titrant, 0.02 N.

16.3.1 Place 3.723g of analytical reagent grade disodium ethylenediamine tetraacetate dihydrate, $Na_2C_{10}H_{14}O_8N_2*2H_2O$, in a 1 L volumetric flask and dilute to the mark with DI water. Store in polyethylene. Standardize daily (18.1.1)

16.4 Hydrochloric acid solution, 1 + 1.

17.0 STANDARDS

17.1 1000 mg/L $CaCO_3$ spiking standard.

17.1.1 Place 1.000g of anhydrous calcium carbonate in approximately 500 mLs of DI water.

17.1.2 Add, a little at a time, 1 + 1 HCl until all of the $CaCO_3$ has dissolved.

17.1.3 Add 200 mLs of DI Water.

17.1.4 Boil for a few minutes to expel CO_2.

17.1.5 Cool.

17.1.6 Bring to a 1L volume in a volumetric flask with DI Water.

17.1.7 The standard expires 1 year from preparation date.

17.2 2500 mg/L $CaCO_3$ LCS standard.

17.2.1 Place 2.500g of anhydrous calcium carbonate in approximately 500 mLs of DI water.

17.2.2 Add, a little at a time, 1 + 1 HCl until all of the $CaCO_3$ has dissolved.

17.2.3 Add 200 mLs of DI Water.

17.2.4 Boil for a few minutes to expel CO_2.

17.2.5 Cool.

17.2.6 Bring to a 1L volume in a volumetric flask with DI Water.

17.2.7 The standard expires 1 year from preparation date.

18.0 STANDARDIZATION OF REAGENTS

18.1 EDTA titrant, 0.02 N.

18.1.1 Pipette 10 mLs of the 1000 mg/L spiking standard into a beaker containing 40 mLs of DI water.

18.1.2 Add 1 mL of buffer solution.

18.1.3 Add 1-2 drops of indicator.

18.1.4 Titrate slowly with continuous stirring until the last reddish tinge disappears. The end point color is blue.

18.1.5 Repeat step 18.1.4 two more times.

18.1.6 Calculate the Normality of the EDTA titrant using the average of the three titrations. The following formula is used to calculate normality:

$$N \text{ of EDTA} = 0.2 / \text{mLs of EDTA}$$

18.1.7 Standardization must be performed at least monthly.

19.0 QUALITY CONTROL (QC) REQUIREMENTS

19.1 Frequency of QC:

19.1.1 A method blank (MB) and a laboratory control sample (LCS) are analyzed every 20 samples.

19.1.2 A sample duplicate and matrix spike are analyzed for every 10 samples.

19.2 Acceptance limits:

19.2.1 Matrix relative percent differences (RPD): limits 0-10%.

19.2.2 Matrix spike recoveries (% recovery: limits 90%-110%.

19.2.3 LCS recoveries (% recovery): limits 90% - 110%.

19.2.4 MB criteria: less than the RL.

19.3 Handling out-of-control situations:

19.3.1 Notify the group leader, team task leader, or PM immediately.

19.3.2 If the MB should fall outside of the control limits, the entire batch must be reanalyzed.

19.3.3 If the LCS recovery should fall outside of the SPC limits, the entire batch must be reananlyzed.

19.3.4 Samples may exhibit matrix interferences, which cause matrix QC to fall outside of the SPC limits. When this occurs, the analyst should determine that this is the case and narrate the interferences in the case narrative system in AlphaLIMS.

19.3.5 The analyst should document in the case narrative the specific QC that is out-of-control and cross-reference data from any subsequent reanalysis.

20.0 RUN SEQUENCE

20.1 Method Blank (MB)

20.2 Laboratory Control Sample (LCS)

20.3 Samples 1 through x where x < 10

20.4 Sample x duplicate

20.5 Sample x spike

20.6 Repeat steps 20.3 - 20.5 for every group of ten samples in the batch.

21.0 PROCEDURE

21.1 The sample should require < 15 mLs of EDTA titrant and the titration should be completed within 5 minutes of buffer addition. If the sample requires >/= 15 mLs of EDTA titrant, a smaller sample volume shall be used.

21.2 Place 25 mLs of sample in a titration vessel and dilute to approximately 50 mLs with DI water.

21.3 Add 1 to 2 mLs of buffer solution.

21.4 Add 1 to 2 drops of indicator solution.

21.5 Titrate slowly with continuous stirring with standard EDTA titrant until the last reddish tint disappears. The solution is normally blue at the end point.

21.6 To create the LCS, dilute 5 mLs of the 2500 mg/L LCS standard to 25 mLs total volume with DI Water. This creates a 500 mg/L LCS working standard.

21.7 To spike samples, add 5 mLs of the 1000 mg/L spiking standard to 25 mLs of sample. This creates a 200 mg/L spike.

22.0 CALCULATIONS

22.1 The following formula is used to calculate Total Hardness (mg/L as CaCO3):

Hardness = (A x N x 50,000) / mLs of sample

Where: A = mLs of EDTA titrant
N = normality of EDTA titrant

23.0 DATA REVIEW, VALIDATION, AND APPROVAL PROCEDURES

23.1 Data review is performed In accordance with GL-GC-E-092 for Data Packaging and Validation.

24.0 DATA TRANSMITTAL

24.1 When a batch is given General Chemistry departmental "DONE" status, it is made available to reporting personnel.

25.0 RECORDS MANAGEMENT

Data is maintained in AlphaLIMS.

26.0 ROUTINE INSTRUMENT/EQUIPMENT MAINTENANCE

26.1 Refer to GL-LB-E-004 for Temperature Monitoring and Documentation Requirements for Refrigerators, Freezers, Ovens, Incubators, and other Similar Devices.

27.0 LABORATORY WASTE HANDLING AND DISPOSAL

For the proper disposal of sample and reagent wastes from this procedure, refer to GL-LB-G-001, Laboratory Waste Management Plan.

28.0 METHOD VERIFICATION

28.1 Method detection limit studies are performed in accordance with the GL-LB-E-001 for the determination of Method detection limits.

29.0 REFERENCES

29.1 Standard Methods, 22nd Edition, Method 2340C-97, 2012.

30.0 HISTORY

Revision 8: Updated SPC limits and MB Blank Criteria
Revision 7: Updated standard method reference.
Revision 6: Removed Ammonium Hydroxide, 1N from reagents section and updated steps for creating the LCS.
Revision 5: Changed volume requirements and deleted EPA 130.2 references.
Revision 4: Added equipment list and standardization frequency.

VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

GEL LABORATORIES, LLC

QUALITY ASSURANCE PLAN

(GL-QS-B-001 REVISION 34)

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SECTION 1
INTRODUCTION

Section 1 - Introduction

GEL Laboratories, LLC (GEL) is a privately owned environmental laboratory dedicated to providing personalized client services of the highest quality. Our mission is to be the "Analytical Firm of First Choice."

GEL was established as an analytical testing laboratory in 1981. Now a full service lab, our analytical divisions use state of the art equipment and methods to provide a comprehensive array of organic, inorganic, radiochemical, and bioassay analyses and related support services to meet the needs of our clients.

This Quality Assurance Plan provides an overview of our quality assurance program for analytical services. Outlined in this plan are the responsibilities, policies, and processes essential to maintaining client satisfaction and our high quality of performance. The Director of Quality Systems is responsible for revising, controlling, and distributing the QAP. It is updated/reviewed at least annually.

Everyone on our staff is expected to understand the policies, objectives, and procedures that are described in this plan and to fully appreciate our commitment to quality and their respective roles and responsibilities with regard to quality. We also expect any analytical subcontractors we employ to perform in accordance with the quality assurance requirements delineated in this plan. All GEL employees are required to participate in Annual Quality Systems training.

This Quality Assurance Plan (QAP) has been prepared according to the standards and requirements of the US Environmental Protection Agency (EPA), ANSI/ISO/IEC 17025-2017, and the TNI (The NELAP Institute) Standards adopted in August, 2009.

1.1 Quality Policy

GEL's policy is "to provide high quality, personalized analytical services that enable our clients to meet their environmental needs cost effectively."

We define quality as "consistently meeting the needs and exceeding the expectations of our clients." As such, we consistently strive to:

- meet or exceed client and regulatory requirements
- be technically correct and accurate
- be defensible within contract specifications
- provide services in a cost-effective, timely and efficient manner

At GEL, quality is emphasized at every level—from the Chairman and CEO to the newest of employees. Management's ongoing commitment to good professional practice and to the quality of our testing services to our customers is demonstrated by their dedication of personnel and resources to develop, implement, assess, and improve our technical and management operations.

The purpose of GEL's quality assurance program is to establish policies, procedures, and processes to meet or exceed the expectations of our clients. To achieve this, all personnel that support these services to our clients are introduced to the program and policies during their initial orientation, and annually thereafter during company-wide training sessions.

GEL's management is committed to compliance with and continual improvement of our quality assurance program. The program is designed to comply with the guidelines and specifications outlined in the following:

- TNI 2009
- ASME/NQA-1
- ISO/IEC 17025-2017
- QAPPs, U.S. EPA QA/R5
- Department of Energy Order 414.1B, 414.1C and 414.D
- ANSI N42.23-1996 Measurement and Associated Instrument Quality Assurance for Radioassay Laboratories
- ANSI N13.30-2011 Performance Criteria for Radiobioassay
- DOE STD 2019

- Performance Criteria for Radiobioassay- ANSI N13.30-1996.
- Energy Reorganization Act, 1974, Section 206, 10 CFR, Part 21
- MARLAP
- U.S. Department of Defense (DoD), Department of Energy (DOE) Consolidated Quality System Manual (QSM) for Environmental Laboratories, Revision 5.3, May 2019
- 10 CFR Part 21- Reporting of Defects and Noncompliance
- 10 CFR Part 61- Licensing Requirements for Land Disposal of Radioactive Waste
- NRC REG Guide 4.8
- NRC REG Guide 4.15

1.2 Quality Goals

GEL's primary goals are to:

- Ensure that all measurement data generated are scientifically and legally defensible, of known and acceptable quality per the data quality objectives (DQOs), and thoroughly documented to provide sound support for environmental decisions.
- Ensure compliance with all contractual requirements, environmental standards, and regulations established by local, state and federal authorities.

Additional goals include:

- A comprehensive quality assurance program to ensure the timely and effective completion of each measurement effort.
- A commitment to excellence and improvement at all levels of the organization.
- Early detection of deficiencies that might adversely affect data quality.
- Adequate document control.
- Effective quality assurance objectives for measurement systems and for quality data in terms of accuracy, precision, completeness, and comparability through the use of proven methods.
- The establishment of procedures that demonstrate that the analytical systems are in a state of statistical control.
- The implementation of corrective actions and improvements to ensure the integrity of data.
- Reduction of data entry errors through comprehensive automated data handling procedures.
- The development and implementation of good laboratory and standard operating procedures (SOPs).
- Ability to customize quality assurance procedures to meet a client's specific requirements for data quality.
- Good control of instruments, services, and chemical procurement.
- A continuously capable laboratory information management system (AlphaLIMS).
- Validated and documented computer hardware and software.
- All employees who have access to the AlphaLIMS system are required to participate in computer security awareness training annually.

1.3 Key Quality Elements

A sound quality assurance program is essential to our ability to provide data and services that consistently meet our high standards of integrity. The key features of our program are:

- An independent quality assurance (QA) validation and Quality Systems Department.
- A formal quality policy and QAP.
- Management review.
- Stated data quality objectives.
- A comprehensive employee training program.
- Ethics policy and education program.
- Internal audits and self-evaluations.
- A closed-loop corrective action program.
- State-of-the-art facilities and instruments.
- Adherence to standard operating procedures.
- EPA/NIST traceable reference materials.
- Electronically based document control.
- Chain of custody and electronic sample tracking.
- Inter-laboratory comparison programs.
- Formal laboratory accreditations.
- The evaluation of subcontractor laboratories.

- Statistical controls for analytical precision and accuracy.
- Replicate, method blank, matrix spike, tracer yield, internal standards, and surrogate measurements.
- The preventive maintenance of instrumentation and equipment.
- Independently prepared blind standard reference materials.
- Multi-level review processes.
- Focus on client satisfaction.
- Electronic tracking of client commitments, nonconformance's and corrective actions.
- Trend analysis of nonconforming items.

1.4 Management Reviews

The effectiveness of the Quality System is reviewed at least annually by Senior Management. These reviews address issues that impact quality, and the results of the reviews are used to develop and implement improvements to the system. Records of the review meetings are maintained as quality documents.

1.5 Disposition of Client Records

In the event that the laboratory should change ownership, the responsibility for the maintenance and disposition of client records shall transfer to the new owners. In the unlikely event that the laboratory ceases to conduct business, clients shall be notified and asked to provide instructions as to how their records should be returned or disposed. If a client does not provide instructions, those records will be maintained and disposed in a manner consistent with regulations and good laboratory practices for quality records.

1.6 Supporting Documents

Our laboratory operations and the quality of our analytical data comply with the specifications described in the documents listed in Appendix A.

1.7 Definitions

Applicable definitions are listed in Appendix B.

SECTION 2
ORGANIZATION, MANAGEMENT, AND PERSONNEL

Section 2 - Organization, Management, and Personnel

The chart found in Appendix C depicts our corporate organization, chain of command and flow of responsibility. The illustration in this appendix is designed to ensure the overall quality and cost efficiency of our company's analytical products and services.

Our structure is based on customer-focused divisions that follow a project from the point of initial contact to the final invoicing of work. These divisions include expertise in project management, sample receipt and custody, sample preparation and analysis, data review, and data packaging. An independent Quality Systems Management Department monitors the adherence of these divisions to the Quality Assurance Program.

The general responsibilities associated with the following position levels are discussed in this section:

- Chairman and Chief Executive Officer (CEO)
- President
- Chief Operating Officer (COO)
- Chief Financial Officer (CFO)
- Quality Systems Director
- Laboratory Directors
- Project Managers
- Group Leaders
- Laboratory and Technical Staff
- Information Systems Manager
- Environmental Manager
- Radiation Safety Officer
- Director of Human Resources

An overview of GEL's employee training protocol is also provided at Section 2.12.

2.1 Chairman and CEO, President, Chief Financial Officer and Chief Operating Officer

Operational responsibility rests with GEL's owners, CFO and COO. James M. Stelling and Joseph M. Hodgson Jr. are owners and serve respectively as Chairman and CEO, and President. Carey J. Bocklet occupies the position of COO. Laurie Herrington occupies the position of CFO. As the highest level executives, their philosophical approach to quality, technology and customer service keeps GEL unique. They are also part of a Leadership Team that works to create a workplace environment that attracts and retains highly qualified professionals.

As Chairman and CEO, Mr. Stelling oversees the Executive Committee and leads management in implementing total quality initiatives that ensure quality services that meet stringent criteria of excellence. He holds a Bachelor of Science in Commerce from the University of Virginia.

Joseph M. Hodgson Jr. is GEL's President. He has overall operational responsibility and operates the laboratory according to corporate policies, applicable licenses and regulations. He is also responsible for Strategic Planning, Marketing and Business Development. In addition, he has primary responsibility for the development and administration of our analytical testing and environmental consulting services. Mr. Hodgson Jr. holds a Bachelor of Science in Business from Wake Forest University.

The Chief Operating Officer is Carey J. Bocklet. Ms. Bocklet is responsible for the daily operations of the laboratories and client services. Ms. Bocklet holds a Bachelor of Science in Chemical Engineering, and a Master of Science in Business Administration, both from Clemson University.

Laurie Herrington is GEL's Chief Financial Officer and oversees GEL's financial management. She is responsible for contracts administration, invoicing, purchasing, payroll, accounts payable, and receivable, inventory control, property control and financial forecasting. Ms. Herrington holds a Bachelor of Science in Accounting and Business Administration from the College of Charleston. Ms. Herrington also has her licenses as a Certified Public Accountant and a Certified Fraud Examiner.

Together, the Chairman and CEO, President, COO and CFO form GEL's Executive Committee. Their responsibilities include the following:

- Ensuring that the individuals who staff our technical and quality positions have the necessary education, training, and experience to competently perform their jobs.
- Ensuring that all staff members receive ancillary training, as needed, to enhance performance in assigned positions.
- Budgeting, staffing, managing, and equipping the laboratory to meet current and future analytical program requirements.
- Overseeing the implementation and overall effectiveness of our Quality Assurance Plan, health and safety initiatives, and environmental programs.
- Managing production and cost control activities.
- Ensuring development of capabilities in response to new or revised regulations, instrumentation and procedures, and quality assurance initiatives.

2.2 Technical Laboratory Co-Directors

To enhance our responsiveness to clients through dedicated expertise and teamwork, our laboratory is divided into two major divisions, Chemistry and Radiochemistry, each with its own Technical Laboratory Director.

The Technical Directors report to the Executive Committee and are ultimately responsible for the technical content and quality of work performed within each division. They are also responsible for strategic planning, profitability and growth, personnel management and business development. Other responsibilities include:

- Monitoring and meeting profitability and growth objectives of the division.
- Establishing and implementing short and long range objectives and policies that support GEL's goals.
- Defining the minimum level of qualification, experience, and skills necessary for positions in their divisions.
- Establishing and implementing policies and procedures that support our quality standards.
- Ensuring that technical laboratory staff demonstrates initial and continuing proficiency in the activities for which they are responsible.
- Documenting all analytical and operational activities of the laboratory.
- Supervising all personnel employed in the division.
- Ensuring that all sample acceptance criteria are verified and that samples are logged into the sample tracking system, properly labeled, and stored.
- Documenting the quality of all data reported by the division.
- Developing internal mechanisms and measurements to improve efficiency.
- Overseeing activities designed to ensure compliance with laboratory health and safety requirements.
- Allocating the resources necessary to support an effective and ongoing quality assurance program.
- Representing the company to the public and to clients.
- Ensuring the appropriate delegation of authorities during periods of absence.
- Ensuring compliance to the ISO 17025:2017 Standard.

2.3 Quality Systems Director

Our Quality Systems Director (QSD) reports directly to the CEO. The QSD manages the design, implementation and maintenance of our quality systems in a timely, accurate, and consistent manner.

In addition to having responsibility for the initiation and recommendation of corrective and preventive actions, the QSD is responsible for:

- Establishing, documenting, and maintaining comprehensive and effective quality systems.
- Developing and evaluating quality assurance policies and procedures pertinent to our laboratory functions, and communicating these with the division directors and managers.

- Ensuring that the operations of the lab are in conformance with the Quality Assurance Plan and meet the quality requirements specific to each analytical method.
- Ensuring that laboratory activities are in compliance with local, state, and federal environmental laws and regulations.
- Reviewing project-specific quality assurance plans.
- Ensuring that quality control limits are established and followed for critical points in all measurement processes.
- Initiating internal performance evaluation studies using commercially purchased certified, high-purity standard reference materials.
- Performing independent quality reviews of randomly selected data reports.
- Conducting periodic audits to ensure method compliance.
- Conducting or arranging periodic technical system evaluations of facilities, instruments and operations.
- Overseeing and monitoring the progress of nonconformance's and corrective actions.
- Communicating system deficiencies, recommending corrective action to improve the system, and defining the validity of data generated during out of control situations.
- Preparing and updating quality assurance documents and reports to management.
- Coordinating inter-laboratory reviews and comparison studies.
- Overseeing Stop Work Orders in out-of-control situations.
- Administering accreditation and licensing.
- Administering our document control system.
- Providing guidance and training to laboratory staff as requested.
- Evaluating subcontractors and vendors that provide analytical and calibration services.
- Designating quality systems authorities in times of absence to one or more appropriately knowledgeable individuals.
- Overseeing notification if required for compliance with Energy Reorganization Act, 1974, 10 CFR, Part 21, should data recall be necessary.
- Ensuring that the laboratory has policies to avoid involvement in activities or relationships which might negatively affect confidence in the laboratory's competence, impartiality, judgement or operational integrity.
- Ensuring that management and personnel are free from undue internal and external pressures and influences that may adversely affect their impartiality, affecting the quality of their work, by mitigating pressures.
- Ensuring that employee competence measurements are established and monitored.

2.4 Quality Systems Review

The effectiveness of the Quality System is reviewed on a regular basis during meetings of the Leadership Team, which may be as often as weekly, but not less than quarterly. These meetings address issues that impact quality, and the subsequent discussions are used to design and implement improvements to the system. At least annually, a management assessment of GEL's Quality System is conducted and reported. The QSD maintains records of these assessments.

2.5 Manager of Client and Support Services

Project Managers (PMs) serve as primary liaisons to our clients. PMs, under the guidance of the Manager of Client and Support Services, manage the company's interaction with clients. They are the client's first point of contact and have responsibility for client satisfaction and for communicating project specifications and changes to the appropriate laboratory areas.

Additional responsibilities include:

- Retaining clients and soliciting new work.
- Managing multiple sample delivery orders and preparing quotes.
- Working with clients to define analytical methodologies, quality assurance requirements, reports, deliverables, and pricing.

- Overseeing sample management and informing laboratory staff of the anticipated arrival of samples for analysis.
- Conducting a review of client documents (i.e. quotes, invoices, routine and specialized reports).
- Working with the accounting team on invoicing and collection issues.
- Working with the Laboratory Directors, Production Manager, and Group Leaders to project workloads and determine schedules.

2.6 Group Leaders

Group Leaders are a critical link between project management, lab personnel, and support staff. They report to the Technical Directors and have the following responsibilities:

- Planning and coordinating the operations of their groups to meet client expectations.
- Scheduling sample preparation and analyses according to holding times, quality criteria, and client due dates.
- Ensuring a multi-level review of 100% of data generated by their groups.
- Coordinating nonconformances and corrective actions in conjunction with the Quality Systems Management team.
- Serving as technical resources to their groups, including data review.
- Managing special projects, reviewing new work proposals, and overseeing the successful implementation of new methods.
- Monitoring and controlling expenses incurred within their groups such as overtime and consumables.
- Providing performance and career development feedback to their group members.

2.7 Laboratory and Technical Staff - General Requirements

At GEL, every effort is made to ensure that the laboratory is sufficiently staffed with personnel who have the training, education, and skills to perform their assigned jobs competently.

Depending upon the specific position, laboratory personnel are responsible for:

- Complying with quality assurance and quality control requirements that pertain to their group and/or technical function.
- Demonstrating a specific knowledge of their particular function and a general knowledge of laboratory operations.
- Understanding analytical test methods and standard operating procedures that are applicable to their job function.
- Documenting their activities and sample interactions in accordance with analytical methods and standard operating procedures.
- Implementing the quality assurance program as it pertains to their respective job functions.
- Identifying potential sources of error and reporting any observed substandard conditions or practices.
- Identifying and correcting any problems affecting the quality of analytical data.
- Identifying and performing all client specific requirements outlined in the special requirements on the pull sheet of every batch.

2.8 Information Systems Manager

The Information Systems Manager reports directly to the COO. The responsibilities of this position include management of the Computer Services Team and AlphaLIMS, our laboratory information management system.

The combined responsibilities of the Information Systems Team, performing under the leadership of the Information Systems Manager, include the:

- Development and maintenance of all software and hardware.
- Translation and interpretation of routines for special projects.
- Interpretation of general data and quality control routines.
- Optimization of processes through better software and hardware utilization.

- Customization, testing and modification of data base applications.
- Maintenance and modification of our computer modeling, bar coding, CAD, statistical process control, project management, and data packaging systems.
- Development and maintenance of client and internal electronic data deliverables.
- Validation and documentation of software used in processing analytical data.

2.9 Environmental Manager

The Environmental Manager oversees our physical facility, laboratory and radiation safety programs, and instrumentation. This position reports to the COO, and manages and supervises the functions and staff assigned to these areas.

Responsibilities of the Environmental Manager include:

- Planning, evaluating, and making recommendations for facility maintenance, additions and renovations.
- Overseeing building renovations and new construction activities.
- Implementation of the Chemical Hygiene and Radiation Safety programs.
- Installing, maintaining, repairing, and modifying analytical instrumentation.
- Providing technical expertise and training in instrumentation operation, calibration, and maintenance.
- Monitoring and ensuring regulatory compliance for waste management operations and off-site disposal.

2.10 Radiation Safety Officer

The Radiation Safety Officer (RSO) reports to the COO. The RSO is responsible for the administration and execution of GEL's Radiation Protection Program. This person provides technical guidance and leadership for all issues concerning radiation health and safety as well as direct operations to ensure compliance with South Carolina Department of Health and Environmental Control (SCDHEC) regulations for radioactive materials.

Responsibilities of the RSO include:

- Establishing and enforcing policies consistent with the principles and practices designated to maintain all exposure to ionizing radiation "As Low As Reasonably· Achievable" (ALARA).
- Supervising Radiation Protection Specialists in the execution of radiological surveys and maintenance of the Radioactive Material License inventory.
- Executing the Personal Dosimetry, Air Effluent Monitoring, and Sealed Radioactive Source Leak Test Programs.
- Developing procedures and protocols to establish and maintain compliance.
- Providing training for staff in proper radiation protection practices.

2.11 Director of Human Resources

The Director of Human Resources reports directly to the CEO. The DHR manages the design, implementation, and ongoing development of our Human Resources. Responsibilities of the DHR include:

- Administration, orientation, and indoctrination of all new employees.
- Administration and compliance with Federal, State, and Local employment regulations.
- Sourcing candidates for all functional positions to maintain and strengthen the technical services provided by GEL.
- Management of occupational health and safety as it relates to Federal, State, and OSHA regulations.

2.12 Employee Training

To ensure that our clients receive the highest quality services possible, we train our employees in the general policies and practices of the company, as well as the specific operating procedures relative to their positions. We conduct and document this training

according to GL-HR-E-002 for Employee Training and GL-QS-E-017 for Maintaining Technical Training Records.

New employees participate in a company orientation shortly after they are hired. During orientation they receive information on quality systems, ethics/data integrity, laboratory safety, and employment practices. Each new employee is also provided a manual that reiterates our policies on equal opportunity, benefits, leave, conflicts of interest, employee performance, and disciplinary action. Employees can access standard operating procedures, the Quality Assurance Plan, Safety, Health, and Chemical Hygiene Plan, and the Laboratory Waste Management Plan on GEL's Intranet.

Other training provided on an ongoing basis may include:

- Demonstration of initial proficiency in analytical methods and training to SOPs conducted by a trainer who has been documented as qualified and proficient in the process for which training is being provided.
- Demonstration of continued analyst proficiency is updated continuously, using the most recent data available in AlphaLIMS. Proficiency is demonstrated using the same processes as those used for initial Demonstration of Capability. (Refer to Section 8.3.1.)
- Company-wide, onsite training.
- Courses or workshops on specific equipment and analytical techniques.
- University courses.
- Professional and trade association conferences, seminars, and courses.

Documentation of employee training is the joint responsibility of the employee and the applicable Group Leader. If an SOP is revised during the course of the year, training to the revised SOP must be documented.

2.13 Ethics and Data Integrity

As our corporate vision statement explains, "We are a company that values: Excellence as a way of life, Quality Service, a Can-Do attitude, and a fundamental commitment to Ethical Standards." Employees attend ethics education programs that focus on the high standards of data integrity and ethical behavior mandated by our company and expected by our clients.

The annual ethics training includes:

- Specific examples of unethical behaviors for the industry and for the laboratory.
- Explanation of Internal Auditing for unethical behaviors and practices.
- GEL use of electronic audit functions using instrument and AlphaLIMS software.
- Explanation of GEL's Ombudsman policy for reporting inappropriate activities.
- Examples of consequences of inappropriate or unethical behaviors/practices.
- Examples of impartiality from commercial, financial or other pressures, both external and internal.

All employees sign an Ethics and Data Integrity Agreement that reflects their commitment to always perform their duties with these high standards. (Refer to Appendix F.) During the initial and continuing Ethics and Data Integrity training, GEL's policy on confidential reporting of potential integrity issues is thoroughly discussed. Potential business or data integrity issues are handled and reviewed in a confidential manner until such time as a follow-up evaluation, full investigation, or other appropriate actions have been completed and issues clarified. All investigations are confidentially processed by GEL's QSD, or other members of GEL's Laboratory Management staff under the direction of the QSD. All investigations that result in finding of inappropriate activity are properly documented and include any disciplinary actions involved, corrective actions taken, and all appropriate notifications of clients. The QSD is responsible for updating GEL's Executive Committee on the progress of integrity investigations during regularly scheduled meetings.

2.14 Confidentiality

The laboratory maintains the confidentiality and proprietary rights of information including the type of work performed and results of analysis. Laboratory personnel and staff are informed of this policy and sign a confidentiality agreement.

A confidentiality statement accompanies the electronic transfer of data from GEL via telefacsimile (fax) or electronic mail systems (email). Government affiliated auditing agencies have access to pertinent laboratory records. However, contract, third party, and client auditors have access only to those records that may be applicable to their inspection and shall not be granted access to client records that may be considered in conflict with their interests, unless prior authorization has been given by the submitting client. Confidential information may be purged of references to client identity, project and/or sample identity by the laboratory so that records may be provided to other entities (e.g. auditors) for review.

2.15 Impartiality

The laboratory is committed to Impartiality in producing valid results derived under its range of activities or scope of work. Results are provided accurately, objectively, clearly and in a report format which includes all the information necessary for the interpretation of the results. All information required by the method used and agreed with the customer is reported. The laboratory strives to maintain impartiality from commercial, financial or other pressures which might compromise impartiality. In addition to internal management structure mitigating undue pressures on employees, the laboratory reviews requests and tenders for possible risks to impartiality prior to bidding on work.

Our Core Values, along with procedures, plans, and policies outlined in this Quality Assurance Plan, scheduled management meetings, and monitoring of key performance indicators help in the management of risks on an on-going basis.

SECTION 3
QUALITY SYSTEMS

Section 3 - Quality Systems

Our Quality Systems include all quality assurance (QA) policies and quality control (QC) procedures necessary to plan, implement, and assess the work we perform. GEL's QA Program establishes a quality management system (QMS) that governs all of the activities of our organization.

GEL's quality management system is designed to conform to the requirements specified in the standards referenced in Appendix A. Essential elements of our quality management system are described in this section and Appendix E.

3.1 Quality Systems Team

The Quality Systems Team monitors risks to impartiality, confidentiality and other undue influences which could adversely affect confidence in the laboratory's competence, judgement or operational integrity. This team monitors conformity to the range of activity under which it performs. Risks which may affect the validity of results are identified, monitored and assessed as to the potential impact on the validity of the results. This group is responsible for recording and managing customer complaints through the laboratory non-conformance reporting system. .

Following is a summary of the responsibilities of each position, in addition to the duties discussed in section 2.3

3.1.1 Quality Systems Director

- Reports to the CEO
- Demonstrates strict adherence to and support of the company ethics policy
- Serves as management's representative for quality
- Responsible for the implementation and maintenance of the QMS
- Supervises the Quality Systems Team and their functions
- Initiates and recommends preventive action and solutions to quality problems
- Implements appropriate action to control quality problems until solutions are implemented and verified to be effective
- Verifies that effective solutions are implemented
- Demonstrates knowledge of the Quality System as defined by NELAC, TNI, NUPIC, ISO/IEC 17025, DOECAP DoD ELAP, and DOELAP.

3.1.2 Quality Systems Lead Auditor

- Reports to the Quality Systems Director
- Demonstrates strict adherence to and support of the company ethics policy.
- Demonstrates knowledge of the Quality System defined under NELAC, TNI, DOECAP,DoD ELAP, DOELAP, NUPIC and other quality standards such as ISO/IEC 17025-2017.
- Plans, schedules and participates in GEL's client audits, internal audits, and subcontractor audits
- Conducts conformance audits as necessary to verify implementation and closure of audit action items
- Serves as liaison to client and third party auditors
- Coordinates laboratory responses to audit reports and prepares final response
- Monitors progress of corrective actions
- Prepares and monitors progress of internal and subcontractor audit reports

3.1.3 Quality Assurance Officers

- Report to the Quality Systems Director
- Demonstrate strict adherence to and support of the company ethics policy.
- Demonstrate the ability to evaluate data objectively without outside influence
- Have documented training and/or experience in QA/QC procedures and knowledge of the Quality system as defined under NELAC, TNI and ISO 17025.
- Have knowledge of analytical methods

- Assist in the conduct of internal and supplier audits and requests for pricing reviews
- Administer corrective actions and nonconformances
- Monitor and respond to client -identified nonconformances and technical inquiries
- Implement and maintain statistical process control (SPC) system
- Ensure the monitoring of balances and weights, and temperature regulation of ovens, water baths, and refrigerators
- Coordinate the monitoring of DI water system and volatile organics storage coolers
- Maintain Method Detection Limit studies
- Write or review quality documents and standard operating procedures under the direction of the QS Director
- Provide training in quality systems and good laboratory practices.
- Manage laboratory certification processes
- Coordinate the receipt and disposition of external and internal performance evaluation samples.

NOTE: Once PE samples have been prepared in accordance with the instructions provided by the PE vendor, they are managed and analyzed in the same manner as environmental samples from clients. The analytical and reporting processes for PE samples are not specially handled.

3.1.4 Quality Systems Specialists/Document Control Officer

- Reports to the Quality Systems Director
- Demonstrates strict adherence to and support of the company ethics policy.
- Assist the team as directed with respect to Records Management, Document Control, Laboratory Certification, temperature and weight calibrations, logbook review, training documentation, and nonconformances, etc.

3.2 Quality Documents

Our Quality Systems policies and procedures are documented in this and other supporting documents. GEL's management approves all company quality documents. Pre-approval is secured for any departures from such documents that may affect quality.

In addition, to the QA Plan, Quality Systems allows for QA Project Plans (QAPjP) and includes standard operating procedures and any other quality assurance program requirements defined by individual contracts. The QA Plan describes the quality standards that we apply to our laboratory operations. We use Quality Assurance Project Plans to specify individual project requirements. The QA Plan and supporting documents are verified to be understood and are implemented throughout the laboratory fractions to which they apply.

Finally, our Standard Operating Procedures (SOPs) are used to describe in detail those activities that affect quality. SOPs are prepared, authorized, changed, revised released, and retired in accordance with GL-ADM-E-001. SOPs are accessible electronically via GEL's Intranet.

3.3 Document Control

The control of quality documents is critical to the effective implementation of our Quality Program. We define and control this process in accordance with GL-DC-E-001 for Document Control. Responsibilities for document control are divided between the Group Leaders and the Document Control Officer (DCO).

Group Leaders are responsible for:

- Supporting the development and maintenance of controlled documents that apply to their respective departments.
- Reviewing all quality documents annually for continued validity.
- Ensuring documentation that the affected employees are aware of revisions to documents or manuals.

The Computer Services Team is responsible for:

- Electronic maintenance of all records required for control, re-creation, and maintenance of analytical documentation.
- Maintenance of electronic copies of archived data and the electronic log of how they were determined.

The DCO is responsible for:

- Demonstrating strict adherence to and support of the company ethics policy.
- Managing the system for the preparation, authorization, change, revision, release, and retirement of the Quality Manual, QAP, project plans, and standard operating procedures.

- Ensuring that current controlled documents are accessible via GEL's Intranet.
- Managing a system to document current revision numbers and revision dates for all distributed documents and manuals.
- Managing a system to identify the nature of document revisions.
- Maintaining hard or electronic copies of obsolete documents.
- Maintaining electronic or hard copy originals of all controlled documents.

Revisions to controlled quality documents are made by replacing individual sections or the entire document, as determined by the DCO.

3.4 Controlled Document Review

Internally generated controlled documents undergo a multi-level review and approval process before they are issued. These levels include a procedural review, technical and/or quality review and final authorization of the appropriate manager or director if necessary. To ensure that new or revised standard operating procedures are not implemented prematurely, SOPs are effective upon the date of the final approval signature.

3.5 Quality Records

Quality records provide evidence that specified quality requirements have been met and documented. We generate them in accordance with applicable procedures, programs, and contracts. Quality records include but are not limited to:

- Observations
- Calculations
- Calibration data
- Certificates of analysis
- Certification records
- Chains of custody
- Audit records
- Run logs, instrument data, and analytical logbooks
- Instrument, equipment, and building maintenance logs
- Material requisition forms
- Monitoring logs
- Nonconformance reports and corrective actions
- Method development and start-up procedures including method detection limit studies
- Technical training records
- Waste management records
- Standard logs
- Software validation documentation
- Standard Operating Procedures (SOPs)
- Sample collection and field data

Our quality records are:

- Documented in a legible manner.
- Indexed and filed in a manner conducive to ready retrieval.
- Stored in a manner that protects them from loss, damage, and unauthorized alterations.
- Accessible to the client for whom the record was generated.
- Retained and disposed in the identified time period.

The generation, validation, indexing, storage, retrieval, and disposition of our quality records are detailed in GL-QS-E-008 for Quality Records Management and Disposition. The quality records of subcontracted services are also required to meet the conditions established in this SOP.

3.6 Internal and Supplier Quality Audits

We conduct internal audits annually to verify that our operations comply with the requirements of our QA program and those of our clients. We perform supplier audits as necessary to ensure that they too meet the requirements of these programs. Both internal and supplier audits are conducted in accordance with GL-QS-E-001 for the Conduct of Quality Audits.

3.6.1 Audit Frequency

Internal audits are conducted at least annually in accordance with a schedule approved by the Quality Systems Director. Supplier audits are contingent upon the categorization of the supplier, and may or may not be conducted prior to the use of a supplier or subcontractor (Refer to GL-QS-E-001.) Type I suppliers and subcontractors, regardless of how they were initially qualified, are re-evaluated at least once every three years.

Additional internal and supplier audits may be scheduled if deemed necessary.

3.6.2 Audit Team Responsibilities

Internal and supplier audits are conducted by qualified staff under the direction of the Lead Auditor or Quality Systems Director. A qualified audit team member shall have the technical expertise to examine the assigned activities.

We do not allow staff to audit activities for which they are responsible or in which they are directly involved. It is the responsibility of the Lead Auditor to ensure that such conflicts of interest are avoided when the audit team is assembled.

The Leadership Team has a significant role in the internal audit process, including:

- Provision of audit personnel
- Empowerment of the audit team with authority to make the audit effective
- Development and implementation of timely corrective action plans

3.6.3 Identification and verification of OFIs

Opportunities for Improvement are identified conditions that have potential to improve the quality of products or services. Several examples of objective evidence are used to support an OFI, which might be classified as an, observation, and/or recommendation.

The Lead Auditor may initiate an OFI and may reference a Nonconformance Report (NCR) or Corrective Action Request and Report (CARR) The OFI, is then entered into the NCR system per GL-QS-E-012 for NCR Database Operation.

Implementation of any changes or action is verified as effective prior to implementation. The OFI may be verified for continued effective implementation during the next scheduled audit.

3.7 Managerial and Audit Review

Our Leadership Team reviews the audit process at least annually. This ensures the effectiveness of the corrective action plan and provides the opportunity to introduce changes and improvements.

We document all review findings and corrective actions. Implementation plans and schedules are monitored by the Quality Systems Team.

3.8 Nonconformances

Processes, materials, and services that do not meet specifications or requirements are defined as nonconforming. Such nonconformances can include items developed in-house or purchased from vendors, samples received from clients, work in progress, and client reports.

At GEL, we have a nonconformance reporting system (NCR) that helps us prevent the entry of defective goods and services into our processes and the release of nonconforming goods and services to our clients. Our NCR system provides a means for documenting the disposition of nonconforming items and for communicating these to the persons involved in the process affected by the adverse condition(s).

Nonconformances are documented according to GL-QS-E-004 for the Documentation of Nonconformance Reporting and Dispositioning and Control of Nonconforming Items. We regularly review SOPs, client complaints, and quality records, including completed NCRs, to promptly identify conditions that might result in situations or services that do not conform to specified quality requirements.

Our Quality Group processes, categorizes and trends nonconformances. Trending information may be provided to the Leadership Team and Group Leaders of the affected areas.

3.9 Corrective Action

There are two categories of corrective action at GEL. One is corrective action implemented at the analytical and data review level in accordance with the analytical SOP. The other is formal corrective action documented by the Quality Systems Team in accordance with GL-QS-E-002. Formal corrective action is initiated when a nonconformance reoccurs or is so significant that permanent elimination or prevention of the problem is required.

We include quality requirements in most analytical SOPs to ensure that data are reported only if the quality control criteria are met or the quality control measures that did not meet the acceptance criteria are documented.

Formal corrective action is implemented according to GL-QS-E-002 for Conducting Corrective/Preventive Action and Identifying Opportunities for Improvement

and documented according to GL-QS-E-012 for NCR Database Operation.

Any employee at GEL can identify and report a nonconformance and request that corrective action be taken. Any GEL employee can participate on a corrective action team as requested by the QS team or Group Leaders. The steps for conducting corrective action are detailed in GL-QS-E-002.

In the event that correctness or validity of the laboratory's test results is doubted, the laboratory will take corrective action. If investigations show that the results have been impacted, affected clients will be informed of the issue in writing within 5 calendar days of the discovery.

GEL will notify all affected customers of any data quality issues resulting from nonconforming work within 15 business days of discovery. GEL will provide and submit records of the corrective actions to resolve the nonconformance(s) to the customer(s) with 30 business days. This procedure will also be followed to notify GEL's accrediting body if the laboratory experiences any instances of inappropriate and prohibited laboratory practices. GEL will perform these procedures in accordance with SOP GL-QS-E-002 Conducting Corrective/Preventative Action and Identifying Opportunities for Improvement.

3.10 Performance Audits

In addition to internal and client audits, our laboratory participates in annual performance evaluation studies conducted by independent providers. We routinely participate in the following types of performance audits:

- Proficiency testing and other inter-laboratory comparisons.
- Performance requirements necessary to retain certification (Appendix D).
- Evaluation of recoveries of certified reference and in-house secondary reference materials using statistical process control data.
- Evaluation of relative percent difference between measurements through SPC data.

We also participate in a number of proficiency testing programs for federal and state agencies and as required by contracts. It is our policy that no proficiency evaluation samples be analyzed in any special manner.

Our annual performance evaluation participation generally includes a combination of studies that support the following:

- US Environmental Protection Agency Discharge Monitoring Report, Quality Assurance Program (DMR-QA). Annual national program sponsored by EPA for laboratories engaged in the analysis of samples associated with the NPDES monitoring program. Participation is mandatory for all holders of NPDES permits. The permit holder must analyze for all of the parameters listed on the discharge permit. Parameters include general chemistry, metals, BOD/COD, oil and grease, ammonia, nitrates, etc.
- Department of Energy Mixed Analyte Performance Evaluation Program (MAPEP). A semiannual program developed by DOE in support of DOE contractors performing waste analyses.
- ERA's MRAD-Multimedia Radiochemistry Proficiency test program. This program is for labs seeking certification for radionuclides in wastewater and solid waste. The program is conducted in strict compliance with USEPA National Standards for Water Proficiency study.
- ERA's InterLaB RadCheM Proficiency Testing Program for radiological analyses. This program completes the process of replacing the USEPA EMSL-LV Nuclear Radiation Assessment Division program discontinued in 1998. Laboratories seeking certification for radionuclide analysis in drinking water also use the study. This program is conducted in strict compliance with the USEPA National Standards for Water Proficiency Testing Studies.
- Water Pollution (WP). Biannual program for waste methodologies. Parameters include both organic and inorganic analytes.
- Water Supply (WS): Biannual program for drinking water methodologies. Both organic and inorganic parameters are included.

At GEL, we also evaluate our analytical performance on a regular basis through statistical process control acceptance criteria. Where feasible, this criterion is applied to both measures of precision and accuracy and is specific to sample matrix.

We establish environmental process control limits at least annually. In Radiochemistry, quality control

evaluation is based on static limits rather than those that are statistically derived, unless specified by regulatory programs such as Drinking Water. Our current process control limits are maintained in AlphaLIMS. GEL maintains client-specific and program-specific control limits and reporting requirements in the LIMS. Examples of client or program specific limits may be found in documents such as the DoD QSM tables in Appendices B and C of DOD-DOE QSM version 5.2, and in the HASQARD Standard which are available as Quality Systems documents.

We also measure precision through the use of matrix duplicates and/or matrix spike duplicates. The upper and lower control limits (UCL and LCL respectively) for precision are plus or minus three times the standard deviation from the mean of a series of relative percent differences. The static precision criteria for radiochemical analyses are 0 – 20% for activity levels exceeding the contract required detection limit (CRDL).

Accuracy is measured through laboratory control samples and/or matrix spikes, as well as surrogates and internal standards. The UCLs and LCLs for accuracy are plus or minus three times the standard deviation from the mean of a series of recoveries. The static limit for radiochemical analyses is 75 – 125%, except as specified by the Drinking Water regulations. Specific Instructions for out-of-control situations are provided in the applicable analytical SOP.

3.11 Control Charts

Per the U.S. Department of Energy, Quality Systems for Analytical Services (DOE QSAS): Control charts are a graphical representation of data taken from a repetitive measurement or process. Control charts may be developed for various characteristics, (e.g. mean, standard deviation, range, etc.) of the data. Per MARLAP "A control chart has two basic uses:

- As a tool to judge if a process was in control.
- As an aid in achieving and maintaining statistical control.

For applications related to radiation detection instrumentation or radiochemical processes, the mean (center line) value of a historical characteristic (e.g. mean detector response), subsequent data values and control limits placed symmetrically above and below the center line are displayed on a control chart."

For GEL's Chemistry, Radiochemistry, and Bioassay laboratories, the Computer Services Team (CST) developed a program where Group Leaders are sent email notifications that provide LCS failures by compound/analyte name. This assists the Group Leader with monitoring out of control situations due to laboratory contamination or analyst error. This program sends notifications once a week.

Each Group Leader may utilize programs in LIMS where they can review trending data as control charts by work order or by the SPC program.

GEL's QA Officer or designee shall review control charts during the period when the LIMS SPC program queries data points for analyses that require dynamic SPC limits for quality control parameters. This is performed on a biannual basis. At this time, any out of control conditions will be identified and a corrective action initiated. The QA Officer shall be able to stop unsatisfactory work or prevent the reporting of results generated from this program.

Dynamic SPC limits for control parameters are generally developed when more than 20 data points are available for review. Data points may be determined as outliers based on the process knowledge of the procedure being evaluated and the professional opinion of the data reviewer.

During their annual system review, management will evaluate the need to consolidate any redundant procedures and/or policies to help eliminate any confusion for work processes.

3.12 Essential Quality Control Measures

Some quality control measures are method-specific. There are, however, general quality control measures that are essential to our quality system. These quality measures include:

- Monitoring of negative and positive controls
- Defining variability and reproducibility through duplicates
- Ensuring the accuracy of test data including calibration and/or continuing calibrations, use of certified reference materials, proficiency test samples, etc.
- Evaluating test performance using method detection limits and quantitation limits or range of applicability such as linearity
- Selecting the appropriate method of data reduction

- A copy of GEL's Ethics and Integrity Agreement is provided in Appendix F.

SECTION 4
FACILITIES

Section 4 – Facilities

Our laboratory is designed with a full-service approach to handling environmental needs. The layout provides dedicated space for radiochemical analyses, bioassay analysis, organic extractions, semi-volatile organic analyses, volatile organic analyses, metals analyses, general chemistry analyses, and air analyses.

The laboratory and support offices occupy approximately 85,000 square feet engineered to meet the stringent quality control and utility requirements of the modern environmental laboratory. Records are temporarily stored on-site then warehoused in a climate-controlled building off-site. The diagram in Appendix H depicts the layout of the laboratories.

Discussed in this section are:

- Facility security
- Utility services and deionized water
- Prevention of contamination
- Assessment of contamination

4.1 Facility Security

Our facility features secured laboratory and storage areas. Restricted entry assures sample integrity and client confidentiality, which satisfies clients and potential national security interests.

Visitors cannot gain entry without being escorted through the laboratory by authorized personnel. A designated sample custodian and a bar-coded chain-of-custody provide a second level of security.

4.2 Utility Services

Each defined laboratory area is equipped with the following utilities:

- Cold water
- Hot water
- Deionized water
- Compressed air
- Natural gas
- Vacuum
- 110 Volt AC
- 208 Volt AC (at selected stations)
- Specialty gases (as required)

4.2.1 Deionized Water

We have two independent deionized water (DI) systems. One serves radiochemistry while the other serves the remaining laboratories. DI water is made from city water flowing through a reverse osmosis system and a deionization system capable of producing 5 gallons per minute of Type I laboratory water.

We monitor compliance according to GL-LB-E-016 for The Collection and Monitoring of the DI Water Systems. Our monitoring activities and frequencies can be found in Table 1 of the SOP.

4.2.2 Specialty Gasses

The specialty compressed gasses may be required by specific analytical systems. Each specialty compressed gas system is monitored for background contamination that would negatively impact the efficiency of the operating system. Monitoring is generally conducted through use of routine instrument control samples which are introduced to the operating system prior to instrument calibrations and throughout the analytical process. Requirements for the purity of the gasses are identified in the instrument operating manuals and standard operating procedures.

4.3 Prevention of Contamination

Work areas that are free of sample contaminants, constituents and measurement interferences are important to the generation of quality data. With this in mind, we designed our laboratories to prevent contamination and reinforce this design with good laboratory practices.

In addition to keeping our work areas free of dust and dirt accumulations, policies and features that prevent or minimize contamination include:

- An air conditioning system that controls the environment of individual laboratories for optimum performance of sensitive instruments and to eliminate potential cross contamination.
- Segregation of volatile and semi-volatile laboratories to minimize potential contamination associated with the use of commonly required solvents.

- Negative and positive pressure air locks to isolate selected laboratories to prevent the entry of airborne contaminants.
- Fume hoods to remove fumes and reduce the risk of aerosol and airborne contaminants and personal safety hazards are monitored in accordance with GL-FC-E-003 for Local Exhaust Ventilation Systems.
- Restricted access to the volatiles laboratory (authorized personnel only).
- Designated area for glassware preparation wherein all glassware used in sample prep and analysis is cleaned according to GL-LB-E-003 for Glassware Preparation.
- Segregated storage areas for volatiles and radioactive samples.
- Production, use, and monitoring of Type I DI water.
- Tracking and trending of any significant sample and/or reagent spills using the AlphaLIMS NCR system, allowing efficient analysis of any potential contamination.

4.4 Assessment of Contamination Levels

We evaluate contamination resulting from the following sources on the basis of quality assurance and quality control data derived from the analytical method and method blanks.

- Sample containers
- Reagent water
- Reagents and solvents
- Sample storage
- Chemical and physical interference
- Constituent carryover during analysis

Contamination in each of the volatile storage coolers is monitored by the weekly analysis of water blanks. Two DI water blanks are placed in each monitored cooler at the beginning of each month with one being analyzed each week. If the concentration of any target analyte exceeds the PQL, this is verified (with the second blank for that week) and corrective action is implemented to eliminate the source of contamination, evaluate the effect of samples stored in the cooler, and to notify clients. SOP GL-OA-E-058 discusses these practices in detail.

SECTION 5
EQUIPMENT AND REFERENCE MATERIALS

Section 5 – Equipment and Reference Materials

GEL's ability to efficiently generate data that are reproducible, accurate, and legally defensible is attributable to our use of high-quality instruments, equipment, and reference materials.

Provided in this section are:

- GEL's policies governing instruments, equipment, and reference materials
- Identification of instrumentation and support equipment
- Procurement protocol

5.1 General Policies

It is our policy to purchase instrumentation, equipment and high-quality reference materials that meet or exceed the method and regulatory requirements for the analyses for which we are accredited. If we need to use instruments or equipment not under our permanent control, we ensure that it also meets these standards.

Instrumentation and equipment are placed into service on the basis of ability to meet method or regulatory specified operating conditions such as range and accuracy. All laboratory instrumentation and testing equipment is maintained in accordance with standard operating procedures (SOPs).

Instrumentation and equipment is used in a manner that assures, where possible, that measurement uncertainty is known and consistent with specified quality requirements. Instruments and equipment are taken out of service and segregated or labeled as such under the following conditions:

- Mishandling and/or overloading
- Results produced are suspect
- Demonstrated defect or malfunction

Tagged or segregated instruments and equipment remain out of service until repaired and shown by test, calibration, or verification to perform satisfactorily. Instruments that are in service and normally calibrated prior to and during use are not tagged.

Each item of equipment, including reference materials is, if appropriate, labeled, marked or otherwise identified to indicate its calibration status. We maintain records for each major item of equipment, instrumentation, and all reference materials significant to quality performance. These records are often in the form of maintenance logs, which are kept in accordance with GL-LB-E-008 for Basic Requirements for the Use and Maintenance of Laboratory Notebooks, Logbooks, Forms, and Other Recordkeeping Devices.

Documentation included in these records may include but is not limited to:

- Equipment name
- Manufacturer's name
- Type identification
- Serial number or other unique identification
- Date received and date placed in service (if pertinent)
- Current location
- Condition when received (if known)
- Manufacturer's instruction, where available
- Dates and results of calibrations and or verifications
- Date of next calibration and/or verification, where written procedures do not specify frequency
- Details of maintenance carried out to date and planned for the future
- History of any damage, malfunction, modification or repair

5.2 Instrumentation and Support Equipment

Appendix G lists the instruments we use for the analysis of environmental, radiochemical and bioassay samples. Where feasible, our instruments are equipped with autosamplers that improve efficiency and facilitate consistent sample introduction to the sample detector. They are also connected to an area network to facilitate data transfer.

Devices that may not be the actual test instrument but are necessary to support laboratory operations are referred to as support equipment. We also maintain this equipment in proper working order. Support equipment utilized at GEL includes:

- balances
- ovens
- refrigerators
- freezers
- incubators
- water baths
- temperature measuring devices
- volumetric dispensing devices
- muffle furnaces
- distillation apparatus
- grinders and homogenizers
- hot plates and heating mantles
- ultraviolet sterilizers.

Guidelines for the required calibration and evaluation of this equipment are discussed in Section 7.

We perform radiochemical and bioassay analytical services in accordance with the instrumentation and reference methods approved by the Department of Energy (DOE), the Environmental Measurements Lab (EML), the Environmental Protection Agency (EPA), ASTM, and Los Alamos Health and Environmental Chemistry (LAHEC). Modifications to these methods may be appropriate as a result of Performance Based Measurement Systems (PBMS).

SOPs are used to describe our procedures for all routine analyses performed by our labs. These procedures include step-by-step instructions for sample collection, storage, preparation, analysis, instrument calibration, quality control, disposal, and data reporting.

5.3 Procurement and Control of Purchased Items

Materials, equipment, and services that affect the quality of our products are designated as Quality Materials, Equipment, and Services and are only purchased from approved suppliers. We approve and document suppliers according to GL-QS-E-001 for the Conduct of Quality Audits.

At GEL, we maintain documentation of specific quality requirements for Quality Materials and Services. Records that document the quality of a product or service may include:

- certificates of analysis and traceability
- verifications of chemical quality
- inspections of equipment or materials
- verifications or inspections of vendor product specifications

Our procedure for requisitioning supplies, instruments, equipment and other common use material is described in GL-RC-E-002 for Material Requisition. These requests typically include:

- The date and name of person(s) requesting materials
- Account, department, project number to which the material is to be billed
- Recommended supplier or vendor
- Additional information necessary to expedite the purchase request
- Specifications that could affect the quality of products and services
- Vendor's material part number
- Amount of material needed
- Description of material
- Cost per unit
- Person(s) authorizing the purchase
- Time frame in which the material is needed

The equipment, instruments, and reference materials we purchase are inspected upon receipt in accordance with GL-RC-E-001 for the Receipt and Inspection of Material and Services. This inspection is to verify that procured items meet the acceptance criteria defined in the procurement documentation. Staff performing initial inspection routinely:

- Open and inspect all items for damage
- Compare the items with the issued purchase order or contract for catalog or part number, description or procurement specification, quality requirement, and acceptance criteria
- Label items with a limited shelf life with the date received
- Determine if the items conform to the specifications agreed to by the vendor.

The individual responsible for the technical acceptance of the item provides procurement and receiving staff with the proper acceptance documentation. Items found not to conform to quality standards are returned to the supplier, identified as nonconforming or disposed according to the established procedures in GL-

QS-E-004 for AlphaLIMS Documentation of Nonconformance Reporting and Dispositioning and Control of Nonconforming Items. These nonconforming items may also include those identified as suspect/counterfeit items as identified in DOE guide DOE G 414.-3 for use with DOE 414.1B, C and D.

SECTION 6
HEALTH AND SAFETY

Section 6 – Health and Safety

GEL maintains a safe work environment and promotes healthy work practices. Our corporate Safety, Health, and Chemical Hygiene Plan was developed by a resident certified industrial hygienist. Procedures outlined in the plan are consistent with Occupational Safety and Health Administration, CERCLA, the Environmental Protection Agency, and SCDHEC.

All employees are trained in the safety practices applicable to their job functions. This training is conducted in accordance with GL-HR-E-002 for Employee Training.

Discussed in the section are:

- Fire safety and safety equipment
- Safety equipment and procedures related to handling radioactive samples

6.1 Fire Safety

Our facility is equipped with a fire alarm system designed to detect smoke in all areas of the facility. Certain high-risk areas, such as, the cold and ambient storage areas, organic sample preparation lab, hazardous waste lab, and solvent storage are additionally equipped with automatic halon systems. Fire blankets and dry chemical extinguishers are located at strategic points throughout the lab. We routinely inspect these extinguishers in accordance with GL-FC-E-004. Lab personnel are trained in the proper use and selection of fire extinguishers.

In order to decrease the risk of fire, bulk solvents are stored in a halon-protected storage room.

6.2 Evacuation

In the unlikely event of a fire (or other emergency), we have defined evacuation routes depicted in Appendix H. This diagram is posted in pertinent areas of the facility and designated staff members serve as evacuation leaders for the work groups.

6.3 Safety Equipment

Safety equipment, including safety glasses, lab coats, safety goggles, protective gloves, hard hats, and coveralls, is available to all employees as needed. We also provide respirators when needed to those who have completed training in the use of this specialized equipment.

Eyewashes and overhead showers are located throughout the laboratory. We routinely inspect these as directed in GL-FC-E-002 for Testing Emergency Eyewash and Shower Equipment.

6.4 Radiation Safety

Since GEL specializes in the handling of radioactive material, we have health physics procedures to ensure its safe handling. While lab personnel do not encounter significant levels of radiation requiring personal monitoring, a Dosimetry Program is in effect utilizing personal dosimeters for designated personnel. These dosimeters are exchanged quarterly and records of exposure are maintained. Instructions for the proper use of dosimeters are addressed in GL-RAD-S-009 for Personnel Dosimetry.

We take special precautions to ensure that samples are safely processed. Upon receipt, trained personnel use a survey meter to screen all samples for the presence of radioactivity. Protocols for the receipt of radioactive samples and for surveying suspected or known radioactive samples are detailed in GL-RAD-S-007 for Receiving Radioactive Packages and GL-RAD-S-001 for Radiological Surveys. This process is described in Section 9.

Upon leaving a radiologically controlled area, personnel check their hands and feet for potential contamination. This is done utilizing detection instrumentation that employs Geiger-Mueller or scintillation technologies. In addition, stations with portable detection instruments are set up for personnel frisking and in-process contamination surveys.

Key areas throughout the facility are surveyed:

- Laboratory analytical areas (Monthly smears)

- Radioactive Sample Storage Areas (Monthly smears and exposure rate)
- Sample Receipt and Waste Handling Areas (Monthly smears and exposure rate)
- Unrestricted and Radioactive Material Prohibited Areas (Quarterly smears)

SECTION 7
MEASUREMENT, TRACEABILITY, AND CALIBRATION

Section 7 – Traceability and Calibration

Traceability of measurements and the calibration of testing equipment are imperative to our ability to produce accurate and legally defensible data. As such, we have implemented procedures to ensure that equipment calibration and measurement verification are traceable to nationally recognized standards obtained from the National Institute of Standards and Technology (NIST) or accredited reference material producer (RMP) with traceability to NIST. Reference materials purchased outside the United States must be traceable back to each country's national standards laboratory or another national or international reference organization such as ILAC, APLAC and/or IAAC. The RMP may also have established acceptability by its approval as an ISO Guide 34 RMP. Commercial suppliers of radiochemistry reference standards/sources must conform to ANSI N42.22 and must be accompanied by a certificate of calibration consistent with ANSI N42.22-1995, section 8.

Where possible, calibration certificates provide traceability to national and/or international standards of measurement.

Calibration certificates provide measurement results and any associated uncertainty of measurement, and/or a statement of compliance with the identified specification. Calibration certifications are maintained as quality records.

When traceability to a national standard is not applicable, verification of measurement is achieved through inter-laboratory comparisons, proficiency tests, or independent analyses.

The following measurement and traceability practices are described in this section:

- Calibration criteria for support equipment
- General requirements
- Balances
- Temperature-sensitive devices and temperature monitoring
- Air displacement pipets
- Calibration criteria for instruments
- Calibration verification
- Initial calibration verification
- Continuing calibration verification

7.1 Calibration Criteria for Support Equipment

This section addresses calibration protocols for support equipment, including balances, temperature – sensitive equipment, and air displacement pipets. The general criteria applicable to the calibration of support equipment are as follows:

- Equipment is maintained in proper working order. Records of all maintenance activities including service calls are kept.
- Calibrations or re-verifications over the entire range of use, using NIST-traceable references when available, are conducted either quarterly, annually or biennially.
- The laboratory is allowed to re-verify some standards, sources and reagents to extend their expiration dates. However these reverifications must meet method acceptance criteria for their specific method and intended use. This has been GEL's process for numerous years and the laboratory has established a track record for both the reference materials and the producers. The reference materials verified/re-verified by the process have been subjected to numerous interlaboratory comparisons and cross-checked by use of different methods over a period of many years.
- If results of calibration and verification are not within the specifications for the equipment's application, then:
  1. The equipment is removed from service until repaired
  2. Under certain conditions, a deviation curve may be prepared. All measurements are corrected for the deviation, recorded and maintained.

- Prior to use each day, balances, ovens, freezers, refrigerators, incubators, and water baths are checked with NIST-traceable references (where possible) in the expected range of use.
- If prescribed by the test method, additional monitoring is performed for a device used in a critical test (such as an incubator or water bath).
- Support equipment is used only if the reference standard specifications (provided by the supplier or described in the analytical method) are met.
- Reference standards of measurement such as Class S or equivalent weights or traceable thermometers may be used for calibration when demonstrated that their performance as reference standards will not be invalidated.
- Reference standards of measurement are calibrated by a body that can provide, where possible, traceability to a national standard.
- Reference standards and measuring and testing equipment are, subject to in-service checks between calibrations and verifications, in accordance with ANSI/ISO/IEC 17025-2017.
- Reference materials, where possible, are traceable to national or international standards of measurement, or to national or international standard reference materials.
- Mechanical volumetric dispensing devices, except Class A glassware, are checked monthly for accuracy.

7.1.1 Balances

Our balances are under a service contract for annual calibration, maintenance, and cleaning. Each balance is labeled with a serial number, service date, date of next service, and signature or initials of the service technician.

Balances are set up, calibrated, and operated in the range required by the analytical method in accordance with GL-LB-E-002 for Balances. Prior to using a balance, the analyst is responsible for checking its calibration.

Calibration and calibration verification are performed using weights that are or have been calibrated against Class S or equivalent weights. These weights are traceable to NIST and calibrated biennially by a calibration service provider that meets the requirements of the ANSI/ISO/IEC 17025-2017 standard.

Calibration and calibration verification are recorded in the electronic balance calibration logbook. If the calibration or calibration verification does not meet the specified acceptance criteria, the balance is recalibrated. If the calibration criteria are still not met, the balance is removed from service and tagged as such.

7.1.2 Refrigerators, Freezers, Incubators, Ovens, Water Baths, and Similar Devices

Careful control of temperature is often central to the production of acceptable data. Temperature excursions beyond the established limits may invalidate a procedure and the associated data. Constant monitoring in accordance with GL-LB-E-004 for Temperature Monitoring and Documentation Requirements for Refrigerators, Freezers, Ovens, Incubators, and Other Similar Devices assures us that regulatory and/or method temperature requirements are being met.

We measure temperatures with thermometers that are verified either quarterly or annually against a NIST-traceable thermometer. The NIST traceable thermometers are independently verified at least annually by a verification service that meets the requirements of the ANSI/ISO/IEC 17025-2017 standard. The protocol for thermometer verification is described in GL-QS-E-007. We monitor the temperature of the following equipment according to GL-LB-E-004:

- Refrigerators and freezers used to store samples, standards, and other temperature-sensitive materials
- Incubators
- Ovens
- Water baths

We monitor the temperatures of refrigerators and freezers prior to use on each working day. The temperatures of ovens, water baths, and other devices used as part of an analytical process must be

monitored prior to, during, and immediately after use. Incubators and other devices used for other specialized analytical methods may require more frequent monitoring as specified in the corresponding SOP.

Temperature measurements are documented on logs specific to each piece of equipment. These logs may be paper or recorded electronically in LIMS. The logs may be posted on or near each refrigerator, freezer, water bath, oven, or other temperature control device. Electronic monitoring logbooks for refrigerators, freezers, and coolers with temperature probes are found in AlphaLIMS. Each log includes the following information:

- Date and time of each measurement
- Acceptance limits for device being monitored
- Whether device conforms with specifications at time of measurement
- Name, location, and number of device being monitored
- Notation of any out-of-control condition
- Any corrective action

When the process to maintain and document temperatures within acceptance limits does not conform to specifications appropriate action is then taken to document the nonconformance. According to GL-QS-E-004 for AlphaLIMS Documentation of Nonconformance Reporting and Dispositioning and Control of Nonconforming Items. Any corrective action taken to bring the equipment back into acceptable use is discussed.

Examples of nonconformances are:

- Failure to maintain process temperature within acceptance limits
- Failure of device to achieve calibration
- Total failure of temperature control device
- Failure to monitor the temperature as required

7.1.3 Air Displacement Pipets

We calibrate air displacement pipets in accordance with GL-LB-E-010 for Maintenance and Use of Air Displacement Pipets. As specified in the SOP, the calibration of an air displacement pipet is verified daily prior to use, based on a single point measurement.

The acceptance criteria for each measurement are based on the standard deviation of the calibration measurements. Tolerance limits for commonly used verification volumes and accuracy and precision checks are included in the pipet calibration logbook. Calibrations and daily calibration verifications are traceable to each pipet using the unique identification found on its label.

If a pipet does not meet the calibration tolerance limits, it is removed from service until it again demonstrates compliance after being cleaned and/or repaired. Analysts whose jobs may require the use of air displacement pipets are trained in their proper use and calibration.

7.2 Instrument Calibrations

To ensure that the data generated by an instrument are accurate, we calibrate the instrument using standards containing known concentrations of target analytes. We verify the accuracy of calibration standards by analyzing an additional standard containing the target analytes. This initial calibration verification standard (ICV) originates from a second source. Verification that the instrument response is reliable and has not changed significantly from the current calibration curve is accomplished by the analysis of a continuing calibration verification (CCV) standard. Some analytical methods employ the use of CCVs at varying concentrations.

Traceability of calibration, calibration verification, and other quality control standards to the recognized standard is documented per GL-LB-E-007 for Laboratory Standards Documentation. Preparation and Verification of Radioactive Standards is described in GL-RAD-M-001. Individual identification numbers are assigned to each source standard and each subsequent intermediate and working standard prepared.

The identification number makes it possible to trace a standard to a parent standard and ultimately to the source standard. The date each standard is prepared, the protocol used in the preparation, the person preparing the standard, and the standard's expiration date are documented in the appropriate standards log, usually maintained in AlphaLIMS. The information is accessible via the standard ID number.

We record standard and reagent ID numbers on instrument run logs, analytical logbooks, sample preparation logs, and instrument raw data. Calibration standards that are used in the analysis of a particular sample or group of samples can be traced to NIST, US EPA, or other nationally recognized standards.

Calibration procedures for specific instruments, and the frequencies of performance for defined methods, are described in the applicable operating or analytical SOP. Calibration is discussed in general terms in GL-QS-E-014 and includes standard laboratory practices and formulas used for determinations made by these practices. General guidelines include:

- Verification of initial calibrations with a standard obtained from a second source (unless one is not available).
- Analysis of verification standards (ICV and CCV) with each initial calibration within 15% of the true value unless historical data have demonstrated that wider limits are applicable.
- Preparation of calibration curves as specified in the reference method.

If a test method does not specify the number of calibration standards, the minimum number is two, not including blanks, with one at the lowest quantitation limit. The reference SOP must establish the initial calibration requirements.

7.3 Calibration Verification

Unless otherwise specified by the method, regulatory program or demonstrated through historical data, the recovery of target analyte(s) in calibration verification standards shall be between 85 – 115%. We discuss additional requirements below.

7.3.1 Initial Calibration Verification (ICV)

- If an initial calibration curve is not established on the day of analysis, the integrity of the curve should be verified each day of use or every 24-hour period. Verification requires the initial analysis of a blank and standard from a second source. The standard concentration should be at the method-defined level. If not specified, a standard at a mid-level concentration may be used.
- If the initial calibration verification does not meet acceptance criteria, the analytical procedure is stopped and evaluated, and appropriate corrective measures are taken. Initial calibration verification must be acceptable before any samples are analyzed.

7.3.2 Continuing Calibration Verification (CCV)

Additional standards called CCVs are analyzed after the initial calibration curve or the integrity of the initial calibration curve is accepted. CCVs are analyzed at a frequency of 5% or every 12 hours, whichever is more frequent. If an instrument consistently drifts outside the acceptance criteria before the next calibration, the frequency is increased.

CCVs may be from the same source as the calibration standards or from a second source. The concentration is determined by the anticipated or known concentration of the samples and/or method-specified levels. At least one CCV shall be at a low-level concentration.

To the extent possible, we bracket the samples in each interval (every 20 samples or every 12 hours) with CCV concentrations closely representing the lower and middle range of reported sample concentrations. If this is not possible, the standard calibration checks should vary in concentration throughout the range of the data being acquired.

If the recovery of a CCV does not meet the acceptance criteria and routine corrective actions fail to produce a second consecutive check within acceptance criteria, a new initial calibration curve should be constructed. Analytes of interest found in corresponding environmental samples may be reported, however, only if all of these criteria are met:

1. CCV recovery for target analyte exceeds the acceptance criteria (biased high)
2. Target analyte in the environmental sample is not detected at a concentration exceeding the level required by client contract (i.e., MDL, PQL).

   Non-detects that meet these criteria are also referred to as “passable non-detects.”

If samples are found to contain target analytes that exceed the associated quantitation limits, and the CCV recovery does not meet the acceptance criteria, the

affected samples are re-analyzed. This occurs only after a new calibration curve has been established, evaluated, and accepted.

7.4 Bioassay Instrument Calibration and Frequency

Our Bioassay instruments are calibrated at the frequency of the instrument's use, stability, and method requirements. The calibration procedure for each instrument is described in the corresponding analytical SOP and is performed by those individuals proficient in the analyses described in the SOP.

SECTION 8
ANALYTICAL METHODS AND STANDARD OPERATING PROCEDURES

Section 8 – Analytical Methods and Standard Operating Procedures (SOPs)

We provide a wide array of parameters including volatile organics, extractable organics, metals, general inorganic/wet chemistry, radiochemistry, and radiobioassay. The procedures we use to determine these parameters are consistently executed due to our extensive system of SOPs and our training requirements for analytical staff.

A list of our SOPs and the analytical methods they represent (if applicable) is provided in Appendix I. Discussed here are:

- Selection of analytical methods
- Standard operating procedures
- Method validation and initial demonstration of capability
- Sample aliquots
- Data verifications
- Standard and reagent documentation and labeling (Refer to Section 10.1)
- Computers and data requirements

8.1 Selection of Analytical Method

Project Managers are ultimately responsible for selecting the test codes and methods assigned to a client based on client requirements and sample collection techniques. In selecting methods, our goal is to meet the specific needs and requirements of the client while providing data that are scientifically valid.

When the use of a specific test method is mandated, only that method is used. If the analysis cannot be performed by the client-requested method, we notify the client. We do not perform method substitutions without the client's consent. We recommend that clients who submit data to regulatory agencies also obtain the agency's approval of method modifications.

When clients have specific process or reporting deviations from GEL's standard practices, the laboratory may document the deviations in contracts, case narratives and/or with specific work instructions from the Project Management Team to the laboratory. Approval of the deviations is made after consideration of all safety and quality concerns have been resolved by GEL's management.

A Project Management AlphaLIMS Manual (GL-CS-M-001) is available to assist PMs and PMAs in selecting test codes and methods and communicating the client's analytical and data reporting specifications.

8.2 Standard Operating Procedures (SOPs)

We determine each parameter by the protocol detailed in the corresponding SOP. The defined protocol originates from the analytical method or methods referenced in the SOP and may incorporate regulatory and client requirements. Descriptions of the methods we employ can be found in:

- EPA SW-846
- EPA/600/479/020
- Official Methods of Analysis of the Association of Official Analytical Chemists (AOAC)
- American Society for Testing and Materials (ASTM)
- Standard Methods for the Examination of Water and Wastewater (SM)
- South Carolina Department of Health and Environmental Control (SCDHEC)
- Code of Federal Regulations (CFR) Titles 40 and 49
- Department of Energy Environmental Measurements Laboratory (EML)
- Los Alamos Health and Environmental Chemistry (LAHEC)
- DOE
- DoD
- HASL

In addition to these references, a number of our radiochemistry procedures were developed in conjunction with Florida State University (FSU) under the guidance of Dr. Bill Burnett.

Laboratory sections have access to GEL's SOPs to ensure that each operational system and analytical procedure is performed in a uniform manner. SOPs are controlled according to GL-DC-E-001 for Document Control and are posted on the Intranet by the Document Control Officer.

We write and issue SOPs in accordance with GL-ADM-E-001 for the Preparation, Authorization, Change, Revision, and Release of Standard Operating Procedures. A technical and/or quality review is made of each new or revised SOP prior to its implementation.

Technical reviews ensure that procedures are technically sound and method-compliant, and are conducted by a senior analyst, group leader, or data reviewer. The quality review is an independent review by a member of the Quality Systems team and ensures that the quality requirements of the method, regulatory agencies, and GEL are adequately and accurately identified.

SOPs are modified when:

- Instruments or equipment change
- An error is identified
- Improvements in technology and/or reagents need to be incorporated
- Reference methods are revised or discontinued

Proposed revisions are submitted for review on Documentation Initiation and Revision Request (DIRR) forms. Changes are not implemented without a technical and quality review.

We review our technical SOPs annually and revise them as necessary. Analytical SOPs either contain or reference other SOPs that contain:

- reference method
- applicable matrix or matrices
- method detection limit
- scope and application including parameters to be analyzed
- method summary
- definitions
- interferences and limitations
- specific safety requirements
- required equipment and supplies
- reagents and standards
- sample collection, preservation, shipment, and storage
- quality control
- calibration and standardization
- procedure
- calculations
- method performance
- pollution prevention
- data assessment and acceptance criteria for quality control measures
- corrective actions for out of control or unacceptable data
- waste management
- references
- tables, diagrams, flowcharts, validation data
- identification of any modifications we have made to the published procedure

8.3 Method Validation and Initial Demonstration of Capability

Method validation requirements for Radiochemistry are documented and maintained in accordance with GL-RAD-D-002, Analytical Methods Validation for Radiochemistry.

An initial demonstration of method performance is required before a new analytical method is implemented and any time that there is a significant change in instrumentation or methodology. Exempted from this requirement are any tests for which spiking solutions are not available. Analyses that are exempt include those for determining:

- total dissolved, total suspended, total volatile, and total solids
- pH
- color
- free liquids
- temperature
- dissolved oxygen
- turbidity

We conduct the initial demonstration as described in Section 8.3.1. Records of initial demonstration are maintained in accordance with GL-QS-E-008 for Quality Records Management and Disposition. These records are available upon request.

After we demonstrate our ability to perform a specific analysis, we continue to demonstrate method performance through the analysis of laboratory control samples and performance evaluation samples.

If spiking solutions or quality control samples are not available, an analyst is trained by a qualified trainer to conduct the analysis. Analyst capability and proficiency is evaluated by the appropriate Group Leader before the

analyst is qualified to perform the analysis on client samples. The evaluation is documented and maintained according to GL-QS-E-017 for Maintaining Technical Training Records.

Method Validation must also occur when substantive modifications are made to stoichiometry, technology, mass tuning acceptance criteria, quantitation ions, compressing digestion or extraction timeframes, reducing reagent or solvent volumes, changing solvents or compressing instrument.

8.3.1 Procedure for Initial and Continuing Demonstrations of Capability (IDOC and CDOC)

We conduct initial demonstrations of capability for mandated analytical or EPA reference test methods following the procedure outlined below. This procedure is adapted from the EPA test method published in 40 CFR part 136, Appendix A and the 2003 NELAC and 2009 TNI Standards. IDOCs are completed whenever there is a change in instrument type, method or personnel. CDOCs are updated constantly in the laboratory AlphaLIMS.

Step 1: A quality control sample is obtained from an outside source (if possible). If one is not available, the sample may be prepared internally using stock standards that are prepared independently from those used in instrument calibration. The concentration is not known to the analyst.

Step 2: The QC sample is diluted in a volume of clean matrix. Sufficient volume of the diluted QC sample is prepared so that at least four aliquots of the required method are analyzed. Alternatively, four matrix spike samples may be evaluated for levels of precision and accuracy.

Step 3: Four aliquots of the diluted quality control sample are prepared and analyzed according to the analytical test method. This may occur concurrently or over a period of days.

Step 4: With the results obtained from the analysis of the diluted QC sample, the average recovery (x) in the appropriate reporting units (such as µg/L) and the standard deviation of the population sample (n-1) (in the same units) are calculated for each parameter of interest.

Step 5: For each parameter, the standard deviation (s) and the average recovery (x) are compared to the corresponding acceptance criteria for precision and accuracy in the test method (if applicable) or in laboratory-generated acceptance criteria. If "s" and "x" for all parameters meet the acceptance criteria, analysis of samples may begin. If any one parameter exceeds the acceptance range, the performance is unacceptable for that parameter.

Step 6: When one or more tested parameters fail one or more of the acceptance criteria, we locate and correct the source of the problem and repeat the test for every parameter of interest.

Other options for successful IDOCs are the following:

- PT Study- successful analysis of a PT Sample. If 4 LCSs cannot be performed, successful analysis of a PT sample may be used to demonstrate capability to perform a test. The PT sample may be single-blind to the analyst or double blind to the laboratory.
- Supervised Analysis- where other options are not practical, supervised analysis of a procedure may be used to demonstrate capability.
- Analysis of authentic sample with results statistically matching those obtained by another trained analyst.
- Other – this option may be used for certain personnel having sufficient analytical skills to develop a new procedure, as deemed appropriate by the supervisor or Quality Assurance personnel.

8.4 Sample Aliquots

When obtaining aliquots from a sample, it is imperative that the subsamples be representative of the parent sample. This ensures that the results obtained from the analysis of the aliquots are representative of the entire parent sample, not just the subsample. We employ different techniques to obtain subsamples. GEL's SOP for subsampling is GL-LB-E-029.

We can obtain representative aliquots of soil samples for the determination of metals through quartering. This involves the repeated quartering of the sample until the resulting quarter is equivalent to the amount of sample needed for analysis. Quartering may not be appropriate for obtaining subsamples for volatiles or other analyses where potential contamination or loss of target analytes is a concern.

Water samples are inverted several times prior to the collection of a subsample. This ensures a thorough mix and is absolutely required for the accurate

determination of analytes like total and total suspended solids.

The appropriate techniques for obtaining sample aliquots for designated analyses are discussed in the applicable SOPs.

8.5 Data Verification

All of the data we include in final reports to our clients undergoes extensive data verification. At GEL, we have a multi-level review process that takes place in all areas of the laboratory beginning with sample login. This process and the responsibilities of each level of review are delineated in a number of procedures, including GL-GC-E-092 for General Chemistry Data Review and Packaging, GL-MA-E-017 for Metals Data Validation, and GL-RAD-D-003 for Data Review, Validation, and Data Package Assembly.

8.5.1 Sample Login:

Samples are analyzed by the methods and for the target analytes identified when samples are logged into our database. If there is an error in this entry that is not promptly identified, the incorrect analytical method may be used or certain analytes may not be determined.

To prevent this, the person who enters the information into the database is generally the client's assigned Project Manager or PM Assistant. This entered information is reviewed against the client confirmation letter and/or chain of custody. If errors are identified, they are immediately corrected.

8.5.2 Data Validation in the Laboratory

The multi-level review process in our laboratory includes initial review by the analyst, a second review by a peer, and a final review by a group leader or data reviewer. Where appropriate based on personnel and client needs, the industrial division institutes two levels of review.

Our analytical data reviews ensure that:

- The analytical procedures comply with current SOPs.
- Quality control samples are analyzed at the frequency specified in the SOP or client specifications.
- The acceptance criteria for quality control samples are met, including recoveries of matrix spikes and laboratory control samples, the relative percent difference for matrix duplicates, matrix spike duplicates, laboratory control sample duplicates, and concentrations of target analytes in the method blank.
- Instrument data, run logs, and logbooks are reviewed to ensure that all method quality control criteria were met (e.g., calibration, initial calibration verifications, and continuing calibration verifications).
- Documentation is sufficient to reconstruct the analytical procedure.
- Data are maintained according to GL-LB-E-008 for Basic Requirements for the Use and Maintenance of Laboratory Notebooks, Logbooks, Forms, and Other Recordkeeping Devices.
- Raw data are in agreement with the computer generated batch sheets and data reports.
- The calculations, dilution factors, concentration reported, and nominal concentrations are verified.
- Comments, qualifiers, or nonconformances for noncompliant or questionable data are documented.
- Data generated when the analytical process appears to be out of statistical control are not reported.

8.5.3 Validation of Data Reports and Packages

Before we report data to the client, we review the requested data report for package accuracy, completeness, and client-specifications. Responsibilities for review are dependent upon the type of report or package being generated. (Refer to Section 11 for Laboratory Report Formats.)

When a client is receiving a certificate of analysis or certificate of analysis and Quality Control Summary Report, the Project Manager (PM) or Project Manager Assistant (PMA) reviews the information for accuracy, completeness and the addition of pertinent comments made by the laboratory about the analysis or sample. The PM or PMA also reviews data for consistency as described in the Project Management AlphaLIMS Manual, GL-CS-M-001. For Bioassay results, the package is then reviewed for completeness by validator, team or group leader as described in GL-RAD-B-026.

If a client requests a case narrative, our data validators review the analyst-prepared case narrative for

accuracy and to assure its consistency with the information included on the certificate of analysis and Quality Control Summary Report. If a client requests a more detailed level of data package up to and including a CLP-like package, every laboratory fraction of data is reviewed by that fraction's data validator. The data are then compiled into a final data package. The Quality manager or designee will review a minimum of 10% of all data packages for technical completeness and accuracy on a quarterly basis and if data quality issues are discovered during the review, the client will be notified with fifteen (15) business days of the discovery of the issue.

8.6 Standard and Reagent Documentation and Labeling

The documentation and labeling of standards and reagents is addressed in GL-LB-E-007 and GL-RAD-M-001 for Laboratory Standards Documentation, and in Section 10.1 of the QAP, Recordkeeping System and Design.

8.7 Computer and Electronic Data Related Requirements

Our Information Management System (IT) SOPs describe the way in which we manage our software programs and hardware systems. Control of software development and modification activities is described in GL-IT-E-003 for Requirements, Design, Operation, Validation, and Removal of Hardware and Software Systems Used by the GEL Group, Inc. All development and revision activities are validated, and revision activities are validated, verified, and controlled with revision software or other procedures prior to production use.

Analytical software that is purchased from a vendor is validated and verified in accordance with GL-IT-E-005 for Requirements, Design, Operation, Validation, and Removal of Applications Used by The GEL Group, Inc. Documentation requirements are also described in this SOP.

Electronic signature requirements for confidentiality of records are described in GL-IT-E-001 for Instrument Technology Program for Good Laboratory and Good Manufacturing Practices.

SECTION 9

SAMPLE HANDLING, ACCEPTANCE, RECEIPT, AND INTERNAL CHAIN OF CUSTODY

Section 9 – Sample Handling, Acceptance, Receipt, And Internal Chain of Custody

The way we receive and handle samples is critical to providing our clients with data that are of the highest quality and are legally defensible. We have strict policies that govern the acceptance and receipt of a sample, sample handling and integrity, maintenance of the internal chain of custody, and storage of the sample upon completion of the required analytical processes. This section describes the policies and practices that we employ, including the following:

- Agreements to perform analysis
- Proper labeling of submitted samples
- Chains of custody
- Sample receipt procedures
- Sample receipt procedures for radioactive samples
- Sample tracking
- Sample storage
- Sample disposal

9.1 Agreement to Perform Analysis

Before we accept samples, we should have an agreement with the client that specifies the analytical methods, the number of samples to be analyzed, the price for the analysis, the date by which the client must receive results, and the reporting format. Any special requirements the client may have, such as non-routine methods and reporting limits, should be part of that agreement.

An agreement to perform analysis should be in one of three forms, further detailed in our Analytical Services Reference Manual and the SOPs for Delegated Authority to Commit the Company and Request for Proposal (RFP) and Contract Review (GL-CO-E-002 and GL-CO-E-003):

- Client confirmation letter (CCL) between the client and project manager for a specific group of samples. This letter includes the cost, turn-around time, requested analysis, sample matrix, number of samples, and type of client report.
- Sample acceptance by the Project Manager from an established client based on previously agreed conditions and confirmed by the client's submission of the sample(s).
- Contractual agreement for analytical services over a designated time period or project that delineates the specifications agreed upon.
- When the laboratory agrees to perform analyses with exceptional departures from normal processes, these exceptions are clearly defined in the client-laboratory agreement.

9.2 Sample Labels and Chain of Custody Forms

Once an agreement is established, we assume joint responsibility with the client to ensure that the samples submitted are properly labeled and accompanied by full and complete documentation that includes chain of custody and, where possible, material safety data sheets. Samples that are submitted without proper documentation may be refused.

Sample labels should include the:

- client's sample identification
- location, date, and time of collection
- collector's name
- chemical preservatives used
- constituents of interest (if space permits)

When requested, we ship labeled sample containers with appropriate preservatives and a chain of custody to the client for use during sample collection. There are several advantages to using these containers, including:

- Dedication of appropriate type sample container for the intended analyte or analytical method.
- Proper sample preservation for analytical test
- Traceability of bottle lot number to the manufacturer's certification that the containers are clean and show no signs of contamination.
- If a manufacturer cannot provide a certificate of cleanliness for radiochemistry parameters, a gross alpha-beta screen can be performed on the lot of containers being used. This is mandatory for containers used in support for SDWA programs.

Chain of custody forms include the following information and are initiated at the time of sample collection:

- name and address of client
- client sample identification
- date and time of sample collection
- sample matrix
- description of sampling site location
- number of containers
- methods, chemical and physical constituents for which the analyses are to be conducted
- preservatives
- date and signature of person who collected the sample
- date of transfer and signature of person relinquishing sample to the laboratory.

When our Field Services personnel collect samples, our standard chain of custody form and certified containers are automatically used. Our standard chain of custody forms are also available to our clients and are included with each shipment of pre-labeled and preserved containers. GEL chain of custody forms should always be used unless otherwise agreed to by contract.

9.3 Sample Conditions

In addition to properly documenting sample container labels and the chain of custody form, we need to make sure that samples meet the established requirements for analytical testing. This is particularly critical for samples that are being analyzed to meet regulatory requirements.

Samples should be collected in the appropriate type of container, preserved as directed, and stored in the conditions specified in the analytical method or established regulatory guidelines. In addition, samples should be submitted with sufficient time to conduct the specified analysis within the regulatory or method holding time. Aliquots should be of sufficient volume to perform the requested analyses. A summary of these conditions and holding times for routine analyses can be found in Appendix J.

9.4 Sample Receipt

Samples submitted to us are received in a central sample receiving area by our sample custodian or login clerk. Every sample is subject to the protocols established in GL-SR-E-001 for Sample Receipt, Login and Storage.

Our sample custodian acknowledges receipt of a sample by signing the chain of custody and recording the date and time custody was transferred from the client to the laboratory. The date, time, and person receiving the sample are also recorded on a standard or client-specific Sample Receipt Review (SRR) form.

The sample custodian is also responsible for noting the condition of a sample upon its arrival. This information may be recorded on both the sample chain of custody and the Sample Review Receipt form. As detailed in GL-SR-E-001, the sample custodian should:

- Inspect all sample containers for integrity.
- Document any unusual physical damage or signs of tampering with custody seals.
- Place any samples that appear to be leaking or have unusual odor under the fume hood while notifying the responsible project manager.
- Review the chain of custody submitted by the client for completeness.
- Compare descriptions and other information on the sample container labels to that listed on the chain of custody.
- Verify the sample is within the regulatory holding time for the analyses.
- Measure and record the temperature of sample aliquots that are to be used for analyses requiring thermal preservation.
- Measure and record the pH of all sample aliquots submitted for analyses that require chemical preservation to a specific pH.
- Verify that there are adequate sample aliquots for the requested analyses.
- Verify that appropriate sample containers were used for requested analyses.

If the sample custodian discovers any abnormalities or departures from standard conditions, the PM is

informed immediately. The PM will then notify the client as quickly as possible so that a decision can be made to proceed with the analysis or submit another sample or additional sample aliquots.

Common abnormalities or departures from standard conditions include:

- Sample containers with signs of damage, leaking, or tampering.
- Incomplete/missing chain of custody.

NOTE: If a nonradioactive sample has no chain of custody, the sample custodian should initiate one. "INITIATED ON RECEIPT" should be documented on the chain of custody.

- Discrepancies between the information on the chain of custody and the sample container labels.
- Method or regulatory holding time is exceeded.
- Sample is not preserved to the method or regulatory-required pH.
- The sample container does not meet method or regulatory criteria.
- The sample temperature exceeds or falls below the thermal preservation regulation or method requirement of 0° ≤ 6° C.

NOTE: If a sample is hand delivered to the laboratory immediately after collection with evidence that the chilling process has begun (arrival on ice), the sample shall be deemed acceptable.

- Radioactivity that exceeds that allowed by our radioactive license. (The handling of radioactive samples is discussed in 9.5.)

Samples that are not appropriate for the requested analyses or have no full test specifications require:

- Retention of all correspondence and records of conversations concerning the final disposition of the sample.
- Full documentation on the chain of custody and Sample Receipt Review form of the nonconforming condition and a decision to proceed with analysis.
- Documentation that the analysis is qualified appropriately on the final report.

9.5 Receipt of Radioactive Samples

The radioactive samples we receive are subject to the same monitoring identified in 9.4 when radioactivity levels do not exceed the level permitted by our license. Special procedures governing the receipt of radioactive samples are described in the GL-RAD-S-007 for the Receiving Radioactive Packages. These procedures prevent the inadvertent spread of radioactive contamination.

Because we cannot exceed the limits of our radioactive license, it is imperative that our clients notify us of impending shipments of radioactive samples. We reserve the right to refuse and return any radioactive sample where the radioactivity:

- Exceeds our permitted level by itself or in combination with other samples already on site; or
- Exceeds our administrative level of 25 mrem/hr.

The following special requirements for receiving radioactive samples are applicable:

- Only designated staff trained in the proper handling of radioactive materials handle radioactive samples.
- If a sample is labeled as radioactive, the custodian will immediately inform the Radiation Safety Officer (RSO) before opening the sample.
- The radioactivity of the sample will be measured by scanning the exterior surface of the cooler using a survey meter calibrated in Mr/hr. Refer to GL-RAD-S-001 for our Radiological Survey Procedures.
- If the radioactive level of the exterior of the cooler exceeds 0.5 Mr/hr, the RSO will be notified before the cooler is opened.
- If the radioactivity level of a sample or group of samples is found to exceed 25 mrem/hr, the RSO will be notified immediately. The client will be contacted and arrangements will be made to return the sample(s) or reduce the per sample exposure.
- If a chain of custody is not submitted with a sample, it will be placed on hold until a chain of custody is submitted.
- The inside of the cooler will be surveyed to ensure that no leakage or contamination has occurred.

- Each sample container will be surveyed and the highest reading will be documented on the Radioactive Shipment Inventory.

9.6 Sample Tracking

We track the samples we receive by a unique laboratory identification number that is automatically assigned when information pertaining to the sample is first entered into our database. Pursuant to GL-SR-E-001, the following information is entered for each sample received:

- client and/or project code
- client sample ID
- sample matrix
- equivalent laboratory sample matrix
- type of report format specified by client
- date and time of collection
- date received
- initials of person making entries
- number of containers submitted for the sample
- requested analyses
- pertinent observations or comments affecting the sample analysis or rejection

As soon as this information is entered, AlphaLIMS automatically assigns a unique number to the sample and its containers. We use the number to track the location of a sample container and to link to any subsamples and subsequent digestates and extracts.

The unique laboratory identification number is printed on a durable barcode label that contains the client identification, sample date and time. Once labeled, the sample container's identification number is uploaded into the database by scanning the barcode. Information included in the database at the time of sample scanning is the container's storage location, bottle type and volume, physical characteristics of the bottle, preservative, and the initials of the person entering this information. Entering of this information into the database is an important part of initiating our electronic internal chain of custody.

9.7 Internal Chain of Custody

Chain of custody procedures ensure traceability and sample integrity. Our legal and evidentiary chain of custody protocol establishes a continuous record of the physical possession, storage, and disposal of sample containers, collected samples and aliquots, and sample digestates or extracts.

The internal chain of custody starts with the scanning of a container's barcode label into an electronic database while identifying the location of the sample and the person having custody, or placing the sample in a secured storage area. If we supply the containers, the chain of custody may begin when the containers are provided to the client.

With regard to the internal chain of custody, a sample is defined as being in someone's custody if:

- It is in one's actual physical possession
- It is in one's view after being in one's physical possession
- It is in one's possession and then is locked up so that no tampering may occur
- It is kept in a secured area restricted to authorized personnel only

The protocol for ensuring sample integrity using the internal chain of custody is detailed in GL-LB-E-012 for Verifying the Maintenance of Sample Integrity. The electronic internal chain of custody works in conjunction with the chain of custody submitted by the client with a sample to:

- Account for all time associated with a sample, its subsamples, and extracts or digestates from the time the sample is received at GEL to its disposal
- Identify all individuals who physically handled the sample
- Provide evidence that the sample was stored in accordance with method and regulatory protocols

The electronic internal chain of custody is stored in AlphaLIMS so that information demonstrating the proper maintenance of custody can be provided to the client on the data reports or electronic data deliverables.

9.8 Sample Storage

In order to ensure the maintenance of sample integrity, all aliquots are stored in secured areas designated for sample storage. The storage location of

each sample aliquot can be tracked using the internal chain of custody. Areas designated for sample storage include:

- Main cooler where most samples requiring maintenance at a temperature range of 0° ≤ 6° C are stored.
- Volatile coolers for samples to be analyzed for volatile contaminants.
- Radioactive cooler for segregation of radioactive sample aliquots requiring refrigeration.
- Ambient storage for non-radioactive samples not requiring refrigeration.
- Ambient storage for radioactive samples.

The temperature of each refrigerated storage unit is monitored daily and documented per GL-LB-E-004 for Temperature Monitoring and Documentation Requirements for Refrigerators Freezers, Ovens Incubators, and Other Similar Devices. In addition, the main and radioactive coolers are monitored twenty-four hours a day by temperature sensors that are connected to our main security system. If the temperatures exceed the required range, the security system notifies the facilities manager or his designee immediately. This allows corrective actions to be initiated promptly.

Prior to and immediately after analysis, samples and their digestates and extracts are stored in compliance with the requirements of the requested analytical methods and GL-SR-E-001 for Sample Receipt, Login, and Storage. If a single aliquot is supplied for analyses by several methods, the most stringent analytical storage requirements are applied to the sample.

If samples are to be analyzed for volatile organic compounds, they are stored in designated volatile coolers that are maintained at a temperature range of 0° ≤ 6° C. No sample aliquots are stored in these refrigerators unless they are to be analyzed for volatiles. These storage units are monitored on a weekly basis for contamination by the analysis of volatile cooler storage blanks.

At the beginning of each month, two 40 mL vials are filled with treated deionized water, which is used for volatile method blanks and placed in each monitored cooler. Each week, two vials may be analyzed by EPA 8260B and the data are reported to the Quality Department. If the analysis reveals evidence of potential contamination, appropriate corrective actions are immediately implemented. SOP GL-OA-E-058 discusses the laboratory practices pertaining to monitoring and testing for VOA contamination.

Sample aliquots for non-volatile analysis, which also should be maintained at 0° ≤ 6° C, are stored in the main cooler unless they are radioactive. In order to reduce the chance of contamination, radioactive samples are stored in a designated cooler.

Sample aliquots to be analyzed for biochemical oxygen demand (BOD) are also delivered to the bacteriology laboratory and stored in the designated BOD cooler. This cooler is also maintained at 0° ≤ 6° C. After initiation of this analysis, the sample aliquots are returned to the main cooler.

After all analyses are complete and results are submitted to the client, sample aliquots are transferred to the sample archive area. They are stored in this area until they are disposed.

Radioactive and non-radioactive samples remain segregated in archive to reduce the risk of contamination.

9.9 Sample Disposal

Our policies concerning sample disposal are described in the Laboratory Waste Management Plan, GL-LB-G-001 and can be divided into two categories: those governing the disposal of sample laboratory waste, and those governing the disposal of residual client sample after the completion of all analyses.

9.9.1 Sample Laboratory Waste

Unless otherwise requested by contract, laboratory waste is collected in designated satellite containers found in sample collection and accumulation areas. These areas are monitored by both the waste department and analysts trained in waste collection. Wastes are segregated based on the type of hazard they present. I.e. radioactive, acid, base solvent, etc. when containers are full, the waste department is notified and the containers are removed from the laboratory for disposal. Direction for disposal activities, such as

packaging, shipping, and disposal site selection are provided in the Laboratory Waste Management Plan (GL-LB-G-001).

9.9.2 Residual Client Sample

Unused client sample material that is not consumed during the sample preparation or analytical procedures is either disposed of in accordance with the Laboratory Waste Management Plan (GL-LB-G-001) or at the client's request, returned in accordance with GEL's SOP GL-SR-E-002 for Transportation and Shipping of Samples and Pre-preserved Sample Containers.

It is our policy to hold samples for a minimum of sixty days after invoicing and before disposal, unless otherwise specified by contract or if the sample is part of litigation. If the sample is part of litigation, disposal of the physical sample shall occur only with concurrence of the affected legal authority, sample data user, and/or client.

When sample analyses are complete and regulatory and/or contractual holding times have expired, samples are moved from their storage locations to the radioactive or non-radioactive archives. Samples that are to be returned to the client or held for an extended time period are segregated from the other samples. Radioactive and non-radioactive samples remain segregated.

When internal or client-specified storage time expires, samples with like matrices are composited into appropriate containers. The composites are then subject to the same treatment and disposal protocol. Samples that are approved for disposal are scanned into our LIMS and assigned the status of "Disposed."

SECTION 10
RECORDS

Section 10 – Records

Our quality records provide the documentation we need to support analytical results and conclusions. Documented evidence that quality assurance and quality control requirements have been met is critical to providing data that fulfill the specifications of applicable procedures, programs, and contracts.

As described in Section 3 of this Quality Assurance Plan (QAP), quality records include but are not limited to:

- Observations
- Calculations
- Calibration data
- Certificates of analysis
- Certification records
- Chains of custody
- External, supplier, and internal audits
- Run logs
- Instrument data and analytical logbooks
- Instrument, equipment and building maintenance logs
- Material requisition forms
- Monitoring logs
- Nonconformance reports
- Corrective actions
- Method development and start-up procedures including MDL studies
- Training records
- Waste management records
- Standard logs
- Software validation
- Standard operating procedures (SOPs)
- Sample collection and field data

Our procedures provide a legal and evidentiary chain of custody and are described in Section 9 of this QAP. Described in this section are:

- Record keeping system and design
- Records management and storage
- Sample handling records
- Records of support activities
- Analytical records
- Administrative records

10.1 Recordkeeping System and Design

We manage, maintain and store our quality records according to GL-QS-E-008 for Quality Records Management and Disposition. The protocols established in this document work in conjunction with those for specific types of records addressed in other SOPs to govern our record keeping system. Our record keeping system allows the historical reconstruction of all laboratory activities that produced analytical data.

We facilitate historical reconstruction by maintaining the following records and information, from the time a sample is received until it is disposed.

- A master list of all employee signatures and initials is maintained in Human Resources. This allows the identification of any GEL personnel who accept, handle, analyze, prepare, review, store, or dispose of a sample, its subsamples, associated data and reports, and other related documentation.
- If we provide bottles and containers to a client or sampling personnel, these records are kept in accordance with GL-SR-E-002 Transportation and Shipping of Sample and Pre-preserved Sample Containers. These electronic and paper records include:
  - Supplier and lot numbers of containers and/or bottles provided
  - Certifications that the containers are free of contaminates that may bias the analyses
  - Addition of preservatives and identity of person responsible for this preservation.
  - Barcode of containers supplied to a particular client or for a specific field-sampling event.

The person or agency responsible for collecting a sample is documented on the chain of custody and entered into AlphaLIMS. Other records supporting the acceptance of a sample include:

- Date and time of sample receipt

- Person accepting sample
- Condition of sample upon receipt
- Client-confirmation letter and/or sample quote
- Client chain of custody
- Electronically generated sample ID numbers specific to each sample aliquot and linked to the client's sample description, sample collection and receipt information, and analyses to be performed.
- Identification of each person who has custody of a sample, its subsamples, extracts, or digestates. (This is provided through the internal chain of custody procedures described in Section 9.)

Documentation that materials purchased for use in the analysis or preparation of samples meet specifications is maintained in accordance with GL-RC-E-001 for Receipt and Inspection of Material and Services.

Records of equipment calibrations are maintained and traceable by date and ID number to a specific analysis. These records include certifications of calibration and service that have been initialed or signed.

Our thermometers are verified against a NIST traceable thermometer and records of this verification are maintained as described in GL-QS-E-007 for Thermometer Verification. Records of the daily and monthly calibration verifications of our analytical balances are kept in accordance with GL-LB-E-002 for Balances. The calibration records for our air-displacement pipets are maintained in pipet calibration logs specific to each pipet according to GL-LB-E-010 for Maintenance and Use of Air Displacement Pipets.

When methods and/or regulations specify that samples, subsamples, extracts, and/or digestates be stored at designated temperatures, or when the method, itself, has temperature sensitive steps, we document those temperatures on monitoring logs at the frequency defined in the corresponding SOPs. We can trace the specific storage location of a sample through the internal chain of custody.

We require that the initials of all personnel responsible for monitoring temperatures be recorded in the temperature monitoring logs pursuant to GL-LB-E-004 for Temperature Monitoring and Documentation Requirements for Refrigerators, Freezers, Ovens, Incubators and Other Similar Devices. The logs are reviewed for completeness in accordance with GL-QS-E-005 for Review of Monitoring Device Logs.

Documentation on the instruments and equipment used for the analysis of samples is recorded in run logs, laboratory logbooks, instrument data and/or sample preparation logs. Routine or corrective maintenance that is performed on equipment or instruments is recorded in the maintenance log specific to the instrument. We document these records in accordance with GL-LB-E-008 for Basic Requirements for the Use and Maintenance of Laboratory Notebooks, Logbooks, Forms and Other Recordkeeping Devices.

The standards containing known quantities of target analytes that we use in instrument calibration, calibration verification, and as quality control samples, such as matrix spikes and laboratory control samples, are documented according to GL-LB-E-007 and GL-RAD-M-001 for Laboratory Standards Documentation. These records contain the following information.

- Protocol by which each standard was prepared
- Traceability of each child standard to its parent
- Date each standard was prepared
- Initials of person preparing the standard
- Expiration dates
- Concentration of each standard

This information allows us to document that the standards used were prepared in accordance with the established protocol, produced using source standards that meet the method and regulatory criteria, and used prior to their expiration date.

If required, reagents used in the preparation, dilution, and analysis of samples are verified to be free of interferences or target analytes. We record these verifications in the Reference Material in LIMS in accordance with GL-LB-E-008 for Basic Requirements for the Use and Maintenance of Laboratory Notebooks, Logbooks, Forms and Other Recordkeeping Devices.

Analytical and sample preparation methods applied to each sample aliquot are documented via the internal chain of custody, method information, and information recorded in lab notebooks, sample preparation logs, run logs, and instrument data. The laboratory protocol we employ during analysis is dictated by the SOP in effect at the time the sample was analyzed or prepared by a specific method.

Run logs, laboratory notebooks, instrument data and sample preparation logs are used to document the preparation and analysis of samples and the associated instrument calibrations. These logs and notebooks are governed by GL-LB-E-009 for Run Logs and GL-LB-E-008 for Basic Requirements for the Use and Maintenance of Laboratory Notebooks, Logbooks, Forms, and Other Recordkeeping Devices. As stated in these SOPs, sample preparation and analytical records that are not electronically generated should be:

- Legible
- Recorded in permanent ink
- Corrected using one line marked through the error, initialed and dated
- Initialed by the responsible party

We maintain electronic records for each analytical batch. These records include the ID numbers of each client and quality control sample prepared and/or analyzed together, the method of preparation and analysis, and the matrix of the samples included in the batch.

Through our electronic statistical process control system (SPC), the acceptance criteria applied for all quality control (QC) samples are stored and maintained. The acceptance limits for target analytes are method, matrix, and time-period specific, which allow us to regenerate the criteria applied to QC samples associated with identified client samples.

Our Quality Systems Team maintains the records of nonconformances and corrective actions associated with specific samples, batches, and processes. We maintain these records according to GL-QS-E-004 for the Documentation of Nonconformance Reporting and Dispositioning and Control of Nonconforming Items; and GL-QS-E-002 for Conducting Corrective/Preventative Action and Identifying Opportunities for Improvement.

Electronic data records are maintained in a secured database designed to protect the integrity of the data. Data that are uploaded directly from instruments and that are manually entered are backed up by a second system.

Permanent records of electronic data deliverables are maintained along with the corresponding sample preparation and analytical data review records. This documentation includes the initials of the reviewer and date of the review.

Records of the data we report to our clients are maintained in a manner that protects client confidentiality, as well as any potential national security concerns. These records include copies of certificates of analysis, quality control summary reports, case narratives, CLP forms, and other information we provided to the client. The copies may be paper or electronic. The majority of the data packages submitted to Federal clients are stored electronically prior to being submitted to the client.

Records of samples being disposed or returned to the client are documented in accordance with GL-SR-E-002 for Transportation and Shipping of Samples and Pre-Preserved Sample Containers. Such records include the date samples are returned or disposed, the destination of the samples, and name of the person transferring the samples.

10.2 Record Storage

We store quality records in compliance with GL-QS-E-008 for Quality Records Management and Disposition. The records are:

- Stored in a secured area to maintain data integrity and protect client confidentiality, including any national security concerns.
- Kept in areas where they are protected from fire loss, environmental deterioration, and, in the case of electronic records, electronic or magnetic sources.
- Indexed and filed in a manner allowing for ready retrieval.

- Accessible to the client for whom the record was generated.
- Retained for an identified period of time that equals or exceeds ten years as determined by applicable law and client contract requirements.

Electronic data records are stored on compact disks.

All of the hardware and software we need to reconstruct data is maintained according to GL-IT-E-003 for Requirements, Design, Operation, Validation and Removal of Hardware and Software Systems Used by the GEL Group, Inc. Records that are stored or generated by network or personal computers have either hard copy or write-protected backup.

10.3 Sample Handling Policy

Records of all procedures applicable to samples are maintained in our possession. These records include documents that pertain to:

- Preservation, including sample container and holding time
- Sample identification, receipt, acceptance or rejection, and login
- Sample storage and tracking including shipping receipts, transmittal forms, routing and assignment records
- Sample preparation (ID codes, cleanup and separation protocols, volumes, weights, instrument printouts, meter readings, calculations, reagents)
- Sample analysis
- Standard and reagent origin, receipt, preparation, and use
- Equipment receipt, use, specification, operating conditions and preventative maintenance
- Instrument calibration frequency and acceptance criteria
- Data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions
- Method performance criteria including expected quality control requirements
- Quality control protocols
- Electronic data security, software documentation and verification, software and hardware audits, backups and records of any changes to automated data entries
- Automated sample handling systems
- Disposal of hazardous samples

10.4 Records of Laboratory Support Activities

In addition to sample handling records, we maintain the following:

- Original raw data for calibrations, samples and quality control measures, including worksheets and data output records (chromatograms, strip charts, and other instrument readout records)
- A written description of or reference to the specific method used, including the computational steps used to translate parameter observations into a reportable analytical value
- Copies of final reports
- Archived standard operating procedures
- Correspondence relating to project-specific laboratory activities
- Corrective action reports, audits and audit responses
- Proficiency test results

10.5 Analytical Records

We document and maintain analytical records, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs according to GL-LB-E-008 for Basic Requirements for the Use and Maintenance of Laboratory Notebooks, Logbooks, Forms, and Other Recordkeeping Devices, and GL-LB-E-009 for Run Logs.

The information that is documented in analytical records includes:

- Laboratory sample ID code
- Date and time of analysis
- Instrument ID and operating conditions/parameter (or reference to such data)

- Method of analysis
- All calculations
- Dilutions
- Initials of analyst or operator
- Units of measurement

Our policy is to produce and maintain analytical records that are:

- Accurate
- Reviewed and verified
- Legible and understandable
- Traceable and authentic to their source
- Grouped in a contemporary manner with data entered and information recorded as it is obtained

10.6 Administrative Records

A number of pertinent records are maintained by Human Resources or Quality Systems, including:

- Staff qualifications and experience.
- Training records, including initial demonstrations of proficiency. (Refer to procedure GL-HR-E-002 for Employee Training.)
- A log of names, initials and signatures for individuals having responsibility for initialing laboratory records.

We monitor continuing demonstrations of proficiency through AlphaLIMS per GL-HR-E-002 for Employee Training.

SECTION 11
LABORATORY REPORT FORMAT AND CONTENTS

Section 11 – Laboratory Report Format and Contents

Accurate data are of little benefit to a client unless they are reported in a format that is easy to interpret and provides all pertinent information relating to the analysis of a sample. At GEL, we have developed certificate of analysis report formats that meet the different needs of our clients, yet provide all of the information necessary to satisfy regulatory requirements while allowing for the interpretation of the data. Each format provides accurate, clear, unambiguous and objective data.

In addition to a certificate of analysis, a client can request and receive an extended data package. This package may include any of the following: certificates of analysis; summaries of quality control; case narratives; instrument data; sample preparation data; measurement traceability and calibration information; and electronic data deliverables. If clients require the reporting of data following the established contract laboratory protocol (CLP), we can provide a CLP-like data package that will meet their needs.

It is important that the certificate of analysis format and data package requirements be discussed with the client prior to our acceptance of the samples. Project Managers and contract staff are responsible for establishing an agreement with the client concerning data reporting and the potential cost to the client for data packages and/or specialized reporting. Our analytical data are reported to three significant figures unless otherwise required by client contract.

Laboratory reports and data packages are stored and transmitted in a manner that protects client confidentiality and potential matters of national security. No reports or data packages are released to persons or organizations outside GEL without the expressed consent of the client. If directed by a regulatory agency or subpoenaed to submit documents to a court of law, we will notify the client of the demand and the records being released.

The following elements of report formats and data packages are described in this section:

- Certificates of analysis (C of A)
- Quality control summary reports (QCSR)
- Analytical case narratives
- Electronic data deliverables (EDDs)
- Types of data packages and reporting formats
- Review of data packages and reports

11.1 Certificates of Analysis

We have two primary C of A report formats, Level 1 and Level 2. Both contain the following information when applicable:

- Title
- GEL address and phone number
- Name of PM or person serving as the primary client contact
- Barcode identification of the C of A
- Number of page and total number of pages
- Name and address of client, where appropriate
- Project name or code if applicable
- Client-provided sample description
- Unique laboratory ID number for the sample
- Sample matrix
- Characterization and condition of the sample where relevant
- Date of receipt of sample
- Date and time of sample collection, if provided
- Date and time of sample analysis, reanalysis, and/or sample preparation
- Initials of analyst and person responsible for sample prep
- Analytical batch number
- Sample analysis and preparation methods (or unambiguous description of any non-standard method used)
- Reference to sampling procedure
- Additions to or deviations or exclusions from the test method, and other information relevant to a specific test, such as environmental conditions and the use and meaning of data qualifiers
- Nonconformances that affect the data
- Whether data are calculated on a dry weight or wet weight basis

- Identification of the reporting units, such as µg/L or mg/kg
- Statement of the estimated uncertainty of the test result, if applicable
- Signature and title of the person(s) accepting responsibility for the content of the C of A
- Date C of A was issued
- Clear identification of data provided by outside sources, such as air temperature or ambient water temperature
- Identification of the reporting detection limit (RDL) or practical quantitation limit (PQL) for each analyte, if applicable.

If a portion of the sample analysis is subcontracted, the C of A will identify the subcontractor or applicable accreditation number, and the data that was determined by the subcontracting laboratory

Level 2 Certificates of analysis contain the following additional information:

- Dilution factors
- Method detection limits
- Surrogate recoveries and the acceptance criteria for all organic analyses
- Estimated concentrations determined for nondetects and appropriate "U" and "J" qualifiers for nondetects and concentrations that fall between the MDL and PQL respectively.

Once issued, a C of A is not altered unless a subsequent C of A is identified as a revised report.

11.2 Quality Control Summary Report (QCSR)

We prepare and analyze samples in groups of twenty or less. The quality control data that demonstrate the sample preparation and/or analytical efficiency of the batch are summarized on a QCSR. The data reported on the QCSR may be limited to a sample delivery group contained in the batch or may include all quality control for the batch. Information reported on QCSR includes:

- Quality control sample ID number
- Type of quality control sample
- Concentrations determined, where applicable, for method blanks, matrix spikes, matrix spike duplicates, matrix duplicates, laboratory control samples, serial dilutions, and laboratory control sample duplicates
- Acceptance criteria for matrix spikes, matrix spike duplicates, matrix duplicates, laboratory control samples, and laboratory control sample duplicates
- Nominal concentrations of matrix spikes, matrix spike duplicates, LCSs, and LCS duplicates
- Concentration of parent sample for the matrix spikes, matrix spike duplicates, or sample duplicates
- Percent recoveries for LCS and matrix spikes
- Relative percent differences for the matrix spike duplicates, matrix duplicates, and LCS duplicates
- Analytical batch number with which the quality control data is associated
- Parent sample numbers for matrix spikes, matrix duplicates, and matrix spike duplicates
- Sample or sample delivery group ID
- Project code
- Date issued, page numbers/total number of pages
- Identification of recoveries or relative percent differences that do not meet the acceptance criteria

11.3 Analytical Case Narratives

Analytical case narratives are written by an analyst or data validator to describe the overall conditions affecting the analysis of a batch or a specific sample in the batch. Case narratives usually include:

- Sample delivery group ID number
- Analytical batch number
- Methods of preparation and analysis
- Sample matrix
- Initial of person preparing and/or reviewing the narrative
- Specific sample ID numbers
- Identification and description of batch quality control samples including parent sample identification
- Affirmation that all sample preparation conditions specified by the method or regulatory agencies were met or identification of specific deviations
- Affirmation that all analysis criteria specified by the method or regulatory agencies were met or identification of specific deviations

- Instrumentation employed if applicable and verification of its calibration
- Summary of batch quality control as compared to acceptance criteria
- Identification of nonconformances
- Pertinent comments and observations of factors that affect sample data quality

11.4 Electronic Data Deliverables (EDDs)

Electronic data deliverables are generated according to client specifications. EDDs use programs supplied by the client or created internally by our EDD team. Internally generated EDDs are usually written in Perl and/or PL/SQL.

11.5 Types of Data Packages and Reports

We offer three levels of data reports and the ability to design packages to meet the needs of our clients. The levels of data reports are summarized in Table 1.

Table 1: Data Report Formats

Level	Contents
1	Level 1 C of A
2	Level 2 C of A plus QCSR
3	Level 2 plus Case Narrative

If a client so requests, the above reports can be accompanied by EDDs, case narratives, copies of associated nonconformance reports, and other support documentation. The client's specific requirements are communicated to the laboratory and data reviewers through AlphaLIMS.

GEL's SOP GL-CS-E-002 for The Internal Review of Contractually Required Quality Criteria for Client Package Delivery defines preparation and review of the package.

If a client requests a CLP-like data package, and we agree to provide one, it is compiled in accordance with GL-LB-E-013 for CLP-Like/DOE Data Package Assembly and Revision. If a client does not request a full CLP-like data package but asks for data to be provided on CLP forms generated from software, we follow the applicable procedures in GL-LB-E-013.

11.6 Review of Data Reports, EDDs, and Data Packages

Level 1 and Level 2 data reports are reviewed for accuracy and completeness by the PM or PMA. Level 3 and CLP-like data packages are reviewed in the laboratory by a data reviewer, who is responsible for reviewing specific fractions of the data package for accuracy, consistency, and completeness in accordance with the SOP for that lab area.

No data package fraction is to be provided to the data packaging team without the approval of the appropriate data reviewer.

CLP-like data packages are reviewed in compliance with the basic protocol. Specific requirements are described in GL-LB-E-013 for the CLP-Like/DOE Data Package Assembly and Revision.

11.7 State Specific Reporting Criteria

Some state agencies require laboratories who perform drinking water analyses in support of Clean Water Act programs to communicate specific results to clients and/or agencies in some circumstances. If samples are found to contain concentrations of target analytes above those required by Federal or State regulations, the state must be informed. Please see Appendix K for state specific reporting criteria for drinking water programs.

SECTION 12
SUBCONTRACTING ANALYTICAL SAMPLES AND OUTSIDE SUPPORT SERVICES

Section 12 – Subcontracting Analytical Samples and Outside Support Services

We provide a full array of organic, inorganic, and radiochemical analyses. The subcontracting of samples to other facilities, while infrequent, may occur when:

- The client has requested analytical services for which we are not certified or do not offer as a routine product.
- The regulatory or method holding times and/or client due dates are in danger of not being met as the result of instrument malfunction or the unexpected influx of a large group of samples.

No samples are subcontracted without the client's consent. The laboratories selected to receive subcontracted samples are expected to meet the following criteria:

- Demonstrated technical capability to provide data that meet and conform to our quality standards.
- Established certification, if available, for the requested analyses.
- Successful proficiency evaluation results, if available.
- Commitment to meet time requirements for delivery of results to the client.
- Agreement to provide all documentation requested in conjunction with the analysis.
- NELAP, or ISO/IEC 17025 accreditation for the analysis if required by the client.

We audit potential subcontractors for technical and administrative compliance as directed in GL-QS-E-001 for Conduct of Quality Audits. An audit may be in the form of a book audit or an on-site review.

If there is evidence of a technical, administrative, or quality deterioration, the laboratory is removed from our list of approved subcontractor laboratories pending further evaluation, which may include an on-site audit. Once the laboratory again demonstrates compliance with GEL's standards, it can be reclassified as an approved subcontractor laboratory.

At GEL, we have a multi-faceted and trained staff. There are occasions, however, when it may be necessary to obtain the services of professionals outside of GEL. This may be due to such things as sample workload, introduction of a new instrument or method requiring special knowledge, or employee leaves of absence.

Any outside support services or service personnel are subject to the same scrutiny as a subcontract laboratory. If a service fails to meet our standards for excellence, the appropriate parties are promptly notified. If immediate corrections are not implemented and services are not of adequate quality to maintain confidence, the contract is canceled.

SECTION 13
CLIENT SATISFACTION

Section 13 – Client Satisfaction

Meeting the needs and expectations of our clients is essential to meeting our commitment to be the environmental laboratory of first choice. An important part of meeting this commitment involves receiving and resolving client concerns and complaints.

Client complaints that question the quality of laboratory data or data deliverables are directed to Quality Systems. These concerns are responded to with input from the laboratory, EDD team or data packaging group as may be needed.

The types of complaints, area(s) affected, and any impacts on quality are trended on a quarterly basis. This information is available to members of the Leadership Team and other managers and group leaders.

We use AlphaLIMS to monitor client complaints, nonconformances and corrective actions. Every complaint is entered into the system upon receipt and assigned an internal and external due date. The external due date is often established by client contract. The internal due date allows time for the Quality Systems Team to review the response and transmit it to the client on or before the due date.

If we notice a trend that significantly affects the quality of our data, a corrective action is initiated following GL-QS-E-002 for Conducting Corrective/Preventive Action and Identifying Opportunities for Improvement. The implementation and verification of the corrective action affirms an effective and permanent solution.

The Quality Systems Team promptly audits those areas of activity or responsibility for which a complaint or concern has been stated.

APPENDIX A: REFERENCES

- The NELAC Institute, TNI Standard, 2016.
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- 40 CFR Part 136, August 19, 2014 Guidelines Establishing Test Procedures for the Analyses of Pollutants.
- 40 CFR Part 141- National Primary Drinking Water Regulations, July 1, 2009, Subpart C-Monitoring and Analytical Requirements
- DOE Orders 414.1B 414.1C, and 414.D Quality Assurance, U.S. Department of Energy.
- EPA Requirements for Quality Assurance Project Plans (QAPPs), US EPA QA/R5.
- EPA 815-R-05-004 EPA Manual for the Certification of Laboratories Analyzing Drinking Water.
- Model Statement of Work for Analytical Laboratories, Prepared for Department of Energy NNSA Service Center by Analytical Quality Associates, Rev 7, November 2006.
- Specifications and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs, American National Standard ANSI/ASQC E4-2004.
- Measurement Associated Instrument Quality Assurance for Radiobioassay Laboratories ANSI N42.23-1995.
- US Department of Defense (DoD) Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD/DOE QSM Version, 5.3, May 2019.
- MARLAP- Multi-Agency Radiological Laboratory Analytical Protocols
- 10 CFR Part 21- Reporting of Defects and Noncompliance
- 10 CFR Part 61- Licensing Requirements for Land Disposal and Radioactive Waste
- NRC REG Guide 4.15 and NRC REG Guide 4.8
- ISO/IEC 17025-2017
- DOE G 414/1-3, 11-3-04, Suspect/ Counterfeit Items Guide for use with 10 CFR 830 Subpart A. Quality Assurance Requirements, and DOE O 414.B, Quality Assurance.

APPENDIX B: DEFINITIONS

The following definitions are used throughout the text of our Quality Systems Plan. These definitions were reprinted from "Definitions for Quality Systems," NELAC, July 1, 1999. For most entries, the original source of each definition is provided.

AlphaLIMS: GEL's Laboratory Information Management System.

Acceptance Criteria: Specified limits placed on characteristics of an item, process, or service defined in the requirement documents. (ASQC)

Accreditation: The process by which an agency or organization evaluates and recognizes a program of study or an institution as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. In the context of the National Environmental Laboratory Accreditation Program (NELAP), this process is a voluntary one. (NELAC)

Accuracy: The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (Glossary of Quality Assurance Terms, QAMS, 8/31/92)

Aliquot: A discrete, measured, representative portion of sample taken for analysis. (DoD, EPA QAD Glossary)

Analyst: The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality. (NELAC)

Analyte: The specific chemicals or components for which a sample is analyzed; may be a group of chemicals that belong to the same chemical family, and are analyzed together. (EPA Risk Assessment Guide for Superfund, OSHA Glossary)

Analytical Detection Limit: The smallest amount of an analyte that can be distinguished in a sample by a given measurement procedure throughout a given confidence interval. (NELAC Quality Systems Committee)

Analytical Reagent (AR) Grade: Designation for the high purity of certain chemical reagents and solvents given by the American Chemical Society. (NELAC Quality Systems Committee)

Analytical Sample: Any solution of media introduced into an instrument on which an analysis is performed excluding instrument calibration, initial calibration verification (ICV), initial calibration blank (ICB), continuing calibration verification (CCV), and continuing calibration blank (CCB)

ANSI: American National Standards Institute–this consensus standards body approves standards as a guide to aid the manufacturer, the consumer and the general public who may be concerned with its scope and provisions.

Audit: A systematic evaluation to determine the conformance to quantitative and qualitative specifications of some operational function or activity. (EPA-QAD)

Batch: Environmental samples prepared and/or analyzed together with the same process and personnel using the same lot(s) of reagents. A **preparation batch** is composed of one to 20 environmental samples of the same NELAC-defined matrix, meeting the above-mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) that are analyzed together as a group using the same calibration curve or factor. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples. (NELAC Quality Systems Committee)

Blank: A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subject to the usual analytical and measurement process to

establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. (ASQC)

Blind Sample: A subsample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process. (NELAC)

Calibrate: To determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter or other device, or the correct value for each setting of a control knob. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements. (NELAC)

Calibration: The set of operations that establish, under specified conditions, the relationship between values indicated by a measuring device, or the correct value of each setting of a control knob. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements. (NELAC)

Calibration Curve: The graphical relationship between the known values, such as concentrations, of a series of calibration standards and their analytical response. (NELAC)

Calibration Standard: A substance or reference material used to calibrate an instrument. (QAMS)

Certified Reference Material (CRM): A reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation that is issued by a certifying body. (ISO Guide 30 – 2.2)

Chain of Custody: A record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number of and types of containers; the mode of collection; collector; time of collection; preservation; and requested analyses. (NELAC Quality Systems Committee)

Commercial Grade Items: When applied to analytical services provided to nuclear power plants licensed pursuant to 10 CFR Part 50, commercial grade item means a structure, system, or component, or part there of that affects its safety function, that was not designed and manufactured as a basic component. In the laboratory operations, commercial grade items may include calibration standards, quality control standards, reagents, instrument software conducting calculations, calibration services for support instrumentation, and other process controls, verifying their acceptability by inspections, tests, validation, or analyses by the purchaser or third-party dedicating entity (such as NIST, A2LA, NPL and TNI). This activity assures that a critical characteristic is acceptable. Commercial grade items do not include items where the design and manufacturing process require in-process inspections and verifications to ensure that defects or failures to comply are identified and corrected. These types of items are considered Consumables.

When applied to facilities and activities licensed pursuant to 10 CFR Parts 50, commercial grade item means an item that is:

(245) Not subject to design or specification requirements that are unique to those facilities or activities;

(ii) Used in applications other than those facilities or activities; and

(iii) To be ordered from the manufacturer/supplier on the basis of specifications set forth in the manufacturer's published product description (for example, a catalog).

It is the responsibility of the purchaser to identify the vendor type, grade, and use of the purchased item

Confirmation: Verification of the presence of a component through the use of an analytical technique that differs from the original test method. These may include: (NELAC)

Second column confirmation
Alternate wavelength
Derivatization
Mass spectral interpretation
Alternative detectors or
Additional cleanup procedures

Continuing Calibration Blank (CCB): Aliquot of reagent water or other blank matrix that is analyzed after each CCV. The CCB is used to determine whether the analytical sequence is in control during sample analysis.

Continuing Calibration Verification Standard (CCV): An aliquot of reagent water or to the blank matrix to which known quantities of the method analytes are added in the laboratory. The CCV is analyzed exactly like a sample periodically throughout the sequence. Its purpose is to determine whether the analytical sequence is in control during the sample analysis. It may be prepared from the same source as the calibration standards and is usually of varied concentrations.

Control Limits: A range within which specified measurement results must fall to be compliant.

Corrective Action: Action taken to eliminate the causes of an existing nonconformity, defect, or other undesirable situation in order to prevent recurrence. (ISO 8402)

Data Audit: A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality (i.e., that they meet specified acceptance criteria). (NELAC)

Data Reduction: The process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useful form. (EPA-QAD)

Detection Limit: The lowest concentration or amount of the target analyte that can be determined to be different from zero by a single measurement at a stated degree of confidence. Refer to Method Detection Limit. (NELAC)

Document Control: The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly, and controlled to ensure use of the correct version at the location where the prescribed activity is performed. (ASQC)

Duplicate Analyses: The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA-QAD)

Holding Times (Maximum Allowable Holding Times): The maximum times that samples may be held prior to analysis and still be considered valid. (40 CFR Part 136)

Initial and Continuing Demonstrations of Capability: Procedures to establish the ability of the laboratory to generate acceptable accuracy and precision which is included in many of the EPA's analytical test methods. In general, the procedure includes the addition of a specified concentration of each analyte in each of four separate aliquots of laboratory pure water or authentic samples. These are carried through the analytical procedure and the percentage recovery and the standard deviation are compared to specified limits. (40 CFR Part 136, 2003 NELAC)

Internal Standard: A known amount of standard added to a test portion of a sample and carried through the entire measurement process as a reference for evaluating and controlling the precision and bias of the applied analytical test method. (NELAC)

Initial Calibration Blank (ICB): An aliquot of reagent water or other blank matrix that is analyzed after each ICV. The ICB is used to determine whether there is carryover contamination after injection of the mid-level ICV.

Initial Calibration Verification (ICV): A solution of method analytes of known concentrations that is used to fortify an aliquot of blank or sample matrix. The ICV is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check laboratory performance with externally prepared test materials.

Instrument Performance Check Solution (IPC): A solution of one or more method analytes, surrogates, internal standards, internal standards, or other test substances used to evaluate the performance of the instrument system with respect to a defined set of criteria.

Internal Standard (ISTD): A known amount of standard added to a portion of the sample extract as a reference for evaluating and controlling the precision and bias of the applied analytical method.

Interferents: Substances that affect the analysis for the element of interest.

ISO/IEC 17025: The International Organization for Standardization and International

Ectrotechnical Commission form this specialized system for worldwide standardization. Members of ISO or IEC participate in the development of International Standards through technical committees established by their organization to deal with particular fields of activity. Other international organizations, government and non-government, also take part in development of these standards. The ANSI/ISO/IEC 17025-2017 is approved as an American National Standard and covers general requirements for the competence of testing and calibration laboratories.

Laboratory: A body that calibrates and/or tests.

1. In cases where a laboratory forms part of an organization that carries out other activities besides calibration and testing, the term "laboratory" refers only to those parts of that organization that are involved in the calibration and testing process.
2. As used herein, the term "laboratory" refers to a body that carries out calibration or testing at or from a permanent location, from a temporary facility, or a mobile facility. (ISO 25)

Laboratory Control Sample (LCS): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes from a source independent of the calibration standards or a material containing known and verified amounts of analytes. It is generally used to establish intra-laboratory or analyst specific precision and bias to assess the performance of all or a portion of the measurement system. (NELAC)

Laboratory Duplicate: Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently. (NELAC Quality Systems)

Limit of Detection (LOD): The lowest concentration level that can be determined by a single analysis and with a defined level of confidence to be statistically different from a blank. See also Method Detection Limit. (Analytical Chemistry, 55, p.2217, Dec. 1983, modified)

Limit of Quantitation (LOQ): The lowest concentration level of the initial calibration curve used to quantitate an analyte. (DoD clarification) The LOQ must be $\geq$ 3X the LOD, and is usually not more than 10X the LOD.

Lower Limit of Quantitation (LLOQ): The lowest concentration at which a target analyte can be reliably measured and reported. The LLOQ is $\geq$ the lowest point in the calibration curve and represents a concentration at which both quantitative and qualitative requirements can be consistently demonstrated. The LLOQ is verified at least annually, by typically quarterly, as the LOQ verification. The verifications are performed by extracting and analyzing an LCS spiked at 0.5 to 2 times the LOQ. The LLOQ verification is carried through the same preparation and analytical procedures as environmental samples and QC.

Linear Calibration Range: The concentration range over which the instrument response is linear.

Matrix: The component or substrate that contains the analyte of interest. For purposes of batch determination, the following matrix types shall be used:

- ◊ Aqueous: Any aqueous sample excluded from the definition of a drinking water matrix or saline/estuarine source. Includes surface water, groundwater, and effluents.
- ◊ Drinking Water: Any aqueous sample that has been designated a potable or potential potable water source.
- ◊ Saline/Estuarine: Any aqueous sample from an ocean or estuary, or other salt-water source.
- ◊ Non-aqueous liquid: Any organic liquid with <15% settleable solids.
- ◊ Biological Tissue: Any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.
- ◊ Solids: Includes soils, sediments, sludges and other matrices with >15% settleable solids.
- ◊ Chemical Waste: A product or by-product of an industrial process.
- ◊ Air Samples: Media used to retain the analyte of interest from an air sample such as sorbent tubes or summa canisters. Each medium shall be considered as a distinct matrix. (Quality Systems)

Matrix Spike (MS): Prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency. (Glossary of Quality Assurance Terms, QAMS, 8/31/92)

Matrix Spike Duplicate (spiked sample/fortified sample duplicate): A second replicate matrix spike is prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte. (Glossary of Quality Assurance Terms, QAMS, 8/31/92)

May: Denotes permitted action, but not required action. (NELAC)

Method Blank (MB): A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples containing an analyte of interest through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses. (NELAC)

Method Detection Limit (MDL): The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136 Appendix B)

Must: Denotes a requirement that is required to be met. (Random House College Dictionary)

Negative Control: Measures taken to ensure that a test, its components, or the environment does not cause undesired effects, or produce incorrect test results. (NELAC)

NELAC: National Environmental Laboratory Accreditation Conference. A voluntary organization of state and federal environmental officials and interest groups purposed primarily to establish mutually acceptable standards for accrediting environmental laboratories. A subset of National Environmental Laboratory Accreditation Program (NELAP).

Performance Audit: the routine comparison of independently obtained quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory. (NELAC)

Performance Based Measurement System (PBMS): A set of processes wherein the data quality needs, mandates, or limitations of a program or project are specified and serve as criteria for selecting appropriate test methods to meet those needs in a cost-effective manner. (NELAC)

Positive Control: Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects. (NELAC)

Practical Quantitation Limit (PQL): The lowest level in the calibration curve. With the prep factor applied, the PQL is referred to as the effective PQL.

Precision: The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance, or range, in either absolute or relative terms. (NELAC)

Preservation: Refrigeration and or reagents added at the time of sample collection to maintain the chemical and or biological integrity of the sample. (NELAC)

Proficiency Test Sample (PT): A sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria. (Glossary of Quality Assurance Terms, QAMS, 8/31/92)

Proficiency Testing: A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source. (NELAC, Section 2.1)

Proficiency Testing Program: The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results in comparison to peer laboratories and the collective demographics and results summary of all participating laboratories. (NELAC)

Protocol: A detailed written procedure for field and/or laboratory operation (e.g., sampling, analysis) that must be strictly followed. (EPA-QAD)

Pure Reagent Water: Shall be water in which no target analytes or interferences are present at a concentration that would impact the results when using a particular analytical test method. (NELAC)

Quality Assurance: An integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality within a stated level of confidence. (Glossary of Quality Assurance Terms, QAMS, 8/31/92)

Quality Control: The overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the need of users. (Glossary of Quality Assurance Terms, QAMS, 8/31/92)

Quality Manual: A document stating the quality policy, quality system and quality practices of an organization. This may also be called a Quality Assurance Plan or a Quality Plan. **NOTE:** The quality manual may call up other documentation relating to the laboratory's quality arrangements. (Quality Systems Committee)

Quality System: A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC. (ANSI/ASQC E-41994)

Quantitation Limits: The value at which an instrument can accurately measure an analyte at a specific concentration that includes the maximum or minimum levels, concentrations, or quantities of a target that can be quantified with the accuracy required by the data user. These values establish the upper and lower limits of the calibration range. (NELAC with DoD clarification)

Range: The difference between the minimum and the maximum set of values. (EPA_QAD)

Raw Data: Any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies,

computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments. If exact copies of raw data have been prepared (e.g., tapes that have been transcribed verbatim, dated and verified accurate by signature), the exact copy or exact transcript may be submitted. (EPA-QAD)

Reagent Blank (method reagent blank): A sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps. (Glossary of Quality Assurance Terms, QAMS, 8/31/92)

Reference Material: A material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (ISO Guide 30 -2.1)

Reference Standard: A standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived. (VIM – 6.08)

Relative Percent Difference (RPD): The difference between two duplicate samples, such as a MS/MSD/, LCS/LCSD, or sample/sample DUP. It is determined by taking the difference between the two results and dividing by the average.

Reporting Limit (RL): The level at which a target analyte would meet the data quality objectives of the laboratory and/or a project, which may include establishing compliance with a regulatory and/or action limit. The RL may be equal to the laboratory practical quantitation limit (PQL)

Requirement: Denotes mandatory specification; often designated by the term "shall." (NELAC)

Sample: Portion of material collected for chemical analysis, identified by a single, unique term. A sample may consist of portions in multiple containers, if a single sample is submitted for multiple or repetitive analysis. (DoD)

Safety Related Procured Items: As specified in 10 CFR Part 50 and other Nuclear Power related activities, a basic component includes safety-related analytical services that are associated with the component information in support of an early site permit application or other safety related services identified by the client, whether the services are performed by the laboratory or others. GEL has identified the primary safety related basic component item for these services as:

- ◊ Calibration Standards for Radiochemical Analyses used in the direct issuance of analytical data reported to a Nuclear Facility. These standards are the primary sources (critical characteristic) of calibration for instruments that will provide the analytical results to our client. All Safety Related Procured Items are considered Type I procurement and must meet all specifications as identified in SOP GL-RC-E-002.

Standard Operating Procedure (SOP): A written document that details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and is accepted as the method for performing certain routine or repetitive tasks. (Glossary of Quality Assurance Terms, QAMS, 8/31/92)

Standard Reference Material (SRM): A certified reference material produced by the U.S. National Institute of Standards and Technology and characterized for absolute content, independent of analytical test method. (NELAC)

Statistical Process Control (SPC): Statistically derived limits that establish acceptable ranges for recoveries of analytes of interest, including LCS, MS, MSD, PS, PSD and internal standards.

Stock Standard Solution: A concentrated solution containing one or more method analytes prepared in the laboratory using certified reference materials or purchased from a reputable commercial source.

Selectivity: The capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. (NELAC Quality Systems)

Sensitivity: The capability of a test method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (NELAC Quality Systems)

Serial Dilution: The dilution of a sample by a known factor. When corrected by the dilution factor, the diluted sample should agree with the original undiluted sample within the specified limits. Serial dilution may reflect the influence of interferents.

Shall: Denotes a requirement that is mandatory whenever the criterion for conformance with the specification requires that there will be no deviation. This does not prohibit the use of alternative approaches or methods for implementing the specification so long as the requirement is fulfilled. (ANSI)

Should: Denotes a guideline or recommendation whenever noncompliance with the specification is permissible. (ANSI)

Spike: A known mass of target analyte added to a blank sample or subsample; used to determine recovery efficiency or for other quality control purposes.

Subsample: A portion of the entire sample randomly collected and composited to create weight used for the solvent extraction process. The subsample should be representative of the entire sample.

Surrogate: A substance with properties that mimic the analyte of interest. It is unlikely to be found in environmental samples and is added to them for quality control purposes. (Glossary of Quality Assurance Terms, QAMS, 8/31/92)

Test: A technical operation that consists of the determination of one or more characteristics or performance of a given product, material equipment, organism, physical phenomenon, process or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate. (ISO/IEC Guide 2 – 12.4)

Test Method: An adoption of a scientific technique for a specific measurement problem, as documented in a laboratory SOP. (NELAC)

Tolerance Chart: A chart in which the plotted quality control data is assessed via a tolerance level (e.g. ± 10% of a mean) based on the precision level judged acceptable to meet overall quality/data use requirements instead of a statistical acceptance criteria (e.g. ± 3 sigma). (ANSI N42.23-1995, Measurement and Associated Instrument Quality Assurance for Radiochemistry Laboratories)

Traceability: The property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons. (VIM-6.12)

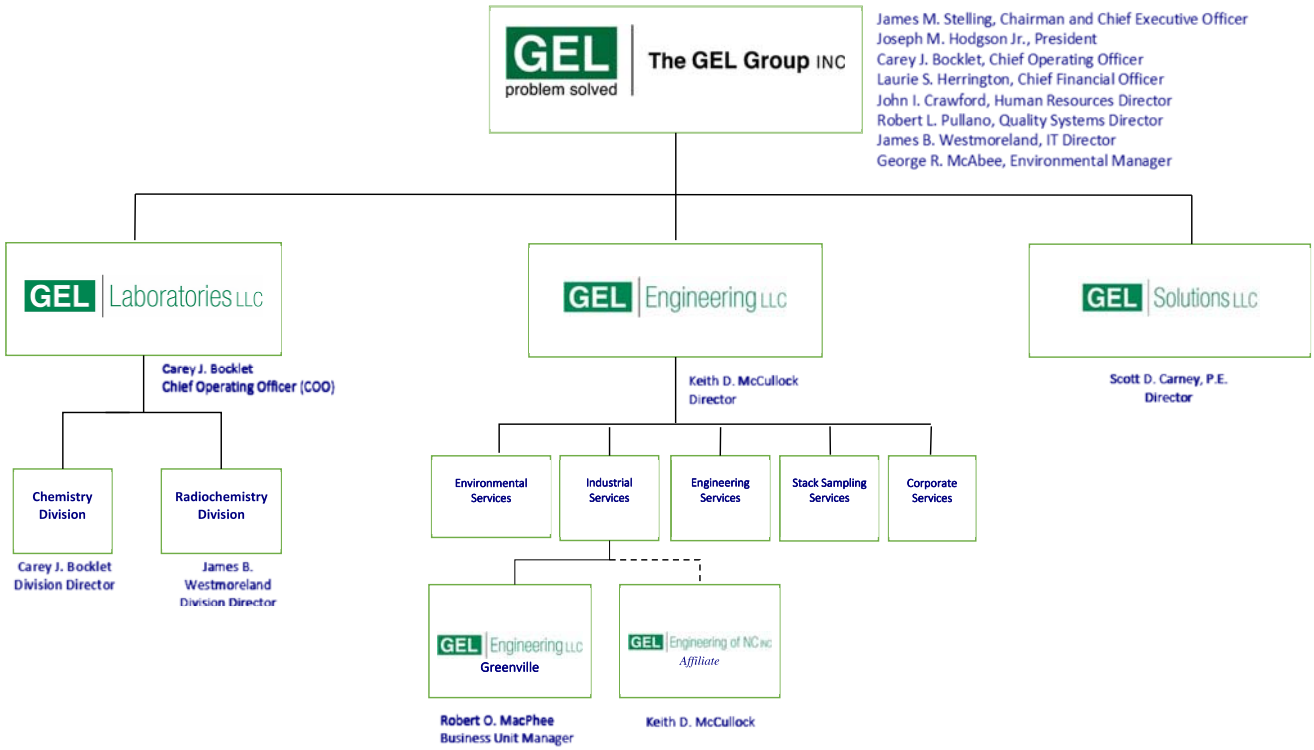
Validation: The process of substantiating specified performance criteria.

Verification: confirmation by examination and provision of evidence that specified requirements have been met. (NELAC)

NOTE: Verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation, or specification peculiar to the management of the measuring equipment.

The result of verification leads to a decision either to restore in service, to perform adjustments, or to repair, or to downgrade, or to declare obsolete. In all cases it is required that a written trace of the verification performed shall be kept on the measuring instrument's individual record.

APPENDIX C: CORPORATE ORGANIZATION CHART



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APPENDIX D: CERTIFICATIONS

GEL Laboratories, LLC maintains environmental laboratory certification in many states, including primary NELAP in Utah and secondary in Florida, Illinois, Kansas, Louisiana, New Hampshire, New Jersey, New York, Pennsylvania, Texas and Virginia. We expand our list of certifications as needed.

Original Scope of Accreditation/Range of Activities are maintained in the Quality Assurance work area. Electronic copies are available in .pdf form on the GEL intranet. *Please call to confirm the status of any certification of interest.*

- **Department of Defense (DoD) Department of Energy (DOE**) Consolidated Quality Systems Manual Version 5.3, May 2019 through American Association for Environmental Laboratory Accreditation (A2LA) (A2LA 2567.01)
- **U.S. Department of Agriculture –** Foreign soil importation permit # P330-18-00302, P330-18-00303
- **National Environmental Laboratory Accreditation Program (NELAP) –** Primary issued through the State of Utah, Department of Health; Secondary issued through the States of Florida, Illinois, Kansas, Louisiana, New Hampshire, New Jersey, New York, Pennsylvania, Texas and Virginia
- **Clinical Laboratory Improvement Amendments (CLIA) –** U.S. Department of Health and Human Services, Certificate of Compliance for Acceptance of Human Specimens (GEL ID: 42D0904046)
- **Alaska Department of Environmental Conservation Contaminated Sites** Laboratory Approval 17-018
- **Alaska Department of Environmental Conservation Laboratory Certification Program, Chemical Analysis in Drinking Water** SC00012
- **Arkansas** Department of Environmental Quality Laboratory Certification Program for Wastewater, Groundwater, Solid Waste Reciprocal Certification to SC DHEC (88-0651)
- **California** Environmental Laboratory Accreditation Program Certification, ELAP (GEL ID: 2940)
    - Sanitation Districts of Los Angeles County (GEL ID: 9255651)
- **Colorado** Department of Public Health and Environment, Reciprocal Certification to SC DHEC Environmental Laboratory Certification Program for Safe Drinking Water Chemistry and Radiochemistry (SC00012)
- **Connecticut** Department of Public Health – Potable Water, Waste Water and/or Trade Waste, Sewage and/or Effluent, Soil and Radiochemistry Reciprocal Certification (GEL ID: PH-0169)
- **Florida** Department of Health, Bureau of Laboratories, Secondary NELAP (GEL ID E87156)
- **Georgia** Department of Natural Resources, Reciprocal Certification to SC DHEC Environmental Laboratory Certification Program for Safe Drinking Water (GEL ID: 967)
- **Hawaii** Department of Health, Safe Drinking Water, reciprocal to Utah NELAP, SC00012
- **Idaho** Department of Health and Welfare, SC00012

- **Illinois** EPA Environmental Laboratory Accreditation for Drinking Water, Wastewater, and Hazardous and Solid Waste, Secondary NELAP (GEL ID: 200029)
- **Indiana** State Department of Health (C-SC-01)
- **Kansas** Department of Health and Environmental Laboratory, Non-potable Water and Solid and Hazardous Waste, Secondary NELAP (GEL ID: E-10332)
- **Kentucky** Department of Environmental Protection for Drinking Water and Waste Water (GEL ID: 90129)
- State of **Louisiana** Department of Health and Hospitals (SC00012), Safe Drinking Water, Secondary NELAP
- State of **Louisiana** Department of Environmental Quality, (03046, AI 33904), Non-drinking water, Secondary NELAP
- State of **Maine** Laboratory Accreditation Program (SC00012)
- **Maryland** Department of Health and Mental Hygiene, Laboratories Administration, Reciprocal Certification to SC DHEC Environmental Laboratory Certification Program for Safe Drinking Water –Radiochemistry (GEL ID: 270)
- **Massachusetts** Department of Environmental Protection, Division of Environmental Analysis – Potable Water, Radiochemistry (GEL ID: M-SC012), Per- and Polyfluoralkyl Substances (PFAS)
- **Michigan** Department of Environmental Quality – Potable Water, Radiochemistry (GEL ID: 9976)
- **Mississippi** State Department of Health NELAP reciprocity
- **Nebraska**, Department of Health and Human Services (GEL ID: NE-OS-26-13)
- **Nevada** Department of Human Resources, Health Division, Bureau of Licensure and Certification, Radiologicals and Non-Radiologicals (GEL ID: SC000122020-1), Nevada Mining
- State of **New Hampshire** Environmental Laboratory Accreditation Program, Secondary NELAP (2054)
- **New Jersey** Department of Environmental Protection, Safe Drinking Water, Solid and Hazardous Waste, and Water Pollution Certification, Secondary NELAP (GEL ID: SC002)
- State of **New Mexico** Environment Department, Drinking Water Bureau, reciprocal to NELAP SC00012
- **New York** Department of Health, Environmental Laboratory Approval Program Certification, Potable Water, Non-potable Waters and Solids/Hazardous Wastes, Secondary NELAP (GEL ID: 11501)
- **North Carolina** Division of Water Quality Lab Certification Program, Waste Waters/Ground Waters. (GEL ID: 233)
- **North Carolina** Department of Health and Human Services, North Carolina State Laboratory Public Health Environmental Sciences, Safe Drinking Water. (GEL ID: 45709)

- **North Dakota** State Department Protection- Bureau of Laboratories, Secondary NELAP (R-158) and TENROM
- **Oklahoma** Department of Environmental Quality, General Water Quality/Sludge Testing Laboratory Dual Certification (GEL ID: 9904)
- **Pennsylvania** Department of Environmental Protection – Bureau of Laboratories, Secondary NELAP (GEL ID: 68-00485)
- **Puerto Rico** Department of Health Recipricol certification to Manual for the Certification of Laboratories Analyzing Drinking Water. PRDOH (SC00012)
- **South Carolina** Department of Health and Environmental Control – Environmental Laboratory Certification Program, Clean Water, Safe Drinking Water, Radiological, and Solid/Hazardous Wastes (GEL ID: 10120001/10120002)
- **South Carolina** Department of Health and Environmental Control (DHEC) Radioactive Material License (License #362)
- **Tennessee** Department of Health – Division of Laboratory Services, Reciprocal Certification to SC DHEC Environmental Laboratory Certification Program, Safe Drinking Water-Radiochemistry and Non-radiochemistry (GEL ID: 02934)
- **Texas** Commission on Environmental Quality, Secondary NELAP (GEL ID: T104704235-20-16)
- **Utah** Department of Health, Division of Epidemiology and Laboratory Services, Safe Drinking Water, Clean Water and Resource and Conservation and Recovery Act Certifications Primary NELAP (Customer ID: SC000122020-32)
- **Vermont** Department of Environmental Conservation, Water Supply Division, Secondary NELAP (VT87156)
- Commonwealth of **Virginia** Department of General Services – Division of Consolidated Laboratory Services, Safe Drinking Water, Clean Water Act and Resource and Conservation Act Certifications, Secondary NELAP (GEL ID: 460202)
- **Washington** State Department of Ecology, Safe Drinking Water, Clean Water and Resource and Conservation and Recovery Act Certifications (GEL ID: C780)

APPENDIX E: ESSENTIAL QUALITY CONTROL REQUIREMENTS

At GEL, we enforce strict adherence to quality control measures. Quality control measures for each type of analysis are delineated in the associated standard operating procedure and include those specified in the identified analytical method. Client requests for additional quality control agreed to by us will be communicated to the laboratory by the Project Manager and performed accordingly.

All quality control measures are assessed and evaluated on an ongoing basis. We use these measures to establish statistically derived quality control acceptance criteria. The acceptance criteria are used to evaluate whether the analytical process is in control and to assist us in establishing the validity of the data. Our procedures for handling out- of-control situations are written in the analytical standard operating procedure.

Method-specific quality measures are described in the appropriate standard operating procedure. Essential but general quality control requirements are summarized in the sections below for chemical testing, including inorganic and organic analyses, and radiochemical testing.

E1 Chemical Testing

This section includes our quality control requirements for inorganic and organic analyses, and discusses:

- Negative controls
- Positive controls
- Analytical variability and reproducibility
- Method evaluation
- Method detection limits
- Data reduction
- Quality of standards and reagents
- Selectivity
- Constant and consistent test condition

E1.1 Negative controls

We implement a negative control at least once per analytical batch of samples having the same matrix, and where, if applicable, the same extraction or preparation method is employed. The negative control is a method blank that we use to determine the presence of contamination. If discovered, we must investigate the source of contamination and take measures to correct, minimize, or eliminate the source if:

1. The concentration of target analyte exceeds the established practical quantitation limit and exceeds a concentration greater than 1/10 of the measured concentration of any sample in the analytical batch;
2. The concentration of a target analyte in the method blank exceeds that present in the samples and is greater than 1/10 of the specified regulatory limit.

If a method blank is indicative of contamination, we must assess each sample in that batch against the above criteria to determine if the data are acceptable. Any sample associated with a contaminated method blank shall be reprocessed for analysis, as needed, or we will report the results with appropriate data qualifiers.

E1.2 Positive Control – Method Performance

E1.2.1 Laboratory Control Sample (LCS)

Purpose: The LCS is used to evaluate the performance of the total analytical system, including all preparation and analysis steps. Results of the LCS are compared to established criteria and, if found to be outside of these criteria, indicates that the analytical system is “out of control.” Any

affected samples associated with an out-of-control LCS shall be reprocessed for re-analysis or the results reported with appropriate data qualifying codes, as necessary.

Frequency: The LCS is analyzed at a minimum of 1 per preparation batch. Exceptions would be for those analytes for which no spiking solutions are available such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, temperature, dissolved oxygen or turbidity. In those instances for which no separate preparation method is used (example: volatiles in water) the batch shall be defined as environmental samples that are analyzed together with the same method and personnel, using the same lots of reagents, not to exceed the analysis of 20 environmental samples.

Composition: The LCS is a controlled matrix, known to be free of analytes of interest, spiked with known and verified concentrations of analytes. **NOTE:** The matrix spike may be used in place of this control as long as the acceptance criteria are as stringent as for the LCS. Alternatively the LCS may consist of a medium containing known and verified concentrations of analytes or as Certified Reference Material (CRM). All analyte concentrations shall be within the calibration range of the methods. The following shall be used in choosing components for the spike mixtures:

The components to be spiked shall be as specified by the mandated test method or other regulatory requirement or as requested by the client. In the absence of specified spiking components the laboratory shall spike per the following:

For those components that interfere with an accurate assessment such as spiking simultaneously with technical chlordane, toxaphene, and PCBs, the spike should be chosen that represents the chemistries and elution patterns of the components to be reported.

For those test methods that have extremely long lists of analytes, a representative number may be chosen. The analytes selected should be representative of all analytes reported. The following criteria shall be used for determining the minimum number of analytes to be spiked.

a) For methods that include 1-10 targets, spike all components;
b) For methods that include 11-20 targets, spike at least 10 or 80%, whichever is greater;
c) For methods with more than 20 targets, spike at least 16 components.

NOTE: Unless otherwise noted in project quality assurance plans or if components interfere with an accurate assessment, all Department of Defense projects will have LCS, MS, and MSD that contain all target analytes.

Evaluation Criteria and Corrective Action: The results of the individual batch LCS are calculated in percent recovery. The laboratory shall document the calculation for percent recovery. The individual LCS is compared to the acceptance criteria as published in the mandated test method. Where there are no established criteria, the laboratory determines internal criteria or utilizes client specified assessment criteria.

An LCS that is determined to be within the criteria effectively establishes that the analytical system is in control and validates system performance for the samples in the associated batch. Samples analyzed along with a LCS determined to be “out of control” should be considered suspect and the samples reprocessed and re-analyzed or the data reported with appropriate data qualifying codes as necessary.

E1.2.2 Sample Specific Controls

The laboratory must document procedures for determining the effect of the sample matrix on method performance. These procedures relate to the analyses of matrix specific Quality Control (QC) samples and are designed as data quality indicators for a specific sample using the designated test method. These controls alone are not used to judge

laboratory performance. Examples of matrix specific QC include: Matrix Spike (MS); Matrix Spike Duplicate (MSD); Post Spike (PS) and Post Spike Duplicate (PSD) sample duplicates; and surrogate spikes.

E1.2.3 Matrix Spike ; Matrix Spike Duplicates, Post Spike ; Post Spike Duplicates :

Purpose:	Matrix specific QC samples indicate the effect of the sample matrix on the precision and accuracy of the results generated using the selected method. The information from these controls is sample/matrix specific and would not normally be used to determine the validity of the entire batch.
Frequency:	The frequency of the analysis of matrix specific samples shall be determined as part of a systematic planning process (e. g. Data Quality Objectives) or as specified by the required mandated test method.
Composition:	The components to be spiked shall be as specified by the mandated test method. Any permit specified analytes, as specified by regulation or client requested analytes shall also be included. If there are no specified components, the laboratory shall spike per the following: For those components that interfere with an accurate assessment such as spiking simultaneously with technical chlordane, toxaphene and PCBs, the spike should be chosen that represents the chemistries and elution patterns of the components to be reported. For those test methods that have extremely long lists of analytes, a representative number may be chosen using the following criteria for choosing the number of analytes to be spiked. However, the laboratory shall insure that all targeted components are included in the spike mixture over a 2-year period. a) For methods that include 1-10 targets, spike all components; b) For methods that include 11-20 targets, spike at least 10 or 80%, whichever is greater; c) For methods with more than 20 targets, spike at least 16 components.
Evaluation Criteria and Corrective Action:	The results from matrix spike/matrix spike duplicate and post spike/post spike duplicate are primarily designed to assess the precision and accuracy of analytical results in a given matrix and are expressed as percent recovery (%R) and relative percent difference (RPD). Results are compared to the acceptance criteria as published in the mandated test method. Where there are no established criteria, the laboratory should determine internal criteria and document the method used to establish the limits. For matrix spike or post spike results outside established criteria, corrective action shall be documented or the data reported with appropriate data qualifying codes.

E1.2.4 Matrix Duplicates:

Purpose:	Matrix duplicates are defined as replicate aliquots of the same sample taken through the entire analytical procedure. The results from this analysis indicate the precision of the results for the specific sample using the selected method. The matrix duplicate provides a usable measure of precision only when target analytes are found in the sample chosen for duplication.
Frequency:	The frequency of the analysis of matrix duplicates may be determined as part of a systematic planning process (e. g. Data Quality Objectives) or as specified by the mandated test method.
Composition:	Matrix duplicates are performed on replicate aliquots of actual samples. The composition is usually not known.
Evaluation Criteria and	The results from matrix duplicates are primarily designed to assess the precision of analytical results in a given matrix and are expressed as relative percent difference (RPD) or another

Corrective Action	statistical treatment (e. g., absolute differences). The laboratory shall document the calculation for relative percent difference or other statistical treatments. Results are compared to the acceptance criteria as published in the mandated test method. Where there are no established criteria, the laboratory shall determine internal criteria and document the method used to establish the limits. For matrix duplicates results outside established criteria corrective action shall be documented or the data reported with appropriate data qualifying codes.

E1.2.5 Surrogate Spikes:

Purpose	Surrogates are used most often in organic chromatography test methods and are chosen to reflect the chemistries of the targeted components of the method. Added prior to sample preparation/extraction, they provide a measure of recovery for every sample matrix.
Frequency	Except where the matrix precludes its use or when not available, or is not a method requirement, surrogate compounds are added to all samples, standards, and blanks for all appropriate test methods.
Composition:	Surrogate compounds are chosen to represent the various chemistries of the target analytes in the method. They are often specified by the mandated method and are deliberately chosen for their being unlikely to occur as an environmental contaminant. Often this is accomplished by using deuterated analogs of select compounds.
Evaluation Criteria and Corrective Action:	The results are compared to the acceptance criteria as published in the mandated test method or determined using statistical process controls (SPC). Where there are no established criteria, the laboratory determines internal criteria and documents the method used to establish the limits. Surrogates outside the acceptance criteria must be evaluated for the effect indicated for the individual sample results. The appropriate corrective action may be guided by the data quality objectives or other site specific requirements. Results reported from analyses with surrogate recoveries outside the acceptance criteria include appropriate data qualifiers.

E1.3 Method Evaluation

The following procedures, as described in the other sections of the QAP, are in place in order to ensure the accuracy of the reported result:

- Procedure for initial demonstration of analytical capability performed initially (prior to the analysis of any samples) and if there is a significant change in instrument type, personnel, matrix or test method. Refer to Section 8.
- Procedures for initial and continuing calibration protocols as specified in Section 7.
- Procedures for utilizing proficiency test samples to evaluate the ability of a procedure and/or analyst laboratory to produce accurate data as specified in Section 3.

E1.4 Method Detection Limits

Method detection limits (MDLs) are determined as described in GL-LB-E-001 for The Determination of Method Detection Limits. This procedure is based on that established in 40 CFR Part 136, Appendix B.

Where possible, MDL studies are conducted for both aqueous and solid matrices and biological tissues using a clean matrix appropriate to the test method (such as laboratory pure reagent water or Ottawa sand). MDL studies for the majority of routine parameters are conducted by:

- analyzing a minimum of seven replicates of the lowest calibration standard
- determining the standard deviation of the seven replicates

- multiplying the standard deviation by 3.143 (based on six degrees of freedom and representing a 99% confidence level) to obtain the calculated MDL.

If the MDL study is being conducted for a new method or target analyte, the following steps are taken:

- the MDL is estimated based on information provided in the method or analytical experience
- a standard with a concentration three to five times the estimated MDL is prepared and analyzed a minimum of seven times
- the MDL is calculated as above based on the standard deviation and degrees of freedom
- the MDL is evaluated for reasonableness by verification through analysis of a prepared standard solution two to three times the calculated MDL.

MDL studies are not performed for any target analyte for which spiking solutions are not available such as total volatile solids, pH, color, temperature, dissolved oxygen, or turbidity.

Practical quantitation limits (PQLs) are determined by either multiplying the MDL by approximately 2 to 10 or are equal to that of the lowest calibration standard. Concentrations of a target analyte determined to be greater that it's PQL are defined as quantitative results. All quantitative reported results are bracketed by calibration or calibration verification standards.

All MDL studies conducted by the laboratory are submitted to the Quality Group for an independent review. Upon acceptance of the MDL study, the MDLs reported to clients via our computer system are updated unless otherwise specified by contract. PQLs are also updated as directed by the new MDLs or changes to procedures.

All data pertaining to the study and the calculation of MDLs is stored on the production file system for data packages for four years and then archived to DVD.

GEL uses an industry standard approach to establishing radiochemistry and radiobioassay MDA (minimum detectable activity). This approach is based on MARLAP guidance for posteriori determination of MDA. The approach incorporates real time events that affect the observed sensitivity for every measurement performed in the laboratory. GEL recognizes for EPA radiological drinking water samples, that a MDL study is required similar to chemical constituents tested for drinking water.

GEL will follow the source document EPA 815-R-05-004 EPA Manual for the Certification of Laboratories Analyzing Drinking Water. In Chapter VI Critical Elements for Radiochemistry, section 1.5 of this document and alternate procedure is given for radiological constituents.

The analyst should prepare and measure a sample set of at least four reagent blanks and four laboratory fortified blanks that have the radioanalyte of interest added to quantitation levels appropriate for drinking water samples, the activity level added to the laboratory fortified blanks should be between the radioanalyte's MCL and its required detection limit. To be deemed an acceptable demonstration of proficiency, the mean recoveries and the standard deviation of the recoveries of the replicate measurements should be consistent with the requirements for accuracy and precision described in Section 7.7, and reagent blank measurements must have a mean result below the detection limit for each analyte measured with the method.

E1.5 Data Reduction

The procedures for data reduction, such as use of linear regression, are documented in the individual analytical standard operating procedures. GEL's policy governing the manual integration of chromatographic data is detailed in GL-LB-E-017, Procedure and Policy for Manual Integration. Manual integrations of chromatographic peaks can only be performed in accordance with GL-LB-E-017. This ensures that the integrations are done in a consistent and technically justifiable manner while meeting the requirements set forth under the Good Automated Laboratory Practices.

SOP GL-QS-E-014, Quality Assurance Measurement Calculations and Processes, discusses the use of laboratory data in statistical determinations and includes discussion of Estimation of Total Analytical Uncertainty, Statistical Process Control (SPC) Limits, and Calibration of Instrumentation. Understanding of the procedures used for data generation and reduction is an important part of an analyst demonstrating proficiency in an analytical procedure. All analysts and technicians responsible for generating curves and using curve-generated data are trained to this SOP per GEL annual and interim training requirements.

E1.6 Quality of Standards and Reagents

The quality of standards used in instrument calibration or quality control samples and reagents used in sample preparation and/or analysis must meet the criteria described in Section 7. In methods where the purity is not specified, analytical grade reagents are used. Reagents of lesser purity than those specified by the test method are never used. Upon receipt and prior to use, the labels on the container are checked to verify that the purity of the reagents meets the documented requirements of the particular test method.

The quality of water sources is monitored and documented as described in Section 4. The quality of water used in sample preparation or analysis meets the method-specified requirements. The type of water available in the laboratory is described in Section 4.

E1.7 Selectivity

Absolute and relative retention times aid in the identification of components in chromatographic analyses and in evaluation of the effectiveness of a column in separating constituents. The procedures governing retention time widows are documented in the applicable analytical SOP and meet all regulatory and method requirements.

In addition to retention time windows, the acceptance criterion for mass spectral training is also documented in the appropriate analytical SOP. In all cases, the acceptance criteria meet or exceed those specified in the analytical methods.

Unless stipulated in writing by the client, confirmations are performed to verify the compound identification of positive results detected on a sample from a location that has not been previously tested by our laboratory. Such confirmations are performed on a second column for organic tests such as pesticides, herbicides, or acid extractable or when recommended by the analytical test method except when the analysis involves the use of a mass spectrometer. All conformation is documented.

E1.8 Constant and Consistent Test Conditions

GEL's implementation of standard operating procedures that specify quality criteria including initial and continuing calibrations assures that our test instruments consistently operate within the specifications required of the application for which the equipment is used.

In addition to the specifications applied to instrumentation, glassware used for sample preparation or analysis is cleaned in a manner that reduces the potential for positive or negative interferences. Glassware is prepared in accordance with GL-LB-E-003 for Glassware Preparation.

This SOP details the procedures used to clean the following groups of glassware:

- That used for the determination of metals
- Reusable bottles and plastic ware
- Bottles sued for the determination of biochemical oxygen demand (BOD)
- Glassware used in the determination of organic compounds
- That used for the determination of methylene blue active substances (MBAS)
- Glassware used in the determination of total organic halides (TOX)
- Glassware used in the analyses of samples for total Kjeldahl nitrogen (TKN) and total phosphorous
- Generic glassware used in all other analyses

If the method specifies that the glassware be stored in a particular manner, this requirement is documented in the appropriate analytical SOP.

Section E2 Radiochemical Analysis

This section describes the general quality control applied to radiochemical analysis. The specific quality control criteria applied to each analysis are delineated in the corresponding SOP. Detector Capabilities, Relative Bias, Relative Precision, and methods of calculating results for periodic Quality Control Determinations are discussed in the appropriate SOPs.

Discussed in this section are:

- Negative controls
- Positive controls
- Test variability/reproducibility
- Tracers and carriers
- Method evaluation
- Radiation measurement system calibration
- Data reduction
- Quality of standards and reagents
- Test conditions

E2.1 Negative Controls

Method blanks serve as the primary negative controls providing a means of assessing the existence and magnitude of contamination introduced via the analytical scheme. A method blank is analyzed at a frequency of one per preparation or analytical batch and is one of the quality control measures used to assess batch acceptance.

The activity level determined for each target in the method blank is assessed against the specific acceptance criteria specified in the applicable SOP. These criteria are based on a designated sample aliquot size and include appropriate calculations to compare the blank to activity levels determined for different sizes of sample aliquots.

The activity level of any target analyte in the method blank should be less than or equal to the contract required detection limit. The method blank may exceed this limit if the activity is less than 5% that of the lowest sample activity in the batch.

If the method blank acceptance criteria are not met, the specified corrective action and contingencies delineated in the SOPs are followed. Any failures of method blanks to meet the acceptance criteria are documented in the laboratory report and through GEL's nonconformance reporting system specified in GL-QS-E-004 for the Documentation of Nonconformance Reporting and Dispositioning and Control of Nonconforming Items.

The activity levels determined for method blanks are not subtracted from those obtained for the samples in the associated preparation or analytical batch. Correction factors such as instrument background and analyte presence in the tracer may, however, be applied to all analyzed samples including both client samples and internal quality control samples.

E2.2 Positive Controls

Positive controls routinely employed in radiochemical analyses include both laboratory control samples (LCS) and matrix spikes (MS).

The laboratory standards used to prepare LCS and MS are from a different source than those used in instrument calibration, except when the calibration has been verified with a different source. This requirement may be superseded by client specific contract requirements. The activity levels of target analytes in the LCS and MS exceed ten times the prior detection limit and are less than one hundred times this detection limit. If a radiochemical method,

however, has more than one reportable analyte isotope, the LCS and MS need to only include one of the analyte isotopes.

Gamma spectroscopy is the exception to this guideline requiring the LCS and MS to contain isotopes representing the low, medium, and high-energy range of the analyzed gamma spectra.

E2.2.1 Laboratory Control Sample (LCS)

Laboratory control samples are analyzed at a minimum of once per preparation or analytical batch containing twenty or less samples.

The recovery of target analytes in the LCS is compared to the acceptance criteria specified in the applicable analytical SOP. If the recovery of the LCS does not fall within the acceptance range, the corrective actions and contingency steps specified in the SOP are implemented. These steps include the completion of an internal nonconformance report in accordance with GL-QS-E-004 and noting the failure on the laboratory report.

E2.2.2 Matrix Spike (MS)

Matrix spikes are analyzed at a minimum of once per preparation or analytical batch containing twenty samples or less under the following conditions:

- The analytical method does not utilize an internal standard or carrier
- There is a physical or chemical separation process
- There is sufficient sample volume provided for the analysis.

The target analyte recoveries are one of the quality control measures used to assess batch acceptance. The recovery of target analytes in the MS is compared to the acceptance criteria specified in the applicable analytical SOP. If the recovery of the MS does not fall within the acceptance range, the data associated with that matrix spike are qualified accordingly.

E2.3 Test Variability/Reproducibility

The reproducibility of measurements is evaluated by the use of matrix duplicates. Matrix duplicates are analyzed once per preparation or analytical batch of twenty samples. The relative percent difference (RPD) obtained between the activity levels obtained for the sample and its duplicate is evaluated against the range in the SOP.

E2.4 Tracers and Carriers

Two additional quality control measures specific to radiochemical analysis are tracers and carriers. If the analytical method requires a tracer or carrier, each sample result will be associated with a tracer recovery that is calculated and reported. For radiochemistry procedures requiring gravimetric or radiometric recovery (tracer yields), the acceptable limits are 15% - 125%. These limits may vary for specific clients and/or projects. If the applicable limits are not met, the corrective actions delineated in the SOP are implemented.

E2.5 Method Evaluation

GEL evaluates the radiochemical preparation and analytical methods to ensure the accuracy of the reported result. This evaluation includes initial demonstrations of capability as described in Section 8 and the analysis of proficiency test samples as described in Section 3. The suppliers of proficiency test samples conform to the requirements of ANSI N42.22 and ISO/IEC 17025-2017.

E2.6 Radiation Measurement System Calibration

It is not generally necessary or practical to calibrate radiochemical instrumentation each day of use due to its stability and the time-consuming nature of some of the measurements. There are, therefore, significant differences in the calibration requirements for radiochemical instrumentation from that used for chemical analyses.

Calibration differences include but are not limited to the following:

- The requirement in Section 7 for the determination of the appropriate number of standards for initial calibration is not applicable to radiochemical methods. If the radiochemical method requires multiple standards for initial calibration, the number of standards is included in the applicable SOP.
- If linear regression or non-linear regression is used to fit standard response or calibration standard results to a calibration curve, the correlation coefficient is determined. This differs from Section 7.
- The requirement identified in Section 7 for the bracketing of quantitative results by calibration or calibration verification standards is not applicable to radiochemical analyses due to the non-correlated event nature of decay counting instrumentation.
- As indicated in Section 7, the LCS may fill the requirements for the performance of an initial calibration and continuing calibration verification standard. The calibration verification acceptance criteria are the same as specified for the LCS (75 -125%).
- Background calibration measurements are made on a regular basis and monitored using control charts. These values are subtracted from the total measured activity in the determination of the sample activity. The frequency of these measurements is indicated in the SOP GL-RAD-I-010.
- Instrument calibration shall be performed with reference standards as defined in Section E3.8.
- The frequency of calibration shall be addressed in the governing SOPs.

E2.7 Data Reduction

All sources of method uncertainties and their propagation must be traceable to reported results. This is performed under the guidance of the ISO "Guide to the Expression of Uncertainty in Measurement" and the NIST Technical Note 1297 on "Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results." Details of calculations and equations used in reporting Radiochemistry analytical results may be found in GL-RAD-D-003 for Data Review, Validation and Data Package Assembly.

E2.8 Quality of Standards and Reagents

The reference standards we use are obtained from the National Institute of Standards and Technology (NIST), EPA, or suppliers providing NIST standards. Reference standards should be accompanied by a certificate of calibration whose content is described in ANSI N42.22 – 1995, Section 8, Certificates. All reagents used shall be analytical reagent grade or better.

E2.9 Test Conditions

GEL adheres to written procedures that minimize the possibility of cross contamination between samples. This prevents incorrect analysis results from the cross contamination. Procedures are in place, for example, to separate known radioactive and nonradioactive samples from the time of sample receipt to analysis and sample disposal.

Instrument performance checks are performed on a regular basis and monitored with control charts. This ensures that the instrument is operating properly and that the calibration has not changed. The same check source used in the preparation of the control chart at the time of calibration is used in the performance checks of the instrument. The sources must provide adequate counting statistics for a relatively short count time and should be sealed or encapsulated to provide loss of activity and contamination of the instrument and laboratory personnel.

Instrument performance checks include checks on the counting efficiency and the relationship between channel number and alpha or gamma ray energy.

APPENDIX F: ETHICS AND DATA INTEGRITY AGREEMENT

The GEL Group Inc.

ETHICS and DATA INTEGRITY AGREEMENT

I. I, (Name), state that I understand the high standards of integrity required of me with regard to the duties I perform and the data I report in connection with my employment at The GEL Group, Inc.

II. I agree that in the performance of my duties at The GEL Group, Inc.:

A. I shall not intentionally report data values that are not the actual values obtained,

B. I shall not intentionally report data that does not meet method or procedural specifications unless that data is properly qualified through comments or other notations in the analytical report.

C. I shall not intentionally report dates and times of data analyses that are not the actual dates and time of data analyses; and

D. I shall not intentionally represent another individual's work as my own.

III. I agree to inform a Group Leader, Manager, Director, or member of the Executive Committee of The GEL Group, Inc. of any accidental or intentional reporting of non-authentic data by myself or other employees in a timely manner.

IV. I will not knowingly participate in any questionable activities or violations of the Procurement Integrity Act during purchasing or sales activities. I will report any questionable activities to a Group Leader, Manager, Director, or member of the Executive Committee of The GEL Group, Inc. This includes discussions on analytical, consulting, and geophysical services pricing and contracts, vendor pricing, or other essential business information to anyone outside of The GEL Group, Inc. family.

This Ethics and Data Integrity Agreement has been explained to me by the Director of Quality Systems, my Group Leader, or at a training session, at which time I have been provided the opportunity to ask questions on any part of this agreement that I did not understand. It has also been explained to me that any violation of this agreement conducted during work performed under a subcontract or direct contract to a government agency could subject me to potential prosecution.

I understand that violation of this policy subjects me to disciplinary action, up to and including termination of my employment with The GEL Group Inc.

Employee Signature: _____ Date: _____

Trainer Signature: _____ Date: _____

APPENDIX G: EQUIPMENT LIST

SEMI-VOLATILE ANALYSIS-INSTRUMENTATION

#	Equipment	Model #	Purchase Date	ID/Serial #
1	Agilent 6890N Gas Chromatograph/ 5973 Mass Spectrometer w/ 7683 Autosampler Tower	5973	September-05	CN10521005/US52440275 MSD1
1	Agilent 7890A Gas Chromatograph/ 5975C Inert Mass Spectrometer w/ 7683 Autosampler Tower	5975	April-09	CN10848121/US83131300 MSD2
1	Agilent 7890A Gas Chromatograph/ 5975C Inert Mass Spectrometer w/ 7683 Autosampler Tower	5975	April-09	CN10821032/US83131355 MSD3
1	Agilent 7890A Gas Chromatograph/ 5975C Inert Mass Spectrometer w/ 7683 Autosampler Tower	5975	November-07	CN10727001/US90704000 MSD4
1	Hewlett Packard 6890 Gas Chromatograph/ 5973 Mass Spectrometer w/ 7683 Autosampler Tower	5973	May-97	US00023050/US82311233 MSD5
1	Hewlett Packard 6890 Gas Chromatograph/ 5973 Mass Spectrometer w/ 7683 Autosampler Tower	5973	May-97	US00025502/US82311417 MSD6
1	Agilent 7890 Gas Chromatograph with MMI/ 5977A Mass Spectrometer w/ 7693 Autoinjector	5977A	June-15	CN15233175/US1523M414 MSDA

SEMI-VOLATILE ANALYSIS-INSTRUMENTATION

1	Hewlett Packard 6890 Gas Chromatograph/ 5973 Mass Spectrometer w/ 7683 Autosampler Tower	5973	May-97	US00028102/US82311610 MSD8
1	Agilent 6890N Gas Chromatograph-FID w/ CTCH5500 Headspace Autosampler	6890	July-08	CN10805007 FID8
1	Agilent 6890N Gas Chromatograph-FID w/ 7683B Autosampler	6890	March-08	CN10805005 FID6
1	Agilent 6890N Gas Chromatograph-FID w/ 7683B Autosampler	6890	June-08	CN10811015 FID7
1	Agilent 6890N Gas Chromatograph-FID w/ 7683B Autosampler	6890	July-07	US10604037 FID5
1	Hewlett Packard 6890 Gas Chromatograph-FID w/ 6890 Autosampler	6890	March-98 (Installed 4/11/2011. Old MSD2 GC)	US0009213 FID9
1	Hewlett Packard 6890 Gas Chromatograph-Dual ECD w/ 7683 Autosampler	6890	March-98	US00023402 ECD1
1	Hewlett Packard 6890 Gas Chromatograph-Dual ECD w/ LEAP PAL RSI Autosampler	6890	March-98	US00028911 ECD2

SEMI-VOLATILE ANALYSIS-INSTRUMENTATION

1	Agilent 7890A Gas Chromatograph-Dual Micro ECD w/ 7693 Autosampler	7890A	March-10 (Purchased from CFA December-11)	CN10842125 ECD3
1	Hewlett Packard 6890 Gas Chromatograph-Dual ECD w/ 7673 Autosampler	6890	November-97	US00009591 ECD5
1	Hewlett Packard 6890 Gas Chromatograph-Dual ECD w/ 7683 Autosampler	6890	November-97	US00023343 ECD6
1	Hewlett Packard 6890 Gas Chromatograph-Dual ECD w/ 7673 Autosampler	6890	November-97	US00010134 ECD7
1	Agilent 6890 Gas Chromatograph-Dual ECD w/ 7683 Autosampler	6890	July-98	US10133016 ECD8
1	Agilent 7890A Gas Chromatograph-Dual Micro ECD w/ 7693 Autosampler	7890A	July-10	CN10261088 ECD9
1	Agilent 1260 Infinity II HPLC w/ DAD	1260	March-19	DEAEK04846, DEAEQ24263

1	Agilent 1100 HPLC w/ DAD	1100	2000?	US80603453, DE91609651
1	Ohaus Adventurer Analytical Balance		July-05	8912414746

LIQUID CHROMATOGRAPHY/HPLC

#	Equipment	Model #	Purchase Date	ID/Serial #
7	LC/MS/MS	Quattro Ultima (2)	May-02	D99SM9012R (LC) VB150 (MS)
		ABSCiex 4000 (3)	Sep-05	DE91608981 (LC) V04290402 (MS)
		ABSciex QTRAP 5500 (5)	Dec-14	L20435252316(LC) L20435252317(LC) AU212181403 (MS)
		ABSciex 5500 (6)	Nov-16	L20435453570(LC) L20435453571(LC) BB214331608 (MS)
		ABSciex 5500 (7)	Apr-17	L20435453807(LC) L20435453808(LC) BB215361701 (MS)
		ABSciex 5500 (8)	Nov-17	L20435553978(LC) L20435553979(LC) BB231241708 (MS)
		ABSciex 5500 (9)	Apr-19	L20435654577(LC) L20435654578(LC) BB230071702 (MS)
1	Shimadzu Column Heater (5)	CTO-20AC	Dec-14	L20215251917
1	Shimadzu Degasser (5)	DGU-20A	Dec-14	L20705263668

LIQUID CHROMATOGRAPHY/HPLC				
1	ABSciex PAL Autosampler (5)	MXY013-02A	Dec-14	326966
1	Shimadzu Column Heater (6)	CTO-20AC	Nov-16	L20215452696
1	Shimadzu Degasser (6)	DGU-20A	Nov-16	L20705366534
1	CTC Analytics PAL Autosampler (6)	MXY04-01A	Nov-16	380940
1	Shimadzu Column Heater (7)	CTO-20AC	Apr-17	L20215452846
1	Shimadzu Degasser (7)	DGU-20A	Apr-17	L20705467839
1	ABSciex PAL Autosampler (7)	MXY013-02A	Apr-17	141417
1	Shimadzu Column Heater (8)	CTO-20AC	Nov-17	L20215452696
1	Shimadzu Degasser (8)	DGU-20A	Nov-17	L20705568380
1	ABSciex PAL Autosampler (8)	MXY013-02A	Nov-17	410574
1	Shimadzu Column Heater (9)	CTO-20AC	Apr-19	L20215653401
1	Shimadzu Degasser (9)	DGU-20A	Apr-19	L20705670383
1	ABSciex PAL Autosampler (9)	MXY013-02A	Apr-19	423934
1	Agilent ALS	1100	Sep-05	JP13212623
1	Agilent Degasser	1100	Sep-05	US82404465
1	Agilent Column Heater	1100	Sep-05	DE11120879
1	Agilent Column Heater	1100	Apr-07	DE23919817
1	Hewlett Packard Quantum Pump	1100	Oct-99	DE91607770
1	Hewlett Packard ALS	1100	Oct-99	DE14913984
1	Hewlett Packard DAD	1100	Oct-99	JP03925183
1	Hewlett Packard Degasser	1100	Oct-99	US72103603

LIQUID CHROMATOGRAPHY/HPLC				
1	Hewlett Packard Column Heater	1100	Oct-99	DE91606066
1	Hewlett Packard Quantum Pump	1100	Nov-99	US80603453
1	Hewlett Packard ALS	1100	Nov-99	DE54627302 DE14904242
1	Agilent HPLC with DAD and FLD	1100	Nov-99	JP63203519
1	Hewlett Packard Degasser	1100	Nov-99	DE91609651
1	Hewlett Packard Column Heater	1100	Nov-99	DE33224733
1	Aglient Quantum Pump	1100	Jun-05	DE23909584
1	Agilent ALS	1100	Jun-05	DE91608331 DE92001137
1	Agilent HPLC with DAD and FLD	1100	Jun-05	JP13211588
1	Agilent Degasser	1100	Jun-05	DE33235932
1	Agilent Column Heater	1100	Jun-05	DE23919852
1	Aglient Quantum Pump	1100	Jun-07	US64401050
1	Aglient ALS	1100	Jun-07	DE43603083
1	Agilent DAD	1100	Jun-07	JP73016466
1	Agilent Degasser	1100	Jun-07	US82404303
1	Agilent Column Heater	1100	Jun-07	00119266EK
1	OHAUS Analytical Balance	CQ10R11-2E1	N/A	61476

1	Organomation N-EVAP112	9125	Nov-16	

VOLATILE ORGANIC ANALYSIS				
#	**Equipment**	**Model #**	**Purchase Date**	**ID/Serial #**
1	Hewlett Packard Gas Chromatograph/Mass Spectrometer Chemstation with OI 4760/Eclipse/4100 Sample Processor	5973	Oct-99 Jul-19	A917447740 US91911845 (US7119196) VOA1 E921410286
1	Hewlett Packard Gas Chromatograph/Mass Spectrometer Chemstation with 0I 4760 Eclipse/4100 sample Processer	5973	Nov-98 Jul-19	G107466806P US71191097 (US00023264) VOA9 E919410643
1	Aglient Gas Chromatograph/Mass Spectrometer Chemstation with OI 4760 Eclipse/4100 Sample Processor	5973	Apr-09 May-19	A910447548 US71191093/US00026073 VOA4 B521413051
1	Hewlett Packard Gas Chromatograph/Mass Spectrometer Chemstation with OI 4560/Arcon Autosampler	5975	Aug-06	K736460761 12534 US61332879(CN10848050)(VOA5)
VOLATILE ORGANIC ANALYSIS				

#	Equipment	Model #	Purchase Date	ID/Serial #
1	Hewlett Packard Gas Chromatograph/Mass Spectrometer Chemstation with 0I 4660 Eclipse/Arcon Autosampler	5973	Jan-98	K523466628P 12548 US71191112(US00010331)VOA8
1	Hewlett Packard Gas Chromatograph/Mass Spectrometer Chemstation with OI Eclipse/Arcon Autosampler	5975C	Apr-09	(E911466523P) VOA2 MS0901W017 US83131318/CN10606080
1	Hewlett Packard Gas Chromatograph/Mass Spectrometer Chemstation with OI 4560/Arcon Autosampler	5973	Jul-04	M948460722 41581 US71191113(US00028288)VOA3
1	Agilent Gas Chromatograph/Mass Spectrometer Chemstation	5977A	July-15	US51523M408/CN15173066 VOAC
1	Agilent Gas Chromatograph/Mass Spectrometer Chemstation with OI 4560/Arcon Autosampler	5975	Sep-05	A920447563 US52430466(CN10525054)VOA6 E919410645
1	Agilent Gas Chromatograph/Mass Spectrometer Chemstation with OI Eclipse/Arcon Autosampler	5975	Apr-09	E911466524P CN10848117 (VOAA) MS0901W018

				US83131219
1	Hewlett Packard Gas Chromatograph/Mass Spectrometer Chemstation with OI Eclipse/Arcon Autosampler PID/FID Detectors	6890	June-11	US0026725 (B431466149P) (VOAB) FID=1471 PID=54500 Archon 13382
1	Agilent Flame Ionization Detector /Chemstation with OI 4560	6890N	Aug-08	CN10813002 (VOC4)
1	OHAUS Toploading Balance	AV812N	N/A	B323410747
1	Sartorius Toploading Balance	CP622	N/A	19452583

ORGANICS EXTRACTIONS

Equipment	Model #	Purchase Date	ID/Serial #
Tekmar Sonic Distribution	600		22461D
J2 Scientific GPC	Accup-MP5	Jul-05	05C-1159-4-0

ORGANICS EXTRACTIONS

Equipment	Model #	Purchase Date	ID/Serial #
Zymark Turbovap	Turbovap II	May-96	TV9612N6726 TV9631N6975 TV9628N6939 TV9809R7994 TV0146N10597 TV0146N10596 TV0146N10598 TV0146N10595 TV1346N20168 TV1246N17453
Soxtherms	SOX416/SE416	Jan-05 Nov-16	4041427 4040014 4040019 4040018 SX2033 SX2050 1/846516004 1/8465160005 1/8465160006 1/8465160007
Turbovap II Biotage	Turbovap II	Feb-18	180600348 174600239 180200293 180600353 180600350 180300303

N-Evaps Organomation	115 1205	Jun-93 Jun-95	2812 6184 2038 11634
2- OHAUS Adventurer Toploading Balance	AX2202	July-2019	B843654719 B843654723
Sartorius AG Toploading Balance	LP8200P	N/A	14908834

METALS ANALYSIS

#	Equipment	Model #	Purchase Date	ID/Serial #
3	Perkin Elmer Mercury Analyzer	FIMS 100 FIMS 100 FIMS 100	Feb-14 Oct-18 Nov-19	101S14020102 101S18092701 101S19081301
1	AA WINLAB (Software)	6.5.0.0266	Feb-14	NA
2	Syngistix for AA (software)	Ver 3.1	10/18/2018 11/20/2019	NA
1	PS Analytical Atomic Fluorescence Mercury Analyzer	10.035	Aug-17	606
1	Millennium (Software)	NA	Aug-17	NA

METALS ANALYSIS

4	Perkin Elmer Inductively Coupled Plasma Mass Spectrometer	ELAN 9000 NexION 300 NexION 350 NexION 350X	Apr-10 May-14 Aug-14 Mar-18	AJ13141002 81VN4031301 85VN4061701 85XN7111002
4	Perkin Elmer ICPMS (Software)	2.4 SP3	Apr-10 May-14 Aug-14 Mar-18	NA
4	Perkin Elmer Inductively Coupled Plasma Spectrometer	7300DV 8300DV AVIO500 AVIO500	Mar-10 Apr-14 Feb-18 Jan-20	077C0022701 078S1403012 081S1711281 081S1911213
1	Winlab 32 (software)	Ver 3.1.0	Apr-19	NA
3	Syngistix (software)	Ver 3.0	Mar-10 Mar-18 Jan-20	NA
1	Thermo Orion 3Star	3Star	Dec-17	9731
1	Thermo Orion pH meter	420	Prior to 2008	065576
1	OHAUS Balance	AV313	Jan-14	B351136893
1	Sartorius Balance	CP22025	Prior to 2008	14509268

3	OHAUS Balance	AX423/E AX423/E AX2202	1/19/2019 1/19/2019 10/28/2019	B848841591 B843640835 B925840821
4	TCLP Tumblers	NA	Prior to 2008	T 101 T 104 T 105 T 106
3	Environmental Express HotBlock	SC100	Prior to 2008	Various units
11	Environmental Express HotBlock	SC154	Prior to 2008	Various units
2	Torrey Pines Scientific Hotplate	HP51	Prior to 2008	08301024 08301025
1	U.S. Filter Modulab Water System	M00100	Prior to 2008	LW2264
1	Barnstead NANOpure Diamond	D11901	Aug-02	1190030186870
1	Thermo Centrifuge	CL30	Apr-08	307070484

GENERAL CHEMISTRY

#	Equipment	Model #	Purchase Date	ID/Serial #
1	OI Analytical, TOC 1030S	OI1030S	Oct-15	A536733677
2	OI Analytical, TOC 1030W	OI1030W	Apr-15 Jan-16	P504730315 P550730559P

GENERAL CHEMISTRY

#	Equipment	Model #	Purchase Date	ID/Serial #
2	ATOC (software)		Apr-15	NA
2	Horizon Speed Vap II	9000 9000	Oct-01 Apr-02	01-337 01-340
2	Lachat QuikChem 8000	8000 series	Jul-01 Jul-02	A83000-1910 A83000-2077
1	Lachat QuikChem 8500	8500 series	Jan-06	60900000344
3	Omnion (software)	3.0.218 3.0.218 3.0.219	Jul-01 Jul-02 Jan-06	NA
2	ThermoSpectronic	20D+	Nov-03 Aug-06	3DUD255001 3DUJ199004
2	Mitsubishi Total Organic Halogen Analyzer	AOX-200	Jul-10 Mar-16	E7B00117 E7BA0376
2	Dionex Ion Chromatograph	ICS-3000	Apr-09 Apr-09	09030720 09030721
1	Dionex Ion Chromatograph	ICS-5000	Jul-10	10060501
3	Dionex Ion Chromatograph	Aquion	Jun-16 Aug-19 Mar-20	160540166 190640129 200240003
1	Dionex Ion Chromatograph	ICS-1600	Jul-14	14060002

GENERAL CHEMISTRY

#	Equipment	Model #	Purchase Date	ID/Serial #
7	Chromeleon (software)	7.2.1	Jul-14	NA
1	Turbidimeter	430T	Nov-19	19430781
1	Titrino Karl Fischer Moisture Analyzer	870KF	Mar-20	1870001031535
2	TKN Block Digestor	TKN100	Jul-16 May-17	2016TKNBC115 2017TKNBC133
2	Fried Electronics Stirring Hotplates	MH1-3x2	Jul-17	0568 0569
1	YSI Dissolved Oxygen Meter	5000	Apr-15	15D100827
1	IEC Clinical Centrifuge	Clinical	Prior to 2008	428-17189
1	Rapid Tester Setaflash	RT-00001	May-14	142271
1	VWR Oven	1370FM	Prior to 2008	101399
2	Yamato	DX602C DX600	3/20/2020 Prior to 2008	J1812255 A7700008
1	Vulcan Furnace	3-550PD	Apr-15	DKZ1316115V
2	HACH COD Reactor	95600-00	Jan-94	911005731C 9807000017919
1	Orion Conductivity Meter	A212	Dec-17	X37231

GENERAL CHEMISTRY

#	Equipment	Model #	Purchase Date	ID/Serial #
1	Parr 6200 Calorimeter	Parr 6200	Aug-14	M40303
6	Sartorius Balance	1872 BP2100S BA210S BA221S LC4800-P ED2200S	Prior to 2008	40245216 3410156 90710197 90606741 410010032 25150025
2	OHAUS Balance	PA 114 AX124	Jan-11 Apr-17	8331440032 B649420569
1	Brookfield Viscometer	LVDVE	Apr-05	E6515383
1	Beckman Centrifuge	TJ-6	Prior to 2008	4359
1	VWR Centrifuge	Clinical 200	Nov-11	68105001
5	Simple Cn Hotblocks	SC6002	Apr-09 Apr-09 Jan-09 Jan-09 Dec-18	5388DIS1012 5388DIS1016 5873DIS1030 5873DIS1036 2018DISW1225
2	BOD incubator	2020 818	Jan-99 Jan-10	10059509 26AW-9
1	Thermo Orion Star A111	A111	Sep-15	J10067
1	Thermo Orion Star A111	A111	Feb-14	J06078

1	Electronic Digital Caliper	62379-531	Dec-18	191986678
1	Setaflash Series 8 Active Cool	82100-2 U	Aug-19	1059732
2	MicroBlock R Distillation	EMD 1920-107	Nov-17 Dec-18	2262 2386

RADIOCHEMISTRY/BIOASSAY				
#	**Equipment**	**Model #**	**Purchase Date**	**ID/Serial #**
96	Canberra Alpha Spectrometers for Alpha Spectroscopy System (environmental)	7401	1992 to 1995	Various
156	Canberra Alpha Analyst Spectrometers with PIPS Detectors (environmental)	7200	1988 to 2009	Various
144	Canberra Alpha Analyst Spectrometer with PIPS Detectors (bioassay)	7200	1988-2009	Various
1	Perkin Elmer Automatic Gamma Counter	1480	Jun-05	4800440
1	Gamma Products G5400W Low background Alpha/Beta Counting System with 4 detectors	G5420-400T	Jan-17	121603

RADIOCHEMISTRY/BIOASSAY				
4	Compaq/DEC Alpha Work Stations for Alpha/Gamma Data Management System	500AU 500AU 500AU DS-10 DS-10	Nov-98 Nov-98 Jan-04 May-06 Mar-09	N188806229 4006DP9Z1060 AY93206555 AY30703843 (spare)
2	Protean Automatic Proportional Counter (Bioassay)	WPC 9550	Oct-2003 Jul-2004	EMC 0329438 924233
11	Protean Multi-Detector (40) Proportional Counter	MDS-16	Apr-02 Jul-2005 Oct-05 Mar-02	10751,10752,10753,10754 0525767,0525768 0531474,0531474 311437,311438, 0021910
4	Protean Multi-Detector (16) Proportional Counter	MDS-16	Feb-09	9115168, 169, 170,171
2	Tennelec LB-4100 Proportional Counter with 32 detectors	LB4100	Jun-93 Dec-98	18483 21938
1	Tennelec LB-4100 Proportional Counter with 16 detectors	LB4100	2010	70562
1	Gas Flow Proportional Counter with 4 detectors	G5420-400T	Jan-17	121603

RADIOCHEMISTRY/BIOASSAY				
8	Beckman Liquid Scintillation Counters	LS6000 LS6500 LS6500 LS6000 LS6500 LS6500 LS6000 LS6000	Jun-93 Jun-93 Apr-94 Mar-03 Oct-03 Dec-98 Dec-98 Jan-14	7065155 7067083 7067404 7060655 7070506 7069123 7060656 7069693
1	Perkin Elmer Inductively Coupled Plasma Mass Spectrometer	ELAN9000	Jun-10	AJ13351006
2	Perkin Elmer Liquid Scintillation Counter – Wallac Guardian (environmental)	1414	1997 2010	4140127 4140421
2	Protean Automatic Proportional Counter	WPC 4550		2910 1111
1	Perkin Elmer Liquid Scintillation Counter – Wallac Guardian (bioassay)	1414	1998	4140299
2	Perkin Elmer Quantulus	1220	1998 2009	2200082 DG06095168
2	Ortec – Alpha Spectrometers	alpha ensemble-8	2010	10235232 10230971
7	Ortec – Alpha Spectrometers	octete-pc	2010	177, 182, 217, 266, 264, 144, 176

RADIOCHEMISTRY/BIOASSAY				
#	**Equipment**	**Model #**	**Purchase Date**	**ID/Serial #**
1	Perkin Elmer Liquid Scintillation Counter – Rackbeta	1219	2010	206147
1	Perkin Elmer ICPMS	ELAN 9000	2010	AJ13271005
1	Broad-Energy Germanium Detector(Carbon Comp. Window)	BE3825	2006	3068173
1	High Purity Germanium Coaxial Detector	GEM90210-P	1990	30-TP30546-A
1	High Purity Germanium Coaxial Detector	GEM-35190	2004	CV-P122204CA
1	High Purity Germanium Coaxial Detector	GEM35	2007	CV-PO42407CA
1	High Purity Germanium Coaxial Detector	GEM35P4-83	2008	CV-TP011608CA
1	High Purity Germanium Well Detector	GCW3523	1994	3941466
1	Low Energy Germanium Detector (Beryllium Window)	GL1015	1988	488926
1	Low Energy Germanium Detector (Beryllium Window)	GL1010S	1990	10902649
1	Low Energy Germanium Detector (Beryllium Window)	GL2820R	1995	1954119
1	Low Energy Germanium Detector (Beryllium Window)	GL2820R	1998	3984452

RADIOCHEMISTRY/BIOASSAY				
#	Equipment	Model #	Purchase Date	ID/Serial #
1	Low Energy Germanium Detector (Beryllium Window)	GL2020R	2007	9078304
1	Low Energy Germanium Detector (Carbon Comp. Window)	GL2020-S	1992	12922782
1	N-Type High Purity Germanium Coaxial Detector	GMX 45225-P-S	1990	37-TN11260A
1	N-Type High Purity Germanium Coaxial Detector	GMX30200-P	1990	30-TN10348
1	N-Type High Purity Germanium Coaxial Detector	NIG3019	1991	PGT2461
1	P-Type High Purity Germanium Coaxial Detector (Bioassay)	IGC3919	1993	2605
1	Reverse-Electrode Coaxial Germanium Detector (Beryllium Window)	GR3019	1986	9861606
1	Reverse-Electrode Coaxial Germanium Detector (Beryllium Window)	GR2020	1991	1912509
1	Reverse-Electrode Coaxial Germanium Detector (Carbon Comp. Window)	GR3520	1993	8932581
1	Reverse Electrode Coaxial Germanium Detector	GR3021	1992	3922553

RADIOCHEMISTRY/BIOASSAY				
#	**Equipment**	**Model #**	**Purchase Date**	**ID/Serial #**
1	Reverse Electrode Coaxial Germanium Detector (Beryllium Window)	GR4019	1996	1966073
2	Standard Electrode Coaxial Germanium Detector	GC3519	1991	9912854, 11912876
2	Standard Electrode Coaxial Germanium Detector	GC3520	1992 2000	12922955 2007152
4	Standard Electrode Coaxial Germanium Detector	GC2018	1992	9923035 9923043 10923049 10923050
1	Standard Electrode Coaxial Germanium Detector	GC3018	1993	5933088
1	Standard Electrode Coaxial Germanium Detector	GC3519	1994	1943234
1	Standard Electrode Coaxial Germanium Detector	GC8021	1994	8943324
1	Standard Electrode Coaxial Germanium Detector (Bioassay)	GC3519	1994	1943199

RADIOCHEMISTRY/BIOASSAY				
1	Standard Electrode Coaxial Germanium Detector	GC3519	1992 2005	3922907 7059000
8	Standard Electrode Coaxial Germanium Detector	GC4019	1995 2001 2006 2007	6953489 6953483 6953542 10017452 10017444 9069163 9069175 10079344
3	Standard Electrode Coaxial Germanium Detector	GC4020	2005 2006	10059017 10059015 4069118
4	Standard Electrode Coaxial Germanium Detector	GC4520	2009	4099544 4099570 10099624 11099639
1	N-Type High Purity Germanium Coaxial Detector	GMX35195-P-S	1991	34-TN-20891A

RADIOCHEMISTRY/BIOASSAY				
8	Ludlum Alpha Scintillation Detector	Ludlum-182	2007 Mar-17 Jul-17 Jan-17	PR086493 PR140731 PR101846 PR078964 PR364855 PR139590 PR286612 PR286613
1	Perkin Elmer Automatic Gamma Counter	Model 2480	Oct-17	DG12095812
1	Sartorius Balance	A200S		38080204
1	Sartorius Balance	CP2201		18150253
2	Sartorius Balance	CP323S		18550299 15750050
1	Sartorius Balance	CP 2202S		17955156
1	Sartorius Balance	HD 2000 D		39020004
2	Sartorius Balance	I 12000 S		40109033 39039003
1	Sartorius Balance	L2200S		38110007
1	Sartorius Balance	BP3100S		51204863

RADIOCHEMISTRY/BIOASSAY				
1	Sartorius Balance	U5000D		36080009
1	Sartorius Balance	R 300S		38110047
1	Sartoris Balance	LC6200S		30503875
1	Sartoris Balance	LC3201D		60108592
1	Sartoris Balance	TE313S		16107662
1	Sartoris Balance	ENTRIS5201		34104035
3	Sartoris Entirs Balance	ENTRIS5201-1S		35602249 35602240 33005595
1	Sartoris Entris Balance	ENTRIS224-15		33604148
1	Sartoris Entris Balance	ENTRIS52202-1S		33010896
1	Mettler Analytical Balance	AE160		C31514
1	Mettler Analytical Balance	AE163		F33394

RADIOCHEMISTRY/BIOASSAY				
2	Mettler Analytical Balance	AE200		F30560 1113021018
1	Mettler Analytical Balance	AE240		L28658
1	Mettler Analytical Balance	AE50		1113092273
1	Mettler Analytical Balance	PM16-N		N39169
1	Mettler Analytical Balance	PM 4600		J93763
1	OHAUS Toploader Balance	RD6RM		2525244
2	Mettler Analytical Balance	Model AT261	Jan-05	M64061

HIGH RAD ALIQUOT ROOM				
#	**Equipment**	**Model #**	**Purchase Date**	**ID/Serial #**
1	Adventurer Pro	AV2102	Oct-14	B440101411

LABORATORY INFORMATION MANAGEMENT SYSTEMS

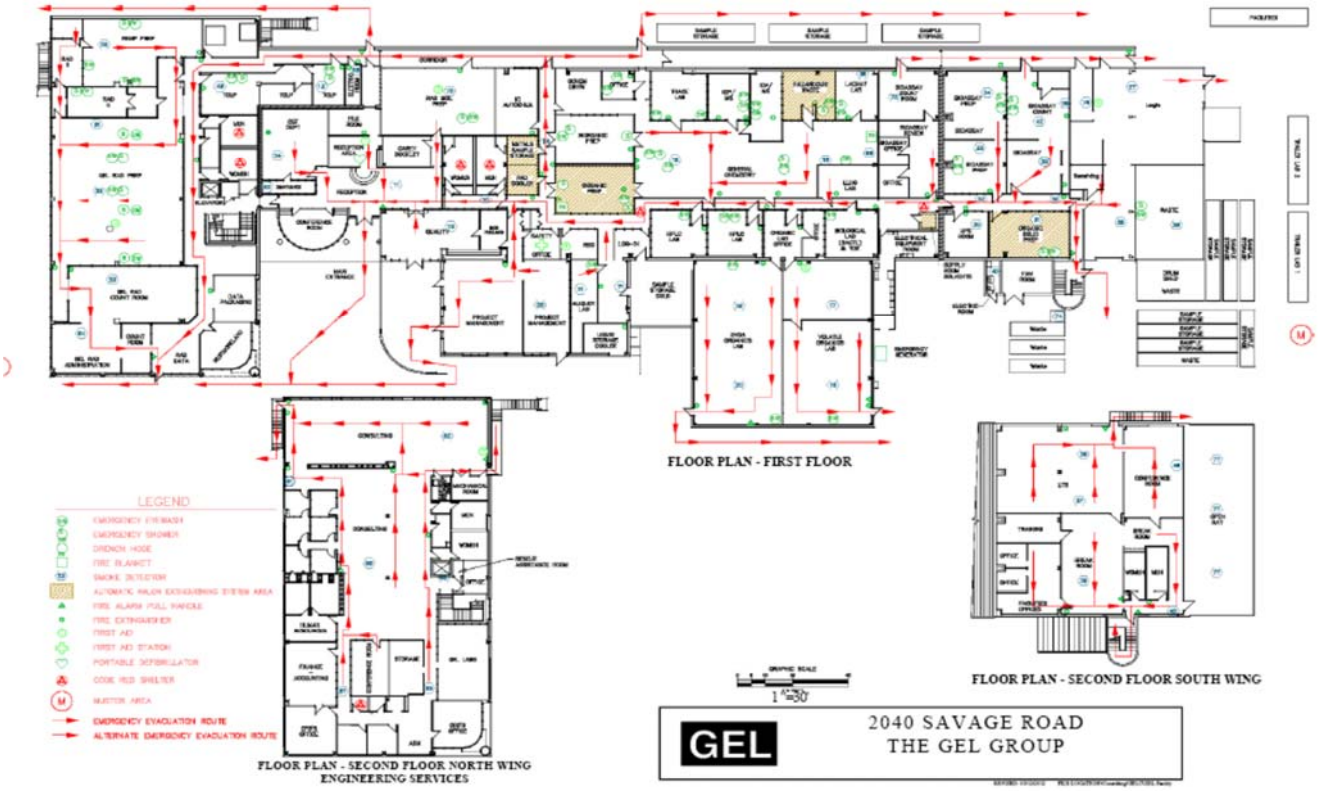
#	Equipment	Model #	Purchase Date	ID/Serial #
1	DELL Poweredge 2950 2 X 3.0Ghz 2GB ram	2950	2007	DG2CNB1
1	HP9000 Dclass, HP-UX 10.20, 2 cpu, 256 MB RAM, (hpclp1) 50GB Disk (mirrored and RAID%), Raid tower, 100 Mbps Eth card, Target Software	N/A	Nov-97	A3480A
1	HP9000 Dclass, HP-UX 10.20, 2 cpu, 256 MB RAM, (104ilroy) 50GB Disk (mirrored and RAID5), Raid tower, 100 Mbps Eth card, Target Software	N/A	Nov-97	A3480A
1	HP9000 Dclass, HP-UX 10.20, 2 cpu, 256 MB RAM, (prdsvr07) 50GB Disk (mirrored and RAID5), Raid tower, Target Software	N/A	Nov-97	A3480A
1	Sun V890 (prodsvr01) 8X1.5Ghz) 128GB ram (mirrored and raid5)	V890	2007	0529AM019F
1	Sun V890(standbysvr01) 4X1.35Ghz 32GB (mirrored and rad5)	V890	2008	0526AM02F

1	HP-Prolient DL380 Gen9 (vmhost01) 2-10QuadCoreX2.40GHz 352GB	DL380	2009	MXQ6290GNC
1	HP-Prolient DL380 Gen9 (vmhost02) 2-10QuadCoreX2.40GHz 352GB	DL380	2009	MXQ6290GNH
1	HP-Prolient DL380 Gen9 (vmhost03) 2-QuadCoreX2.83GHz 352GB	DL362	2009	MXQ6290GP5
1	HP2012i (san01) DC Modular Smart Array	2012i	2009	3CL904C108
1	HPE Nimble Hybrid Storage Array	HF40	2019	5UM9390073
1	EMC Storage Array Network (SAN)	VNX5200	Jan-2015	APM00145036951

UNIVERSAL POWER SUPPLY

#	Equipment	Model #	Purchase Date	ID/Serial #
1	Mitsubishi	9900B	~10/10/17	16-7M85443-01

APPENDIX H: FACILITIES WITH EVACUATION ROUTES



APPENDIX I: STANDARD OPERATING PROCEDURES AND ANALYTICAL METHODS

Standard Operating Procedures and Analytical Methods

SOP #	SOP Title	Methods
GL-ADM-E-001	Preparation, Authorization, Advance Change, Revision, Release, and Retirement of Standard Operating Procedures	N/A
GL-AP-E-001	Invoicing Analytical Lab Numbers	N/A
GL-CO-E-001	Revising GEL Laboratories Catalog of Analytical Services	N/A
GL-CO-E-002	Delegated Authority to Commit the Company	N/A
GL-CO-E-003	Request for Proposal (RFP) and Contract Review	N/A
GL-CS-E-002	Internal Review of Contractually Required Quality Criteria for Client Package Delivery	N/A
GL-CS-E-005	Electronic Data Deliverables	N/A
GL-CS-E-006	Subcontracting Analytical Services	N/A
GL-CS-E-008	Prelogin, Login, and Login Review	N/A
GL-CS-M-001	Project Management AlphaLIMS Manual	N/A
GL-DC-E-001	Document Control	N/A
GL-FC-E-001	Facility Security	N/A
GL-FC-E-002	Testing Emergency Eyewash and Shower Equipment	N/A
GL-FC-E-003	Local Exhaust Ventilation Systems	N/A
GL-FC-E-004	Inspection of Fire Extinguishers	N/A
GL-GC-E-001	Total Dissolved Solids	EPA 160.1, 2540C
GL-GC-E-004	General Chemistry Standards, Definitions, and Preparation	N/A
GL-GC-E-007	Total Organic Halogen (TOX) and Adsorbable Organic Halides on Liquid Samples Using the Mitsubishi AOX-200 Analyzer	1650C, 9020B
GL-GC-E-008	pH	EPA 150.1, 9040B/9040C, 9041A, 9045C/9045D 4500-H^+ -00
GL-GC-E-009	Conductivity and Salinity	EPA 120.1, 9050A, SM 2510B-97, SM 2520B-10
GL-GC-E-010	Paint Filter Test	EPA 9095A/9095B
GL-GC-E-011	Total Solids	2540B, 2540G-2011
GL-GC-E-012	Total Suspended Solids	SM 2540D
GL-GC-E-028	Carbonaceous Biochemical Oxygen Demand (CBOD)	EPA 405.1, 5210B-01
GL-GC-E-029	Corrosivity Toward Steel	1110(M), 1110A(M)
GL-GC-E-032	Carbon Dioxide (Total and Free) by Calculation	4500-CO_2 D
GL-GC-E-033	Alkalinity: Total, Bicarbonate, Carbonate, Hydroxide, and Phenolphthalein	EPA 310.1(M), 2320B-97
GL-GC-E-035	Volatile Suspended Solids	EPA 160.4, 2540E
GL-GC-E-036	Color by Visual Comparison	2120B

Standard Operating Procedures and Analytical Methods		
SOP #	**SOP Title**	**Methods**
GL-GC-E-037	Turbidity	180.1, 2130-B
GL-GC-E-040	Pretreatment of Cyanide Amenable to Chlorination	EPA 335.1, 9010B/9010C, 9012A/9012B 4500-CN^- G-99
GL-GC-E-044	Colorimetric Determination of Hexavalent Chromium	7196A, 3500-Cr D, 3060A
GL-GC-E-045	Biochemical Oxygen Demand (BOD)	EPA 405.1, 5210B
GL-GC-E-047	Methylene Blue Active Substance	EPA 425.1, 5540C
GL-GC-E-048	Heating Value Determination by Bomb Calorimeter	ASTM D 240-14, 4809-13, E 711-87(M)
GL-GC-E-052	Sulfide (Methylene Blue Method)	EPA 376.2(M), HACH 8131, 4500 S^{2-} D
GL-GC-E-056	Sulfite	4500-SO_3^{2-} B-2000, EPA 377.1
GL-GC-E-057	Volatile Solids and % Ash Procedure for Water Samples	EPA 160.4, 2540E
GL-GC-E-058	Volatile Solids and % Ash Procedure for Solid and Semisolid Samples	2540G
GL-GC-E-059	Dissolved Oxygen Analysis by Membrane Electrode Method	4500-O^- G, EPA 360.1
GL-GC-E-061	Chemical Oxygen Demand (COD) Digestion Reactor Method	EPA 410.4, 5220-D, HACH 8000
GL-GC-E-062	Total Carbon and Total Organic Carbon Analysis Using the OI Analytical 1030S TOC Solids Module	9060 (M) 9060A (M), 5310-B
GL-GC-E-064	Density	ASTM D5057
GL-GC-E-065	Specific Gravity	ASTM D5057
GL-GC-E-066	Flashpoint by Setaflash	1020, ASTM D 3278-78
GL-GC-E-067	Cyanide Sample Distillation	9012, 9010 335.3, 335.4, 335.2-M, 4500-CN^- C
GL-GC-E-068	Viscosity	ASTM D2161 (Mod), ASTM D2983 (Mod), Brookfield Viscometer
GL-GC-E-069	Reactive Cyanide and Sulfide	SW-846 Chap 7.3.3, Chap 7.3.4
GL-GC-E-071	Total Phosphorous and Total Kjeldahl Nitrogen Sample Preparation	EPA 365.4, 351.2, 4500N_{org}-D-2011
GL-GC-E-072	Ammonia-Nitrogen Sample Preparation	EPA 350.1, 4500-NH_3^- B
GL-GC-E-073	Free Cyanide Analysis by Microdiffusion	ASTM D 4282
GL-GC-E-074	Extractable Organic Halides (EOX)	SW-846 9023
GL-GC-E-076	Total Residue Chlorine	4500-Cl G
GL-GC-E-077	Cyanide Weak Acid Dissociable Sample Preparation and Analysis	EPA 335.4, 4500-CN^-I
GL-GC-E-079	Bomb Preparation Method for Solid Waste	5050
GL-GC-E-082	Acid-Soluble Sulfides	9030B, 9034
GL-GC-E-086	Ion Chromatography (IC)	EPA 300.0, 9056

Standard Operating Procedures and Analytical Methods

SOP #	SOP Title	Methods
GL-GC-E-087	Percent Water by Karl Fischer Titration	ASTM E203-08
GL-GC-E-090	Acidity	2310B
GL-GC-E-091	Wavelength Calibration Verification of Thermospectronic Spectrophotometers	N/A
GL-GC-E-092	General Chemistry Data Review and Packaging	N/A
GL-GC-E-093	Total, Total Inorganic and Total Organic Carbon (TOC) using the OI Analytical Model 1010 TOC Analyzer	EPA 415.1, 9060, 9060A 5310B-2011
GL-GC-E-094	N-Hexane Extractable Material (HEM; Oil and Grease) and Silica GEL Treated N-Hexane Extractable Material (SGT-HEM Non-Polar Material) in Aqueous Matrices	1664, 1664B
GL-GC-E-095	Cyanide Analysis by Lachat QuikChem 8000 FIA	CLP 335.2-M, 335.3. 335.4, 9010, 9012, 4500-CN^- E
GL-GC-E-096	Perchlorate by Ion Chromatography (IC)	EPA 314.0
GL-GC-E-100	Total Hardness by Titration	SM 2340C-97
GL-GC-E-102	Total Recoverable Phenol by the Lachat QuikChem FIA+ 8000 Series	EPA 420.4, 9066
GL-GC-E-103	Total Phosphorus by the Lachat Quickchem FIA+ 8000 Series Instrument	EPA 365.4, 4500 P H
GL-GC-E-104	Total Kjeldahl Nitrogen (TKN) Using the Lachat QuikChem FIA+ 8000 Series Instrument	EPA 351.2, 4500 N_{org} D
GL-GC-E-106	Ammonia Determination by the Lachat Quickchem FIA + 8000 Series	EPA 350.1 Rev 2, 4500-NH_3 H
GL-GC-E-107	Inorganic Calculations	N/A
GL-GC-E-123	Column Settling	EM 1110-02-5027
GL-GC-E-127	Modified Elutriate Test	N/A
GL-GC-E-128	Nitrate/Nitrite (NO_3+NO_2) Analysis Using The Lachat QuickChem FIA + 8000 Series Instrument	EPA 353.2, 4500-NO_3^-
GL-GC-E-130	Percent Ash Determined at 775 C Procedure for Solid and Semisolid Samples	ASTM D 482-03 (M)
GL-GC-E-132	Hexavalent Chromium Analysis Using the Lachat Quikchem FIA +8000 Series Instrument	SM 3500-Cr B, SW-846 7196A
GL-HR-E-002	Employee Training	N/A
GL-IT-E-001	Information Technology Program for Good Laboratory and Good Manufacturing Practices	N/A
GL-IT-E-002	Computer Systems Team Roles and Responsibilities	N/A
GL-IT-E-003	Requirements, Design, Operation, Validation and Removal of Hardware and Software Systems Used by the GEL Group, Inc.	N/A
GL-IT-E-004	Change Control Requirements for Hardware and Software	N/A
GL-IT-E-005	Requirements, Design, Operation, Validation and Removal of Applications Used by The GEL Group, Inc.	N/A

Standard Operating Procedures and Analytical Methods

SOP #	SOP Title	Methods
GL-IT-E-006	Change Control Requirements for Applications	N/A
GL-IT-E-007	User Roles and Responsibilities for Personnel Using Computer Services	N/A
GL-IT-E-009	Archive and Retrieval of Systems Information	N/A
GL-IT-E-010	Backup of Computer Controlled Instrumentation	N/A
GL-IT-E-011	System Security and Virus Protection	N/A
GL-IT-E-012	Application Tools used by Computer Services Personnel	N/A
GL-IT-E-013	GEL Electronic Processes and LIMS Audit System	N/A
GL-IT-E-014	Disaster Recovery	N/A
GL-IT-E-015	Operation of LIMS Database Primary and Failover Servers	N/A
GL-LB-E-001	The Determination of Method Detection Limits and Method Quantitation Limits	N/A
GL-LB-E-002	Balances	N/A
GL-LB-E-003	Glassware Preparation	N/A
GL-LB-E-004	Temperature Monitoring and Documentation Requirements for Refrigerators, Ovens, Incubators, and Other Similar Devices	N/A
GL-LB-E-005	Data Review and Validation	N/A
GL-LB-E-006	Toxicity Characteristic Leaching Procedure Preparation	SW-846 1311
GL-LB-E-007	Laboratory Standards Documentation	N/A
GL-LB-E-008	Basic Requirements for the Use and Maintenance of Laboratory Notebooks, Logbooks, Forms and Other Recordkeeping Devices	N/A
GL-LB-E-009	Run Logs	N/A
GL-LB-E-010	Maintenance and Use of Air Displacement Pipets	N/A
GL-LB-E-012	Verifying the Maintenance of Sample Integrity	N/A
GL-LB-E-013	CLP-Like/DOE Data Package Assembly and Revision	N/A
GL-LB-E-016	The Collection and Monitoring of the DI Water Systems	N/A
GL-LB-E-017	Procedure and Policy for Manual Integration	N/A
GL-LB-E-018	Instrument Clock Verification	N/A
GL-LB-E-020	Tuning of High Intensity Ultrasonic Processor	N/A
GL-LB-E-023	Waste Extraction Test (WET)	N/A
GL-LB-E-024	Synthetic Precipitation Leaching Preparation	EPA 1312
GL-LB-E-026	Container Suitability Testing	N/A
GL-LB-E-027	Bioassay Kit Delivery and Retrieval	N/A
GL-LB-E-029	Laboratory Sub-Sampling	N/A
GL-LB-E-030	Silica Gel and Air Filter Removal and Replacement	N/A
GL-LB-E-031	Sample Compositing	N/A
GL-LB-E-032	The Distribution of High Risk and Limited Volume Samples	N/A

Standard Operating Procedures and Analytical Methods

SOP #	SOP Title	Methods
GL-LB-E-033	Proper Peak Identification for Organics	N/A
GL-LB-E-034	Laboratory Filtration Samples	N/A
GL-LB-G-001	Laboratory Waste Management Plan	N/A
GL-LB-N-001	Safety, Health and Chemical Hygiene Plan	N/A
GL-LB-S-001	Disaster Preparedness and Recovery Plan	N/A
GL-LB-E-X-001	Facility Closure Plan	N/A
GL-MA-E-006	Acid Digestion of Total Recoverable or Dissolved Metals in Surface and Groundwater Samples for Analysis by ICP or ICP-MS	3005A
GL-MA-E-008	Acid Digestion of Total Metals in Aqueous Samples and Extracts for Analysis by ICP and ICP-MS	3010A
GL-MA-E-009	Acid Digestion of Sediments, Sludges, and Soils	3050B, 6010, 6020
GL-MA-E-010	Mercury Analysis Using the Perkin Elmer Automated Mercury Analyzer	245.1, 245.2, 7470A, 7471A, 7471B
GL-MA-E-013	Determination of Metals by ICP	EPA 200.7, 6010B, 6010C, 6010D
GL-MA-E-014	Determination of Metals by ICP-MS	6020, 6020A, 6020B, EPA 200.8,
GL-MA-E-016	Sample Preparation for Total Recoverable Elements by EPA Method 200.2	EPA 200.2
GL-MA-E-017	Metals Data Validation	N/A
GL-MA-E-018	Mercury Analysis using the PS Analytical Millennium Automated Mercury Analyzer	EPA 1631 Rev E
GL-MA-E-020	Acid Digestion of Personal Cassette Filters for Analysis by ICP	NIOSH 7303
GL-OA-E-001	Establishing Retention Time Windows for GC and HPLC Analysis	SW-846 8000
GL-OA-E-003	Non-Volatile Total Petroleum Hydrocarbons by Flame Ionization Detector	8000, 8015, 3541, 3580
GL-OA-E-004	Volatile Total Petroleum Hydrocarbons by Flame Ionization Detector	5030, 5035, 8000, 8015
GL-OA-E-009	Analysis of Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry	8270, EPA 625.1
GL-OA-E-010	Extraction of Semivolatile and Nonvolatile Organic Compounds from Soil, Sludge, and Other Miscellaneous Solid Samples	3500, 3550, 8270, 8081, 8082, 8015, 8310
GL-OA-E-011	Analysis of Chlorophenoxy Acid Herbicides by ECD	8000, 8151A
GL-OA-E-013	Extraction of Semivolatile and Nonvolatile Organic Compounds from Groundwater, Wastewater, and Other Aqueous Samples	3510, 8270, 8081, 8082, 8015 8310, 608.3, 625.1, AK102, AK103
GL-OA-E-015	The Extraction of Herbicides from Groundwater, Wastewater, and Other Aqueous Samples	8151

Standard Operating Procedures and Analytical Methods		
SOP #	**SOP Title**	**Methods**
GL-OA-E-020	Percent Moisture	ASTM D2216-05
GL-OA-E-022	Volatile Organic Compounds by Gas Chromatograph/Mass Spectrometer Applicable to EPA Method 524.2	EPA 524.2
GL-OA-E-026	Volatile Organic Compounds (VOC) by Gas Chromatograph/Mass Spectrometer	EPA 624.1
GL-OA-E-027	The Extraction of Herbicides from Soil and Sludge Samples	8151
GL-OA-E-030	Polynuclear Aromatic Hydrocarbons	8310
GL-OA-E-033	Nitroaromatics and Nitramines by High Performance Liquid Chromatography (HPLC)	8000, 8330A, 3535
GL-OA-E-036	Florisil Cleanup of Organochlorine Pesticide Solvent Extracts	3510, 3620, 3550, 8081,
GL-OA-E-037	Sulfuric Acid/Permanganate Cleanup of PCB Solvent Extract	3550C, 3665A, 8082,
GL-OA-E-038	Volatile Organic Compounds (VOC) by Gas Chromatograph/Mass Spectrometer	8260, 5030, 5035, 8000, 3585, SM 6200
GL-OA-E-039	Closed-System Purge-and-Trap Collection and Extraction Volatile Organics in Soil and Waste Samples	EPA 5035, 3585
GL-OA-E-040	Polychlorinated Biphenyls	8000, 8082, 608.3
GL-OA-E-041	Organochlorine Pesticides and Chlorinated Hydrocarbons	8000, 8081, 608.3
GL-OA-E-044	Organics Validation	N/A
GL-OA-E-045	Sulfur Clean-up	3660B
GL-OA-E-046	Common Industrial Solvents, Glycols, and Various Organic Compounds by Flame Ionization Detector	8000, 8015
GL-OA-E-047	Gel Permeation Cleanup of Solvent Extracts	3640A, 3510C, 3550C, 8270, 8081, 8082
GL-OA-E-049	Silica Gel Cleanup Using Solid Phase Silica Gel Extraction Cartridges	3550C, 3510C, 3630C, 3541
GL-OA-E-050	The Extraction of Semi-Volatile and Nonvolatile Organic Compounds from Oil	3580, 8015, 8081, 8082, 8081, 8270
GL-OA-E-054	The Determination of Gasoline Range Organics Using Flame Ionization Detection Per Alaska Method AK101	AK101
GL-OA-E-055	The Determination of Diesel Range and Residual Range Organics	AK102. AK 103, 3510C, 3550B
GL-OA-E-056	Definitive Low Level Analysis of Nitroaromatic Explosives Utilizing Liquid Chromatography/Mass Spectrometry/Mass Spectrometry (LC/MS/MS) by SW-846 Method 8321 Modified (8321M)	8321A(M), 8000, 8330(M), , 8330B(M)
GL-OA-E-058	Volatile Storage Blanks	N/A
GL-OA-E-059	Analysis of 1,2-Dibromoethane (EDB) and 1,2-Dibromo-3-Chloropropane (DBCP) in Water by GC/ECD Using Methods 504.1 or 8011	EPA 504.1, 8011
GL-OA-E-061	Haloacetic Acids in Water	EPA 552.2

Standard Operating Procedures and Analytical Methods

SOP #	SOP Title	Methods
GL-OA-E-063	Massachusetts Method for the Determination of Extractable Petroleum Hydrocarbons	Massachusetts EPH
GL-OA-E-064	Dissolved Gases in Water by Flame Ionization Detector (FID)	RSK-175
GL-OA-E-065	Reagent/Solvent/Standards Screening for Organic Prep	N/A
GL-OA-E-066	Automated Soxhlet Extraction	EPA 3541,
GL-OA-E-067	Definitive Low Level Perchlorate Analysis Utilizing Liquid Chromatography/Mass Spectrometry/Mass Spectrometry (LC/MS/MS) by EPA Method 6850 Modified (6850M)	6850(M), 8000
GL-OA-E-068	The Processing, Extraction, and Analysis of Nitroaromatics, Nitroamines, and Nitrate Esters by SW-846 8330B	8330B, 3535
GL-OA-E-070	Solid-Phase Extraction	EPA 3535
GL-OA-E-071	The Pre-Extraction Processing of Soil Samples Collected Using Multi-Incremental Sampling (MIS) Techniques	EPA 8330B
GL-OA-E-073	Analysis of 1,4-Dioxane in Drinking Water by Solid Phase Extraction (SPE) and Gas Chromatography/Mass Spectrometry	EPA 522
GL-OA-E-074	Massachusetts Volatile Petroleum Hydrocarbons by Photoionization and Flame Ionization Detectors	N/A
GL-OA-E-075	Washington Method for the Determination of Extractable Hydrocarbons	WA EPH
GL-OA-E-076	The Extraction and Analysis of Per and Polyfluroalkyl Substances Using LCMSMS	DOD QSM Table B-15 V., 5.3; ASTM D79698-17A; 537 Version, 1.1
GL-OA-E-078	The Extraction and Analysis of Cannabinoids by QuEChERS and High Performance Liquid Chromatography	GEL Developed Method
GL-OA-E-079	The Extraction of Herbicides using Solid Phase Extraction	GEL Developed Method
GL-OA-E-080	The Analysis of Naphthalene Sulfonate Using High Performance Liquid Chromatography	GEL Developed Method
GL-QS-B-001	Quality Assurance Plan	N/A
GL-QS-E-001	Conduct of Quality Audits	N/A
GL-QS-E-002	Conducting Corrective/Preventive Action and Identifying Opportunities for Improvement	N/A
GL-QS-E-003	Training and Qualifying Quality Assurance Audit Personnel	N/A
GL-QS-E-004	AlphaLIMS Documentation of Nonconformance Reporting and Dispositioning and Control of Nonconforming Items	N/A
GL-QS-E-005	Review of Monitoring Device Logs	N/A
GL-QS-E-007	Thermometer Verification	N/A

Standard Operating Procedures and Analytical Methods		
SOP #	**SOP Title**	**Methods**
GL-QS-E-008	Quality Records Management and Disposition	N/A
GL-QS-E-011	Method Validation and Initial and Continuing Demonstrations of Capability	N/A
GL-QS-E-012	Client NCR Database Operation	N/A
GL-QS-E-013	Handling of Proficiency Evaluation Samples	N/A
GL-QS-E-014	Quality Assurance Measurement Calculations and Processes	N/A
GL-QS-E-015	Use of Logos and Describing Accredited Status	N/A
GL-QS-E-016	Identification and Implementation of New and Revised Methods	N/A
GL-QS-E-017	Maintaining Technical Training Records	N/A
GL-QS-E-018	Communication of Substantial Nonconforming Safety Related Services	N/A
GL-QS-E-019	Trending of Performance Evaluation Data	N/A
GL-RAD-A-001	The Determination of Gross Alpha And Gross Non-Volatile Beta in Water	900.0, 9310
GL-RAD-A-001B	The Determination of Gross Alpha And Gross Non-Volatile Beta in Soil, Filters, Solid Matrices and Direct Count Air Filters	900.0(M), 9310
GL-RAD-A-001C	The Determination of Gross Alpha in Water by Co-precipitation	520/5-84-006 Method 00-02
Gl-RAD-A-001D	The Determination of Gross Alpha Gross Non-Volatile Beta in Drinking Water	600/4-80-032 Method 900.0
GL-RAD-A-002	The Determination of Tritium	600/4-80-032, 906.0(M)
GL-RAD-A-003	The Determination of Carbon-14 in Water, Soil, Vegetation and Other Solid Matrices	N/A
GL-RAD-A-004	The Determination of Strontium 89/90 in Water, Soil, Milk, Filters, Vegetation and Tissues	905.0(M), DOE RP501 Rev1(M), HASL 300(M)
GL-RAD-A-005	The Determination of Technitium-99 Using ICP-MS	HASL 300(M) TC-02-RC, DOE RP550(M), ASTM C 1387-03(M), ASTM 1476-00(M)
GL-RAD-A-006	The Determination of Radiometric Iodine	901.1(M), HASL 300(M) I-01
GL-RAD-A-007	The Determination of Radon-222 in Water	SM 7500 Rn-B
GL-RAD-A-008	The Determination of Radium-226	903.1(M), HASL 300(M) Ra-04-RC
GL-RAD-A-009	The Determination of Radium-228 in Water and Solids	904.0(M)
GL-RAD-A-010	Total Alpha Radium Isotopes in Soil and Water	900.1(M)
GL-RAD-A-011	The Isotopic Determination of Americium, Curium, Plutonium, and Uranium	DOE RP800 1997(M), HASL-300 U-02-RC(M), HASL-300 Am-05-RC(M) HASL-300 Pu-11-RC(M)
GL-RAD-A-013	The Determination of Gamma Isotopes	901.1 (M), HASL-300 (M) Sec. 4.5.2.3, HASL-300 Ga-01-R
GL-RAD-A-015	Digestion for Soil	N/A

Standard Operating Procedures and Analytical Methods

SOP #	SOP Title	Methods
GL-RAD-A-016	The Determination of Radiometric Polonium	EPA 600/4-80-032
GL-RAD-A-017	The Determination of Iodine-131 in Drinking Water	902.0, 7500 I- B
GL-RAD-A-018	The Determination of Lead-210 in Liquid and Solid Matrices	N/A
GL-RAD-A-019	Determination of Phosphorus-32 in Soil and Water	N/A
GL-RAD-A-020	The Determination of Promethium-147 in Soil and Water	N/A
GL-RAD-A-021	Soil Sample Preparation for the Determination of Radionuclides	N/A
GL-RAD-A-021B	Soil Sample Ashing for the Determination of Radionuclides	N/A
GL-RAD-A-022	The Determination of Ni-59 and Ni-63	N/A
GL-RAD-A-026	The Preparation of Special Matrices for the Determination of Radionuclides	N/A
GL-RAD-A-028	Radium-226 in Drinking Water by EPA Method 903.1	EPA 903.1
GL-RAD-A-029	The Determination of Strontium-89/90 in Drinking Water by EPA Method 905.0	EPA 905.0
GL-RAD-A-030	Determination of Radium-228 in Drinking Water	904.0, 9320
GL-RAD-A-031	The Determination of Selenium	N/A
GL-RAD-A-032	The Isotopic Determination of Neptunium/Thorium	N/A
GL-RAD-A-033	Determination of Chlorine-36 in Solid and Liquid Samples	N/A
GL-RAD-A-035	The Isotopic Determination of Plutonium-241	HASL-300 Pu-11-RC(M)
GL-RAD-A-036	The Isotopic Determination of Americium, Curium, and Plutonium in Large Soil Samples	DOE RP800(M) HASL-300 Am-05-RC(M) HASL-300 Pu-11-RC(M) HASL-300 Pu-12-RC(M)
GL-RAD-A-037	Radium-226 and Radium-228 in Drinking Water by Sulfate Precipitation and Gamma-Ray Spectrometry	N/A
GL-RAD-A-038	The Isotopic Determination of Thorium	DOE RP800(M), HASL-300(M) Pu-02-RC, Pu-03-RC
GL-RAD-A-040	The Determination of Fe-55 in Liquid and Solid Matrices by Liquid Scintillation Counter	N/A
GL-RAD-A-041	The Determination of Total Activity in Solids and Liquids	N/A
GL-RAD-A-044	Total Alpha Radium Isotopes In Drinking Water	903.0, 9315, HASL 300(M)
GL-RAD-A-046	The Determination of Radium-224 and Radium-226 by Alpha Spectroscopy	N/A
GL-RAD-A-047	48 Hour Rapid Gross Alpha Test	ECLS-R-G-A, EPA 600/4-80-032, 900.0(M)
GL-RAD-A-048	The Determination of Calcium-45 in Soils and Waters	N/A
GL-RAD-A-049	The Determination of Sulfur-35	NAS-NS-3054
GL-RAD-A-050	The Determination of Tritium in Drinking Water Samples	600/4-80-032, 906.0
GL-RAD-A-051	The Rapid Determination of Strontium 89/90 by Cerenkov Counting	N/A

Standard Operating Procedures and Analytical Methods		
SOP #	**SOP Title**	**Methods**
GL-RAD-A-052	The Determination of Organically Bound Tritium	600\4-80-032, 906.0
GL-RAD-A-053	Isotopic Determination of Plutonium in Large Water Resin Samples	HASL 300 Pu-11-RC
GL-RAD-A-054	The Determination of Strontium-90 in Brine	N/A
GL-RAD-A-055	The Preparation of Environmental Samples for Isotopic Uranium Analysis Via ICP-MS	N/A
GL-RAD-A-056	The Determination of Gross Alpha and Beta by Liquid Scintillation Counter	N/A
GL-RAD-A-058	The Rapid Determination of Strontium 89/90 by Gas Flow Proportional Counting	N/A
GL-RAD-A-059	The Determination of Technetium-99 Using Analytical Grade 1X8 Resin	N/A
GL-RAD-A-060	The Preparation of Vegetation and Filter Samples Via Organic Destruction and Strong Acid Leach for Radiochemistry Metals Analysis	N/A
GL-RAD-A-063	The Determination of Radium-228 Using DGA Cartridges	N/A
GL-RAD-A-064	The Determination of Fe-55 in Liquid and Solid Matrices Using DGA Resin	N/A
GL-RAD-A-065	The Determination of Carbon-14 in Atmospheric Screening Cartridges	N/A
GL-RAD-A-066	The Determination of Radiometric Polonium Using DGA Cartridges	N/A
GL-RAD-A-067	The Determination of Radiometric of Tritium and Carbon 14 in Combustible Materials Using Pyrolysis	N/A
GL-RAD-A-068	The Determination of Americium, Curium, Plutonium, Uranium, and Thorium in Liquid and Solid Matrices Using Eichrom Resin	N/A
GL-RAD-A-069	Determination of Neptunium Using AG Anion Resin	N/A
GL-RAD-A-070	The Preparation of Environmental Samples for Isotopic Uranium Using DGA Resin Via ICP-MS	N/A
GL-RAD-B-001	The Sequential Determination of Isotopic Americium, Curium, Californium, Plutonium, Strontium and Uranium in Urine	N/A
GL-RAD-B-002	The Determination of Polonium-210 or Radium-226 in Bioassay Samples	N/A
GL-RAD-B-005	Management of Blank Populations	N/A
GL-RAD-B-008	The Determination of Gross Alpha Activity in Nasal Swipes	N/A
GL-RAD-B-009	Bioassay Countroom Alpha Spectroscopy Instrument Standardization and Performance	N/A
GL-RAD-B-010	The Determination of Thorium in Fecal Samples	N/A
GL-RAD-B-011	The Determination of Tritium in Urine	EPA 906
GL-RAD-B-012	The Ashing of Fecal, Bone, and Tissue Samples	N/A

Standard Operating Procedures and Analytical Methods

SOP #	SOP Title	Methods
GL-RAD-B-013	Sequential Determination of Americium, Plutonium, Strontium, Plutonium-241, and Uranium in Fecal, Bone, and Tissue Samples	N/A
GL-RAD-B-014	The Preparation of Synthetic Urine and Fecal Material	N/A
GL-RAD-B-016	The Determination of Technetium-99 in Urine	N/A
GL-RAD-B-020	The Determination of Ni-59 and Ni-63 in Urine	N/A
GL-RAD-B-022	The Determination of Gross Alpha and Gross Non-volatile Beta in Urine	EPA 900.0, 9310, EERF 00-01, USGS R-1120-76
GL-RAD-B-023	The Determination of Carbon-14 in Urine	EERF C-01(M)
GL-RAD-B-024	Managing Statistical Data in the Bioassay Laboratory	N/A
GL-RAD-B-025	The Combination and Preservation of Urine Samples	N/A
GL-RAD-B-026	Bioassay Data Review, Validation and Data Package Assembly	N/A
GL-RAD-B-027	Specific Gravity in Urine	ASTM D5057
GL-RAD-B-029	The Determination of Radiometric Iodine in Urine	N/A
GL-RAD-B-030	The Preparation and Determination of Gamma Isotopes in Urine and Fecal Samples	600/4-80-032
GL-RAD-B-031	Bioassay Quality Control Package Assembly	N/A
GL-RAD-B-033	Bioassay Count Room Alpha Spectrometry Instrument Calibration	N/A
GL-RAD-B-034	The Determination of Metals by ICP-MS	N/A
GL-RAD-B-035	The Preparation of Urine Samples for Total Uranium Analysis by ICP-MS	N/A
GL-RAD-B-036	Initial Installation and Returning to Service of Repaired Instrumentation	N/A
GL-RAD-B-038	The Determination of Neptunium in Fecal Samples	N/A
GL-RAD-B-039	The Determination of Iron-55 in Urine	N/A
GL-RAD-B-040	The Determination of Radium-224 and Radium-226 by Alpha Spectroscopy in Bioassay Sample	N/A
GL-RAD-B-041	The Sequential Determination fo Isotopic Thorium and Neptunium in Urine	N/A
GL-RAD-B-042	The Isotopic Determination of Thorium and Neptunium and Fecal Samples	N/A
GL-RAD-D-002	Analytical Methods Validation for Radiochemistry	N/A
GL-RAD-D-003	Data Review, Validation, and Data Package Assembly	N/A
GL-RAD-D-005	REMP Quality Control Package Assembly	N/A
GL-RAD-D-006	Equations Used in Data Reduction for Environmental Radiochemistry	N/A
GL-RAD-I-001	Gamma Spectroscopy System Operation	N/A

Standard Operating Procedures and Analytical Methods

SOP #	SOP Title	Methods
GL-RAD-I-004	Beckman LS-6000/6500	N/A
GL-RAD-I-006	LB4100 Gross Alpha/Beta Counter Operating Instructions	N/A
GL-RAD-I-007	Ludlum Lucas Cell Counter	N/A
GL-RAD-I-008	VAX/VMS Quality Control Software Program	N/A
GL-RAD-I-009	Alpha Spectroscopy System	N/A
GL-RAD-I-010	Counting Room Instrumentation Maintenance	N/A
GL-RAD-I-012	Managing Statistical Data in the Radiochemistry Laboratory	N/A
GL-RAD-I-013	Column Preparation	N/A
GL-RAD-I-014	WALLAC Guardian Model 1414	N/A
GL-RAD-I-015	WPC 9550 Gross Alpha/Beta Counter: Operating Instructions	N/A
GL-RAD-I-016	Multi-Detector Counter: Operating Instructions	N/A
GL-RAD-I-017	Wallac 1220 Quantalus Liquid Scintillation Counter	N/A
GL-RAD-I-018	Operation of Wallac 1480 Gamma Wizard	N/A
GL-RAD-I-019	Management of Blank Populations	N/A
GL-RAD-I-021	G5400W Series Alpha/Beta Counting System Operating Instructions	N/A
GL-RAD-M-001	Preparation and Verification of Radioactive Standards	N/A
GL-RAD-M-003	Restoring Data from Magnetic Tape for Bioassay and Alpha Spectroscopy	N/A
GL-RAD-S-000	Radiation Safety Plan for GEL Laboratories, LLC	N/A
GL-RAD-S-001	Radiological Surveys	N/A
GL-RAD-S-002	Radiation Related Emergencies	N/A
GL-RAD-S-003	Administration of the Radioactive Material License Inventory	N/A
GL-RAD-S-004	Radioactive Material Handling	N/A
GL-RAD-S-006	Radiation Worker Training	N/A
GL-RAD-S-007	Receiving Radioactive Packages	N/A
GL-RAD-S-009	Personnel Dosimetry	N/A
GL-RAD-S-010	The Handling of Biological Materials	N/A
GL-RAD-S-013	Air Sampling for Radioactivity	Guide 825
GL-RAD-S-014	Release of Laboratory Coats	N/A
GL-RAD-S-015	The Acceptance and Classification of Radioactive Material	N/A
GL-RAD-S-016	Radiation Work Permits	N/A
GL-RAD-S-018	Laboratory Analysis of High Activity (RAD 3) Samples	N/A
GL-RC-E-001	Receipt and Inspection of Material and Services	N/A
GL-RC-E-002	Material Requisition	N/A

Standard Operating Procedures and Analytical Methods		
SOP #	**SOP Title**	**Methods**
GL-SR-E-001	Sample Receipt, Login, and Storage	N/A
GL-SR-E-002	Transportation and Shipping of Samples and Pre-Preserved Sample Containers	N/A
GL-SR-E-003	The Inspection, Cleaning and Screening of Sample Coolers	N/A
GL-SR-E-004	Control of Foreign Soils	N/A
GL-SR-E-005	Wipe Test	N/A
GL-SVR-D-001	Design Specifications for the Network Infrastructure	N/A
GL-SVR-D-002	Design Specifications for the Mail Server	N/A
GL-SVR-D-005	Design Specifications for Backupsvr01	N/A
GL-SVR-E-001	Network Infrastructure	N/A
GL-SVR-E-002	The Mail Server	N/A
GL-SVR-E-005	Backupsvr01	N/A
GL-SVR-R-001	System Requirements for Network Infrastructure	N/A
GL-SVR-R-002	System Requirements for The Mail Server	N/A
GL-SVR-R-005	System Requirements for Backupsvr01	N/A

APPENDIX J: SAMPLE STORAGE AND PRESERVATION REQUIREMENTS
STORAGE AND PRESERVATION

Parameter	Container [1]	Preservation	Holding Time[2]	Min. Volume[5]
INORGANICS				
Acidity	P,G	0 ≤ 6° C	14 days	25 mL / NA
Adsorbable Organic Halides (AOX)	G, amber	0 ≤ 6° C, HNO_3 to pH < 2, zero headspace	>3 days and < 6 months from collection	50 mL / 1 g
Alkalinity	P,G	0 ≤ 6° C	14 days	50 mL / NA
Biochemical Oxygen Demand (BOD) and Carbonaceous Oxygen Demand (CBOD)	**P,G**	**0 ≤ 6° C**	**48 hours**	**500 mL / NA**
Bromide	P,G	None required	28 days	10 mL / 4 g
Carbon Dioxide	**P,G**	**0 ≤ 6° C**	**Immediate**	**50 mL / NA**
Chemical Oxygen Demand (COD)	P,G	0 ≤ 6° C, H_2SO_4 to pH < 2	28 days	2 mL / NA
Chlorine by Bomb Calorimeter	P,G	0 ≤ 6° C	None	NA / 0.5 g
Chloride	P,G	None required	28 days	10 mL / 4 g
Color	**P,G**	**0 ≤ 6° C**	**48 hours**	**50 mL / NA**
Conductivity	P,G	0 ≤ 6° C	28 days	25 mL / NA
Corrosivity by pH	**P,G**	**None**	**Immediate**	**25 mL / 5 g**
Corrosivity to Steel	P,G	None	None	290 mL / NA
Cyanide amenable to chlorination	P,G	0 ≤ 6° C, NaOH to pH > 12, 0.6 g ascorbic acid [3]	14 days [4]	50 mL / NA
Cyanide, Reactive Releasable	G, amber	Zero headspace	7 days liquids, 28 days solids	10 mL / 10 g
Cyanide, total, available, free or Weak Acid Dissociable	P,G	0 ≤ 6° C, NaOH to pH > 12, 0.6 g ascorbic acid [3]	14 days [4]	50 mL / 1 g
Density	P,G	0 ≤ 6° C	7 days	NA / 10 g
Dissolved Oxygen	**G (bottle and top)**	**None, Zero headspace**	**Immediate**	**300 mL / NA**
Extractable Organic Halides (EOX)	G, amber	Zero headspace, 0 ≤ 6° C	**28 days**	**25 mL**
Flashpoint	Metal, G	None	None	25 mL / 2 g Setaflash
Fluoride	P,G	None Required	28 days	25 mL / 4 g
Fluorine by Bomb	P,G	0 ≤ 6° C	None	NA/ 0.5 g
Hardness (EDTA titration)	P,G	0 ≤ 6° C, HNO_3 to pH < 2	6 months	50 mL / NA
Hardness (calculation)	P,G	HNO_3 to pH < 2	6 months	50 mL / NA
Heating Value	P,G	0 ≤ 6° C	None	1 mL / 0.5 g
Nitrogen-Ammonia	P,G	0 ≤ 6° C, H_2SO_4 to pH< 2	28 days	20 mL / 5 g
Nitrate – Liquids	**P,G**	**0 ≤ 6° C**	**48 hours**	**10 mL**

Nitrate – Solids	P,G	0 ≤ 6° C	28 days for extraction, 48 hrs from extraction to analysis	4 g
Nitrite - Liquids	**P,G**	**0 ≤ 6° C**	**48 hours**	**10 mL**
Nitrite - Solids	P,G	0 ≤ 6° C	28 days for extraction, 48 hrs from extraction to analysis	4 g
Nitrate/Nitrite	P,G	0 ≤ 6° C, H_2SO_4 to pH < 2	28 days	4 mL / 4 g
Nitrogen - Total Kjeldahl and Organic	P,G	0 ≤ 6° C, H_2SO_4 to pH < 2	28 days	20 mL / 5 g
Oil and Grease	G	0 ≤ 6° C, HCl or H_2SO4 to pH < 2	28 days	1000 mL
Orthophosphate -Liquids	**P,G**	**Field filter immediately, 0 ≤ 6° C**	**48 hours**	**10 mL**
Orthophosphate – Solids	P,G	0 ≤ 6° C	28 days for extraction, 48 hrs from extraction to analysis	4 g
Paint Filter Liquids Test	Any	None	None	100 g
Percent (%) Moisture	P,G	0 ≤ 6° C	None	2 mL / 5 g
Perchlorate by Ion Chromatography	P,G	0 ≤ 6° C	28 days	10 mL / 4g
Total Phenols	G,	0 ≤6° C, H_2SO_4 to pH < 2	28 days	50 mL / 1 g
pH	**P,G**	**None if within 15 mins of collection,** 0 ≤ 6° C when shipped to lab	**Immediate**	**25 mL / 5 g**
Total Phosphorus	P,G	0 ≤ 6° C, H_2SO_4 to pH < 2	28 days	20 mL / 1 g
Residual Chlorine	**P,G**	**None Required**	**Immediate**	**25 mL / NA**
Residue, Total	P,G	0 ≤ 6° C	7 days	100 mL / NA
Residue, Filterable (TDS)	P,G	0 ≤ 6° C	7 days	70 mL / NA
Residue, NonFilterable (TSS)	P,G	0 ≤ 6° C	7 days	1000 mL
Residue, Volatile and Fixed (% Ash)	P,G	0 ≤ 6° C	7 days	25 mL / 1 g
Salinity	P,G	0 ≤ 6° C	28 days	25 mL / NA
Specific Gravity	P,G	0 ≤ 6° C	7 days	50 mL / NA
Sulfate	P,G	0 ≤ 6° C	28 days	10 mL / 4 g
Sulfide	P,G	0 ≤ 6° C, add ZnAc and NaOH to pH > 9	7 days	200 mL / 20 g
Sulfide, Reactive Releasable	G, amber	Zero headspace, 0 ≤ 6° C	7 days liquids, 28 days solids	10 mL / 10 g
Sulfide, Acid-Soluble	P,G	Zero headspace, 0 ≤ 6° C Liquids: ZnAc and NaOH to pH > 9. Solids: Fill surface with 2N ZnAc	7 days liquids, 365 days solids	200 mL / 20 g
Sulfite	**P,G**	**EDTA[9]**	**Immediate**	**50 mL / NA**

Sulfur by Bomb	P,G	0 ≤ 6° C	None	NA / 0.5 g
Surfactants	**P,G**	**0 ≤ 6° C**	**48 hours**	**100 mL / NA**
Total Organic Carbon (TOC), also applies Dissolved Organic Carbon (DOC), Total Carbon (TC) and Total Inorganic Carbon (TIC)	G, amber	0 ≤ 6° C, HCl or H_2SO_4 to pH < 2	28 days	50 mL / 5 g
Total Organic Halides (TOX)	G	0 ≤ 6° C, H_2SO_4 to pH < 2, Zero headspace	28 days	50 mL / 1 g
Total Petroleum Hydrocarbons	G	0 ≤ 6° C, H_2SO_4 to pH < 2	28 days	1000 mL / NA
TCLP (Toxicity Characteristic leaching Procedure) and Synthetic Precipitation Leaching Procedure (SPLP)	P,G depending on test	0 ≤6° C, depends on test	14 days, VOA 14 days, SVOA 28 days Mercury 180 days non-Hg metals	105 g or 130 g for full TCLP list
Turbidity	**P,G**	**0 ≤ 6° C**	**48 hours**	**50 mL / NA**
Viscosity	P,G	0 ≤ 6° C	None	7 mL
Metals – Liquids (except chromium VI, Boron, Silica and mercury)	P, (G as long as no B or Si is required)	HNO_3 to pH < 2	6 months	20 mL
Boron-Liquids	P, Teflon or Quartz	HNO_3 to pH <2	6 months	50 mL
Silica- Liquids	P or Quartz	0 ≤ 6° C	28 days	50 mL
Metals – Solids[8] (except chromium VI and mercury)	P, (G as long as no B or Si is required)	None	6 months	2 g
Chromium VI – Liquids	**P,G**	**0 ≤ 6° C**	**24 hours**	**25 mL**
Chromium VI - Liquids	P,G	0 ≤ 6° C, $(NH_4)_2SO_4$, pH = 9.3 to 9.7	28 days	25 mL
Chromium VI - Solids[8]	P,G	0 ≤ 6° C	30 days to digestion, 7 days from digestion to analysis	1 g
Mercury - Liquids	P,G	HNO_3 to pH < 2	28 days	50 mL
Mercury - Solids[8]	P,G	0 ≤ 6° C	28 days	2 g
Mercury – Low Level Liquids	P,G	HCl or BrCl	90 days when preserved w/in 48 hrs or oxidized w/in 28 days	50 mL

ORGANICS				
Method AK101-Solids[7]	Amber G	4 ± 2 °C, zero headspace, methanol	14 days	4 oz[7]
Method AK101-Liquids	Amber G	4 ± 2 °C, HCl < 2	14 days	3x40 mL
Method AK102-Liquids	Amber G	4 ± 2 °C, HCl or H_2SO_4 to pH < 2	14 days	1000 mL

Method AK102/103-Solids	Amber G	4 ± 2 °C	14 days for extraction 40 days after extraction for analysis	4 oz
MADEP EPH - Liquids	Amber G	4 ± 2 °C, HCl < 2	14 days	4 oz
MADEP EPH – Solids	Amber G	4 ± 2 °C	14 days	1000 mL
MADEP VPH – Liquids (ambient purge) Trip Blank Required	G, teflon-lined septum	4 ± 2 °C, HCl < 2	14 days	3x40 mL
MADEP – VPH Liquids (Heated Purge) Trip Blank Required	G, teflon-lined septum	4 ± 2 °C, Add 0.40 – 0.44g trisodium phosphate dodecahydrate to pH>11	14 days	3x40 mL
MADEP VPH – Solids Trip Blank Required	G, Teflon-lined septum	1mL MeOH/g sample at sampling or within 48 hrs, 4 ± 2 °C	28 days	60mL vials add 25g sample, 40 mL vials add 15 g sample
BTEX – Liquids	G, Teflon-lined septum	0 ≤ 6° C, zero headspace, HCl to pH < 2, 0.008% $Na_2S_2O_3$ [3]	14 days [6]	3x40 mL
BTEX - Solids[8]	**G, Teflon-lined septum**	**0 ≤ 6° C**	**48 hours for preservation and 14 days for analysis**	**3x5 g EnCores or 2 low and 1 high level vials**
Volatiles - Drinking Water, Wastewater/groundwater (except 2-CLEVE, acrolein, and acrylonitrile)	G, Teflon-lined cap	0 ≤ 6° C, zero headspace, HCl to pH < 2	14 days	3x40 mL
Volatiles (including 2 CLEVE) - Wastewater	G, Teflon-lined cap	0 ≤ 6° C, zero headspace, unpreserved	7 days [6]	3x40 mL
Volatiles - (acrolein and acrylonitrile)	G, Teflon-lined cap	0 ≤ 6° C, zero headspace, unpreserved	3 days [6] by EPA 624.1 7 days [6] by EPA 8260	3x40 mL
Volatiles - Solids[8]	**EnCore Sampler**	**0 ≤ 6° C**	**48 hours for preservation 14 days for analysis**	**3x5 g EnCores**
Volatiles - Concentrated Waste	G, teflon-lined septum	None	14 days	1x40 mL
Base/Neutral and Acid Extractables and 1,4-Dioxane – Liquids	Amber G, Teflon-lined cap	0 ≤ 6° C, 0.008% $Na_2S_2O_3$ [3]	7 days for extraction 40 days after extraction for analysis	1000 mL / 50 g
Base/Neutral and Acid Extractables and 1,4-Dioxane-Solids[8]	G, Teflon-lined cap	0 ≤ 6° C	14 days for extraction 40 days after extraction for analysis	1000 mL / 50 g
Base/Neutral and Acid Extractables - Concentrated Waste	G, Teflon-lined cap	None	7 days for extraction 40 days after extraction for analysis	1000 mL / 50 g
TPH-GRO	G, Teflon-lined cap	0 ≤ 6° C, HCl to pH < 2, zero headspace	14 days	3x40 mL

TPH-DRO	G, Teflon-lined cap	0 ≤ 6° C, HCl to pH < 2	7 days for extraction (Liquids) 14 days for extraction (Solids) 40 days after extraction to analysis	1000 mL / 50 g
Chlorinated Herbicides - Liquids	Amber G, Teflon-lined cap	0 ≤ 6° C, 0.008% $Na_2S_2O_3$ [3]	7 days for extraction 40 days after extraction for analysis	1000 mL
Chlorinated Herbicides - Solids[8]	G, Teflon-lined cap	0 ≤ 6° C	14 days for extraction 40 days after extraction	50 g
Organochlorine Pesticides by SW-846 EPA 8081 Liquids	Amber G, Teflon-lined cap	0 ≤ 6° C, 0.008% $Na_2S_2O_3$	7 days for extraction 40 days after extraction for analysis	1000 mL
Organochlorine Pesticides by SW-846 EPA 8081 Solids	G, Teflon-lined septum	0 ≤ 6° C	14 days for extraction 40 days after extraction for analysis	50g
Organochlorine Pesticides and PCBS by EPA 608.3 only	Amber G, Teflon-lined cap	0 ≤ 6° C, 0.008% , $Na_2S_2O_3$ [3], NaOH and H_2SO_4preserve to pH 5.0 -9.0 (for prep >72 hrs and < 7 days)	**Unpreserved Prep within 72 hrs** Preserved prep within 7 days 40 days after extraction for analysis	**1000 mL / NA**
PCBs- Liquids	Amber G, Teflon-lined cap	0 ≤ 6° C, 0.008% $Na_2S_2O_3$ [3]	365 days for extraction 40 days after extraction for analysis	1000 mL
PCBs- Solids	Wide-mouth glass	0 ≤ 6° C	365 days for extraction 40 days after extraction for analysis	50g
PCBs in Oil	G, Teflon-lined cap	None	365 days for extraction 40 days after extraction for analysis	1x40 mL
Solvents, Glycols, Alcohols and Acetates -- Liquid	G, Teflon-lined septum	0 ≤ 6° C, zero headspace or 0 ≤ 6° C, zero headspace HCl to pH < 2	7 days unpreserved 14 days preserved	1 x 40mL
Solvents, Glycols, Alcohols and Acetates -- Solids	G, Teflon-lined septum	0 ≤ 6° C	14 days	10g
Industrial Solvents	G, Teflon-lined septum	0 ≤ 6° C	14 days	1x40 mL

1,4-Dioxane in Drinking Water by EPA 522	G, Teflon-lined septum	<10°C during transport, Sodium sulfite (50mg/L), sodium bisulfate (1g/L)	28 days for extraction at 0 ≤ 6° C (not frozen) and 28 days after extraction for analysis at -5° C, protected from light	100 mL to 500 mL
Dioxin Screen	G, Teflon-lined cap	0 ≤ 6° C	7 days for extraction 40 days after extraction for analysis	1000 mL / 50 g
EDB and DBCP	G, Teflon-lined septum	0 ≤ 6° C, 0.4% $Na_2S_2O_3$	14 days	3x40 mL / NA
Polynuclear Aromatic Hydrocarbons	Amber G, Teflon-lined septum (Liquids), Teflon-lined cap (Solids)	0 ≤ 6° C	7 days for extraction (Liquids) 14 days to extraction (Solids) 40 days to analysis after extraction	1000 mL / 30 g
Nitroaromatics and Nitroamines	Amber G, Teflon-lined septum	0 ≤ 6° C	7 days for extraction 40 days after extraction for analysis	1000 mL / 2 g
Nitroaromatics and Nitroamines by MIS Prep (solid samples)	Protect from light	0 ≤ 6° C until air drying 22 ± 4° C (or cooler) after drying	14 days for extraction, 40 days after extraction for analysis	Entire Sample
RDX Breakdown	Amber G, Teflon-lined septum for liquids and Teflon-lined cap for solids	0 ≤ 6° C	7 days to extraction for liquids 14 days to extraction for solids 40 days to analysis after extraction	1000 mL / 2 g
Low Level Perchlorate	P	0 ≤ 6° C , headspace required	28 days	10 mL / 2 g
Haloacetic Acids	G, amber, Teflon-lined septum	0 ≤ 6° C , zero headspace, ammonium chloride	14 days to extraction, 7 days after extraction for analysis	3x40 mL
Dissolved Gases	G, Teflon-lined septum	0 ≤ 6° C, HCl to pH < 2, zero headspace	7 days if unpreserved, 14 days if preserved	2x40 mL

Perfluorinated Alkyl Acids PFAS	HDPE Bottle - unlined polyethylene screw cap	0 ≤ 10° C for liquids, 0 ≤ 6° C for solids, 1.25g Trizma® (Drinking Water only)	14 days from collection to extraction, 28 days from extraction to analysis (liquids) 28 days from collection to extract and analyze (solids)	250 mL/10 g
RADIOCHEMISTRY				
Americium – Liquids	P,G	HNO_3 or HCl to pH < 2	6 months	1000 mL
Americium – Solids[8]	P,G	None	6 months	20 g
Calcium-45 – Liquids	P,G	HNO_3 or HCl to pH < 2	6 months	500 mL
Calcium-45 - Solids[8]	P,G	None	6 months	20 g
Carbon-14 Liquids & Solids[8]	P,G	None	6 months	500 mL / 20 g
Cesium 134 – Drinking Water	P,G	HCl to pH < 2	6 months	2000 mL
Chlorine-36 Liquids & Solids[8]	P,G	None	6 months	500 mL / 20 g
Curium - Liquids	P,G	HNO_3 or HCl to pH < 2	6 months	1000 mL
Curium - Solids[8]	P,G	None	6 months	20 g
Gamma Isotopes - Liquids	P,G	HNO_3 or HCl to pH < 2	6 months	2000 mL
Gamma Isotopes - Solids[8]	P,G	None	6 months	200 g
Gross Alpha & Beta – Liquids	P,G	HNO_3 or HCl to pH < 2	6 months	500 mL
Gross Alpha & Beta, Rapid - Liquids	**P,G**	**HNO_3 or HCl to pH < 2**	**48 – 72 hrs**	**500 mL**
Gross Alpha & Beta - Solids[8]	P,G	None	6 months	20 g
Iodine-129 - Liquids & Solids[8]	P,G	None	6 months	1000 mL / 50 g
Iodine -131 - Liquids	**P,G**	**None**	**8 days**	**1000 mL**
Iron 55 -Liquids	P,G	HNO_3 or HCl to pH < 2	6 months	500 mL
Iron 55 - Solids[8]	P,G	None	6 months	20 g
Lead-210 – Liquids	P,G	HNO_3 or HCl to pH < 2	6 months	1000 mL
Lead-210 - Solids[8]	P,G	None	6 months	200 g
Neptunium - Liquids	P,G	HNO_3 or HCl to pH < 2	6 months	1000 mL
Neptunium - Solids[8]	P,G	None	6 months	20 g
Nickel-59 – Liquids	P,G	HNO_3 or HCl to pH < 2	6 months	1000 mL
Nickel-59 – Solids[8]	P,G	None	6 months	20 g
Nickel-63 - Liquids	P,G	HNO_3 or HCl to pH < 2	6 months	1000 mL
Nickel-63 - Solids[8]	P,G	None	6 months	20 g
Phosphorus-32 –Liquids	P,G	HNO_3 or HCl to pH < 2	6 months	1000 mL
Phosphorus-32 - Solids[8]	P,G	None	6 months	20 g
Plutonium – Liquids	P,G	HNO_3 or HCl to pH < 2	6 months	1000 mL
Plutonium - Solids[8]	P,G	None	6 months	20 g
Polonium - Liquids	P,G	HNO_3 or HCl to pH < 2	6 months	1000 mL
Polonium - Solids[8]	P,G	None	6 months	20 g
Promethium-147/Samarium-151 – Liquids	P,G	HNO_3 or HCl to pH < 2	6 months	1000 mL
Promethium-147/Samarium-151 - Solids[8]	P,G	None	6 months	20 g
Radium-223 – Liquids	P,G	HNO_3 or HCl to pH < 2	6 months	2000 mL

Radium-224 – Liquids	P,G	HNO_3 or HCl to pH < 2	6 months	2000 mL
Radium-226 – Liquids	P,G	HNO_3 or HCl to pH < 2	6 months	1000 mL
Radium-228 – Liquids	P,G	HNO_3 or HCl to pH < 2	6 months	1000 mL
Radon-222 – Liquids	**G**	**None, Zero headspace**	**4 days**	**2x40 mL**
Selenium-79 – Liquids	P,G	HNO_3 or HCl to pH < 2	6 months	500 mL
Selenium-79 - Solids[8]	P,G	None	6 months	20 g
Strontium-89/90 – Liquids	P,G	HNO_3 or HCl to pH < 2	6 months	1000 mL
Strontium-89/90 - Solids[8]	P,G	None	6 months	20 g
Sulfur-35 - Liquids	P,G	None	6 months	500 mL
Sulfur-35 - Solids[8]	P,G	None	6 months	20 g
Technetium-99 – Liquids	P,G	HNO_3 or HCl to pH < 2	6 months	1000 mL
Technetium-99 – Solids[8]	P,G	None	6 months	20 g
Thorium – Liquids	P,G	HNO_3 or HCl to pH < 2	6 months	1000 mL
Thorium - Solids[8]	P,G	None	6 months	20 g
Total Activity Liquids	P,G	HNO_3 or HCl to pH < 2	6 months	100 mL
Total Activity - Solids[8]	P,G	None	6 months	20 g
Total Alpha Radium – Liquids	P,G	HNO_3 or HCl to pH < 2	6 months	500 mL
Total Alpha Radium - Solids[8]	P,G	None	6 months	20 g
Total Uranium - Liquids	P,G	HNO_3 or HCl to pH < 2	6 months	100 mL
Total Uranium - Solids[8]	P,G	None	6 months	20 g
Tritium – Drinking Water	G	None	6 months	250 mL
Tritium – Liquids & Solids[8]	P,G	None	6 months	250 mL / 20 g
Uranium – Liquids	P,G	HNO_3 or HCl to pH < 2	6 months	1000 mL
Uranium - Solids[8]	P,G	None	6 months	20 g

[1] P = Polyethylene; G = Glass

[2] Samples should be analyzed as soon as possible after collection. The holding times listed are maximum times that samples may be held before analysis and be considered valid.

[3] Used only in the presence of residual chlorine.

[4] Maximum holding time is 24 hours when sulfide is present. All samples may be tested with lead acetate paper before pH adjustments in order to determine if sulfide is present. If present, remove by adding cadmium nitrate powder until a negative spot test is obtained. Filter sample and add NaOH to pH 12.

[5] Minimum amount of sample needed to prepare and analyze for the parameter. Some parameters may be combined into one analysis, others may need additional amount if quality control is being requested for site-specific samples. Please check with GELs Project Manager for proper sample amounts based on project specific requirements.

[6] Volatiles Groundwater/Wastewater: If samples are to be analyzed for vinyl chloride, styrene, or 2-chloroethylvinyl ether (2-CLEVE) for soil or water, separate samples must be collected without acid preservation and analyzed within 7 days. For aqueous samples to be analyzed for acrolein and acrylonitrile, by EPA Method 624.1, the samples are not to be acidified and must be analyzed within 3 days of collection.

[7] Solids Method AK101 2-4 oz amber wide-mouth jars tared and labeled, 1-4 oz amber wide-mouth jar labeled (evaporative loss), 2-25 mL 2.5 ppm surrogated P/T methanol tubes.

[8] Solids matrix typically applies to soils, sludges and sediments. Some tests have been developed for filters, miscellaneous solid waste, plant and animal tissue, also referred to as solids. Contact GEL to verify a particular matrix for the test of interest.

[9] 1mL of 2.5% EDTA solution per 100mL sample

APPENDIX K: STATE SPECIFIC REPORTING CRITERIA

Massachusetts: Drinking Water (Only)

Regulations at 310 CMR 42.13 (5) require that a laboratory have current knowledge of all Federal and Massachusetts standards for all categories in which it has been certified. Within 24 hours of obtaining valid data, a certified laboratory must notify its clients for any results exceeding an EPA-or Department-established maximum contaminant level, maximum residual disinfectant level or reportable concentration.

The laboratory must identify, in writing, those samples needing special reports (e.g. MCL exceedance) when the laboratory subcontracts with another laboratory.

Reports for drinking water samples must contain information relating to the maximum contaminant levels for each analyte. 310 CMR 42.13(3) specifies that with exception of reports submitted to the Department in a format approved by the Department, all reports of finished drinking water analyses must indicate the maximum contaminant level for each analyte measured. This can be accomplished in AlphaLIMS through the permit level in client set up. (Project Managers must enter these values). The maximum contaminant levels should be verified prior or sample log-in. **Please check with Quality Assurance Officer to verify that the information is correct.**

The report must identify, analyses for which the laboratory holds Department certification and which it does not. Regulations at 310 CMR 42.13(3) (b-c) require that such a distinction be made and that the laboratory clearly distinguish in the report between those analyses that it conducted in accordance with Department certification standards and those it did not.

Pennsylvania: Drinking Water (Only)

Any individual (laboratory, sample collection/pic-up facility, consultant, PWS, etc.) providing a sample to an accredited laboratory for SDWA compliance testing purposes must ensure that all relevant, and necessary information is provided along with the sample. Since the laboratory that performs the testing is responsible for reporting and making any notifications (such as MCL violations) to the PWS and the Department, the PWS and sample specific information is both relevant and necessary. If a laboratory chooses, or is required, to subcontract testing to another accredited laboratory, § 109.810(b)(1)(ii) requires that the following information MUST be provided to the subcontract laboratory:

- PWSID# and Name of the System
- Sample Location ID#
- Dates and Times of Sample Collection
- Name and Contact Number of the PWS

The testing laboratory may, if it chooses to, relinquish its authority to report the sample results. However, this relinquishment can only be made to another accredited laboratory and must be made in writing as described in § 109.810(c). The other accredited laboratory, to which the reporting and notification responsibilities are delegated, is then responsible for meeting all of the 25 Pa. Code Chapter 109.810 requirements.

Failure of the testing laboratory to provide verbal and written notification to the Public Water Supply ("PWS") or the Department, or both of an MCL violation with the required timeframes:
The Department requires in § 109.810(b)(1) that the testing laboratory **notify the PWS by telephone within 1 hour of the determination** that an MCL violation has occurred for any SDWA compliance testing result that is at or above the listed MCL for that contaminant. Chapter 252, §§ 252.708(a)(2) and (3) outline the allowable time that may elapse between initial acquisition of the sample result and the final "determination" of the sample result. The time of the determination of the final sample result triggers the start of the clock for the allowable timeframes to provide notification to the PWS and the Department. It is of upmost importance that you understand that leaving a message or voicemail is not considered "notification" of an MCL exceedance. Should the testing laboratory be unable to notify the PWS within 1 hour of the determination, the laboratory must **notify the appropriate DEP regional office by telephone within 2 hours of the determination** of the MCL exceedance. Finally, the testing laboratory is responsible for providing written notification to the Department of any MCL exceedance within 24 hours of the determination.

Failure of the testing laboratory to maintain full and complete records documenting the notification made to the PWS or the Department, or both, when an MCL violation occurs:
The accreditation regulations require that an accredited laboratory maintain accurate and complete records that allow historical reconstruction of the activities undertaken in the laboratory. The testing laboratory must maintain documentation outlining the steps taken to meet the requirements of § 109.810(a)(1) and § 252.708(a)(2) and (3), also known as the acquisition of the initial sample results and the final determination of the sample results to determine compliance with the 1-hour or 2-hour notification requirements. Specifically, the testing laboratory must maintain the following:

- Date and Time of the initial acquisition of the sample result
- Date and Time of the determination of the sample result
- Date and Time of the telephone call(s) to the PWS
- Individual at the PWS to whom the notification was made
- Date and Time of the telephone call(s) to the Department, if required
- Individual at the Department to whom the notification was made, if required
- Any other pertinent information that would be necessary to ensure a complete record

If the testing laboratory delegates the reporting and notification responsibility to another accredited laboratory, as allowed by § 109.810(c), both laboratories must maintain the records to document their activities and must ensure that the notifications occur with the required timelines. It is important to note that the **reporting laboratory has 1 hour from the determination of the result made by the testing laboratory** to notify the PWS of the MCL violation. The 1-hour notification cannot be extended due an intermediate notification from a testing laboratory to a reporting laboratory.

Failure of the laboratory to accurately and fully report the subcontracting testing laboratory's results to the PWS:
It is the laboratory's responsibility to report the final test results of any PA-DEP compliance sample accurately, correctly, unambiguously, and with any specific client instructions or regulations. The laboratory is required to ensure that it reports only those test results that are associated with appropriately collected, handled, stored, prepared, and analyzed samples or report the results with appropriate data qualifiers. In some cases, a laboratory that subcontracts the testing to another accredited laboratory may choose to transcribe the accredited

laboratory's results onto its own letterhead/report format. In these cases, the reporting laboratory is responsible for full, accurate, and complete transcription of all sample results; data qualifiers; sample collection, handling, preparation comments; any case narrative or other applicable comment directly to the PWS.

The Department recommends that laboratories provide the testing laboratory's final test report directly to the PWS instead of the transcribing the results. The Department also reminds all laboratories that only results that are associated with acceptable sample collection, storage, handling, preparation, analysis, test conditions, and quality control may be reported to DWELR. A laboratory may request permission to report qualified DW results by using the "Request to Report Qualified DW Results" form and submission instructions. Please note that microbiology test results are handled differently than chemistry results. Once the microbiology samples are accepted and the analysis begins, positive microbiology test results can only be invalidated by the Department regardless of the performance of the QC, instrument test conditions, etc.

Failure to maintain an SOP for reporting PA-DEP SDWA compliance samples that meet the requirements of 25 PA. Code Chapter 109:
The Department requires all laboratories accredited to perform SDWA compliance testing to maintain an SOP that meets the requirements of § 109.810(b)(3)(ii), also known as the "SWDA Reporting SOP." The SWDA Reporting SOP must be established initially upon accreditation and updated annually thereafter. The SOP must include procedures to meet all of the reporting, documentation, notification requirements of § 109.810. At a minimum, the SOP must include:

- The procedure for ensuring that the laboratory obtains and maintains the information regarding the Public Water Supplier, including PWSID#, name of the PWS, contact name and telephone number for the PWS;
- The procedure for ensuring that the laboratory obtains the sample specific information, including sample location, contaminants(s) of interest, date and time of sample collection;
- The procedure for notifications of MCL exceedances, both telephonic and in writing;
- The procedure for documenting the laboratory's activities related to MCL violations and notifications of such violations;
- The procedure for reporting results to DWELR;
- The telephone numbers for each DEP regional office's main number and after hours emergency response telephone number.

The following is an expert from 25 Pa. Code Chapter 109 as it relates to the requirements for accredited laboratories:

25 Pa. Code Chapter 109, § 109.810. Reporting and notification requirements.

(a) Beginning November 13, 2009, a laboratory accredited under Chapter 252 (relating to environmental laboratory accreditation) shall electronically report to the Department on behalf of the public water supplier and in accordance with the reporting requirements under § 109.701(a) (relating to reporting and recordkeeping), the results of test measurements or analyses performed by the laboratory under this chapter using a secure computer application provided by the Department. In the event of a Department computer application failure, the Department will notify the laboratory of an alternate reporting method. In the event that a laboratory is unable to submit data electronically, due to circumstances beyond its control, the laboratory shall notify the Department prior to the applicable reporting deadline. If the Department

determines that the circumstances were beyond the control of the laboratory, the Department will specify a temporary, alternate reporting method the laboratory shall use to meet the reporting deadline.

(1) Unless a different reporting period is specified in this chapter, these results shall be reported within either the first 10 days following the month in which the result is determined or the first 10 days following the end of the required monitoring period as stipulated by the Department, whichever is shorter.

(2) Beginning November 23, 2009, an accredited laboratory and the public water supplier shall be given until the 10th of the following month to review and update submitted data using a secure computer application provided by the Department. Omissions and data errors remaining after the review period shall be considered reporting violations of the public water supplier.

(b) A laboratory accredited under Chapter 252 shall whenever the results of test measurements or analyses performed by the laboratory under this chapter indicate an MCL, MRDL or treatment technique performance requirements under § 109.202 (relating to State MCLs , MRDLs and treatment technique requirements) is exceeded, or an action level under § 109.1102 (a) (relating to lead and copper) is exceeded, or sample result requires the collection of check or confirmation samples under § 109.301 (relating to general monitoring requirements), or a sample collected under Subchapter M (relating to additional requirements for groundwater sources) is *E. Coli*-positive:

(1) Notify the public water supplier by telephone within 1 hour of the laboratory's determination. If the supplier cannot be reached within that time, notify the Department by telephone within 2 hours of the determination. If is necessary for the laboratory to contact the Department after the Department's routine business hours, the laboratory shall contact the appropriate Department's regional office's after-hours emergency response telephone number and provide information regarding the occurrence, the name of contact person and the telephone number where that individual may be reached in the event further information is needed. If the Department's appropriate emergency number cannot be reached, the laboratory shall notify the appropriate Department regional office by telephone within 1 hour of the beginning of the next business day. Each accredited laboratory shall be responsible for the following:

Obtaining and then maintaining the Department's current after-hours emergency response telephone numbers for each applicable regional office.

(i) Establishing or updating a standard operating procedure by November 8, 2002, and at least annually thereafter to provide the information needed to report the occurrences to the Department. The information regarding the public water system must include, but is not limited to, the PWSID number of the system, the system's name, the contaminant involved in the occurrence, the level of the contaminant found, where the sample was collected, the dates and times that the sample was collected and analyzed, the name and identification number of the certified laboratory, the name and telephone number of a contact person at the laboratory and what steps the laboratory took to contact the public water system before calling the Department.

(2) Notify the appropriate Department district office in writing within 24 hours of the determination. For the purpose of determining compliance with this requirement, the postmark, if the notice is mailed, or the date the notice is received by the Department, whichever is earlier, will be used. Upon approval by the Department, the notice may be made electronically to the Department as long as the information is received within the 24-hour deadline.

(c) A laboratory accredited under Chapter 252 shall meet the requirements under subsections (a) and (b), regarding the results of test measurements or analyses performed by the laboratory under this chapter, unless the laboratory assigns in writing the responsibility for reporting and notification to another accredited laboratory.

(d) A laboratory accredited under Chapter 252 shall be responsible for the accurate reporting of data required under the section to the Department.

VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE FOR THE ISOTOPIC DETERMINATION OF AMERICIUM, CURIUM, PLUTONIUM, AND URANIUM

(GL-RAD-A-011 REVISION 28)

APPLICABLE TO METHODS:
DOE RP800 1997 (Modified)
EML HASL-300 U-02-RC (Modified)
EML HASL-300 Am-05-RC (Modified)
DOE HASL-300 Pu-11-RC (Modified)
EPA SW-846 3050B (Modified)

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1.0 STANDARD OPERATING PROCEDURE FOR THE ISOTOPIC DETERMINATION OF AMERICIUM, CURIUM, PLUTONIUM, AND URANIUM

2.0 METHOD OBJECTIVE, PURPOSE, CODE, AND SUMMARY

2.1 This standard operating procedure provides the necessary instructions to conduct the analysis for isotopic americium, curium, plutonium, and uranium in a variety of liquid, filter and solid matrices. This method also gives specific guidance on determining U-232, Pu-242 and Am-243, which are typically used as isotopic tracers.

2.2 A soil sample is aliquoted and digested according to GL-RAD-A-015, if necessary. The elements are then separated through ion exchange resins. For liquid samples, transuranic elements are scavenged by coprecipitation with iron hydroxide. The precipitate is dissolved, and separation of elements is accomplished through ion exchange resins. The elements are then prepared for the measurement of radioactive isotopes by coprecipitation with neodymium fluoride. The neodymium fluoride precipitate is trapped on a filter, mounted on a metal disk and placed in a partially evacuated chamber for measurement of isotopic alpha emission.

2.3 This method has been modified from the source method from EML Methods Manual HASL-300 U-02-RC, Am-05-RC, and Pu-11-RC and uses similar principles of radiochemical separation and counting. Modifications include chemical separations utilizing Eichrom TEVA and TRU resins to facilitate separation of various elements. There are also variations in the concentrations of acids, as well as the application of these acids.

2.4 This method is also very similar in concept to the source method from the DOE Methods Manual for Evaluating Environmental and Waste Management Samples, 1997 Edition, RP800, “Sequential Separation of Americium and Plutonium by Extraction Chromatography.”

2.5 This method has been modified on the basis of GEL’s Performance Based Measurement System (PBMS).

2.6 This method also contains a special procedure for digestion of samples in accordance with EPA method SW-846 3050B (Modified).

3.0 METHOD SCOPE, APPLICABILITY, AND DETECTION LIMIT

3.1 Method Detection Limit: Typical minimum detectable activity (MDA) for samples analyzed for Am/Cm/Pu/U is 1 pCi/L or 1 pCi/g for all isotopes.

3.2 Analyst training records are maintained as quality records as outlined in GL-QS-E-008. Analyst training and proficiency in the method is outlined in the Quality SOP for the Method Validation and Initial and Continuing Demonstrations of Capability, GL-QS-E-011.

3.3 Applicable matrices to this SOP are liquids, drinking water, vegetation, tissues, air filters, and solids.

NOTE: This method is not an EPA approved method for the analysis of drinking water.

4.0 METHOD VARIATIONS

Some variations may be necessary due to special matrices encountered in the lab. These variations may be used with approval from a Group or Team Leader. Variations to a method will be documented with the analytical raw data.

5.0 DEFINITIONS

5.1 AlphaLIMS: Laboratory Information Management System used at GEL.

5.2 Batch: Environmental samples prepared and/or analyzed together with the same process and personnel using the same lot(s) of reagents.

5.3 Deionized (DI) Water: Type I water, Refer to GL-LB-E-016.

5.4 Laboratory Control Sample (LCS): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes from a source independent of the calibration standards or a material containing known and verified amounts of analytes.

5.5 Laboratory Duplicate (DUP): Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently.

5.6 Matrix Spike (MS): Prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available.

5.7 Matrix Spike Duplicate (MSD): A second replicate matrix spike is prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

5.8 Method Blank (MB): A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples containing an analyte of interest through all steps of the analytical procedures.

5.9 National Institute of Standards and Technology (NIST): For the purpose of this method, the national scientific body responsible for the standardization and acceptability of analyte solutions.

5.10 Solid Reference Material (SRM): A solid material containing known and verified amounts of analytes.

5.11 Tracer: A known quantity of a radioisotope that is added to each sample of a chemically equivalent radioisotope of unknown concentration so that the yield of the chemical separation can be calculated.

6.0 INTERFERENCES

6.1 Internal tracer standards may have ingrown daughters that may interfere with the analysis. For example Th-228 will be present in aged U-232 standard, Fr-221 will be present in Th-229, which will interfere with the curium analysis, and U-232 will be present in Pu-236. These problems are overcome by running separate aliquots of sample for thorium analysis, or by mathematical compensation for the interference.

6.2 Short lived radioactive progeny may ingrow on prepared filters. For example, the Ra-224 alpha peak will be present if the Th-228 parent is present. These interferences are minimized by counting sample as soon as possible after separation chemistry.

7.0 SAFETY PRECAUTIONS AND WARNINGS

7.1 Personnel performing this analytical procedure are trained in and follow the safe laboratory practices outlined in the Safety, Health and Chemical Hygiene Plan, GL-LB-N-001.

7.2 Personnel handling radioactive materials are trained in and follow the procedures outlined in GL-RAD-S-004 for Radioactive Material Handling.

7.3 Personnel handling biological materials are trained in and follow the procedures outlined in GL-RAD-S-010 for The Handling of Biological Materials.

7.4 If there is any regarding the safety of any laboratory practice, **stop immediately**, and consult qualified senior personnel such as a Group or Team Leader.

8.0 APPARATUS, EQUIPMENT, AND INSTRUMENTATION

8.1 Apparatus and Equipment

8.1.1 Silicon surface barrier detectors with associated electronics, vacuum chambers, and data reduction capabilities

8.1.2 Eichrom Technologies TEVA Resin, 100 – 150 µm particle size

8.1.3 Eichrom Technologies TRU Resin, 100 – 150 µm particle size

8.1.4 Vacuum pump and filtration apparatus

8.1.5 Disposable filter funnels (containing 25 mm polypropylene filters with 0.1 µm pore size)

8.1.6 Metal disks, 29 mm diameter

8.1.7 Stainless steel tweezers

8.1.8 Polypropylene centrifuge tube (50 mL)

8.1.9 Sample drying and ashing apparatus

8.1.10 Sample homogenization apparatus

8.1.11 AG 1X8 anion exchange resin, 100 – 200 mesh

8.1.12 Hot plate

8.1.13 Beakers (Glass and Teflon of various sizes)

8.1.14 2.5 cm^3 column

8.1.15 25 mL column funnel extension

8.1.16 Watch glasses (various sizes)

8.1.17 Digestion vessel

8.1.18 Reflux cap

8.1.19 Ribbed watch glass

8.1.20 Hot block

8.1.21 2.0 µm pore size plunger filter (PTF grade)

9.0 REAGENTS AND STANDARDS

9.1 Reagents

9.1.1 Neodymium carrier (500 mg/L)

9.1.2 Neodymium carrier (10,000 mg/L)

9.1.3 Carbon Colorant: Place four 47 mm cellulose nitrate filters in a beaker and add 5 mL concentrated sulfuric acid. Cover and heat on a hot plate with medium-high heat for approximately 2 to 4 hours. Cool dark residue completely. Slurry the residue in DI water and dilute to 1 L with DI water.

9.1.4 Hydrochloric acid (9 M HCl): Add 750 mL of concentrated hydrochloric acid to 100 mL of DI water. Allow to cool and dilute to

1 L with DI water.

9.1.5 Hydrochloric acid (3 M HCl): Add 250 mL of concentrated hydrochloric acid to 500 mL DI water. Allow to cool and dilute to 1 L with DI water.

9.1.6 Hydrochloric acid, concentrated (12 M HCl).

9.1.7 9 M Hydrochloric acid/0.05 M Ammonium iodide: Dissolve 7.24 g of ammonium iodide in 750 mL of concentrated hydrochloric acid and add to 100 mL of DI water. Allow to cool and dilute to 1 L with DI water. PREPARE DAILY.

9.1.8 Hydrochloric acid (6 M HCl): Add 500 mL of concentrated hydrochloric acid to 500 mL of DI water.

9.1.9 6 M Hydrochloric acid/0.52 M Hydrofluoric acid: Add 500 mL of concentrated hydrochloric acid and 18.6 mL of 49% hydrofluoric acid to 300 mL of DI water. Allow to cool and dilute to 1 L with DI water.

9.1.10 25% Hydrazine dihydrochloride: Dissolve 25 g of hydrazine dihydrochloride in 75 mL of DI water and dilute to 100 mL with DI water.

9.1.11 9 M Hydrochloric acid/0.04% Hydrogen peroxide: Add 8 drops of 30% hydrogen peroxide to 1 L of 9 M hydrochloric acid. PREPARE DAILY.

9.1.12 Ethyl alcohol (80% EtOH): Dilute 800 mL ethanol to 1 L with DI water.

9.1.13 Hydrochloric acid (0.1 M HCl): Add 8.3 mL of concentrated hydrochloric acid to 500 mL of DI water. Allow to cool and dilute to

1 L with DI water.

9.1.14 Hydrofluoric acid, concentrated (49% HF)

9.1.15 Hydrogen peroxide (30% H_2O_2)

9.1.16 Iron Carrier (10 mg/mL): Dissolve 62.7 g of $Fe(NO_3)_3 \bullet 6H_2O$ or 72.3 g of $Fe(NO_3)_3 \bullet 9H_2O$ in 800 mL DI water and dilute to 1 L with DI water.

9.1.17 Nitric acid concentrated (16 M HNO_3)

9.1.18 Nitric acid (2 M HNO_3): Add 125 mL of concentrated nitric acid to 500 mL of DI water. Allow to cool and dilute to 1 L with DI water.

9.1.19 Nitric acid (1 M HNO_3): Dilute 62.5 mL concentrated nitric acid to 500 mL of DI water. Allow to cool and dilute to 1 L with DI water.

9.1.20 2 M Nitric acid/1 M Aluminum nitrate: Dissolve 375.13 g of aluminum nitrate nonahydrate, $Al(NO_3)_3 \bullet 9H_2O$, in 300 mL of DI water. Add 125 mL of concentrated nitric acid to the DI water. Allow to cool and dilute to 1 L with DI water.

9.1.21 Titanium (III) chloride, 10-20% reagent

9.1.22 Hydrochloric acid (2 M HCl): Add 167 mL of concentrated hydrochloric acid to 500 mL DI water. Allow to cool and dilute to 1 L with DI water.

9.1.23 Nitric acid (1 M HNO_3): Add 62.5 mL of concentrated nitric acid to 500 mL of DI water. Allow to cool and dilute to 1 L with DI water.

9.1.24 1.25 M Calcium nitrate: Dissolve 205 g of anhydrous calcium nitrate or 295 g hydrated calcium nitrate, $Ca(NO_3)_2 \bullet 4H_2O$, in 500 mL of DI water. Dilute to 1L with DI water.

9.1.25 Phosphoric acid, concentrated (H_3PO_4)

9.1.26 Lanthanum (10,000 mg/L)

9.1.27 Sulfuric acid (0.1 M H_2SO_4): Add 5 mL concentrated sulfuric acid to 800 mL of DI water. Allow to cool and dilute to 900 mL.

9.1.28 Formic acid, concentrated

9.1.29 4 M Ammonium thiocyanate/0.1 M Formic acid: Add 60 g of ammonium thiocyanate and 1.0 mL of concentrated formic acid to a graduated cylinder and dilute to 200 mL with DI water. Prepare fresh daily.

9.1.30 1.5 M Ammonium thiocyanate/0.1 M Formic acid: Add 9.5 g of Ammonium thiocyanate and 0.5 mL of concentrated formic acid to a graduated cylinder and dilute to 100 mL with DI water. Prepare fresh daily.

9.1.31 Substrate suspension: Dilute 4 mL of neodymium chloride (10,000 mg/L), 80 mL of concentrated hydrochloric acid and 40 mL of carbon colorant to 1500 mL with DI water. Add 40 mL 49% hydrofluoric acid while swirling and dilute to 2 L with DI water.

9.1.32 Sulfuric acid, concentrated (18 M H_2SO_4).

9.1.33 Cellulose nitrate filters (47 mm)

9.1.34 Ammonium hydroxide concentrated (14 N NH_4OH)

9.1.35 Acetone

9.2 Standards

9.2.1 NIST traceable standards: Am-241, Am-243, Cm-244, Pu-242, Pu-239, Pu-238, Pu-236, U-232, U-236, U-238.

9.2.2 Refer to GL-RAD-M-001.

10.0 SAMPLE HANDLING AND PRESERVATION

10.1 Samples should be collected in a plastic bottle and preserved to approximately pH< 2 with nitric acid.

10.2 Before beginning an analysis, the analyst should check the sample pH by removing a minimal amount of sample with a transfer pipette and placing it on a pH strip. DO NOT insert pH strip into sample container. If the sample is received with a pH greater than 2, the analyst should contact the Group Leader or Team Leader. If approved by the client, the analyst should adjust the pH with nitric acid to a pH< 2. If the sample pH is adjusted, let the sample sit in the original container for a minimum of 24 hours before analysis. This acidification should be documented on a batch history sheet and attached to the batch paper work.

10.3 If the sample has exceeded the hold time the analyst should contact the Group Leader before continuing with the batch.

10.4 Soil and filter matrices require no preservation and may be shipped in any suitable container.

11.0 SAMPLE PREPARATION

NOTE: Aliquots may be estimated by using the count time estimator spreadsheet.

11.1 Soil Sample Preparation:

11.1.1 If not already done, dry and homogenize the sample by performing GL-RAD-A-021.

11.1.2 Measure an appropriate aliquot of soil (usually 0.2 g to 1.0 g) in a glass container or digestion vessel. If required, the DUP, MS and MSD should be the same aliquot as the appropriate sample referenced. Record all aliquots. Add approximately 4 to 6 drops of iron carrier to the Blank and LCS beakers. The Blank and LCS aliquot should be recorded to be the same aliquot as the largest sample in the batch. For soils and other special matrices, such as vegetation, air filters, tissue, etc., Deionized water is a suitable matrix for use as the MB and LCS aliquot. Iron carrier may also be added to all samples in the batch, if necessary, based on the appearance and iron content of each sample. If Solid Reference Material is required, weigh out approximately 0.1 g into the LCS beaker and record the exact weight.

NOTE: Ins some cases, the matrix or activity of a sample will preclude taking a representative aliquot. In these cases, it may be necessary to prepare an aliquot, preform sample preparation steps, dilute to a known volume, and then taking an aliquot of the dilution. If this method is required proceed to Step 11.1.4.

11.1.3 Add a certified dpm of the appropriate tracer to each of the samples (usually between 5 to 10 dpm). Add a certified dpm (usually between 5 to 10 dpm) of the appropriate spike to the MS, MSD, LCS and LCSD as applicable. Reference the pull sheet for client requirements to determine appropriate tracer and spike.

11.1.3.1 For the determination of isotopic americium/curium, Am-243 is typically used as the tracer, and Am-241/Cm-244 are typically used as the spike.

11.1.3.2 For the determination of isotopic plutonium, Pu-242 is typically used as the tracer and Pu-239 is typically used as the spike. Pu-236 is an acceptable tracer provided no significant impurities are present.

NOTE: If Pu-241 is run in tandem, a separate MS and LCS is required to quantify Pu-241 spike recovery.

11.1.3.3 For the determination of isotopic uranium, U-232 is typically used as the tracer, and U-238 is typically used as the spike.

NOTE: The addition of tracers and spikes should be witnessed by either another analyst qualified on this procedure, a Team Leader or a Group Leader. After adding the tracers and spikes, the witness must initial and record the date of witnessing.

11.1.3.4 When running samples sequentially with Sr, all Sr carriers and spikes should be added prior to leach or digestion. See step 11.10.2 regarding collection procedures for Sr analysis.

11.1.4 If the analysis of the sample calls for quantification of U-232, Pu-242 or Am-243, the following steps shall be taken:

11.1.4.1 The sample will be run normally with the tracer indicated in sections 11.1.3 or 11.2.2.

11.1.4.2 A second run of the sample shall be made with a different tracer isotope such as U-236, Pu-236 or Cm-244. The quantification of the isotope that was normally the tracer can then be made. If there is any quantifiable activity a correction can be made to the initial run by calculating a correction ratio for the tracer recovery of the first run from the second run results.

NOTE: If prescribed to analyze by EPA method 3050B (Modified), proceed Appendix 7.

11.1.5 It is recommended that the samples be ashed in a muffle furnace as specified in GL-RAD-A-021B.

11.1.6 Review Client Special Requirements to determine the methodology for sample dissolution (leaching, HF total dissolution or fusion), or other special analytical needs. If leaching is required, proceed to Step 11.1.7. If HF total dissolution or fusion is required, proceed to GL-RAD-A-015 and then proceed to step 11.2.8.

NOTE: When refractory elements are suspected in the sample matrix, an HF total dissolution is needed. Many clients (for example, clients in the United Kingdom) will require total dissolution. The Client Special Requirements should reflect the proper analytical process.

11.1.7 A separate Am/Cm/Pu aliquot is treated with an aggressive acid leach of 6M or 9M hydrochloric acid depending on matrix and sample aliquot as described in the following steps. Uranium should not be run by this leaching technique.

NOTE: Determining the leaching routine is based on analyst experience with the matrix. The concentration required to obtain the leached sample will vary depending on the type of material, size of aliquot, muffling of sample, and other factors. The influence of these factors generally can be established by good judgment and experience with the materials being tested.

11.1.7.1 Place the sample in a beaker and add approximately 10 to 20mL of appropriate hydrochloric acid concentration per gram of sample with a minimum of 10 mL.

11.1.7.2 Heat the samples on medium heat and cover with a watch glass. Allow to leach for a minimum of 2 hours. Agitate the sample periodically to enhance the leaching process.

11.1.7.3 Allow the sample to partially cool and transfer to a centrifuge tube. Centrifuge the sample to separate the solid and leached portions.

11.1.7.4 Decant the leachate to a clean labeled beaker, and rinse the solid phase with DI water. Centrifuge the sample and decant the leachate into the beaker.

11.1.7.5 Evaporate the solution to dryness on medium heat.

11.1.7.6 Proceed to step 11.2.8.

11.2 Aqueous Sample Preparation:

11.2.1 Add an appropriate aliquot of sample to a labeled beaker. Prepare a Blank and LCS using DI water and a small amount of concentrated nitric acid to a pH < 2. The volume of DI water used should be the same as the largest volume of sample in the batch. If required, the DUP, MS and MSD should be the same aliquot as the appropriate sample referenced. Record all aliquots.

11.2.2 Add a certified dpm of the appropriate tracer to each of the samples (usually between 5 to 10 dpm). Add a certified dpm (usually between 5 to 10 dpm) of the appropriate spike to the MS, MSD, LCS and LCSD as applicable. Reference the pull sheet for client requirements to determine appropriate tracer and spike.

11.2.2.1 For the determination of isotopic americium/curium, Am-243 is typically used as the tracer, and Am-241/Cm-244 are typically used as the spike.

11.2.2.2 For the determination of isotopic plutonium, Pu-242 is typically used as the tracer, and Pu-239 is typically used as the spike. Pu-236 is an acceptable tracer, provided no significant impurities are present.

NOTE: If Pu-241 is run in tandem, a separate MS and LCS is required to quantify Pu-241 spike recovery.

11.2.2.3 For the determination of isotopic uranium, U-232 is typically used as the tracer, and U-238 is typically used as the spike.

11.2.2.4 When running samples sequentially with Sr, all Sr carriers and spikes should be added prior to initial iron precipitation to scavenge actinides. See section 11.10 regarding collection procedures for Sr analysis.

NOTE: The addition of tracers and spikes should be witnessed by either another analyst qualified on this procedure, a Team Leader or a Group Leader. After adding the tracers and spikes, the witness must initial and record the date of witnessing.

11.2.3 If the analysis of the sample calls for quantification of U-232, Pu-242 or Am-243, the following steps shall be taken:

11.2.3.1 The sample will be run normally with the tracer indicated in sections 11.1.3 or 11.2.2.

11.2.3.2 A second run of the sample shall be made with a different tracer isotope such as U-236, Pu-236 or Cm-244. The quantification of the isotope that was normally the tracer can then be made. If there is any quantifiable activity a correction can be made to the initial run by calculating a correction ratio for the tracer recovery of the first run from the second run results.

11.2.4 If samples contain large amounts of sediment that the client requires analyzed with the liquid portion of the sample, proceed to step 11.9.

NOTE: Other sample matrices, such as vegetation, air filters, tissue, etc. are prepared as outlined in GL-RAD-A-026. The analyst must ensure that the appropriate tracer(s) are added to these other matrices as discussed in sections 11.1.3 or 11.2.2.

11.2.5 Add 1 mL of iron carrier (10 mg/mL).

11.2.6 Add concentrated ammonium hydroxide until turbidity persists, or pH > 9. Then add approximately 2 mL in excess. Heat to boiling for approximately 10 minutes or until precipitate breaks into fine particles. Allow to settle and cool.

11.2.7 Decant excess supernate and discard. Collect the remaining precipitate by centrifugation in a 50 mL centrifuge tube and discard the supernate.

NOTE: Exercise care in this step because finely divided material that contains the actinides may also be present in addition to the large iron hydroxide flocks.

11.2.8 Dissolve the precipitate from step 11.2.7 or residue from step 11.1.6 or 11.1.7.5 in 10 to 15 mL of 9 M hydrochloric acid /0.04% hydrogen peroxide solution.

NOTE: If performing analysis of a diluted digestate, aliquot the appropriate aliquot(s) in to 50 mL centrifuge tube(s) and proceed to step 11.1.3 for tracing.

NOTE: Samples may be dissolved with 10 to 15 mL of 9 M hydrochloric acid and then add 1 drop of 30% hydrogen peroxide as an alternative to dissolving with 9 M hydrochloric acid /0.04% hydrogen peroxide. This may also be done by dissolving samples with 10 to 15 mL 9 M hydrochloric acid and adding approximately 1 mL of DI water to approximately 1 mL of 30% hydrogen peroxide, mixing and adding one drop to each sample.

NOTE: If uranium only is required, the load solution is 10 to 15 mL of 9 M hydrochloric acid.

11.2.9 Slurry AG 1x8 anion resin (Cl form 100-200 mesh) in a squirt bottle with DI water. Transfer the resin to a small column to obtain a settled resin bed of approximately 2.5 mL.

11.2.10 Condition the column with 10 mL of 9 M hydrochloric acid.

11.2.11 Pass the sample solution from step 11.2.8 through the column and collect the eluate in a labeled, disposable 50 mL centrifuge tube for americium/curium analysis.

11.2.12 Rinse the column with 5 mL of 9 M hydrochloric acid and collect with the americium/curium fraction. Proceed to step 11.3.

11.2.13 Rinse the column with an additional 15 mL of 9 M hydrochloric acid and collect in a drip pan for disposal.

11.2.14 Elute plutonium by adding 10 mL of 9 M hydrochloric acid /0.05 M ammonium iodide solution, catching the plutonium elution in a labeled, disposable 50 mL centrifuge tube. Proceed to step 11.5 for plutonium microprecipitation for alpha spectroscopy. This elution may be omitted if plutonium analysis is not required.

11.2.15 Rinse the column with 15 mL of 6 M hydrochloric acid /0.52 M hydrofluoric acid and collect in a drip pan for disposal.

11.2.16 Rinse the column with 5 mL of 6 M hydrochloric acid and collect in a drip pan for disposal.

11.2.17 Place a labeled, disposable 50 mL centrifuge tube under each column. Elute uranium from the column using 15 mL of 0.1 M hydrochloric acid. Proceed to step 11.6 for uranium microprecipitation for alpha spectroscopy.

11.3 Americium/Curium Separation via TRU Resin:

NOTE: If sample aliquot is small or liquid sample is clean and free of particulates continue with step 11.3.1 and TRU column work. If not, proceed to step 11.3.5 for additional clean-up steps and TRU column work.

11.3.1 Precondition a 2 mL TRU column with 5 mL of 9 M hydrochloric acid.

11.3.2 Pass the sample solution from step 11.2.12 through the column collecting in a drip pan for disposal.

11.3.3 Rinse the column with 5 mL of 9 M hydrochloric acid collecting in a drip pan for disposal.

11.3.4 Place a labeled, disposable 50 mL centrifuge tube under each column. Elute americium and curium from the column using 20 mL of 3 M hydrochloric acid. Proceed to step 11.4 Americium/Curium microprecipitation for alpha spectroscopy.

11.3.5 Add 0.5 mL of 1.25 M calcium nitrate to the elution from step 11.2.12.

11.3.6 Add 1.0 mL of phosphoric acid. Swirl to mix.

11.3.7 Dilute to approximately 30 mL with DI water.

11.3.8 Add 28-30% ammonium hydroxide to pH of 8 to 10 to precipitate calcium phosphate. Do not over precipitate.

11.3.9 Allow to cool then spin samples in a centrifuge and pour off supernate.

11.3.10 Add approximately 25 mL of DI water to centrifuge tube, cap, and shake vigorously to break up precipitate.

11.3.11 Spin samples in a centrifuge and pour off supernate.

11.3.12 Add 15 mL of 2 M nitric acid/1 M aluminum nitrate to centrifuge tube and dissolve precipitate. Gently heat if necessary. Solution should be clear.

11.3.13 Precondition a 2 mL TRU column with 10 mL of 2 M nitric acid, collecting the rinse in a drip pan for disposal.

11.3.14 Pass the sample solution from step 11.3.12 through the column, collecting the load solution in a drip pan for disposal.

11.3.15 Rinse the column twice with 5 mL of 2 M nitric acid and collect the rinse in a drip pan for disposal.

11.3.16 Rinse the column with 5 mL of 1 M nitric acid and collect the rinse in a drip pan for disposal.

11.3.17 Place a labeled, disposable 50 mL centrifuge tube under each column. Elute americium and curium from the column using 20 mL of 3 M hydrochloric acid.

If rare earth elements are suspected in the sample proceed to step 11.8.1 to separate rare earth elements via TEVA resin, otherwise, continue with step 11.4.

11.4 Americium/Curium Microprecipitation:

11.4.1 Dilute americium elution from step 11.3.4, 11.3.17, or 11.8.11 to approximately 40 mL with DI water. Add 0.1 mL of neodymium carrier (500 mg/L) to the solution and swirl to mix. Add 5 mL of 49% hydrofluoric acid and swirl to precipitate fluorides. Allow solution to sit for approximately 30 minutes, then proceed to step 11.7.1.

11.5 Plutonium Microprecipitation

11.5.1 Dilute plutonium elution from step 11.2.14 to approximately 40 mL with DI water. Add 0.1 mL of neodymium carrier (500 mg/L) and swirl. Add approximately 3 to 4 drops of 25% hydrazine dihydrochloride and swirl to mix. Let the solution sit for approximately 10 minutes, and add 5 mL of 49% hydrofluoric acid. Swirl to mix. Allow solution to sit for approximately 30 minutes, then proceed to step 11.7.1.

11.6 Uranium Microprecipitation:

11.6.1 Dilute uranium elution from step 11.2.17 to approximately 40 mL with DI water. Add 0.1 mL of neodymium carrier solution (500 mg/L) and swirl to mix. Add 0.5 mL of titanium (III) chloride solution and allow the sample to sit for approximately 30 seconds. Add 5 mL of 49% hydrofluoric acid to precipitate fluorides. Allow the solution to sit for approximately 30 minutes, then proceed to step 11.7.1.

11.7 Sample Filtration:

11.7.1 Place a disposable filter funnel on the filter support screen. Wet the filter with 80% ethyl alcohol and apply vacuum.

11.7.2 Add 5 mL of substrate suspension. After solution has passed through filter, add another 5 mL of substrate suspension.

11.7.3 Add 1 mL of the carbon colorant.

11.7.4 Filter the fluoride precipitated solution through the filter paper. Rinse the centrifuge tube with approximately 5 mL DI water and pass through filter.

11.7.5 Rinse the funnel with 80% ethyl alcohol.

CAUTION: Directing a stream of liquid onto the filter will disturb the distribution of the precipitate on the filter and render the sample unsuitable for alpha spectrometry resolution.

11.7.6 Without turning off the vacuum, remove the funnel.

11.7.7 Turn off vacuum and remove filter. Mount filter on a labeled 29 mm flat planchet. Ensure that the filter is centered and as flat as possible on the planchet.

NOTE: Care should be taken not to touch the active area of the filter with tweezers.

11.7.8 Place the mounted filter under a heat lamp for approximately 5 minutes or allow to air dry completely prior to alpha spectrometry measurement.

11.7.9 Submit samples for Alpha Spec counting.

NOTE: After Alpha Spec counting and review is complete, if Pu-241 analysis is required, proceed to SOP GL-RAD-A-035 step 11.2.30.

11.8 Separation of Americium from the Rare Earth Elements via TEVA Resin:

11.8.1 Transfer the elution from Step 11.3.17 to a clean beaker and add 0.3 mL of lanthanum. Gently cook dry on low heat.

11.8.2 Once the samples have cooled, add 5 mL of concentrated nitric acid and approximately 2 mL of 30% hydrogen peroxide. Heat on a hot plate at low heat to dryness. Cool and repeat.

11.8.3 Dissolve residue in approximately 1 ml of 0.1 M sulfuric acid. Evaporate until a very small amount of acid remains.

11.8.4 Dissolve residue in approximately 1 mL of concentrated formic acid. Evaporate until a very small amount of acid remains.

11.8.5 Repeat step 11.8.4, and evaporate the sample under low heat until the beaker is gently dry.

11.8.6 Dissolve the sample in 10 mL of 4 M ammonium thiocyanate/0.1 M formic acid. Be sure that the 4 M ammonium thiocyanate/0.1 M formic acid is prepared fresh daily.

11.8.7 Condition a TEVA column with 5 mL of 4 M ammonium thiocyanate/0.1 M formic acid, collecting the rinse in a drip pan for disposal.

11.8.8 Load the sample onto the TEVA column, collecting the load in a drip pan for disposal.

11.8.9 Rinse the beaker with 5 mL of 4 M ammonium thiocyanate/0.1 M formic acid and add to the column, collecting the rinse in a drip pan for disposal.

11.8.10 Rinse lanthanum and other rare earth elements from the column with 10 mL of 1.5 M ammonium thiocyanate/0.1 formic acid, collecting the rinse in a drip pan for disposal. Be sure that the 1.5 M ammonium thiocyanate/0.1 formic acid is prepared fresh daily.

11.8.11 Place a labeled, disposable 50 mL centrifuge tube under each column. Elute americium with 20 mL of 2 M hydrochloric acid.

11.8.12 Proceed to Step 11.4 to precipitate and filter samples.

11.9 Samples Containing Large Amounts of Sediment

NOTE: When aliquoting samples that contain large amounts of sediment, ensure that the sample is thoroughly homogenized.

11.9.1 Evaporate to dryness on medium to low heat.

11.9.2 Muffle in a furnace at approximately 550˚ C for a minimum of 2 hours.

11.9.3 If uranium analysis is required, leach for approximately 30 minutes and proceed to step 11.1.6.

11.9.4 If americium, curium, or plutonium analyses are required, proceed to step 11.1.7.

NOTE: Samples requiring americium extraction MUST be separated from rare earth elements via TEVA resin.

11.10 Preparation technique for Sr samples run in tandem with americium, plutonium, or uranium analysis.

11.10.1 Supernate from step 11.2.7 should be decanted into a clean, labeled beaker for further Sr analysis. Do not discard.

11.10.2 Elution from steps 11.2.11 and 11.2.12 should be collected in a clean, labeled centrifuge tube.

11.10.3 If americium/curium analysis is not required sample should be combined with supernate from step 11.10.1. Then proceed to appropriate SOP for Sr analysis.

11.10.4 If americium/curium analysis is needed proceed to step 11.3. Elution from steps 11.3.2 and 11.3.3 should be collected in a clean, labeled centrifuge tube. Elution should be combined with supernate from step 11.10.1. Then proceed to appropriate SOP for Sr analysis.

12.0 QUALITY CONTROL REQUIREMENTS

NOTE: Client contractual QC requirements override the requirements in this section.

12.1 Analyst and Method Verification Requirements

Refer to GL-RAD-D-002 for instructions concerning the validation of analytical methods.

12.2 Method Specific Quality Requirements

12.2.1 A method blank (MB) should accompany each batch of 20 or less samples. The reported value of the blank should be less than or equal to the contract required detection limit (CRDL).

12.2.2 The tracer added to all samples is used to calculate the method recovery. The method recovery of all samples should be between 15-125% when compared to the reference standard.

12.2.3 A duplicate (DUP) sample should be run with each batch of 20 or less samples. The relative percent difference (RPD) between the actual sample and the DUP should be less than or equal to 20% if both the sample and DUP results are greater than 5 times the minimal detectable concentration (MDC), or 100% if they are both less than 5 times MDC. If both results are less than the MDC, then limits are not applicable.

12.2.4 A laboratory control sample (LCS) should be run with each batch of 20 or less samples. The recovery of the LCS should fall between 75-125%.

12.3 Actions Required if the Quality Control Requirements Are Not Met

If any of the QC criteria from 12.2.1 through 12.2.4 cannot be satisfied, the analyst should inform the Group Leader and initiate a Data Exception Report (DER) as outlined in GL-QS-E-004.

13.0 INSTRUMENT CALIBRATION AND PERFORMANCE

For direction on calibration and instrument performance refer to GL-RAD-I-009.

14.0 ANALYSIS AND INSTRUMENT OPERATION

For analysis and instrument operation refer to GL-RAD-I-009.

15.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

For maintenance of system refer to GL-RAD-I-010.

16.0 DATA RECORDING, CALCULATION, AND REDUCTION METHODS

Data recording, calculation, and reduction take place in accordance with GL-RAD-D-003 and GL-RAD-D-006.

17.0 DATA REVIEW, APPROVAL, AND TRANSMITTAL

Refer to GL-RAD-D-003 for instructions concerning the data review process, approval, and transmittal.

18.0 RECORDS MANAGEMENT

Records generated as a result of this procedure are maintained as quality documents in accordance with GL-QS-E-008 for Quality Records Management and Disposition.

19.0 LABORATORY WASTE HANDLING AND WASTE DISPOSAL

Radioactive samples and material shall be handled and disposed of as outlined in the Laboratory Waste Management Plan, GL-LB-G-001.

20.0 REFERENCES

20.1 EPA Environmental Monitoring and Support Laboratory. Las Vegas. Radiochemical Analytical Procedures for Analysis of Environmental Samples. March 1979.

20.2 EML Procedures Manual HASL-300, Volume I February 2000, Method U-02-RC, Revision 1.

20.3 DOE Methods Manual for Evaluating Environmental and Waste Management Samples, 1997 Edition, RP800, "Sequential Separation of Americium and Plutonium by Extraction Chromatography."

20.4 Analytical Chemistry. Rapid Determination of Thorium-230 in Mill Tailings by α Spectrometry. UNC Geotech, Grand Junction Projects Office. Steve Donivan, Mark Hollenbach, and Mary Costello. Vol. 59, No. 21, 1987.

20.5 Los Alamos Health and Environmental Chemistry: Analytical Techniques. LA-10300-M Vol. 1, September 1987.

20.6 Special thanks to Dr. Bill Burnett and his associates for assistance in developing this method at Florida State University.

20.7 EML Procedures Manual HASL-300, Volume II February 1997, Method Am-05-RC.

20.8 U.S. Department of Energy, Environmental Measurements Laboratory Procedures Manual HASL-300, Section 4.5.4, Vol. 1, Pu-11-RC, 28th Ed., 1997

21.0 HISTORY

Revision 28; Updated procedure to include total dissolution for UK samples.
Revision 27: Removed reference to the use of Queue sheets
Revision 26: Added Appendix 7 to include steps of digestion of soil using EPA Method 3050B (Modified).
Revision 25: 1 mL of Titanium chloride to 0.5 mL in sample preparation section and checklist.
Revision 24: Updated Appendix 1. Combined steps 11.3.18 with step 11.3.17 for clarification.

APPENDIX 1

AMERICIUM, CURIUM, PLUTONIUM, AND URANIUM

Use a 2.5 cm^3 column with 1X8 anion resin (Cl^- form 100-200 mesh)

COLUMN WORK

____ 10 mL 9 M HCl (Conditioning)

____ Load solution: 10 to 15 mL 9 M HCl / 0.04% H_2O_2 (Catch in C-Tube for Am/Cm)
NOTE: If U only is required the load solution is 10 to 15 mL of 9 M HCl

____ 5 mL 9 M HCl (Catch in C-Tube for Am/Cm then proceed to Appendix 2 or 3 as appropriate for Am/Cm procedure)

____ 15 mL 9 M HCl (Rinse)

____ **Elute** Pu: 10 mL 9 M HCl / 0.05 M NH_4I (Catch in C-Tube then proceed to Appendix 4 Plutonium Precipitation)

____ 15 mL 6 M HCl / 0.52 M HF (Rinse)

____ 5 mL 6 M HCl (Rinse)

____ **Elute** U: 15 mL 0.1 M HCl (Catch in C-Tube)

____ Proceed to Appendix 4 for Uranium Precipitation

APPENDIX 2

AMERICIUM / CURIUM CONTINUATION

AMERICIUM / CURIUM

____ 0.5 mL 1.25 M Calcium nitrate

____ 1.0 mL Phosphoric acid and swirl

____ Dilute to approximately 30 mL with DI water

____ Concentrated NH_4OH to pH of 8 to 10

____ Centrifuge samples and pour off supernate

____ Add approximately 25 mL DI water and shake samples to break up precipitate

____ Centrifuge samples and pour off supernate

____ 10 mL 2 M HNO_3 (Condition 2 mL TRU Resin Column)

____ Load Solution: 15 mL 2 M HNO_3 / 1 M $Al(NO_3)_3$

____ 5 mL 2 M HNO_3 (Rinse)

____ 5 mL 2 M HNO_3 (Rinse)

____ 5 mL 1 M HNO_3 (Rinse)

____ **Elute** Am/Cm: 20 mL 3 M HCl (Catch in C-Tube)

____ Proceed to Appendix 4 for Am/Cm precipitation

APPENDIX 3

AMERICIUM / CURIUM CONTINUATION

AMERICIUM / CURIUM

___ 5 mL 9 M HCl (Condition 2 mL TRU Resin Column)

___ Load solution from Appendix 1 (catch in drip pan)

___ 5 mL 9 M HCl (Rinse)

___ **Elute** Am/Cm: 20 mL 3 M HCl (Catch in C-tube)

___ Proceed to Appendix 4 for Am/Cm precipitation

APPENDIX 4

AMERICIUM / CURIUM PRECIPITATION

____ Dilute elution with DI water to approximately 40 mL

____ 0.1 mL 500 mg/L Neodymium and swirl

____ 5 mL 49% HF and swirl

____ Wait approximately 30 minutes

____ Filter

PLUTONIUM PRECIPITATION

____ Dilute elution with DI water to approximately 40 mL

____ 0.1 mL 500 mg/L Neodymium and swirl

____ Approximately 3 to 4 drops 25% Hydrazine dihydrochloride and swirl

____ Wait approximately 10 minutes

____ 5 mL 49% HF and swirl

____ Wait approximately 30 minutes

____ Filter

URANIUM PRECIPITATION

____ Dilute elution with DI water to approximately 40 mL

____ 0.1 mL 500 mg/L Neodymium and swirl

____ 0.5 mL Titanium chloride and swirl

____ Wait approximately 30 seconds

____ 5 mL 49% HF and swirl

____ Wait approximately 30 minutes

____ Filter

APPENDIX 5

RARE EARTH CLEAN-UP

____ Transfer elution from TRU column to a clean beaker and add 0.3 ml of lanthanum

____ Evaporate to dryness on low heat

____ 5 mL concentrated HNO_3 and approximately 2 mL of 30% H_2O_2. Evaporate to dryness on low heat.

____ 5 mL concentrated HNO_3 and approximately 2 ml of 30% H_2O_2. Evaporate to dryness on low heat.

____ Approximately 1 mL of 0.1 M Sulfuric Acid. Evaporate to dryness on low heat

____ Approximately 1 mL of concentrated Formic Acid. Evaporate to dryness on low heat

____ Approximately 1 mL of concentrated Formic Acid. Evaporate to dryness on low heat

____ 5 mL 4 M Ammonium thiocyanate/0.1 M Formic acid (Condition 2 mL TEVA column)

____ Load Solution: 10 mL 4 M Ammonium thiocyanate/0.1 M Formic acid

____ 5 mL 4 M Ammonium thiocyanate/0.1 M Formic acid (Rinse)

____ 10 mL 1.5 M Ammonium thiocyanate/0.1 M Formic acid (Rinse)

____ **ELUTE** Am: 20 mL of 2 M HCl (catch in C-tube)

____ Proceed to Appendix 4 for Am/Cm precipitation

APPENDIX 6

SAMPLE CLEANUP FROM AN ALPHA SPEC FILTER

1. Remove the filter from the mounting disc by wetting the filter with a small amount of acetone and pulling the filter off the disc using tweezers. Place the filter in a labeled small glass beaker.

2. Add 4-6 drops of iron carrier (10 mg/L), 10 mL of concentrated hydrochloric acid and 1.0 mL of 5% boric acid solution.

3. Fill the bulb end of a disposable pipette with DI water and turn upside down. Place on the filter in the glass beaker to ensure the filter remains submerged.

4. Heat on a hot plate for 30 minutes frequently stirring the filter, flipping it over then back etc. Use the disposable pipette to stir and flip.

5. Remove the filter from the solution and rinse with DI water. Do this over the glass beaker so the DI water rinse falls back into the beaker.

6. Evaporate to dryness.

7. Proceed to section 11.2.8 and perform separations as specified.

APPENDIX 7

SPECIAL PROCEDURE: DIGESTION OF SOILS AND SEDIMENTS BY EPA METHOD 3050B (MODIFIED)

- Complete Steps 11.1.1 thru 11.1.4 of this procedure
- Add 5 mL of [HNO_3] and 5 mL of Type I DI water to the samples and quality control samples
- Gently swirl the sample and acid mixture
- Cover the sample with a reflux cap or watch glass and heat the sample in a hot block at 95° +/- 5° C. Reflux the sample for 10 to 15 minutes
- Remove the samples from the hot block and allow the samples to cool
- Add 5 mL of [HNO_3], return samples to hot block, replace the watch glass, reflux for 30 minutes. If brown fumes are generated, indicating oxidation of the sample by nitric acid, add an additional 5 mL [HNO_3] until no brown fumes are given off by the sample.
- Using a ribbed watch glass or reflux cap, allow the solution to evaporate to approximately 5 mL, without boiling, or heat for 2 hours.
- Remove the sample from the hot block and allow to cool
- Add 2 mL of Type I DI water and 3 mL of 30% H_2O_2, return samples to hot block and allow the peroxide reaction to occur. Continue to add H_2O_2 in 1 mL increments until effervescence subsides. Do not add more than 10 mL of H_2O_2.
- Cover the samples with ribbed watch glass or reflux cap and heat the samples at 95° +/- 5° C for 2 hours, without boiling.
- Remove from hot block and allow the samples to cool
- Cap the samples and shake well
- Filter each sample with a 2.0 µm pore size plunger type filter (PTF grade) or allow to settle overnight.
- Transfer liquid phase to clean labeled centrifuge tube
- Proceed to Step 11.2.6 of this procedure

VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE FOR THE ISOTOPIC DETERMINATION OF THORIUM

(GL-RAD-A-038 REVISION 18)

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1.0 STANDARD OPERATING PROCEDURE FOR THE ISOTOPIC DETERMINATION OF THORIUM

2.0 METHOD OBJECTIVE, PURPOSE, AND SUMMARY

2.1 This standard operating procedure provides the necessary instructions to conduct the analysis for isotopic thorium in a variety of matrices.

2.2 A soil/solid sample is aliquoted and digested, if necessary. The elements are then separated through ion exchange resins. For liquid samples, actinide elements are scavenged by coprecipitation with iron hydroxide. The precipitate is dissolved, and separation of elements is accomplished through ion exchange resins. The elements are then prepared for the measurement of radioactive isotopes by coprecipitation with neodymium fluoride. The neodymium fluoride precipitate is trapped on a filter, mounted on a metal disk and placed in a partially evacuated chamber for measurement of isotopic alpha emission.

2.3 GEL utilizes methods that are derived from established sources. This method is based on the source method from DOE EML Methods Manual HASL 300 PU-02, 03 and uses similar principles of radiochemical separation and counting. This method is also very similar in concept to the source method from the DOE Methods Manual for Evaluating Environmental and Waste Management Samples, 1997 Edition, RP800: "Sequential Separation of Americium and Plutonium by Extraction Chromatography." This method is also based on the source method EPA 053917 EMSL LV 1979 "Isotopic Determination of Plutonium, Uranium, and Thorium in Water, Soil, Air, and Biological Tissue" and in some cases this method is referenced as EPA RA-LV-PI.

2.4 This method has been modified on the basis of GEL's Performance Based Measurement System (PBMS).

3.0 METHOD APPLICABILITY

3.1 Method Detection Limit (MDL): Typical minimum detectable activity (MDA) for samples analyzed for thorium is 1 pCi/L or 1 pCi/g for all isotopes.

3.2 Analyst training records are maintained as quality records outlined in GL-QS-E-008. Analyst training and proficiency in the method is outlined in GL-QS-E-011 for Method Validation and Initial and Continuing Demonstrations of Capability.

3.3 Applicable matrices to this SOP are liquids, drinking water, vegetation, tissues, air filters, and solids.

NOTE: This method is not an EPA approved method for the analysis of drinking water.

4.0 METHOD VARIATIONS

Some variations may be necessary due to special matrices encountered in the lab. These

variations may be used with approval from a Group or Team Leader. Variations to a method will be documented with the analytical raw data.

5.0 DEFINITIONS

5.1 AlphaLIMS: GEL's Laboratory Information Management System.

5.2 Batch: Environmental samples that are prepared and/or analyzed together with the same process and personnel using the same lot(s) of reagents.

5.3 Deionized (DI) water: Type I water, Refer to GL-LB-E-016.

5.4 Laboratory Control Sample (LCS): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes from a source independent of the calibration standards or a material containing known and verified amounts of analytes.

5.5 Laboratory Duplicate (DUP): Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently.

5.6 Matrix Spike (MS): Prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available.

5.7 Matrix Spike Duplicate (MSD): A second replicate matrix spike is prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

5.8 Method Blank (MB): A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples containing an analyte of interest through all steps of the analytical procedures.

5.9 National Institute of Standards and Technology (NIST): For the purpose of this method, the national scientific body responsible for the standardization and acceptability of analyte solutions.

5.10 Solid Reference Material (SRM): A solid material containing known and verified amounts of analytes.

5.11 Tracer: A known quantity of a radioisotope that is added to each sample of a chemically equivalent radioisotope of unknown concentration so that the yield of the chemical separation can be calculated.

5.12 Refer to the SOP GL-QS-B-001 the Quality Assurance Plan for the additional lab-wide definitions.

6.0 INTERFERENCES

6.1 Internal tracer standards may have ingrown daughters that may interfere with the analysis. For example, Th-228 will be present in aged U-232 standard. This

problem is overcome by not performing thorium and uranium analysis in tandem.

6.2 Short-lived radioactive progeny may ingrow on prepared filters. For example, the Ra-224 alpha peak will be present if the Th-228 parent is present. These interferences are minimized by counting samples as soon as possible after separation chemistry.

7.0 SAFETY PRECAUTIONS AND WARNINGS

7.1 Personnel performing this analytical procedure are trained in and follow the safe laboratory practices outlined in the Safety, Health and Chemical Hygiene Plan, GL-LB-N-001.

7.2 Personnel handling radioactive materials are trained in and follow the procedures outlined in GL-RAD-S-004 for Radioactive Material Handling.

7.3 Personnel handling biological materials are trained in and follow the procedures outlined in GL-RAD-S-010 for The Handling of Biological Materials.

7.4 If there is any Question regarding the safety of any laboratory practice, **stop immediately,** and consult qualified senior personnel such as a Group or Team Leader.

8.0 APPARATUS, EQUIPMENT, AND INSTRUMENTATION

8.1 Apparatus and Equipment

8.1.1 Silicon surface barrier detectors with associated electronics, vacuum chambers, and data reduction capabilities

8.1.2 Vacuum pump and filtration rig

8.1.3 Disposable filter funnels (containing 25 mm filters with 0.1 µm pore size)

8.1.4 Metal disks, 29 mm diameter

8.1.5 Stainless steel tweezers

8.1.6 Polypropylene centrifuge tube (50 mL)

8.1.7 Sample drying and ashing apparatus

8.1.8 Sample homogenization apparatus

8.1.9 Analytical Grade 1X8 anion exchange resin, 100-200 mesh

8.1.10 Beakers (Glass and Teflon of various sizes)

8.1.11 2.5 cm^3 column

8.1.12 25 mL column funnel extension

8.1.13 Watch glasses (various sizes)

8.1.14 Hot plate

8.1.15 Pipettes

8.1.16 Balances

8.1.17 Disposable transfer pipettes

8.1.18 pH strips

9.0 REAGENTS AND STANDARDS

9.1 Reagents

9.1.1 Hydrogen Peroxide (30% H_2O_2)

9.1.2 Neodymium (500 mg/L Nd)

9.1.3 Neodymium (10,000 mg/L Nd)

9.1.4 Ethyl alcohol (80% EtOH): Dilute 400 mL ethanol to 500 mL with DI water.

9.1.5 Hydrochloric Acid, concentrated (12 M HCl)

9.1.6 Hydrofluoric Acid, concentrated (49% HF)

9.1.7 Iron Carrier (10 mg/mL): Dissolve 62.7 g of $Fe(NO_3)_3 \cdot 6H_2O$ or 72.3 g $Fe(NO_3)_3 \cdot 9H_2O$ in 800 mL DI water and dilute to 1 L with DI water.

9.1.8 Hydrochloric Acid (9 M HCl): Add 750 mL of concentrated hydrochloric acid to 100 mL of DI water. Allow to cool and dilute to 1 L with DI water.

9.1.9 Hydrochloric Acid (2 M HCl): Add 167 mL of concentrated hydrochloric acid to 500 mL of DI water. Allow to cool and dilute to 1 L with DI water.

9.1.10 Nitric Acid, concentrated (16 M HNO_3)

9.1.11 Nitric Acid (8 M HNO_3): Add 500 mL concentrated nitric acid to 500 mL DI water.

9.1.12 Cellulose Nitrate Membrane filters (47 mm)

9.1.13 Substrate Suspension: Dilute 4 mL of neodymium (10,000 mg/L) and 80 mL concentrated hydrochloric acid to 1500 mL with DI water. Add, while stirring, 40 mL of 49% hydrofluoric acid and dilute to 2 L with DI water.

9.1.14 Sulfuric Acid, concentrated (36 M H_2SO_4)

9.1.15 Ammonium Hydroxide, concentrated (28-30% NH_4OH)

9.1.16 Saturated Boric Acid, 5%: Dissolve 50 g of H_3BO_3 per liter of DI water.

9.2 Standards

9.2.1 NIST traceable standards: Th-229, Th-230, Th-232, Ac-227.

9.2.2 Refer to GL-RAD-M-001.

10.0 SAMPLE HANDLING AND PRESERVATION

10.1 Liquid samples should be preserved to pH < 2 with concentrated nitric acid and collected in a plastic bottle.

10.2 Before beginning an analysis, the analyst should check the sample pH by removing a minimal amount of sample with a transfer pipette and placing it on a pH strip. DO NOT insert pH strip into sample container. If the sample is received with a pH > 2, the analyst should contact the Group Leader or Team Leader. If approved by the client, the analyst should adjust the pH with concentrated nitric acid to a pH < 2. If the sample pH is adjusted, let the sample sit in the original container for a minimum of 24 hours before analysis. This acidification should be documented on a batch history sheet and attached to the batch paperwork.

10.3 If the sample has exceeded the hold time, the analyst should contact the Group Leader before continuing with the batch.

10.4 Soil samples require no preservation and may be shipped in any suitable container.

11.0 SAMPLE PREPARATION

NOTE: Aliquots may be estimated by using the count time estimator.

11.1 Soil/Solids Sample Preparation

11.1.1 If not already done, dry and homogenize the sample by performing GL-RAD-A-021.

11.1.2 Measure an appropriate aliquot of soil/solids (usually 0.2 g to 1.0 g) in a suitable container (glass or Teflon beaker). If required, the DUP, MS and MSD should be the same aliquot as the appropriate sample referenced on the sheet. Record all aliquots. Add approximately 4 to 6 drops of iron carrier to the MB and LCS beakers. The MB and LCS aliquots should be recorded to be the same aliquot as the largest sample in the batch. Iron carrier may also be added to all samples in the batch, if necessary, based on the appearance and iron content of each sample. If solid reference material is required, weigh out approximately 0.1 g into the LCS beaker and record the exact weight.

11.1.3 Add a certified dpm of the appropriate tracer to each of the samples (usually between 5 to 10 dpm). Add a certified dpm (usually between 5 to 10 dpm) of the appropriate spike to the MS, MSD, LCS and LCSD as applicable. Reference the batch pull sheet for client requirements to determine appropriate tracer and spike.

11.1.3.1 For the determination of isotopic thorium, Th-229 is typically used as the tracer, and Th-232 is typically used as the spike.

NOTE: The addition of tracers and spikes should be witnessed by either another analyst qualified on this procedure, a Team Leader or a Group Leader. After adding the tracers and spikes, the witness must initial and record the date of witnessing.

11.1.4 If samples are to be place in a muffle furnace (recommended for miscellaneous solid samples), samples shall be in a glass beaker (not Teflon). Refer to GL-RAD-A021B for instructions.

11.1.5 For thorium analysis, digest as specified in GL-RAD-A-015.

11.1.6 Proceed to step 11.2.6.

11.2 Aqueous Sample Preparation

11.2.1 Add an appropriate aliquot of sample to a labeled beaker. Prepare a MB and LCS using DI water and a small amount of concentrated nitric acid to a pH < 2. The volume of DI water used should be the same as the largest volume of sample in the batch. If required, the DUP, MS and MSD should be the same aliquot as the appropriate sample referenced. Record all aliquots.

11.2.2 Add a certified dpm of the appropriate tracer to each of the samples

(usually between 5 to 10 dpm). Add a certified dpm (usually between 5 to 10 dpm) of the appropriate spike to the MS, MSD, LCS and LCSD as applicable. Reference the batch pull sheet for client requirements to determine appropriate tracer and spike.

11.2.2.1 For the determination of isotopic thorium, Th-229 is typically used as the tracer, and Th-232 is typically used as the spike.

NOTE: The addition of tracers and spikes should be witnessed by either another analyst qualified on this procedure, a Team Leader or a Group Leader. After adding the tracers and spikes, the witness must initial and record the date of witnessing.

NOTE: Other sample matrices, such as vegetation, air filters, tissue, etc., are prepared as outlined in GL-RAD-A-026. The analyst must ensure that the appropriate tracer(s) is added to these other matrices as discussed in section 11.1.3 and 11.2.2.

NOTE: If samples contain large amounts of sediment and client requires inclusion with the liquid portion of the sample, proceed to section 11.3.

11.2.3 Add 1 mL of iron carrier (10 mg/mL).

11.2.4 Add concentrated ammonium hydroxide until turbidity persists, or the pH > 9. Add approximately 2 mL in excess. Heat to boiling for approximately 10 minutes or until precipitate breaks into fine particles. Allow to settle and cool.

11.2.5 Decant excess supernate and discard. Collect the remaining precipitate by centrifugation in a 50 mL centrifuge tube and discard supernate.

NOTE: Exercise care in this step because finely divided material that contains the actinides may also be present in addition to large iron hydroxide flocks.

11.2.6 Dissolve the precipitate from step 11.2.5 or residue from step 11.1.6 in 10 to 15 mL of 8 M nitric acid.

NOTE: If all of the precipitated iron solids do not go into solution, it may be necessary to place samples in a hot water bath for approximately 10-15 minutes before proceeding to column work.

11.2.7 Slurry Analytical Grade 1X8 anion exchange resin in DI water. Transfer the resin to a small column to obtain a settled resin bed of approximately 2.5 mL.

11.2.8 Condition the column with 15 mL of 8 M nitric acid.

11.2.9 Pass the solution from step 11.2.6 through the column, and collect in a

drip pan for disposal.

NOTE: If the samples were traced with Ac-227, annotate the separation date and time when the load solution has passed through the column.

11.2.10 Rinse the column with 5 mL of 8 M nitric acid, and collect in a drip pan for disposal.

11.2.11 Rinse the column with 15 mL of 8 M nitric acid, and collect in a drip pan for disposal.

11.2.12 Elute the thorium from the column by adding 20 mL of 9 M hydrochloric acid, collecting the elution in a labeled, disposable centrifuge tube.

11.2.13 Transfer the sample to a beaker using DI water. Add approximately 4 to 6 drops of iron carrier. Evaporate the sample to dryness on medium heat.

11.2.14 Add 10 mL of concentrated nitric acid and slowly add approximately 2 to 3 mL hydrogen peroxide. Evaporate samples to dryness on medium heat.

11.2.15 Add 2 mL of concentrated hydrochloric acid and evaporate samples to dryness on medium heat.

11.2.16 Dissolve the residue with 4 mL of 2 M hydrochloric acid and transfer the sample to a clean centrifuge tube using DI water.

11.2.17 Add 0.1 mL of 500 mg/L neodymium and swirl. Add 2.0 mL of 49% hydrofluoric acid and swirl. Wait approximately 30 minutes to allow fluorides to coprecipitate with thorium.

11.2.18 Place the disposable filter funnel on the filter rig and apply vacuum.

11.2.19 Rinse the funnel with 80% ethyl alcohol.

11.2.20 Add 5 mL substrate suspension. After solution has passed through filter, add another 5 mL of substrate suspension.

11.2.21 Filter the fluoride precipitated solution through the filter paper. Rinse the centrifuge tube with approximately 5 mL DI water and pass through filter.

11.2.22 Rinse the funnel with 80% ethyl alcohol.

CAUTION: Directing a stream of liquid onto the filter will disturb the distribution of the precipitate on the filter and render the sample unsuitable for alpha spectrometry resolution.

11.2.23 Without turning off the vacuum, remove the funnel.

11.2.24 Turn off vacuum and remove filter. Mount filter on a labeled 29 mm flat planchet. Ensure that the filter is centered and as flat as possible on the planchet.

NOTE: Care should be taken not to touch the active area of the filter with tweezers.

11.2.25 Place the mounted filter under a heated lamp to dry (usually for 10-20 minutes).

11.2.26 Submit samples for alpha spectrometer counting.

11.3 Samples Containing Large Amounts of Sediment:

NOTE: When aliquoting samples that contain large amounts of sediment, ensure sample is thoroughly homogenized.

11.3.1 Dry on medium to low heat.

11.3.2 Muffle in a furnace at approximately 550° C for a minimum of 2 hours.

11.3.3 Proceed to step 11.1.5.

12.0 QUALITY CONTROL SAMPLES AND REQUIREMENTS

NOTE: Client contractual QC requirements override the requirements in this section.

12.1 Method Verification Requirements

Refer to GL-RAD-D-002 for instructions concerning the validation of analytical methods.

12.2 Method Specific Quality Requirements

12.3 Actions Required if the Quality Control Requirements Are Not Met

If any of the QC criteria from 12.2.1 through 12.2.4 cannot be satisfied, the analyst should inform the Group Leader and initiate a Data Exception Report as outlined in GL-QS-E-004.

13.0 INSTRUMENT CALIBRATION AND PERFORMANCE

For direction on calibration and instrument performance, refer to GL-RAD-I-009.

14.0 ANALYSIS AND INSTRUMENT OPERATION

For analysis and instrument operation, refer to GL-RAD-I-009.

15.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

For maintenance of system, refer to GL-RAD-I-010.

16.0 DATA RECORDING, CALCULATION, AND REDUCTION METHODS

Data recording, calculation, and reduction take place in accordance with GL-RAD-D-003 and GL-RAD-D-006.

17.0 DATA REVIEW, APPROVAL, AND TRANSMITTAL

Refer to GL-RAD-D-003 for instructions concerning the data review process, approval, and transmittal.

18.0 RECORDS MANAGEMENT

Records generated as a result of this procedure are maintained as quality documents in accordance with GL-QS-E-008 for Quality Records Management and Disposition.

19.0 LABORATORY WASTE HANDLING AND WASTE DISPOSAL

Radioactive samples and material are disposed as outlined in the Laboratory Waste Management Plan, GL-LB-G-001.

20.0 REFERENCES

20.1 EPA Environmental Monitoring and Support Laboratory, Las Vegas. Radiochemical Analytical Procedures for Analysis of Environmental Samples. March 1979.

20.2 EML Procedures Manual HASL-300, 1982.

20.3 Analytical Chemistry. Rapid Determination of Th-230 in Mill Tailings by Alpha Spectroscopy. UNC Geotech, Grand Junction Projects Office. Steve Donivan, Mark Hollenbach, and Mary Costello. Vol. 59, No. 21, 1987.

20.4 Los Alamos Health and Environmental Chemistry: Analytical TechniQueues. LA-10300-M Vol. 1, September 1987.

20.5 Special thanks to Dr. Bill Burnett and his associates for assistance in developing this method at Florida State University.

20.6 U.S. DOE RP 800:, Methods for Evaluating Environmental and Waste Management Samples, “Sequential Separation of Americium and Plutonium by Extraction Chromatography”, 1997.

20.7 U.S. EPA, EMSL LV 053917 (EPA RA-LV-PI), “ Isotopic Determination of Plutonium, Uranium, and Thorium in Water, Soil, Air, and Biological Tissue”, 1979.

21.0 HISTORY

Revision 14: Added section 2.3 and references for certification.

Revision 15: Updated reference to calculation SOP and DI water.

Revision 16: Technical updates for SOP consistency as part of annual review. Added Appendix 3 regarding clean up steps.

Revision 17: Updated SOP to current practices.

Revision 18: Remove the reference to queue sheets.

APPENDIX 1

THORIUM

Use a 2.5 cm^3 column with 1 x 8 anion resin (Cl^- form 100-200 mesh)

_____ 15 mL 8 M HNO_3 (Condition Column)
_____ 10–15 mL 8 M HNO_3 (Load)
_____ 5 mL 8 M HNO_3 (Rinse)
_____ 15 mL 8 M HNO_3 (Rinse)
_____ **Elute Th:** 20 mL 9 M HCl (Catch in C-Tube)
_____ Transfer to a clean beaker. Add approximately 4-6 drops of Fe carrier and evaporate to dryness on medium heat
_____ 10 mL of concentrated HNO_3 and approximately 2–3 mL H_2O_2. Evaporate to dryness on medium heat.
_____ 2 mL of concentrated HCl and evaporate to dryness on medium heat.
_____ Dissolve with 4 mL of 2 M HCl and transfer to centrifuge tube with DI water.
_____ Proceed to Appendix 2 for thorium precipitation

APPENDIX 2

THORIUM PRECIPITATION

_____ 0.1 mL 500mg/L neodymium and swirl

_____ 2.0 mL 49% HF and swirl

_____ Wait approximately 30 minutes

_____ Filter

APPENDIX 3

SAMPLE CLEANUP FROM AN ALPHA SPEC FILTER

1. Remove the filter from the mounting disc by wetting the filter with a small amount of acetone and pulling the filter off the disk using tweezers. Place the filter in a labeled glass beaker.
2. Add 4-6 drops of iron carrier (10 mg/L), 10 mL of concentrated hydrochloric acid and 1.0 mL of 5% boric acid solution.
3. Fill the bulb end of a disposable pipette with DI water and turn upside down. Place on the filter in the glass beaker to ensure the filter remains submerged.
4. Heat on a hot plate for 30 minutes frequently stirring the filter, flipping it over then back, etc. Use the disposable pipette to stir and flip.
5. Remove the filter from the solution and rinse with 9M HCl. Do this over the glass beaker so the 9M HCl rinse falls back into the beaker.
6. Evaporate to dryness.
7. Convert with concentrated Nitric Acid and evaporate to dryness.
8. Proceed to section 11.2.6 and perform separations as specified.

VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE

FOR

DATA REVIEW, VALIDATION, AND DATA PACKAGE ASSEMBLY

(GL-RAD-D-003 REVISION 44)

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1.0 STANDARD OPERATING PROCEDURE FOR DATA REVIEW, VALIDATION, AND DATA PACKAGE ASSEMBLY

2.0 PURPOSE

This operating procedure is specific to Radiochemistry data reduction, review of analytical results, and assembling data packages.

3.0 DISCUSSION

3.1 Radiochemistry data review is broken into two levels of review for laboratory analysis. The first level (data review) and second level (data review, validation, and technical review) are conducted by separate data reviewers. In addition, at least 10% of batches within each analytical group are reviewed by the respective group leader or designee.

3.2 Data packages are assembled as required by the client. A checklist is generated in AlphaLIMS based on the client setup. The package is then reviewed by the group leader or designee for completeness and to ensure that the method and sample quality control meet the client requirements.

4.0 DEFINITIONS

4.1 Abundance (Photon Intensity): The value, usually expressed in percent, given to a photon of specific energy that is emitted during the decay of a radionuclide. The abundance represents the probability of emission of a specific energy photon when a radionuclide is decaying.

4.2 Abundance Test: The test where the gamma software verifies the presence of 75% of the total abundance of a nuclide in the system library is present. The presence of greater than 75% of the total abundance will cause a nuclide to be identified. The abundance criteria may be reduced to less than 75% for nuclides with several lower abundant photons.

4.3 Counting Uncertainty: The error of the reported result due to the counting statistics of the instrument used for qualification.

4.4 Energy Tolerance: The test where the gamma software checks the energy line in the spectrum to see if it is within the energy tolerance setting. (The standard setting is 1.5 keV.) If it is within this setting, then the line is associated with that nuclide. The energy line can be associated with more than one nuclide.

4.5 False Positive: An isotope that has failed one or more of several tests including half-life, abundance, and energy tolerance.

4.6 Half-Life Test: This is when the software determines that a set number of half-lives, typically 10, have passed. The software considers the nuclide to have effectively decayed away and generates a 0 MDC. This is calculated by taking the amount of time elapsed between the collect date and the time at the mid-point of the sample count. One half-life is equal to the time it takes for ½ of the nuclide to decay away.

4.7 Key Line: The line chosen by the builder of the library to be the prominent line of the

isotope. This line is used in the gamma MDC table for purposes of calculating activity, error and MDC. For non-identified gamma isotopes the key line energy is used as the basis of determining the region used to calculate the activity, error, and the MDC. Usually this line is the most abundant line on a line that is relatively free from interference.

4.8 Organics Forms Link: "Organics_deliverable_pkg.create_sdg" This link is accessed through the following AlphaLIMS links: Hard Copy Data Deliverables, Forms Generation, ORGANICS, Organics forms or Production Reports, Fraction Work Order Status (With Status) link. Select RAD or RAD/REMP as a Fraction and submit. Select the folder under the Drev column next to the desired work order.

4.9 Positively Identified Nuclide: A Nuclide that must be quantified using its own energy line. For a nuclide to be positively identified, no assumption of equilibrium may be made.

4.10 Spectral Library: Also known as system library or reference library. Based on Evaluated Nuclear Structure Data Files (ENSDF) maintained by the National Nuclear Data Center (NNDC) at Brookhaven National Laboratories. These files are viewed using NuDat 2. NuDat 2 is a software product that allows users to search and plot nuclear structure and nuclear decay data interactively. "Radioactive Decay Data Tables", D.C. Kocher (1981) is another source of nuclear decay data that is used when information is not available in ENSDF.

4.11 Summation Interference: When high gamma emission rates are seen, sample summation can occur. Prominent in geometries close to detection and in low energy range (i.e., 10,000 gps (gammas per second) at 88 keV, 15,000 gps at 210 keV), a summation interference can be seen at 88 + 88 = 176 keV, 210 + 210 = 420 keV, 210 + 88 = 298 keV. Frequently referenced as a summation peak.

4.12 Virtual Data: Refers to data that are not printed out, but rather, available to be viewed electronically.

4.13 Virtual Packaging/Virtual Building/Virtual Capture: A process by which AlphaLIMS compiles files necessary to assembling a client's data package or a raw data batch.

4.14 Combined Standard Uncertainty (CSU): Represents the estimated standard deviation of the result. This is synonymous with the 1-sigma Total Propagated Uncertainty (TPU).

4.15 Refer to GL-QS-B-001 the Quality Assurance Plan for additional lab-wide used definitions.

5.0 PROCEDURES

NOTE: Refer to GL-RAD-B-026 for instructions for ICP-MS.

5.1 Gas Flow, LSC and Lucas Cell Data Reduction

5.1.1 Open Microsoft Excel and click on the spreadsheet icon. You will then be prompted for the batch number. You will need to have \\gelsan\sdrive mapped

on your computer.

5.1.2 After the batch number is entered, you will be asked "what type of spreadsheet would you like to make?" The macro that creates the spreadsheets will reference AlphaLIMS for the test method, the matrix, the RDL units, client ID, TPU requirements, client detection limits, and RER/DER equation required. Select the machine that the samples were counted on, and click ok. Proceed to the appropriate section depending upon sample type.

5.1.3 Gas Flow

5.1.3.1 The next screen will ask you to select the GFPC raw data files. Type in the batch number at the prompt. Double click on the batch number to open the file and click cancel when done.

5.1.3.2 The raw data will show up and may have duplicate sample IDs or sample IDs that are not in the batch. If there are duplicate sample IDs for the batch, use the one with the most recent count start time and/or the longest count time. Sort data so sample IDs are in the same order as in the batch. Next click the button to continue making the spreadsheet.

5.1.3.3 The screen will ask you to select a filename to save the raw data. The raw data should be saved as the batch number. Click save.

5.1.3.4 On the next screen, you will have to enter the RDLs, the spike code and volume for both the MS and LCS. Afterwards, click ok and the spreadsheet will be generated.

5.1.3.5 Click the upload button to send the data to AlphaLIMS.

NOTE: (For Gas Flow Beta only analysis) When an alpha count rate of greater than 1 cpm (count per minute) is present in conjunction with beta sample activity greater than MDA, data will be evaluated for possible alpha interference and sample preparation may need to be returned to the lab for additional clean-steps to remove potential alpha interference before recounting the sample.

5.1.3.6 Proceed to step 5.4 to complete Level 1 data review.

5.1.4 LSC

5.1.4.1 Select the raw data file for Beckman instruments by typing in U, the user number, month, day, and rack number; for Quantalus instruments, type U followed by the batch number; and for Wallac instruments, type in the file number beginning with P. This information can be found on the LSC raw data. Double click on the file to open. If there is more than one raw data printout, repeat this step. After all of the raw data have been opened, click cancel.

5.1.4.2 Click continue to proceed.

5.1.4.3 The next screen will ask you to put in the analyst, prep date, aliquot, and the spike codes and volumes for the MS and LCS. All of this can be found on the electronic logbook page and should pull in automatically from AlphaLIMS. In the rack column, make sure that the right rack number is with the correct sample ID. Once data is entered and double checked, click ok to proceed.

5.1.4.4 Review the instrument generated raw data spectra. The target analyte will produce a peak between two specific channels. The channel range can be found on the LSC raw data printout. Check to confirm that the peak on the raw data spectrum corresponds with the known peak for that analyte. Check the spectrum for lumex interference. If the lumex percentage is greater than 15% and the raw data spectrum confirms the presence of an analyte, recount the sample. If the lumex percentage is greater than 15% and the raw data spectrum does not confirm the presence of the analyte, consult your group leader or designee.

5.1.4.5 Click the upload button to send the data to AlphaLIMS.

5.1.4.6 Proceed to step 5.4 to complete Level 1 data review.

5.1.5 Lucas Cell

5.1.5.1 On the next screen, enter the analyst, prep date, spike code and volume, cell, and aliquot. This information is found on the Queue sheet or electronic logbook. Click ok when finished.

5.1.5.2 The date and time screen with show up next. Enter the degas date/time, the ingrowth date/time, and the count date/time, located on the Queue sheet or electronic logbook. Click ok when finished, and the spreadsheet will be generated.

5.1.5.3 Excel will then tell you to enter the background counts and gross counts directly onto the spreadsheet. This is also found on the Queue sheet or electronic logbook. After this is entered, the spreadsheet is complete.

5.1.5.4 Click the upload button to send the data to AlphaLIMS.

5.1.5.5 Proceed to step 5.4 to complete Level 1 data review.

5.2 Alphaspec Data Reduction

5.2.1 Check special requirements on the batch pull sheet.

5.2.2 Transcription check (from Queue sheet or electronic logbook to Alpha GEL system):

5.2.2.1 Follow 15.4 of GL-RAD-I-009 for processing, correcting transcription errors, and uploading in Alpha GEL system.

5.2.2.2 If the batch analytical SOP needs to be changed prior to processing and uploading (i.e. the SOP selected in Alpha GEL doesn't match the one circled on the batch Queue sheet or electronic logbook):

5.2.2.2.1 Find the batch in the "RASP" Queue ("RADH" Queue for high rad samples) in AlphaLIMS. Click on the "Specific Data Entry" link for the desired batch and then click on the "Entry" link. Next to the SOP ID, select the desired SOP, and hit "Submit". Proceed to the "Select Batch to Review" page in AlphaLIMS for the batch. Click on "Edit batch" and hit "Submit". In Alpha GEL, click on "Tools" and then "Clear cached LIMS data". Follow 15.4 of GL-RAD-I-009 and verify that SOP was updated in Alpha GEL prior to uploading.

5.2.3 Check for proper ROI settings-ROI includes all area and no peak tailing outside ROI.

5.2.3.1 The alpha system processes isotopic results using fixed Regions of Interest (ROIs). Whenever practical, the low and high energy markers are set to include all alpha energies associated with the particular isotope, and the isotopic abundance is set in the nuclide ROI. Each spectrum should be reviewed to ensure that all area is included within the ROI on both the high and low end of the peak. If necessary, with Group Leader or designee approval, peaks may be integrated. If peaks are integrated, refer to reference library, adjust isotopic abundance as necessary, comment in the case narrative, and include both original and integrated spectra in the raw data.

5.2.3.2 The Peak Resolution (FWHM) and Peak Centroid are dependent on the location of the high and low energy markers. Proper integration must be completed before continuing to step 5.2.4.

5.2.4 Check for peak shifts.

5.2.4.1 The tracer peak should be ± 50 keV from the expected energy of the tracer isotope. The expected energy for the tracer isotope can be located on each virtual spectrum under the "ENERGY" column in the "NUCLIDE ACTIVITY SUMMARY" section.

5.2.5 Check for resolution.

5.2.5.1 The tracer peak Full Width Half Maximum (FWHM) should be equal to or less than 100 keV for all tracer isotopes except

Th-229 and Ac-227. Broader peak FWHM values may be acceptable if the complete peak area falls within the ROI and there is not excessive peak tailing.

5.2.6 Proceed to step 5.4 to complete Level I data review.

5.3 Gamma Data Reduction

5.3.1 Refer to Appendix 5 for a summary of the assumptions used in generating the gamma libraries. Additional modifications can be made to client specific libraries based on client specifications.

5.3.2 Transcription check (from Queue sheet to Alpha System):

5.3.2.1 Type ED "BATCH#".DAT

5.3.2.1.1 Use PF4 (or minus key on number pad) to add or remove desired sample IDs in the .DAT file

5.3.2.1.2 CTRL/Z ex to exit and save changes

5.3.2.1.3 CTRL/Z quit to quit without saving changes

5.3.2.2 Type @BATCH_PROCESS "BATCH#" or PROC "BATCH#"

5.3.2.2.1 Follow Prompts

5.3.2.3 Sample data may be reprocessed using the following:

5.3.2.3.1 Type COPY "BATCH#".DAT GAMMA.DAT

5.3.2.3.2 Type ED GAMMA.DAT

5.3.2.3.3 Use PF4 (minus key on number pad) to add or remove sample IDs in the .DAT file

5.3.2.3.4 CTRL/Z ex to exit and save changes

5.3.2.3.5 CTRL/Z quit to quit without saving changes

5.3.2.3.6 Type @GAMMA_REPROCESS and follow prompts

5.3.3 Upload to AlphaLIMS (two options)

5.3.3.1 Continue @BATCH_PROCESS or PROC "BATCH#" and follow prompts or

5.3.3.2 @HORIZON_XFER

5.3.3.2.1 Type COPY "BATCH#".DAT UPLOAD.DAT and edit if necessary

5.3.3.2.2 TYPE @HORIZON_XFER and follow prompts

5.3.4 Check for peak shifts (two options)

5.3.4.1 Continue @BATCH_PROCESS or PROC "BATCH#"

5.3.4.2 Check for peak shifts once the virtual data has been uploaded.

5.3.5 The following criteria are used to accept a reported gamma isotope from the

Results Greater Than MDC reports.

5.3.5.1 The peak FWHM should be less than 3 keV (Refer to 5.3.9.4 for additional information).

5.3.5.2 The energy tolerance should be less than 1.5 keV, but can be raised to 2.0 keV with Team Leader or Group Leader approval.

5.3.5.3 The sensitivity setting should be between 3 and 15. The default setting is 3.

5.3.5.4 The confidence level setting should be 5.

5.3.5.5 These settings should not be changed without approval from a group leader.

NOTE: The following steps may be accomplished in the AlphaLIMS link "Select Batch to Review" unless special instructions are included.

5.3.6 Make sure calculations have run.

5.3.7 Calculate nominal concentrations and recoveries for MS and LCS.

5.3.8 Check to ensure that duplication criteria have been met.

5.3.9 Run Results Greater Than MDC Reports and Failed Detection Limit Reports and review to ensure detection limits met and qualify rejected nuclides. Below are typical comments and explanations for rejected nuclides.

5.3.9.1 "Results are considered a false positive due to low abundance." - This is used when the radionuclide does not meet the identification criteria.

NOTE: If the key-line is not present, the software will generate the activity using a forced-activity calculation. In this case the result will always be qualified. If the key-line is present, the result generated is accurate and may be reported without qualification.

5.3.9.2 "Results are considered a false positive due to interference." - Any time there is significant interference from another radionuclide, that has not been corrected by the software, we use this qualifier narrative to reject the result.

5.3.9.3 "Results are considered a false positive due to a short half-life." - Any time 10 half-lives have expired, the software again goes through a forced activity calculation, in this instance, and there should be a 0 value for the MDC as well.

5.3.9.4 "Results are considered a false positive due to high peak width." Any time, a high full-width, half-maximum (FWHM) is recorded (usually greater than 3.0 keV, but can be up to the Gamma analyst's discretion

if a FWHM greater than 3.0 keV can be acceptable) this qualifier narrative is used.

5.3.9.5 "Results are considered a false positive due to a high counting uncertainty." - This narrative comment is used any time the percent error for the peak is greater than 50 percent. It can also be used when the percent error is between 35-50 percent and supporting evidence indicates it is likely the nuclide is not present. One of the most common forms of evidence is the presence or absence of other radionuclides in the same decay chain. If a sample has undergone interference correction, use the interference corrected line activity report. If the percent error is at 2-sigma, the gray area is 70-100% with automatic rejection above 100%; if the percent error is 1-sigma, the gray area is 35% -50% with automatic rejection above 50%.

5.3.9.6 "Results are considered a false positive due to no valid peak. "This narrative comment is used when analyzing the spectrum; the Gamma analyst finds evidence that the software did not correctly identify the peak.

5.3.9.7 "Results are considered a false positive due to an invalid assumption of equilibrium." This comment is used, particularly for Th-230 and U-234, when there is evidence that secular equilibrium does not exist between the nuclide being quantified and the nuclide used to quantify.

5.3.10 The following guidelines are used to reject unidentified lines on the peak search after environmental background subtraction.

5.3.10.1 The line matches the natural fingerprint of the uranium-238 or thorium-232 decay chains (i.e. 63, 75, 93, 239, 295, 352, 511, 609, 1120, etc.).

5.3.10.2 The line matches as a summation peak from two other lines in the spectrum.

5.3.10.3 The line has a net area of less than 20.

5.3.10.4 The line matches as an escape peak from an identified or unidentified nuclide which emits photons greater than 1022 keV.

5.3.10.5 The line has a percent error (% ERR) greater than 50%.

5.3.11 Results greater than the minimum detectable concentration (MDC) that are not requested by the client may or may not be reported based on certain criteria.

5.3.11.1 If the special requirements indicate that all positively identified nuclides must be reported then all positively identified nuclides that are greater than twice the uncertainty plus the minimum detectable

concentration must be reported.

5.3.11.2 If no special requirements indicate what is to be reported then if any of the following are true, do not report: the nuclide is being analyzed by other in-house analyses, another nuclide is being requested that exists in the same decay chain as the nuclide in question, if the radionuclide is naturally occurring, or if the result is less than twice the uncertainty plus the minimum detectable concentration.

5.3.11.3 If the samples analyzed are the first of two counts where the 2nd count is performed after ingrowth and the initial is not, the first count shall report only the requested parms. Any addition shall be made based on the data from the second count.

5.3.12 Apply correct report status for virtual data.

5.3.13 Proceed to step 5.4 to complete Level 1 data review.

5.4 LEVEL I – Review and Verification:

The data reviewer reviews 100% of the raw data and QC data once the data has been uploaded to AlphaLIMS. There may be additional requirements or different limits than stated below depending on the client's needs. The analyst must check for special requirements on the batch pull sheet. Client specific requirements are maintained under Client Profile Specifications in AlphaLIMS. For DoD or DOE clients please refer to their statement of work or the applicable version of DoD QSM, DOE QSAS, or consolidated DoD/DOE Quality System Manual.

This review ensures:

5.4.1 Standardization has been conducted according to the specific standard operating procedure for the analysis.

5.4.2 Analytical blanks have values less than the reported limits of detection or are less than 5% of the lowest activity.

5.4.3 Duplicate analyses are reported as Relative Percent Difference (RPD) or Relative Error Ratio (RER). Where RER is mentioned throughout the SOP the QSAS normalized absolute difference equation (DER D.4 DOE-15) can be used in its place. This equation is equivalent to MARLAP Z_{REP} (18.2). Duplicate RPD analyses are within 20% Relative Percent Difference (RPD) if the result is greater than five times the minimum detectable activity (MDC) and 100% if less than five times the MDC. If one result is above the MDC and the other is equal to or below the MDC, then the RPD can be calculated using the MDC for the result of the one equal to or below the MDC. The RPD must be 100% or less. In the situation where both results are above the MDC but one result is greater than 5 times the MDC and the other is less than 5 times the

MDC, the RPD must be less than or equal to 20%. If both results are equal to or below the MDC, then the limits on RPD are not applicable. The RER/DER acceptance criteria is less than or equal to 3. If the value is between 2 and 3, then it is acceptable to report with an investigation and a comment in the case narrative.

5.4.3.1 For drinking water analysis, the Relative Percent Difference (RPD) between the actual sample and the DUP should be less than or equal to 20% if both the sample and the DUP results are greater than 5 times MDC. If both results are less than 5 times MDC, the Relative Error Ratio should be less than 2. If both results are less than MDC, then limits are not applicable.

5.4.4 Matrix Spikes (MS) and Laboratory Control Samples (LCS) have recoveries between 75 and 125% of the expected value. For Ra-228 and Gross Alpha/Beta (GAB) drinking water analysis, the MS recovery shall fall between 70 and 130%, and the LCS recovery between 80 and 120%. For other drinking water analysis, the MS recovery should fall between 80 and 120%, and the LCS recovery between 90 and 110%.

5.4.5 Transcription of the raw data has been entered correctly in AlphaLIMS.

5.4.6 During the analytical preparation and sample counting events, unusual dilutions, non-routine handling of samples or data exceptions (refer to Appendix 1) can be noted on the Batch History form (refer to Appendix 4 for example) by the technician or analyst performing the procedure. These comments can be entered into the case narrative system in AlphaLIMS during Level I review. Data exceptions that have been attributed to sample matrix problems can only be considered after an evaluation by the group leader or designee.

5.4.7 If the analytical method requires a tracer or carrier, each sample result will be associated with a tracer or carrier recovery that is calculated and reported. The acceptance limits are 15 -125% for tracers and 25-125% for carriers.

These limits may vary for specific clients and/or projects.

5.4.7.1 If the yields determined with elemental carriers are above the acceptance limits and it is suspected that indigenous analytes may be present, an aliquot of the original sample should be tested by the appropriate method and the calculated recovery adjusted accordingly. See group leader or designee for additional guidance.

5.4.8 The data have been initialed and dated or electronically signed.

5.4.9 The required detection limit has been met.

5.4.9.1 When method limitations for Gross Alpha/Beta analysis prevent the

use of a larger sample to meet the detection limit (no more than 100 mg of solid residue should be on the counting planchet), the samples will be counted for a reasonable amount of time in an effort to obtain the lowest possible detection limit.

5.4.9.2 For Drinking Water analysis, ensure that the achieved limits are equal to or less than those specified in 40CFRPart141.25 (C)(1)Table B and 40CFRPart141.25(c)(2)Table C.

5.4.10 Review data for high count rates. If count rates exceed 100 counts per minute, contact the count room supervisor or designee. Review all samples that have counted on the particular detector, since the high count rate sample and before the subsequent background, for false positives.

5.4.11 Review data for significant negative results. The absolute value of the negative result should be less than three sigma Total Propagated Uncertainty (TPU). Contact group leader or designee for any suspect high negative results. Investigate all samples that have counted on the specific detector, and review detector background control charts.

5.4.12 Compare the data to historical results if available. If a discrepancy is identified, contact team leader, group leader, or designee for additional guidance.

5.4.13 If any of the above criteria have not been met, the data will be considered out of control and corrective action must be taken before the data can be sent to the next level of review.

5.4.14 The batch is placed in review and the Compliance Checker automatically runs. The batch checklists are completed electronically in the AlphaLIMS narrative system once excursions identified in the Compliance Checker have been addressed. Refer to Appendix 2 for example. The batch is then sent to the next data reviewer for Level II review.

5.5 LEVEL II - Data Review, Validation, and Technical Review:

Appendix 2 contains an example second review guideline that may be used in addition to the batch checklist during the second review steps.

There may be additional requirements or different limits than stated below depending on the client's needs. The analyst must check for special requirements on the batch pull sheet. Client specific requirements are maintained under Client Profile Specifications in AlphaLIMS. For DoD or DOE clients please refer to their statement of work or the applicable version of DoD QSM, DOE QSAS, or consolidated DoD/DOE Quality System Manual.

This review is conducted by a data reviewer independent of the first data reviewer to ensure that:

5.5.1 Standardization was conducted according to the specific standard operating

procedure for the analysis.

5.5.2 Analytical blanks have values less than the reported limits of detection or are less than 5% of the lowest activity.

5.5.3 Duplicate analyses are reported as Relative Percent Difference (RPD) or Relative Error Ratio (RER). Where RER is mentioned throughout the SOP the QSAS normalized absolute difference equation (DER D.4 DOE-15) can be used in its place. This equation is equivalent to MARLAP Z_{REP} (18.2). Duplicate RPD analyses are within 20% Relative Percent Difference if greater than five times the MDC or 100% if less than five times the MDC. If one result is above the MDC and the other is equal to or below the MDC, then the RPD can be calculated using the MDC for the result of the one equal to or below the MDC. The RPD must be 100% or less. In the situation where both results are above the MDC but one result is greater than 5 times the MDC and the other is less than 5 times the MDC, the RPD must be less than or equal to 20%. If both results are equal to or below the MDC, then the limits on RPD are not applicable. The RER/DER acceptance criteria is less than or equal to 3. If the value is between 2 and 3, then it is acceptable to report with an investigation and a comment in the case narrative.

5.5.4 Matrix Spikes and Laboratory Control Samples have recoveries between 75% and 125% of the expected value. For Ra-228 and GAB drinking water analysis, the MS recovery shall fall between 70 and 130%, and the LCS recovery between 80 and 120%. For other drinking water analysis, the MS recovery should fall between 80 and 120%, and the LCS recovery between 90 and 110%.

5.5.5 Transcription of the raw data has been entered correctly into AlphaLIMS.

5.5.6 Comments have been entered into AlphaLIMS for unusual dilutions, non-routine handling of samples, explanations on data exceptions, etc. Data Exceptions that have been attributed to sample matrix problems can only be considered after an evaluation by the group leader or designee.

5.5.7 Contract required detection limits have been met.

5.5.7.1 When method limitations for Gross Alpha/Beta analysis prevent the use of a larger sample to meet the detection limit (no more than 100 mg of solid residue should be on the counting planchet), the samples will be counted for a reasonable amount of time in an effort to obtain the lowest possible detection limit.

5.5.7.2 For Drinking Water analysis, ensure that the achieved limits are equal to or less than those specified in 40CFRPart141.25 (C)(1)Table B and 40CFRPart141.25(c)(2)Table C.

5.5.8 If the analytical method requires a tracer or carrier, each sample result will be associated with a tracer or carrier recovery that is calculated and reported. The acceptance limits are 15%-125% for tracers, and 25-125% for carriers. These limits may vary for specific clients and/or projects.

5.5.8.1 If the yields determined with elemental carriers are above the acceptance limits, and it is suspected that indigenous analytes may be present, an aliquot of the original sample should be tested by the appropriate method and the calculated recovery adjusted accordingly. See group leader or designee for additional guidance.

5.5.9 The first reviewer has changed the batch status to REVW.

5.5.10 Review data for high count rates. If count rates exceed 100 counts per minute, contact the count room supervisor or designee. Review all samples that have counted on the particular detector, since the high-count rate sample and before the subsequent background, for false positives.

5.5.11 Review data for significant negative results. The absolute value of the negative result should be less than three sigma TPU. Contact group leader or designee for any suspect high negative results. Investigate all samples that have counted on the specific detector, and review detector background control charts.

5.5.12 Compare the data to historical results if available. If a discrepancy is identified, contact team leader, group leader, or designee for additional guidance.

5.5.13 Review the qualifiers to ensure standard or client specific qualifiers have been appropriately applied.

5.5.14 If any of the above criteria have not been met, the data are considered out of control and corrective actions must be taken before the data are approved for completion.

5.5.15 The data reviewer then initials and dates the batch verifying that the data have met all criteria. Electronic signatures are acceptable.

5.5.16 The data reviewer then gives at least 10% of reviewed batches to the respective group leader or designee for review. The group leader or designee reviews the data based on the analytical method and client requirements and ensures completeness of the review process.

5.5.17 The data reviewer changes the batch status to '**DONE**'.

5.5.18 Batches are compiled virtually in LIMS or scanned to a pdf. They are stored by batch ID on the packaging-in-pdf/raw data network folder or directly in AlphaLIMS.

5.5.19 Batches typically include:

5.5.19.1 Batch checklist

5.5.19.2 Queue sheet or electronic logbook page

5.5.19.3 Any e-mail required with package submission

5.5.19.4 Various reportable reports such as: aliquot and blank corrections, hits reports and qualifiers

5.5.19.5 Raw Data/Spreadsheets

NOTE: Not all elements listed above are generated for each batch.

6.0 QUALITY CONTROL REQUIREMENTS

NOTE: Some clients contractually override these Quality Control requirements and may be more or less restrictive. Also, some clients have their own equations that would supersede those in this SOP.

6.1 A Method Blank (MB) accompanies each batch of 20 samples or less. The reported value should be less than the RDL for all target isotopes or less than 5% of the lowest sample activity.

6.2 A Laboratory Control Sample (LCS) is run with each batch of 20 samples or less. The LCS is equivalent to a Fortified Blank in EPA drinking water compliance manual (refer to section 12.9 of this SOP). Some clients also require a Matrix Spike (MS) to be analyzed. The requirement for recovery for both LCS and MS is between 75 and 125%. For Ra-228 and GAB drinking water analysis, the MS recovery shall fall between 70 and 130% and the LCS recovery between 80 and 120%. For other drinking water analysis, the MS recovery should fall between 80 and 120%, and the LCS recovery between 90 and 110%. Recovery is calculated as follows:

$$\text{MS } (\%) = \frac{\text{Spiked result} - \text{Sample result}}{\text{Spike added}} * 100\%$$

$$\text{LCS}(\%) = \frac{\text{observed concentration}}{\text{known concentration}} * 100\%$$

NOTE: For the MS, if the sample result is less than the MDC, then no activity will be subtracted from the spiked result.

6.2.1 If a MSD and/or a LCSD are required, then the Relative Percent Difference (RPD) of the activities shall be calculated according to the equation in 6.3. If the RPD is outside 0 to 20% and meets the spike recovery requirements, then the data will be reported. The comment will be added to the batch case narrative.

6.3 If a sample duplicate is analyzed, it will be reported as Relative Percent Difference (RPD) or Relative Error Ratio (RER/DER) depending on the client's requirements.

The RPD must be 20 percent or less, if both samples are greater than 5 times the MDC.

If both results are less than 5 times MDC, then the RPD must be equal to or less than 100%. If the RPD fails, then calculate the RER/DER. If the RER/DER passes, then report the data with a comment in the narrative. If one result is above the MDC and the other is below the MDC, then the RPD can be calculated using the MDC for the result of the one below the MDC. The RPD must be 100% or less. In the situation where both results are above the MDC but one result is greater than 5 times the MDC and the other is less than 5 times the MDC, the RPD must be less than or equal to 20%. If both results are below MDC, then the limits on % RPD are not applicable. A 0% will be reported on raw data and spreadsheets.

The RER/DER acceptance criteria is less than or equal to 3. If the value is between 2 and 3, then it is acceptable to report with an investigation and a comment in the case narrative.

Relative percent difference (%RPD) is calculated as follows:

$$RPD(\%) = \frac{\text{High Duplicate Result - Low Duplicate Result}}{\text{Average of Results}} * 100\%$$

Relative Error Ratio (RER/DER) is calculated as follows:

$$\text{RER} = \frac{|\text{SampleActivity} - \text{DuplicateActivity}|}{\sqrt{(\text{Sample CSU})^2 + (\text{Duplicate CSU})^2}}$$

6.3.1 For drinking water analysis, the Relative Percent Difference (RPD) between the actual sample and the DUP should be less than or equal to 20% if both the sample and the DUP results are greater than 5 times MDC. If both results are less than 5 times MDC, the Relative Error Ratio should be less than 2. If both results are less than MDC, then limits are not applicable.

6.4 If any of the above criteria cannot be satisfied, recount, reprep or inform the group leader or designee and initiate a data exception report as outlined in GL-QS-E-004 for Documentation of Nonconformance Reporting and Dispositioning and Control of Nonconforming Items as the situation dictates. When a LCS or other QC sample is recounted and the recount passes quality control criteria, the entire batch should be recounted prior to reporting. In some cases, it may be determined that recounting the entire batch would not be appropriate. Please see group leader or designee for final approval.

7.0 DATA PACKAGES

7.1 A data package based on the client's requirements and needs is generated if additional sample analysis information is client requested. Package guidelines are created for each client's requirements as defined by the project name and level assigned in AlphaLIMS. The guidelines can be generated from multiple locations on the Fraction Work Order Status (With Status) page under the Project Checklist column, from "build package" under "view package checklist" or "sign". Refer to Appendix 3 for a guideline. The following are often requested in a data package:

7.1.1 Fractional Case Narrative

7.1.2 Certificates of Analysis

7.1.3 QC Summary

7.1.4 Queue Sheet or Electronic Logbook and Raw Data

7.1.5 Method Calibrations

7.1.6 Continuing Calibrations (Daily Instrument Checks)

7.1.7 Instrument Background and Efficiency Graphs

7.1.8 Quality Control Charts

7.1.9 Method Spiking Sources Certificates

7.1.10 Instrument Runlogs

7.2 Data pertaining to the following sample types are included in the data package unless otherwise noted in the client requirements:

7.2.1 All samples in the SDG or work order

7.2.2 The method blank, matrix spike, matrix spike duplicate, laboratory control sample, and laboratory control sample duplicate.

7.2.3 The parent and sample duplicate

NOTE: The requirement of sample types will vary by client; therefore, not all of the sample types listed above will be included in each batch.

7.3 Packaging files are stored in the packaging_in_pdf network folder or can be obtained electronically directly from AlphaLIMS. Package elements, if not automatically generated by AlphaLIMS, are generated as follows:

7.3.1 Case Narratives

7.3.1.1 Generate the RAD Fraction and Radiochemistry case narrative for the work order from the AlphaLIMS intranet fractional narrative web page or select the "casNarr" link from the organic forms link that is found next to the case narrative description. Then select "create/batch" link.

7.3.1.2 Edit the narrative per client requirements if necessary.

7.3.1.3 After editing, schedule the narrative.

7.3.2 Cover Sheet, Certificates of Analysis (CoAs) and Quality Control Summary (QC Summary)

7.3.2.1 Submit work order or SDG from the organic forms link.

7.3.2.2 The CoAs and QC Summary reports are automatically schedule by selecting the “Make PDF Reports”.

7.3.2.3 Another alternative method is to select appropriate form number and press schedule. Close window and select appropriate form again. Select “view” link. Document will appear in pdf format at which point can be printed to the “coa” or “qc” summary folder if necessary.

7.3.2.4 The Cover Sheet is signed electronically. Select on the appropriate form number and select the “sign” link. Enter in your password and press submit. Close window and select appropriate form again. Select “view” link. Document will appear in pdf format at which point can be printed to a temporary folder if necessary.

7.3.3 Raw Data: Batches from data review teams are stored electronically. These files are accessed from the "raw data" folder. Alternatively, if the batches have been compiled through virtual capture in LIMS, the raw data can be accessed directly in LIMS either through “Select Batch to Review” page or pulled automatically into package during package assembly.

7.3.4 Method Calibrations: Calibrations are performed and reviewed by qualified staff then stored electronically. They can be accessed from the "calibrations" folder or from the “Maintain Valid Value Tables”, “Instrument Calibration” links from the AlphaLIMS intranet web page. To view calibrations, select either the BatchID, Instrument Type, Geometry, Procedure, Instrument, Employee or Start and End date and “view”. Calibrations can also be accessed via the Organic Forms Link and select “view” next to the Method Calibration Data and then select “View Data” and “view” each individual calibration file under the Inst Cal File. If required for the data package, the appropriate calibrations are automatically pulled into the package during package assembly.

7.3.5 Continuing Calibration Data (Daily Instrument Checks): Dailies are generated by the count room operators and stored electronically in the appropriate "dailies" folder or directly in AlphaLIMS.

7.3.6 Background and Efficiency Graphs are generated as follows:

7.3.6.1 Alpha Spec graphs are generated monthly by countroom staff and electronically stored in

\\gelsan\rad_package\packaging_in_pdf\bkg_eff\Alpha Spec. The charts should be reviewed for trends by the Group Leader or designee and the review should be documented in the appropriate instrument maintenance logbook.

7.3.6.1.1 From the alpha system, for a large number of detectors, list the detectors in alpha_cc.dat

7.3.6.1.2 Type @alpha_cc

7.3.6.1.3 Enter the start date for the chart (typically 6 months previous to current date)

7.3.6.1.4 Enter the end date for the chart (typically the day after current date)

7.3.6.1.5 Answer "Are the chambers you want to process listed in ALPHA_CC.DAT? (Y/N)" Entering "Y" will generate charts for all detectors listed in alpha_cc.dat. Entering "N" will require input specific detectors to generate charts.

7.3.6.1.6 Verify all files were generated. Most file are typically 11K or 12K in size, regenerate any missing or incorrect charts.

7.3.6.2 Gamma Spec graphs are generated monthly by countroom staff and electrically stored in \\gelsan\rad_package\packaging_in_pdf\bkg_eff\GAMMA. The charts should be reviewed for trends by the Group Leader or designee and the review should be documented in the appropriate instrument maintenance logbook.

7.3.6.2.1 From the gamma system, for a large number of detectors, list the detectors in gamma_cc.dat

7.3.6.2.2 Type @gamma_cc

7.3.6.2.3 Enter the start date for the chart (typically 6 months previous to current date)

7.3.6.2.4 Enter the end date for the chart (typically the day after current date)

7.3.6.2.5 Answer "Are the chambers you want to process listed in GAMMA_CC.DAT? (Y/N)" Entering "Y" will generate charts for all detectors listed in gamma_cc.dat. Entering "N" will require input specific detectors to generate charts.

7.3.6.3 GFPC, LSC and Lucas Cell graphs are generated automatically by AlphaLIMS based on the work order. They can be found by work order from the Organic Forms Link and select "view" next to the Form Description Background and Efficiency Data. They can also be found in the packaging_in_pdf, prelim_package_pdfs folder and select appropriate work order. The charts should be reviewed for trends by the Group Leader or designee and the review should be documented in the appropriate instrument maintenance logbook. In the event a new graph must be created, the following steps should be used:

7.3.6.3.1 From the "Instrument trending and lockout system" intranetwebpage, select and submit the appropriate instrument type.

7.3.6.3.2 On the new page, open the instrument folder and select the appropriate detector.

7.3.6.3.3 From the instrument maintenance page, click the "view previous graph events" link. Select the "Show Graph" link for the appropriate date required.

7.3.6.3.4 Print the graph to the appropriate "bkg_eff" folder.

7.3.7 Quality Control Charts (QC charts) are automatically generated from AlphaLIMS based on the work order. In the event that new tables and graphs must be created, the following steps should be used:

7.3.7.1 From the "View SPC studies for a work order" intranet web page, submit the work order. Alternatively, select the "view" link from the Organic Forms Link next to the Form Description Quality Control Charts and select "Create".

7.3.7.2 To reprint the table or graph:

7.3.7.2.1 Click the green dot to bring up the table. Click the parmname description to bring up the graph.

7.3.7.2.2 Click on the table or graph (to indicate which window to print).

7.3.7.2.3 Print the document to PDF in a temp folder. DO NOT print or save the document to the "prelim_package_pdfs" folder where the auto-generated files are saved.

7.3.7.3 To regenerate the table or graph:

7.3.7.3.1 Click the appropriate batch number link.

7.3.7.3.2 Click "Yes" to regenerate the graph.

7.3.7.3.3 After the graph has regenerated, follow the steps listed above to reprint the table or graph.

7.3.8 Method Spiking Source Certificates (Standards) typically consist of a Primary Certificate and a Standard Traceability Log or Standard Logbook. A standard verification sheet is required with the certificates if the expiration date of the standard code is more than a year from the preparation date of that same standard code.

7.3.8.1 Primary certificates and verification sheets are stored electronically. Upon need, these are scanned into the appropriate "standards" folder or uploaded to AlphaLIMS via the “Data Menu”, “Maintain Reference Materials”, “Create/Edit/View Reference Materials” links. Enter in appropriate Serial # and submit. Select the Action of “Verification” or “Edit” and browse to find the file and submit. The file may be viewed in pdf format by clicking “View” File.

7.3.8.2 Standard Traceability Logs or Standard Logbooks (standard AlphaLIMS sheets) are automatically generated (see step 7.3.9.3) or can be manually generated when the new data is entered into AlphaLIMS where they can be generated and printed to PDF. To generate a AlphaLIMS sheet:

7.3.8.2.1 From the "Create/Edit/View reference materials" intranet web page, in the "serial #" field, submit the "'standardID'%" (ex: 0159% or 445-96-2%) for the AlphaLIMS sheet needed.

7.3.8.2.2 Under the "Print" column, click the "2nd Log" link on the row of the standard code needed. For Gamma and ICP-MS standards select the “Logbook” link.

7.3.8.2.3 When the log appears, it can be printed to the appropriate "standards" folder in PDF.

7.3.8.3 The Standards may also be generated by clicking on the Organic Forms Link page and selecting the “Create PDF Reports.” They may be viewed by selecting the “radRefmat” link and “View Data”.

7.3.9 Runlogs

7.3.9.1 The run logs are automatically scheduled by selecting the “Make PDF Reports” link from the Organic Forms Link or by selecting the “radrunlog” link and schedule. The pdf may be saved to the “runlogs” folder if necessary.

7.3.9.2 An alternative method to generate runlogs is to select the “Instrument Run Logs Based on Batch ID or Work Order” intranet

web page, submit the appropriate instrument type and batch number. Print the runlog to pdf to the “runlogs” folder if necessary.

7.4 All package components are converted to PDF format by means of scanning, AlphaLIMS generation or printing to PDF then saved to the appropriate folder if needed. The following naming convention shall be used in manually naming package documents.

7.4.1 Background and Efficiency Graphs

7.4.1.1 Alpha Spec: The graphs are named numerically corresponding to the detector number starting with 1001. Ex: Graphs for detector 1 would be named "1001", detector 77 would be "1077" and detector 111 would be “1111”.

7.4.1.2 Gamma Spec: The graphs for the numerically named detectors are as follows: GAM#. The remaining graphs are named by the detector name: WELL, XRAY1, XRAY2, XRAY3, and XRAY4.

7.4.1.3 Graphs for the following are generated and named automatically from AlphaLIMS. In the event a graph must be manually regenerated, it is suggested that it be named by the detector ID as follows:

7.4.1.3.1 Gas Flow Proportional Counters: Ex: 1A, 11B or 4B.

7.4.1.3.2 Liquid Scintillation Counter: Ex: BLUE, GREEN, or RED

7.4.1.3.3 Lucas Cell Detector: Ex: LUCAS1, LUCAS2 or LUCAS3

7.4.2 Case Narratives: "Client abbreviation “_” work order #” ex: SCNM_123456

7.4.3 Certificates of Analysis: "work order #"_coa (minuscule) ex: 123456_coa

7.4.4 Continuing Calibrations (Dailies) must be named as follows:

DDMMMYY_ ANALYSISMETHOD. When weeklies or monthlies are generated, the name is appended as such.

7.4.4.1 Alpha: Ex: 07JAN05_AS; 05DEC05_AS_MONTHLY

7.4.4.2 Gamma : Ex : 11MAR05_GAMMA; 28MAR05_GAMMA_WEEKLY

7.4.4.3 Gas Flow Proportional Counter: Ex: 16JUL05_GFPC

7.4.4.4 Liquid Scintillation Counter : Ex : 18JUN05_LSC

7.4.4.5 Lucas Cell: Ex: 05APR05_LUCAS

7.4.5 Method Calibrations: Dates associated in the naming convention of calibrations are the effective date of the calibration.

7.4.5.1 Alpha Spec: The initial calibration is named Alpha Spectroscopy. The individual detector calibrations are named as "Detector_YearMonthDay_EnergyCalibrationTime".

Ex: 1001_2009DEC04_1137

7.4.5.2 Gamma Spec: The calibrations are suggested to be named as follows: Ex: GAM01_CAN_12jan10, WELL_CAN_23feb10

7.4.5.3 Gas Flow Proportional Counters and Liquid Scintillation Counters: "Parmname"_INSTRUMENT_MMM_DD_YYYY

Ex: 1.) Total Radium_LB4100_MAR_27_2005;

2.) Radium-228_PIC_AUG_18_2005;

3.) Alpha_PIC_NOV_24_2005;

4.) Tritium_BROWN_AUG_1_2005; or

5.) Iron-55_GREEN_OCT_14_2005

NOTE: Regarding spelling and capitalization, the parmname must appear as it would pull from AlphaLIMS, i.e. as seen on a batch pull sheet.

7.4.5.4 Lucas Cell Detector:

LUCAS"DETECTOR#"_MMM_DD_YYYY
ex: LUCAS2_OCT_21_2005

7.4.6 Data Exception Reports: The DER number is the name of the file.

7.4.7 Packages: "Client ID" _ "workorder or SDG"_R ex. LLNL_284777_R or BRKL_29155_R

7.4.8 Quality Control (QC) Summaries: "workorder"_qc (minuscule) ex:123456_qc

7.4.9 Quality Control (QC) Charts: QC charts are generated automatically by AlphaLIMS. In the event a chart needs to be manually regenerated, the file should be saved to a temporary folder for immediate usage. Due to the nature of the temporary files, a file name format is not necessary.

7.4.10 Raw Data Batches: "batchID#"_Parmname ex: 123456_Ra226; 789012_U

7.4.11 Runlogs: batchID# or workorder # ex: 123456

7.4.12 Method Spiking Certificates (Standards):

7.4.12.1 Primary Certificates: "Primary (Standard) ID #" ex: 0661. This file also contains the most current Standard Traceability Log and Verification Sheet which are manually added to the file.

7.4.12.2 Standard Traceability Log (AlphaLIMS Sheet): "Primary ID #"_lims (minuscule) ex: 0661_lims

7.4.12.3 Verification (ver) Sheet: "PrimaryCode"_ver_MMM_DD_YYYY ex: 0661-A_ver_MAR_14_2005 where the date is the verification

date.

7.5 Status Location and Assembling the Package

7.5.1 Status location: The status screen is located under the "status" column on the Fraction Work Order Status (with status) page. When changing the status, the analyst should enter their initials under the comments section.

NEW New Record

PEND Building Package

PKG Finished Building the Package

VAL Reviewing the Package

RECVD Copy of the Package has been transferred electronically from Radiochemistry Data Package Review to Final Packaging.

7.5.2 Assembling the Package

7.5.2.1 Change the status location from OPEN to PEND

7.5.2.2 Build the virtual package using the Organic Forms Link page using the following steps:

7.5.2.2.1 Select the desired work order and click on the Drev folder from the Fraction Work Order Status (With Status) page.

7.5.2.2.2 Schedule or edit the narrative as stated in step 7.3.1.

7.5.2.2.3 Click on the "Make PDF Reports" link.

7.5.2.2.4 If validating the package, electronically sign the Cover Sheet as stated in step 7.3.3.4.

7.5.2.2.5 Select the "Create Fractional Package" link.

7.5.2.2.6 Select the check all button, generate and close window.

7.5.2.2.7 Select the "Create Fractional Package" link again. Reload the screen until "The Total Package" component link at the bottom of the screen has a date and time next to it under Last Generated.

7.5.2.2.8 Select "The Total Package" link which will appear in PDF format.

7.5.2.2.9 The package can be saved as noted in step 7.4.7 to the "packaging_review" folder if extensive changes may be needed; otherwise, the package may be validated from "The Total Package" link.

7.5.3 Ensure the package contains the required relevant documents. This typically involves removing data, especially from alpha and gamma spectroscopy batches, for work orders not pertinent to the Work Order being built or

inserting documents created after building the package.

7.5.3.1 Remove from all raw data batch files any pullsheets, AlphaLIMS reports, batch history sheets, client/PM emails, recount logs, anything stamped DNR "do not report" unless specifically requested by the client to maintain in the package. An exception is to leave any original spectrum for an alphaspec sample that has been manually integrated. The original should have DNR and a notation that it will be manually integrated; both the original and manually integrated spectrum should be included in the package.

7.5.3.2 For alphaspec and gammaspec raw data, remove all spectra/files for samples that are not the QC parent and not in the package SDG.

7.5.4 Review the certificates of analysis and QC Summary for technical mass balance (Gross alpha vs. actinides, Radium-226 vs. Total Radium, etc.).

7.5.5 If the package will not be reviewed at this time, change the status location from PEND to PKG. Otherwise, proceed to 7.5.6.

7.5.6 Ensure bookmarks are formatted correctly and then save package to "packaging_review" folder if needed.

7.6 Reviewing the Fractional Data Package

7.6.1 Once the fractional data package is assembled, a group leader or designee reviews it for client requirement conformity.

7.6.1.1 Change the status location from PKG to VAL if applicable.

7.6.1.2 Review package for client requirement conformity.

7.6.1.3 If the package was modified and saved to the "packaging_review" folder the final package can be made available for client reporting by either step below. If package was validated from "The Total Package" link follow 7.6.1.3.2.

7.6.1.3.1 Transfer a copy to the "packages_in_done" folder. Be sure to create a mini package at this step if also required. From the status screen on the Packages Required in Fraction page, submit the grade card. This step will automatically transfer a copy of the package to the client folder for final deliverables and status the fraction to RECVD.

7.6.1.3.2 Upload in AlphaLIMS to "The Total Package" component if changes were made. Verify that mini package is available if required. Click on the sendToFinal button. This step will automatically transfer a copy of the package

to the client folder for final deliverables and status the fraction to RECVD.

8.0 ALPHALIMS RAW DATA CHANGES

8.1 If any changes are made to the AlphaLIMS raw data after the results have been reported to a client, a Client Nonconformance should be initiated. Refer to GL-QS-E-012 for NCR Database Operation.

8.2 The revised data is reviewed for completion and response entered in the NCR database.

8.3 The client is notified and new Certificates of Analysis, electronic deliverables, and data packages are processed as required.

8.4 A record of the changes is maintained as a quality record.

9.0 CALCULATIONS

Refer to GL-RAD-D-006 for Equations Used in Data Reduction for Environmental Radiochemistry.

10.0 SAFETY, HEALTH, AND ENVIRONMENTAL HAZARDS

Potential hazards with this process include prolonged sitting and video display terminal usage. Refer to the Safety, Health and Chemical Hygiene Plan, GL-LB-N-001, for basic safety and health information.

11.0 RECORDS MANAGEMENT

11.1 Records generated as a result of this SOP and associated management activities are maintained as quality records in accordance with GL-QS-E-008 for Quality Records Management and Disposition.

11.2 The Batch History is scanned in and stored with the raw data batch until the data package has been assembled and reported to the client.

12.0 REFERENCES

12.1 ANSI/ASQC-E4-1994, American National Standard: Specifications and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs, American Society for Quality Control (ASQC), Energy Division, Environmental Waste Management Committee, Milwaukee, 1994.

12.2 Dept. of Defense (DOD), Dept. of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories DOD Version 5.1, January 2017; DOE QSAS Version 3.1 January 2017.

12.3 DOE/TIC-11026 “Radioactive Decay Data Tables,” by D.C. Kocher (1981).

12.4 DOE es/er/ms-5 “Evaluation of Radiochemical Data Usability,” April 1997.

12.5 Doc S501-V&V Rev 1.2 “Genie-2000 Gamma Analysis Model S501 Analysis Engine Validation & Verification” Canberra Industries, Inc. 2002.

12.6 Doc S500-V&V Rev 1.2 “Genie-2000 Basic Spectroscopy Models S500/S502 Analysis Engine Validation & Verification” Canberra Industries, Inc. 2002.

12.7 Cable, J.E., W.C. Burnett, S.C. Moreland, and J.B. Westmoreland. "Empirical Assessment of Gamma-Ray Self Absorption in Environmental Sample Analysis", Radioactivity & Radiochemistry Volume 12, Number 3 (p. 30-39) 2001.

12.8 National Nuclear Data Center at Brookhaven National Laboratory (www.nndc.bnl.gov/nndc/nudat/).

12.9 Manual for the Certification of Laboratories Analyzing Drinking Water. Criteria and Procedures Quality Assurance. Fifth Edition EPA 815-R-05-004 January 2005.

12.10 EL-V1-2009, NELAC Institute: Management and Technical Requirements for Laboratories Performing Environmental Analysis, 2009.

12.11 NUREG 1576, EPA 402-B-04 "Multi-Agency Radiological Laboratory Analytical Protocols Manual", July 2004.

13.0 HISTORY

Revision 41: Revised to reflect current practices for changes identified during annual review
Revision 42: Update Appendix 1. Update Reference Section with current reference to DOD/DOE Versions 5.1 and 3.1.
Revision 43: Revised to include generation of batch checklists; updates to reflect continued automation; clarified gamma review.
Revision 44: Removed reference to definition of High Error (HE) clarified gray area in section 5.3.9.5.

APPENDIX 1: DATA EXCEPTIONS

NOTE: Some clients contractually override these Quality Control requirements and may be more or less restrictive.

Category: Gamma Spectroscopy

Metric	Acceptance Criteria	Corrective Action
Method Blank	< RDL or < 2x Decision level	Recount to confirm out of control condition. If the problem persists, clean the detector and shield and redo the method blank. If samples have positive detections for the isotopes failing to meet the blank requirement, redo the batch. Qualify the data if required.
Replicate (RPD)	RPD 20%, if both results are ≥5x the MDC. RPD 100%, if both results are ≤5x the MDC. RPD 0% (no precision required) if both results below MDC. RPD 20% If one result ≥ 5x the MDC and one result is greater than MDC but < 5x the MDC. *For acceptance criteria not addressed above, see Section 6.3 for requirements.	Recount to confirm out of control condition. Reprep the batch if problem persists. Determine and correct the problem. Qualify the data if required.
Replicate (RER/DER)	RER/DER ≤ 3	Recount to confirm out of control condition. Reprep the batch if the problem persists. Determine and correct the problem. Qualify the data if required. If RER/DER is between 2-3, must investigate and comment findings in case narrative.
MS	75-125% Recovery	Recount to confirm. If problem persists, reprep the batch. Qualify the data if required.
LCS	75-125% Recovery	Recount to confirm out of control condition. If problem persists, reprep the batch. Qualify the data if required.
Gain	+ 3 KeV	Recount to confirm out of control condition. If problem persists, adjust the instrument gain as required and confirm the energy calibration. Qualify the data if required.
FWHM	<3 KeV	Qualify the data if required.

Category: Gas Flow and Liquid Scintillation Counting

Metric	Acceptance Criteria	Corrective Action
Method Blank	< RDL or < 2x Decision level	Recount to confirm out of control condition, reprep the batch if the problem persists. If samples have positive detections for the parameters failing to meet the blank requirement, redo the batch. Qualify the data if required.
Replicate (RPD)	RPD 20%, if both results are ≥5x the MDC. RPD 100%, if both results are ≤5x the MDC. RPD 0% (no precision required) if both results below MDC. RPD 20% If one result ≥ 5x the MDC and one result is greater than MDC but < 5x the MDC. *For acceptance criteria not addressed above, see Section 6.3 for requirements.	Recount to confirm out of control condition. Reprep the batch if problem persists. Determine and correct the problem. Qualify the data if required.
Replicate (RER/DER)	RER/DER ≤ 3	Recount to confirm out of control condition. Reprep the batch if the problem persists. Determine and correct the problem. Qualify the data if required. If RER/DER is between 2-3, must investigate and comment findings in case narrative.
LCS	75-125% Recovery	Recount to confirm out of control condition if problem persists, reprep the batch. Qualify the data if required.
MS	75-125% or not applicable if sample concentration is > 5x spike concentration	Recount to confirm out of control condition. If problem persists and LCS is also failing, redo the batch. Qualify the data if required.
Internal Tracer/Carrier Yield	Carrier: 25-125% Recovery Tracer: 15-125% Recovery	Recount to confirm out of control condition. If problem persists, repeat sample in another analytical batch to achieve acceptable recovery. Qualify the data if required.

Category: Alpha Spectroscopy

Metric	Acceptance Criteria	Corrective Action
Method Blank	< RDL or < 2x Decision level	Recount to confirm out of control condition, reprep batch if the problem persists. If samples have a positive detection for the isotope failing to meet the blank requirement, re-aliquot and repeat the batch. Check and clean the detector and chamber if required. Qualify the data if required.
Replicate (RPD)	RPD 20%, if both results are ≥5x the MDC. RPD 100%, if both results are ≤5x the MDC. RPD 0% (no precision required) if both results below MDC. RPD 20% If one result ≥ 5x the MDC and one result is greater than MDC but < 5x the MDC. *For acceptance criteria not addressed above, see Section 6.3 for requirements.	Recount to confirm out of control condition. Reprep batch if problem persists. Determine and correct the problem. Qualify the data if required.
Replicate (RER/DER)	RER/DER ≤ 3	Recount to confirm out of control condition. Reprep the batch if the problem persists. Determine and correct the problem. Qualify the data if required. If RER/DER is between 2-3, must investigate and comment findings in case narrative.
LCS	75-125% Recovery	Recount to confirm condition. If problem persists reprep the batch. Qualify the data if required.
MS	75-125% or not applicable if sample concentration is > 5x spike concentration	Recount to confirm if problem persists, reprep the batch. Qualify the data if required.
Internal Tracer Yield	15-125% Recovery	Recount to confirm if problem persists, repeat sample to achieve acceptable recovery. If Blank or LCS fail, reanalyze entire analytical batch, starting with preparation. Qualify the data if required.
FWHM	< 50 KeV on the daily pulsar check	Recount to confirm, if problem persists re-precipitate the source. Qualify the data if required.
Gain	± 50 KeV on the tracer peak	Recount to verify. Reprep sample if problem persists. Qualify the data if required.

APPENDIX 2: REVIEW CHECKLISTS
(For Illustrative Purposes Only)

Batch 1660980 Check-list

This check-list was completed on 05-MAY-17 by Jessica Downey
This batch was reviewed by Jessica Downey on 05-MAY-17 and Jessica Davis on 05-MAY-17.

Batch ID: 1660980 **Product:** ASP__THS **Description:** Alpha Spec Thorium GL-RAD-A-038

#	Criteria	Yes	No	Comments
Preparation Information				
1	Was the preservation correct for this analysis?	Yes		
2	Were any of the samples classified as radioactive?	Yes		
Internal Checklist Information				
3	Is aux data correct?	Yes		
4	Have sample historical results been reviewed for this batch?	Yes		
5	Has an aliquot correction been completed for this batch?	Yes		
6	Are instrument source checks within limits?	Yes		
7	Has the method RDL been met?		No	
8	Were the tracer yield requirement of 15-125%, or the client's contract acceptance criteria, met?	Yes		
Technical Information				
9	Were all the samples prepared/analyzed within the required holding time period?	Yes		
Quality Control (QC) Information				
10	Did all QC samples meet the required acceptance limits?	Yes		
Miscellaneous Information				
11	Were manual integrations performed on any sample or QC data files in this batch?		No	
12	Are sample-specific MDA/MDC calculated and reported?	Yes		

Example Second Review Guideline Checklist

(For Illustrative Purposes Only)

Batch #

Included in batch: Queue Sheet or Electronic Logbook, Aliquot Correction Sheet, Balance Interface Sheet, Spreadsheet, Raw Data/Spectrums, Do Not Report stuff, LIMS Report, Reagent Sheet, Batch History Sheet, Pull Sheet, Scan Sheet

1. Aliquot Correction Sheet / Balance Interface Sheet to Queue Sheet or Electronic logbook
   (units the same)

2. Queue Sheet or Electronic Logbook to Spreadsheet transcription
   (Batch #, aliquots, weights, spike & LCS code, prep date, det #, **units circled, instrument used circled**)

3. Raw Data/Spectrums to Spreadsheet
   (correct detectors, # of bkgs, all spectrums)

4. Spreadsheet to AlphaLIMS

5. Check pullsheet for comments and requirements

Things to Check for:

1. RDLs and requirements met (If not statement in narrative)

2. Case narrative (DERs, sample QC, flamed/unflamed, additional comments)

3. All line outs are initialed and dated

4. Write-overs are lined out and re-written

5. No blank spaces

6. Batch checklist is properly filled out

7. Initial and Date pages

8. Send to Done

APPENDIX 3: EXAMPLE PACKAGE GUIDELINES LIST
(For Illustrative Purposes Only)

GEL Laboratories LLC

Final Data Package Review Checklist

GL-CHL-GC-001 Rev 0

Report Date: Tuesday, July 23, 2013

Project: [redacted]

SDG: 329760

Work Order: 329760

Submittal Level: LEVEL6 **PM:** [redacted]

Package Due Date: 08-AUG-13

Rad		Pages:
Case Narrative		
Case Narrative Analytical	Mini Pkg	
CofA Cover Sheet Signature	Mini Pkg	
Analytical DER	Mini Pkg	
Sample Data Summary		
Form01-RAD Sample Data Summary	Mini Pkg	
Quality Control Summary		
Form01QC-RAD QC Sample Data Summary	Mini Pkg	
Form03-RAD Spike Summary	Mini Pkg	
Form06b-Rad SNJV Duplicate RER Data Summary	Mini Pkg	
Form06a-Rad SNJV Duplicate RPD Data Summary	Mini Pkg	
Gas Flow Raw Data		
Analytical Raw Data Run Log? Y Calibration Rpts Y Instrument Rpts Y	Mini Pkg	
Method Calibration Data		
Continuing Calibration Data		
Background Efficiency Data		
Divider-QCCHART		
Quality Control Charts		
StandardsTraceRad		
Divider-Runlogs		
Rad Runlogs		

Comments:

Yes Case Narrative to include NELAP and NELAC certification statement?
Yes Data required to be delivered by 5PM?
Yes For lateness determine include time_zone_offset in calculation?
Yes Fractional Case Narrative?
Pull narrative by WO instead of SDG. Verify that SPRP prep batches include available [redacted] comments when required.
Yes Method Calibration?
Verify correct calibration has been used for GAB
Yes RDL = PQL? If no, RDL = MDL?
Yes Radiochemistry Counting Uncertainty and TPU reported at 2 sigma?
No Show Surrogates on Sample Summary?
Package Level > 2
Yes Use GEL Standard SPC Limits?
Yes Use GEL standard MDLs/PQLs?
Yes Use Package Senddate to help determine if work order is late?

Gas Flow Direct Count- Alpha/Beta (Americium Calibration)

Batch ID	Instrument	Run Date	BKG Date	Cal Date	Geometry	DER ID	Standards
1316458	LB4100A1	22-JUL-13	20-JUL-13	01-MAY-13	2 inch Planchett		
	LB4100A2	22-JUL-13	20-JUL-13	01-MAY-13	2 inch Planchett		
	LB4100A3	22-JUL-13	20-JUL-13	01-MAY-13	2 inch Planchett		
	LB4100B1	22-JUL-13	20-JUL-13	01-MAY-13	2 inch Planchett		
	LB4100B3	22-JUL-13	20-JUL-13	01-MAY-13	2 inch Planchett		
	LB4100C1	22-JUL-13	20-JUL-13	01-MAY-13	2 inch Planchett		
	LB4100D2	22-JUL-13	20-JUL-13	01-MAY-13	2 inch Planchett		
	LB4100D3	22-JUL-13	20-JUL-13	01-MAY-13	2 inch Planchett		
	LB4100E1	22-JUL-13	20-JUL-13	01-MAY-13	2 inch Planchett		
	LB4100E2	22-JUL-13	20-JUL-13	01-MAY-13	2 inch Planchett		
	LB4100F1	22-JUL-13	20-JUL-13	01-MAY-13	2 inch Planchett		
	LB4100F2	22-JUL-13	20-JUL-13	01-MAY-13	2 inch Planchett		
	LB4100F3	22-JUL-13	20-JUL-13	01-MAY-13	2 inch Planchett		
	LB4100F4	22-JUL-13	20-JUL-13	01-MAY-13	2 inch Planchett		
	LB4100I1	22-JUL-13	20-JUL-13	01-MAY-13	2 inch Planchett		

Page 1

GEL Laboratories LLC

Final Data Package Review Checklist

GL-CHL-GC-001 Rev 0

Report Date: Tuesday, July 23, 2013

Project: [redacted]

SDG: 329760

Work Order: 329760

Submittal Level: LEVEL6

Gas Flow Direct Count- Alpha/Beta (Americium Calibration)

Batch ID	Instrument	Run Date	BKG Date	Cal Date	Geometry	DER ID	Standards
1316458	LB4100J2	22-JUL-13	20-JUL-13	01-MAY-13	2 inch Planchett		
	LB4100J3	22-JUL-13	20-JUL-13	01-MAY-13	2 inch Planchett		

Data Package Reviewed By: _____ **Date:** _____

GEL | Laboratories LLC
2040 Savage Road Charleston SC 29407
P.O. Box 30712 Charleston, SC 29417
Main: 843.556.8171 Fax: 843.766.1178
www.gel.com

APPENDIX 4: BATCH HISTORY FORM

(For Illustrative Purposes Only)

BATCH HISTORY CHECKLIST

BATCH # _____
ANALYST _____

_____Verified current revision of SOP
_____Checked Client Specific Requirements (PM comments & Pull Sheet)
_____Queue sheet (completely filled out, all line-outs initialed and dated)
_____Reagent sheet
_____Scan sheet
_____Aliquot correction sheet (if applicable)
_____Radioactive swipes taken (if applicable)
_____Limited sample volume
_____Preserved correctly

DATE Variations from Procedure

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APPENDIX 5: GAMMA ASSUMPTIONS

Liquid Library

Assumptions made regarding half-lives:	
A	B
Zr-95	Nb-95 and Nb-95m
Ru-106	Rh-106
Cs-137	Ba-137m
Ba-140	La-140
Ce-144	Pr-144
Ra-226	Bi-214 and Pb-214
Ra-228	Ac-228
Th-228	Tl-208, Bi-212, Pb-212 and Ra-224
U-235	Bi-211, Pb-211, Rn-219, Ra-223, Ac-227, Th-227 and Th-231
Np-237	Pa-233
U-238	Th-234
Am-243	Np-239
Assumptions made regarding energy lines:	
A	B
Ru-106	Rh-106
Ba-135	Cs-135
Pb-212	Th-228
Th-227	Ac-227
Ac-228	Ra-228
Th-234	U-238

Solid Library

Assumptions made regarding half-lives:	
A	B
Zr-95	Nb-95 and Nb-95m
Ru-106	Rh-106
Cs-137	Ba-137m
Ba-140	La-140
Ce-144	Pr-144
Ra-226	Bi-214 and Pb-214
Th-232	Tl-208, Bi-212, Pb-212, Ra-224, Ac-228, Ra-228 and Th-228
U-235	Bi-211, Pb-211, Rn-219, Ra-223, Ac-227, Th-227, Pa-231 and Th-231
Np-237	Pa-233
U-238	Pa-234, Pa-234m and Th-234
Am-243	Np-239
Assumptions made regarding energy lines:	
A	B
Rh-106	Ru-106
Ba-135	Cs-135
Pb-212	Th-228
Bi-214	Ra-226
Ra-223	Th-231
Th-227	Ac-227
Ac-228	Ra-228 and Th-232
Th-234	U-238

* Nuclides from column A are used to quantify the nuclides in column B

Analysis of

Chlorinated Herbicides by GC Using Methylation Derivatization

Reference Methods: **EPA 8151A,** SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update III, 1997.

MA BWSC-CAM Section VC, Quality Assurance and Quality Control Requirements and Performance Standards for SW-846 Method 8151A, for the Massachusetts Contingency Plan (MCP). Revision 2. May 28, 2004.

1. Scope and Application

Matrices: Aqueous, soil and waste.

Definitions: See Alpha Laboratories Quality Manual Appendix A

Specifically, Method 8151A may be used to determine the following compounds:

Parameter	CAS No.[a]	MOL WT.	WT as methyl ester	Conversion Factor
2,4-D [1, 2, 3]	94-75-7	221.04	235.06	0.94
2,4-DB	94-82-6	326.18	340.18	0.96
2,4,5-TP (silvex) [1, 2, 3]	93-72-1	269.51	283.51	0.95
2,4,5-T [1, 2]	93-76-5	255.48	269.48	0.95
Dalapon	75-99-0	142.96	156.96	0.91
Dicamba	1918-00-9	221.04	235.04	0.94
Dichloroprop	120-36-5	235.06	249.06	0.94
Dinoseb	88-85-7	240.21	254.21	0.95
MCPA	94-74-6	200.62	214.62	0.93
MCPP	93-65-2	214.65	228.65	0.94

[a] Chemical Abstract Service Registry Number

[1] RCRA List

[2] APA List

[3] TCLP List

Method 8151A is a capillary gas chromatographic (GC) method for determining certain chlorinated acid herbicides and related compounds in aqueous, soil and waste matrices.

Because these compounds are produced and used in various forms (i.e., acid, salt, ester, etc.), Method 8151A describes a hydrolysis step than can be used to convert herbicide esters into the acid form prior to analysis. Herbicide esters generally have a half-life of less than one week in soil.

When Method 8151A is used to analyze unfamiliar samples, compound identifications should be supported by at least one additional qualitative technique. Section 9.4.9 provides gas chromatograph/mass spectrometer (GC/MS) criteria appropriate for the qualitative confirmation of compound identifications.

The estimated detection limits for each of the compounds in aqueous and soil matrices are listed in Table 1. The detection limits for a specific waste sample may differ from those listed, depending upon the nature of the interferences and the sample matrix.

The following compounds may also be determined using this method:

Parameter	CAS No.[a]	MOL WT.	WT as methyl ester	Conversion Factor
Acifluorfen	50594-66-6	361.66	375.66	0.96
Bentazon	25057-89-0	240.28	254.28	0.94
Chloramben	133-90-4	206.02	220.02	0.93
DCPA diacid[b]	2136-79-0	303.91	317.91	0.95
3,5-Dichlorobenzoic acid	51-36-5	191.01	205.01	0.93
Picloram	1918-02-1	241.46	255.46	0.94
4-Nitrophenol	100-02-1	139.10	153.10	0.91
Pentachlorophenol	87-86-5	266.34	280.34	0.95

[a] Chemical Abstract Service Registry Number

[b] DCPA monoacid and diacid metabolites included in method scope; DCPA diacid metabolite used for validation studies. DCPA is a dimethyl ester.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Laboratory Services Manager, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the use of gas chromatography and skilled in the interpretation of gas chromatograms. Only experienced analysts should be allowed to work with diazomethane due to the potential hazards associated with its use (explosive, carcinogenic). Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability..

2. Summary of Method

This SOP for Method 8151A details gas chromatographic conditions for the analysis of chlorinated acid herbicides in water, soil, and waste samples. The hydrolysis of esters is also described.

Water, soil, and waste samples are extracted with diethyl ether and then derivitized with diazomethane. The derivatives are determined by gas chromatography with an electron capture detector (GC/ECD). The results are reported as acid equivalents.

Herbicide esters are determined using this method. Hydrolysis conditions for the esters in water and soil extracts are described.

3. Reporting Limits

The sensitivity of Method 8151A depends on the level of interferences, in addition to instrumental limitations. Table 1 lists the estimated GC/ECD detection limits that can be obtained in aqueous and soil matrices in the absence of interferences. Detection limits for a typical waste sample should be greater.

4. Interferences

4.1 Refer to Method 8000 for instrumental interferences.

4.2 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts or elevated baselines in gas chromatograms. All these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis, by analyzing reagent blanks.

4.2.1 Glassware must be scrupulously cleaned. Clean each piece of glassware as soon as possible after use by rinsing it with the last solvent used in it. This should be followed by detergent washing with hot water and rinses with tap water, then with organic-free reagent water. Glassware should be solvent-rinsed with acetone and pesticide-quality hexane. After rinsing and drying, glassware should be sealed and stored in a clean environment to prevent any accumulation of dust or other contaminants. Store glassware inverted or capped with aluminum foil. Immediately prior to use, glassware should be rinsed with the next solvent to be used.

4.2.2 The use of high purity reagents and solvents helps to minimize interference problems.

4.3 Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from waste to waste, depending upon the nature and diversity of the waste being sampled.

4.4 Organic acids, especially chlorinated acids, cause the most direct interference with the determination by methylation. Phenols, including chlorophenols, may also interfere with this procedure.

4.5 Alkaline hydrolysis and subsequent extraction of the basic solution removes many chlorinated hydrocarbons and phthalate esters that might otherwise interfere with the electron capture analysis. However, hydrolysis may result in the loss of dinoseb and the formation of aldol condensation products if any residual acetone remains from the extraction of solids.

4.6 The herbicides, being strong organic acids, react readily with alkaline substances and may be lost during analysis. Therefore, glassware must be acid-rinsed and then rinsed to constant pH with organic-free reagent water. Sodium sulfate must be acidified.

4.7 Sample extracts must be dry prior to methylation otherwise poor recoveries will be obtained.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

5.1 Lab coats, safety glasses, and gloves must be worn when handling supplies, samples, extracts, standards or solvents, and when washing glassware.

5.2 All extract concentration steps must be performed in extraction hoods. All solvent and extract transfers must also be handled in a hood.

5.3 All expired stock standards, working standards, and spent sample extracts must be placed into the waste bucket in the laboratory for future disposal by the Hazardous Waste Manager. The container must be properly labeled with hazard warning labels indicating the container contents.

5.4 Bottles containing flammable solvents must be stored in the flammables cabinet or in the vented cabinets found under the hoods. All ether bottles are to be checked for peroxide immediately upon opening.

5.5 All waste solvents must be transferred to the satellite waste storage containers located in the extraction laboratory. Separate containers are provided for chlorinated and non-chlorinated solvents and must be used accordingly. Under no circumstances are solvents to be poured down the sink drains.

5.6 Inspect all glassware prior to use. Do not use any glassware that is chipped or cracked if it could present a safety hazard. Damaged glassware is put aside for repair; otherwise the piece is discarded. A number is etched onto each KD concentrator tube to keep track of calibration (Refer to SOP/08-04, KD Tube Calibration).

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Aqueous samples are collected in two one-liter amber glass containers. Soil samples are collected in 8oz. glass jars.

6.2 Sample Preservation

Both aqueous and soil/solid samples are stored in refrigerators to maintain a temperature of 2 – 6 °C.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

Both aqueous and soil/solid samples and extracts are stored under refrigeration (2 – 6 ºC) and protected from light. Aqueous samples must be extracted within 7 days from the time of collection; soil samples, within 14 days from collection. Sample extracts must be analyzed within 40 days following extraction.

After the extraction is completed, the sample extract is stored in a stoppered KD tube before being transferred to a crimp-top or screw cap vial for analysis and long-term storage.

7. Equipment and Supplies

7.1 Gas Chromatograph, Hewlett Packard 6890: An analytical system complete with gas chromatograph configured for split-splitless injection and all required accessories including syringes, analytical columns, gases, electron capture detectors (ECD), and data system.

7.2 GC Columns: Alpha utilizes dual-column analyses. The dual-column approach involves either a single injection that is split between two columns that are mounted in a single gas chromatograph, or dual injections of the split extract on a single GC equipped with two columns. Typical column pairs used are listed below. Other columns may be used as long as method performance criteria can be met.

7.2.1 Narrow Bore Column Pair

Column 1: 30m x 0.32mm ID fused silica capillary column chemically bonded with SE-54 (DB-5, SPB-5, RTx-5, or equivalent), 0.25µm film thickness.

Column 2: 30m x 0.32mm ID fused silica capillary column chemically bonded with 50 percent phenyl methylpolysiloxane (DB-1701, or equivalent) 0.25µm film thickness.

7.2.2 Alternate Column Pair

Column 1: STX-CLP 30m x 0.32mm 0.32 um film thickness
Column 2: STX-CLPII 30m x 0.32mm ID 0.25um film thickness

8. Reagents and Standards

Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

NOTE: Store the standard solutions (stock, composite, calibration, internal, and surrogate) at 4±2 °C in Teflon(R)-sealed containers in the dark. When a large volume of standard is prepared, aliquots of that lot are stored in individual small vials. All stock standard solutions must be replaced after one year or sooner if routine QC tests indicate a problem. All other standard solutions must be replaced after six months or sooner if routine QC indicates a problem.

8.1 Reagent water: All references to water in this method refer to reagent water from Alpha's DI water treatment system.

8.2 Stock Standard Solutions: All stock standard solutions are purchased from commercial vendors as ampulated certified solutions. When an ampulated stock solution is opened, it is transferred to a labeled amber screw-cap vial. The expiration date of the stock solution is either the vendor specified expiration date, or 1 year from the date the ampule was opened, whichever is sooner. Typical stock standard concentrations are listed in Table 2

8.2.1 Internal Standard Stock Solution: 4,4'-dibromooctafluorobiphenyl (DBOB), 250 µg/mL in Acetone. Purchased commercially prepared by AccuStandard, Cat # M-8151-IS.

8.2.2 Primary Calibration Stock Standards (Initial Calibration)

8.2.2.1 2,4-Dichlorophenyl acetic acid (DCAA) methyl ester solution: 100µg/mL in Acetone. Purchased commercially prepared by Ultra Scientific, Cat # PPS-166.

8.2.2.2 Methylated Herbicides Mixture: 100 µg/mL in Methanol (except for MCPP and MCPA at 10000µg/mL). Purchased commercially prepared by Ultra Scientific, Cat # HBM-8151M.

8.2.3 Secondary Calibration Stock Standards (Continuing Calibration)

8.2.3.1 2,4-Dichlorophenyl acetic acid (DCAA) methyl ester solution: 100µg/mL in MTBE. Purchased commercially prepared by Accustandard, Cat # M515-SS.

8.2.3.2 Chlorinated Herbicides Methyl Derivatives: 100µg/mL in MTBE (except for MCPP and MCPA at 10000µg/mL. Purchased commercially prepared by Accustandard, Cat # M-8151.

8.2.4 Spiking Stock Standards

8.2.4.1 Surrogate Stock: 2,4-Dichlorophenyl acetic acid (DCAA) {underivatized}, 100µg/mL in Acetone. Purchased commercially prepared by AccuStandard, Cat # M8150B-SS.

8.2.4.2 LCS/MS Stock: Underivatized Chlorinated Herbicides, 100µg/mL in Acetone (except for MCPP and MCPA at 10000µg/mL). Purchased commercially prepared by AccuStandard, Cat # M8151A.

8.3 Calibration Standards: Calibration standards are prepared volumetrically by diluting the appropriate vendor derivatized stock standard(s) (Sections 8.2.2 and 8.2.3) with Hexane. Calibration standards expire 6 months from the date of preparation, or on the earliest expiration date of any of the stock solutions used to prepare the calibration standard. Calibrations are performed at the 6 concentration levels listed in Table 2.

8.3.1 ICAL Surrogate Stock Standard: To a 10mL volumetric flask, add 1mL of DCAA Methyl Ester Solution (Section 8.2.2.1). Bring to volume with Hexane. Final concentration is 10µg/mL.

8.3.2 Intermediate Initial Calibration Standard: To a 10mL volumetric flask, add 1mL of each stock standard listed in Section 8.2.2. Bring to volume using Acetone and Hexane. Final concentration is 10µg/mL for all analytes except MCPP and MCPA at 1000µg/mL.

8.3.2.1 Working Initial Calibration Standards

8.3.2.1.1 Cal Level 1: To a 10mL volumetric flask, add 50µL of Intermediate Initial Calibration Standard (Section 8.3.2) and 50µL of ICAL Surrogate Stock Standard (Section 8.3.1). Bring to volume with Hexane. Final concentration is 0.05µg/mL for all analytes except MCPP and MCPA at 5.0µg/mL.

8.3.2.1.2 Cal Level 2: To a 10mL volumetric flask, add 100µL of Intermediate Initial Calibration Standard (Section 8.3.2) and 100µL of ICAL Surrogate Stock Standard (Section 8.3.1). Bring to volume with Hexane. Final concentration is 0.10µg/mL for all analytes except MCPP and MCPA at 10.0µg/mL.

8.3.2.1.3 Cal Level 3: To a 10mL volumetric flask, add 200µL of Intermediate Initial Calibration Standard (Section 8.3.2) and 200µL of ICAL Surrogate Stock Standard (Section 8.3.1). Bring to volume with Hexane. Final concentration is 0.20µg/mL for all analytes except MCPP and MCPA at 20.0µg/mL.

8.3.2.1.4 Cal Level 4: To a 10mL volumetric flask, add 500µL of Intermediate Initial Calibration Standard (Section 8.3.2) and 500µL of ICAL Surrogate Stock Standard (Section 8.3.1). Bring to volume with Hexane. Final concentration is 0.50µg/mL for all analytes except MCPP and MCPA at 50.0µg/mL.

8.3.2.1.5 Cal Level 5: To a 10mL volumetric flask, add 1.0mL of Intermediate Initial Calibration Standard (Section 8.3.2) and 1mL of ICAL Surrogate Stock Standard (Section 8.3.1). Bring to volume with Hexane. Final concentration is 1.0µg/mL for all analytes except MCPP and MCPA at 100µg/mL.

8.3.2.1.6 Cal Level 6: To a 10mL volumetric flask, add 2.0mL of Intermediate Initial Calibration Standard (Section 8.3.2) and 2mL of ICAL Surrogate Stock Standard (Section 8.3.1). Bring to volume with Hexane. Final concentration is 2.0µg/mL for all analytes except MCPP and MCPA at 200µg/mL.

8.3.2.2 Intermediate Continuing Calibration Standard: To a 10mL volumetric flask, add 1mL of each stock standard listed in Section 8.2.3. Bring to volume with Hexane. Final concentration is 10µg/mL for all analytes except MCPP and MCPA at 1000µg/mL.

8.3.2.2.1 Working Continuing Calibration Standard: To a 20mL volumetric flask, add 400µL of Intermediate Continuing Calibration Standard (Section 8.3.2). Bring to volume with Hexane. Final concentration is 0.2µg/mL for all analytes except MCPP and MCPA at 20µg/mL.

8.4 Internal Standard Solution: 4,4'-dibromooctafluorobiphenyl (DBOB) is used as the internal standard, and is added to all single-component calibration standards and sample extracts to achieve a concentration of 0.25 µg/mL.

8.4.1 Working Internal Standard Solution: To a 10mL volumetric flask, add 1mL of the IS Stock (Section 8.2.1). Bring to volume with Acetone. Final concentration is 25µg/mL. 10µL of this solution is added to the 1mL sample and QC sample extracts to achieve a concentration of 0.25µg/mL for analysis.

8.5 Surrogate Standard: 2,4-Diclorophenylacetic acid (DCAA). The Surrogate standard is added to the calibration standards at the concentrations listed in Table 2, and is spiked into all samples and QC samples prior to extraction.

8.5.1 Working Surrogate Standard Solution: To a 100mL volumetric flask, add 10mL of Surrogate Stock (Section 8.2.4.1). Bring to volume with Acetone. Final concentration is 10µg/mL.

8.6 LCS/MS Spiking Solutions: The LCS/MS spiking solutions are prepared volumetrically by diluting the appropriate free acid stock standards in acetone. The spiking solution concentrations are listed in Table 2.

8.6.1 Working LCS/MS Solution: To a 25mL volumetric flask, add 2.5mL of LCS/MS Stock (Section 8.2.4.2). Bring to volume with Acetone. Final concentration is 10µg/mL for all analytes except MCPP and MCPA at 1000µg/mL.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

A matrix-specific extraction blank is performed with each extraction batch of 20 or fewer samples, according to the extraction SOPs. The extraction blank must not contain any of the reportable analytes at or above the reporting limit. If any reportable analytes are detected in the blank, the entire extraction batch is suspect and re-extraction of all associated samples is required. Appendix 1 contains specific corrective action procedures.

9.2 Laboratory Control Sample (LCS) / LCS Duplicate

A Laboratory Control Sample (LCS) and an LCS Duplicate (LCSD) is extracted with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with all target analytes. The concentrations of the spiking solutions are listed in Table 2. The recovery and RPD acceptance criteria are listed in Table 4. If any recovery criteria are not met, the extract should be reanalyzed. If the criteria are still not met, the entire batch should be re-extracted. If this is not possible, due to insufficient sample or holding time exceedence, the analyst must write up the failure on a narrative sheet for inclusion in the client report. Appendix 1 contains specific corrective action procedures.

9.3 Initial Calibration Verification (ICV)

Prepare calibration standards using the procedures in Section 8.3 and Table 2. The calibration standards are aliquoted into autosampler vials and capped prior to loading onto the autosampler tray. The calibration standards are analyzed and the curve is calculated prior to sample analysis.

9.4 Continuing Calibration Verification (CCV)

A second-source CCV standard must be injected at the beginning of each 12-hour shift prior to conducting any sample analyses. Subsequent continuing calibration standards are injected at intervals of not less than once every twenty samples.

9.5 Matrix Spike (MS)/MS Duplicate

A matrix spike/matrix spike duplicate pair is extracted and analyzed for each batch of 20 or less samples only upon client request. The spike compounds and levels are listed in Table 2. The recovery and RPD acceptance criteria are listed in Table 4. If the recovery criteria are not met, but are met in the LCS, the failure may be attributed to sample matrix effects and must be noted on a narrative sheet for inclusion in the client report. Appendix 1 contains specific corrective action procedures.

9.6 Laboratory Duplicate

Not applicable.

9.7 Method-specific Quality Control Samples

9.7.1 Surrogates

All extracted samples and associated QC are spiked with surrogates at the levels listed in Table 2. The laboratory must evaluate surrogate recovery data from individual samples and QC samples versus the surrogate control limits for both columns listed in Table 3. If the surrogate limits are not met, the extract should be reanalyzed to determine if the failure was due to an instrument problem. If the criteria are still not met, the affected samples should be re-extracted to confirm that the failure was due to sample matrix. If matrix effect is confirmed, this must be noted on a narrative sheet for inclusion in the client report. Appendix 1 contains specific corrective action procedures.

9.8 Method Sequence

Initial calibration:
Instrument Prime
Std Level 1
Std Level 2
Std Level 3
Std Level 4
Std Level 5
Std Level 6
Initial Calibration Verification Standard (ICV)

Daily sequence:

1. Instrument Prime
2. Herbicide Continuing Calibration Standard
3. Extraction Blank
4. Laboratory Control Sample

5. Laboratory Control Sample Duplicate
6. Matrix Spike (Upon Client Request)
7. Duplicate/Matrix Spike Duplicate (Upon Client Request)
8. Samples (typically 4 – 8, however up to 20 may be analyzed)
9. Herbicide Continuing Calibration Standard
10. Repeat 4 – 9 (as needed)

10. Procedure

10.1 Equipment Set-up

10.1.1 Typical Gas Chromatographic Conditions:

Temperature 1:	60 °C	**Detector temperature:**	350 °C
Ramp 1:	9°C/minute	**Carrier gas:**	Helium
Temperature 2:	96°C	**Carrier flow:**	1.7 mL/min
Ramp 2:	21°C/minute	**Carrier mode:**	Constant flow
Temperature 3:	220°C	**Makeup gas:**	Argon/methane (P5)
Ramp 3:	40°C/minute	**Total detector flow:**	55 mL/min
Final Temperature:	280°C	**Injection volume:**	1 µL
Final Time:	15 minutes		
Injector Temperature:	250°C		
Injector Mode:	Spiltless		
Injector Flow:	100mL/min		
Splitless Purge:	On at 0.45 min		
Purge Flow:	95.6 mL/min		

10.2 Initial Calibration

10.2.1 Prepare calibration standards using the procedures in Section 8.3 and Table 2. The calibration standards are aliquoted into autosampler vials and capped prior to loading onto the autosampler tray. The calibration standards are analyzed and the curve is calculated prior to sample analysis.

10.2.2 Calibration Factors

Internal standard calibration techniques are employed in this method.

10.2.2.1 Internal Standard Procedure. In each standard, calculate the response factor (RF) for each analyte, the average RF, and the relative standard deviation (RSD) of the RFs, using the Target data processing software. The calculations are performed automatically, using the formulae listed in Alpha's Quality Manual.

10.2.3 Initial Calibration Criteria

If the RSD for an analyte is $\leq$ 20%, then the response of the instrument for this compound is considered linear over the range and the mean calibration factor (Avg. RF) can be used to quantitate sample results. (Section 9.6.3).

If the RSD for any analyte is > 20%, then linearity through the origin cannot be assumed. The mean response factor cannot be used for quantitation. An alternative calculation may be done by the use of linear regression as long as the correlation coefficient is $\geq$0.99. (The regression equation cannot be forced

through the origin.) The equation for linear regression may be found in Alpha's Quality Manual.

If both of these quantitation methods fail criteria for any compound in the initial calibration, then the system must be reevaluated and a new calibration curve must be analyzed.

10.2.4 Retention Time Windows
The retention time windows used for the identification of target analytes is ± 0.05 minutes. These criteria have been adopted from the EPA CLP Statement of Work (OLM04.2). It has been found that these limits work well, being wide enough to eliminate false-negatives while being tight enough to eliminate false-positives. Windows that are calculated using the procedure recommended in Method 8000 tend to be very narrow, creating the risk of false negative results. The Retention Times are listed in Table 3.

10.2.4.1 The windows listed above are used as guidance, however the experience of the analyst weighs heavily in the interpretation of the chromatograms. For example, it has been observed that certain oil matrices can cause the retention times to shift more dramatically. Additionally, if any positive results are questionable and at a sufficiently high concentration, GC/MS analysis is used for confirmation.

10.2.5 The following compounds often co-elute using the dual column analysis scheme:

- DB-5: 2,4-DB / Dinoseb
- DB-1701: 2,4-D / Pentachlorophenol 2,4-DB / Chloramben

NOTE: 2,4-DB does not co-elute on both channels at the same time.

10.3 Equipment Operation and Sample Processing

10.3.1 The same GC operating conditions used for the initial calibration must be employed for sample analyses, including sample injection volume (Section 9.1.10).

10.3.2 Tentative identification of an analyte occurs when a peak from a sample extract falls within the retention time window for the compound. Each tentative identification is confirmed using a second GC column of dissimilar stationary phase. In particularly difficult matrices, confirmation by GC/MS may be advisable (see Section 9.4.9).

10.3.3 The concentration reported for an identified target analyte in an extract is calculated using the Target data processing software. The Target methods have been configured to utilize the quantitation formulas found in Alpha's Quality Manual. Proper quantitation requires the appropriate selection of a baseline from which the peak area or height can be determined. See the Manual Integration SOP for integration guidelines. Also refer to Section 9.6 for calculations.

10.3.3.1 If the responses exceed the calibration range of the system, dilute the extract and reanalyze.

10.3.4 Each sample analysis must be bracketed with an acceptable initial calibration, calibration verification standard(s) (each 12-hour analytical shift), or calibration standards interspersed within the samples. When a calibration verification

standard fails to meet the QC criteria, all samples that were injected after the last standard that last met the QC criteria must be re-injected.

10.3.5 Sample injections may continue for as long as the calibration verification standards and standards interspersed with the samples meet instrument QC requirements. Standards are analyzed after every 4 – 8 samples to minimize the number of samples that must be re-injected should the standards fail the QC limits. The sequence ends when the set of samples has been injected or when qualitative and/or quantitative QC criteria are exceeded.

10.3.6 Use the calibration standards analyzed during the sequence to evaluate retention time stability. The retention time windows are established using the absolute retention time of each analyte in the mid-concentration standard during the initial calibration as the mid-point of the window. The widths of the windows are defined in Section 9.2.4.

10.3.7 Each subsequent injection of a standard during the 12-hour analytical shift (i.e., those standards injected every 20 samples, or more frequently) must be checked against the retention time windows. If any of these subsequent standards fall outside their absolute retention time windows, the GC system is out of control. Determine the cause of the problem and correct it. If the problem cannot be corrected, a new initial calibration must be performed.

10.3.8 If compound identification or quantitation is precluded due to interference (e.g., broad, rounded peaks or ill-defined baselines are present) cleanup of the extract may be needed. If instrument problems are suspected, rerun the extract on another instrument to determine if the problem results from analytical hardware or the sample matrix. Refer to the extraction SOPs for the procedures to be followed in sample cleanup.

10.3.9 GC/MS Confirmation

GC/MS confirmation may be used in conjunction with either single-column or dual-column analysis if the concentration is sufficient for detection by GC/MS.

10.3.9.1 Full-scan GC/MS will normally require a concentration of approximately 10ng/µL in the final extract for each single-component compound.

10.3.9.2 The GC/MS must be calibrated for the specific target herbicides when it is used for quantitative analysis.

10.3.9.3 GC/MS may not be used for confirmation when concentrations are below the sensitivity of the instrument.

10.3.9.4 GC/MS confirmation should be accomplished by analyzing the same extract that is used for GC/ECD analysis.

10.3.9.5 The base/neutral/acid extract and the associated blank may be used for GC/MS confirmation if the surrogates and internal standards do not interfere and if it is demonstrated that the analyte is stable during acid/base partitioning. However, if the compounds are not detected in the base/neutral/acid extract, then GC/MS analysis of the pesticide extract should be performed.

10.3.9.6 A QC reference sample containing the compound should also be analyzed by GC/MS. The concentration of the QC reference sample must demonstrate that those pesticides identified by GC/ECD can be confirmed by GC/MS.

10.3.10 If a co-elution pair (Section 9.2.5) exists, the target compounds must be reported from the non-coelution channel.

10.4 Continuing Calibration

10.4.1 A second-source CCV standard must be injected at the beginning of each 12-hour shift prior to conducting any sample analyses. Subsequent continuing calibration standards are injected at intervals of not less than once every 10 samples.

10.4.1.1 The calibration factor (for external standard compounds) and response factor (for internal standard compounds) for each analyte to be quantitated must not exceed a ± 15% difference when compared to the initial calibration curve. The Target data processing software automatically calculates the %D for all analytes according to the formula in Alpha's Quality Manual

If this criterion is exceeded, inspect the gas chromatographic system to determine the cause and perform whatever maintenance is necessary before verifying calibration and proceeding with sample analysis.

10.4.1.2 If routine maintenance does not return the instrument performance to meet the QC requirements (Section 10) based on the last initial calibration, then a new initial calibration must be performed. Due to the large number of analytes present, allowances may be made for a CF or RF that drifts out high, as long as there are no positive hits for that particular analyte in any of the associated samples. Any QC failures must be written up by the analyst on narrative sheets for inclusion with the sample data.

10.4.2 Compare the retention time of each analyte in the calibration standard with the absolute retention time windows described in section 9.2.4. The center of the absolute retention time window for each analyte is its retention time in the mid-concentration standard analyzed during the initial calibration. Each analyte in each standard must fall within its respective retention time window. If not, the gas chromatographic system must either be adjusted so that a second analysis of the standard does result in all analytes falling within their retention time windows, or a new initial calibration must be performed and new retention time windows established.

10.4.3 Co-elution pairs (Section 10.2.5) will often separate or come together as the length of the column changes. If a CCV shows a different co-elution pattern than the Initial Calibration, the instrument must be recalibrated for the new pattern(s) and any samples analyzed since the last acceptable calibration verification must be reanalyzed.

10.5 Preventive Maintenance

Routine preventive maintenance should be performed to maintain GC system performance. This includes periodic replacement of injector septa, replacement of injector liner(s), and replacement of injector seals. All maintenance is noted in individual Instrument Maintenance Logbooks.

11. Data Evaluation, Calculations and Reporting

Herbicides are calculated as described in Section 9.4.3, and reported in µg/L or µg/Kg units. After performing technical data review, validating that all QC criteria have been met and confirming all positive hits, the data report is sent electronically to the LIMS computer for generation of the client report. Two levels of review of the data in the LIMS system are preferred prior to release of data. These reviews include analyst review and review by the Department Supervisor or other trained analyst.

11.1 Aqueous Calculation

$$\mu g/L = \frac{(A_s)\,(C_{IS})\,(D)\,(V_f)}{(A_{IS})\,(\overline{RF})\,(V_s)\,(1000)}$$

Where:

A_s = Peak area of analyte
A_{IS} = Peak area of Internal Standard
C_{IS} = Concentration of IS (µg/L)
D = Dilution factor (D = 1 for no dilution)
V_f = Final extract volume (mL)
Vs = Initial volume extracted (mL)

$\overline{RF}$ = Mean response factor

11.2 Soil/Solid Calculation

$$\mu g/Kg = \frac{(A_s)\,(C_{IS})\,(D)\,(V_f)}{(A_{IS})\,(\overline{RF})\,(W_s)\,(1000)}$$

Where:

A_s = Peak area of analyte
A_{IS} = Peak area of Internal Standard
C_{IS} = Concentration of IS (µg/L)
D = Dilution factor (D = 1 for no dilution)
V_f = Final extract volume (mL)
Ws = Initial weight extracted (g)

$\overline{RF}$ = Mean response factor

11.2.1 Response Factor Calculation

$$RF = \frac{(A_s)\,(C_{IS})}{(A_{IS})\,(C_s)}$$

Where:

A_s = Peak area of analyte
A_{IS} = Peak area of Internal Standard
C_s = Concentration of analyte (µg/L)
C_{IS} = Concentration of IS (µg/L)

11.3 Molecular Weight Correction

In addition, conversion factors for the molecular weights have been listed for all analytes reported under EPA Method 8151.

11.3.1 The calibration is performed using the methyl ester compounds; therefore the calculation of the concentration includes a correction for the molecular weight of the methyl ester versus the acid herbicide.

Derivatized herbicides are used for the standard curve, and they are supplied as

methyl esters as opposed to acid herbicide form which is used for the LCS. The difference between the acid form and the methyl ester is + 14 (i.e. In Derivatization, a -CH_3 group replaces the -H, (15-1 = 14)). The factors used for correction (listed in the Table below) vary for each herbicide and are applied to the standard concentration at each level of the calibration within the target processing software.

Example: 2,4-D

2,4-D MW = 221.04 2,4-D Methyl ester MW = 235.06

If you spike 10 ug of 2,4-D, and derivatized it, you end up with 10.634 ug of 2,4-D Methyl ester that you are measure, as you've stripped the -H and added a -CH3, increasing the molecular weight. The true value for the spike is 10 ug as the acid herbicide, and 10.634 ug as the methyl ester. So a spike of 10 ug of acid, would measure 10.634 ug of the methyl ester to get 100% recovery. The true value for the spike would be 10.634 as methyl ester, which you use to divide your measured value by.

If measuring recoveries, the value of the 10 ug of the methyl ester, would be 10 ug measured (as methyl ester)/10.634 ug spiked (as methyl ester) = 94.04%.

If, on the other hand you want to convert your final result using a correction factor, you would use the MW of the acid/MW of the ester (221.04/235.06 = 0.94).

Conversion Factors:

Parameter	CAS No.[a]	MOL WT.	WT as methyl ester	Conversion Factor
2,4-D [1, 2, 3]	94-75-7	221.04	235.06	0.94
2,4-DB	94-82-6	326.18	340.18	0.96
2,4,5-TP (silvex) [1, 2, 3]	93-72-1	269.51	283.51	0.95
2,4,5-T [1, 2]	93-76-5	255.48	269.48	0.95
Dalapon	75-99-0	142.96	156.96	0.91
Dicamba	1918-00-9	221.04	235.04	0.94
Dichloroprop	120-36-5	235.06	249.06	0.94
Dinoseb	88-85-7	240.21	254.21	0.95
MCPA	94-74-6	200.62	214.62	0.93
MCPP	93-65-2	214.65	228.65	0.94

Parameter	CAS No.[a]	MOL WT.	WT as methyl ester	Conversion Factor
Acifluorfen	50594-66-6	361.66	375.66	0.96
Bentazon	25057-89-0	240.28	254.28	0.94
Chloramben	133-90-4	206.02	220.02	0.93
DCPA diacid[b]	2136-79-0	303.91	317.91	0.95
3,5-Dichlorobenzoic acid	51-36-5	191.01	205.01	0.93
Picloram	1918-02-1	241.46	255.46	0.94
4-Nitrophenol	100-02-1	139.10	153.10	0.91
Pentachlorophenol	87-86-5	266.34	280.34	0.95

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Refer to Section 10 for specific corrective actions.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Qualtrax Document ID 1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/08-12 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

Qualtrax ID 1732 MDL/LOD/LOQ Generation

Qualtrax ID 1739 IDC/DOC Generation

Qualtrax ID 1728 Waste Management and Disposal SOP

16. Attachments

Table 1: REPORTING LIMITS

Table 2: STANDARD SOLUTIONS

Table 3: RETENTION TIMES OF CHLORINATED HERBICIDES

Table 4: QC ACCEPTANCE CRITERIA

Appendix 1: Table V C-1 Specific QA/QC Requirements and Performance Standards

TABLE 1

REPORTING LIMITS

Herbicides	RL (Aqueous)	RL (Soil)
2,4-D	1.0 ug/L	33 ug/kg
Dalapon	1.0 ug/L	33 ug/kg
2,4-DB	1.0 ug/L	33 ug/kg
Dicamba	1.0 ug/L	33 ug/kg
Dichloroprop	1.0 ug/L	33 ug/kg
Dinoseb	1.0 ug/L	33 ug/kg
MCPA	100.0 ug/L	3300 ug/kg
MCPP	100.0 ug/L	3300 ug/kg
Silvex (2,4,5-TP)	1.0 ug/L	33 ug/kg
2,4,5-T	1.0 ug/L	33 ug/kg

TABLE 2

STANDARD SOLUTIONS

Compound	Level 6	Level 5	Level 4	Level 3	Level 2	Level 1	Std. Spike Concentration *	Std. LCS Concentration *
	(µg/mL)	(µg/mL)	(µg/mL)	(µg/mL)	(µg/mL)	(µg/mL)	(µg/mL)	(µg/mL)
2,4-D	2.0	1.0	0.5	0.2	0.1	0.05	10	10
Dalapon	2.0	1.0	0.5	0.2	0.1	0.05	10	10
2,4-DB	2.0	1.0	0.5	0.2	0.1	0.05	10	10
Dicamba	2.0	1.0	0.5	0.2	0.1	0.05	10	10
Dicloroprop	2.0	1.0	0.5	0.2	0.1	0.05	10	10
Dinoseb	2.0	1.0	0.5	0.2	0.1	0.05	10	10
MCPA	200	100	50	20	10	5	1000	1000
MCPP	200	100	50	20	10	5	1000	1000
Silvex (2,4,5-TP)	2.0	1.0	0.5	0.2	0.1	0.05	10	10
2,4,5-T	2.0	1.0	0.5	0.2	0.1	0.05	10	10
3,5-Dichlorobenzoic Acid	2.0	1.0	0.5	0.2	0.1	0.05	10	10
4-Nitrophenol	2.0	1.0	0.5	0.2	0.1	0.05	10	10
Chloramben	2.0	1.0	0.5	0.2	0.1	0.05	10	10
Pentachlorophenol	2.0	1.0	0.5	0.2	0.1	0.05	10	10
Bentazon	2.0	1.0	0.5	0.2	0.1	0.05	10	10
Picloram	2.0	1.0	0.5	0.2	0.1	0.05	10	10
DCPA Diacid	2.0	1.0	0.5	0.2	0.1	0.05	10	10
Acifluorfen	2.0	1.0	0.5	0.2	0.1	0.05	10	10
DCAA (Surrogate)	2.0	1.0	0.5	0.2	0.1	0.05	10	10

***** Calculations are based on an initial aqueous sample volume of 1L**,** initial soil sample weight of 30g, and final extract volume of 10mL.

TABLE 3

Typical RETENTION TIMES (minutes) of METHYL DERIVATIVES of CHLORINATED HERBICIDES

ANALYTE	Narrow-Bore Columns	
	Primary Column	Confirmation Column
Dalapon	3.9	4.0
3,5-Dichlorobenzoic Acid	15.9	16.0
4-Nitrophenol	18.3	16.12
DCAA (surrogate)	19.4	18.8
Dicamba	19.9	19.1
Dichloroprop	22.2	21.3
2,4-D	23.2	21.8
DBOB (internal standard)	22.8	22.9
Pentachlorophenol	23.3	23.6
Chloramben	27.2	24.5
2,4,5-TP	25.1	24.4
2,4,5-T	26.2	25.0
2,4-DB	27.2	26.3
Dinoseb	28.6	26.4
Bentazon	29.1	27.0
Picloram	31.2	28.0
DCPA diacid[a]	29.8	29.1
Acifluorfen	35.6	33.4
MCPP	20.5	19.9
MCPA	21.3	20.3

Primary Column: **DB-5:** 30m x 0.32mm ID fused silica capillary column chemically bonded with SE-54 (DB-5, SPB-5, RTx-5 , or equivalent), 0.25µm film thickness.

Confirmation Column: **DB-1701:** 30m x 0.32mm ID fused silica capillary column chemically bonded with 50 percent phenyl methylpolysiloxane (DB-1701, or equivalent) 0.25µm film thickness.

Temperature Program: See Section 10.1

Helium Carrier Flow: 1.7mL/min

Injection Volume: 1µL, splitless, 45 sec delay

Injector Temperature: 250ºC

Detector Temperature: 320ºC

[a] DCPA monoacid and diacid metabolites included in method scope; DCPA diacid metabolite used for validation studies. DCPA is a dimethyl ester.

TABLE 4
QC ACCEPTANCE CRITERIA

	Aqueous		Soil	
Surrogate % Recovery	**Lower Control Limit**	**Upper Control Limit**	**Lower Control Limit**	**Upper Control Limit**
2,4-Diclorophenylacetic acid (DCAA)	30%	150%	30%	150%

	Aqueous % Recovery		Soil % Recovery	
LCS	**Lower Control Limit**	**Upper Control Limit**	**Lower Control Limit**	**Upper Control Limit**
2,4-D	30%	150%	30%	150%
dalapon	30%	150%	30%	150%
2,4-DB	30%	150%	30%	150%
dicamba	30%	150%	30%	150%
dicloroprop	30%	150%	30%	150%
dinoseb	30%	150%	30%	150%
MCPA	30%	150%	30%	150%
MCPP	30%	150%	30%	150%
Silvex (2,4,5-TP)	30%	150%	30%	150%
2,4,5-T	30%	150%	30%	150%

MCP only	**Aqueous % Recovery**		**Soil % Recovery**		**% RPD**	
LCSD	**Lower Control Limit**	**Upper Control Limit**	**Lower Control Limit**	**Upper Control Limit**	**Aqueous**	**Soil**
2,4-D	40%	140%	40%	140%	≤ 25%	≤ 25%
dalapon	40%	140%	40%	140%	≤ 25%	≤ 25%
2,4-DB	40%	140%	40%	140%	≤ 25%	≤ 25%
dicamba	40%	140%	40%	140%	≤ 25%	≤ 25%
dicloroprop	40%	140%	40%	140%	≤ 25%	≤ 25%
dinoseb	40%	140%	40%	140%	≤ 25%	≤ 25%
MCPA	40%	140%	40%	140%	≤ 25%	≤ 25%
MCPP	40%	140%	40%	140%	≤ 25%	≤ 25%
Silvex (2,4,5-TP)	40%	140%	40%	140%	≤ 25%	≤ 25%
2,4,5-T	40%	140%	40%	140%	≤ 25%	≤ 25%

	Aqueous % Recovery		Soil % Recovery		Duplicate and/or MSD	
MS / MSD	**Lower Control Limit**	**Upper Control Limit**	**Lower Control Limit**	**Upper Control Limit**	**Aqueous RPD**	**Soil RPD**
2,4-D	30%	150%	30%	150%	30%	30%
dalapon	30%	150%	30%	150%	30%	30%
2,4-DB	30%	150%	30%	150%	30%	30%
dicamba	30%	150%	30%	150%	30%	30%
dicloroprop	30%	150%	30%	150%	30%	30%
dinoseb	30%	150%	30%	150%	30%	30%
MCPA	30%	150%	30%	150%	30%	30%
MCPP	30%	150%	30%	150%	30%	30%
Silvex (2,4,5-TP)	30%	150%	30%	150%	30%	30%
2,4,5-T	30%	150%	30%	150%	30%	30%

Appendix 1: Table V C-1 Specific QA/QC Requirements and Performance Standards

Massachusetts Department of Environmental Protection Bureau of Waste Site Cleanup	BWSC-CAM	Table V C-1
Quality Assurance and Quality Control Requirements and Performance Standards for SW-846 Method 8151A, Chlorinated Herbicides by Gas Chromatography	28 May 2004	Revision No. 2
	Final	Page 11 of 20

Title: **Specific QA/QC Requirements and Performance Standards for SW-846 Method 8151A**

Required QA/QC Parameter	Data Quality Objective	Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
Retention Time Windows	Laboratory Analytical Accuracy	(1) Prior to initial calibration and when a new GC column is installed. (2) Calculated according to the method. (Section 7.6 of SW-846 8000).	No	NA	NA
Initial Calibration	Laboratory Analytical Accuracy	(1) Minimum of 5 standards. (2) Low standard must be ≤ reporting limit. (3) %RSD should be ≤20 or "r" should be ≥0.99. (4) If regression analysis is used, the curve must not be forced through the origin. (5) Curves must be verified by an independent ICV before analysis. (6) All standards must be derivitized using the same procedures used for samples, whether prepared in the laboratory or purchased from a vendor.	No	Recalibrate as required by method.	Report exceedances in case narrative.
Continuing Calibration (CCAL)	Laboratory Analytical Accuracy	(1) Prior to samples, every 12 hours or every 10 samples, whichever is more frequent, and at the end of the analytical sequence. (2) Concentration level near midpoint of curve. (3) Percent difference or percent drift of calibration factors should be ≤15. (4) Verify all analytes fall within retention time windows. (5) All standards must be derivitized using the same procedures used for samples, whether prepared in the laboratory or purchased from a vendor.	No	(1) Perform instrument maintenance, reanalyze CCAL and/or recalibrate as required by method. (2) Reanalyze "associated samples" if beginning or closing CCAL exhibited low response and associated herbicides were or were not detected in samples. (3) Reanalyze "associated samples" if beginning or closing CCAL exhibited high response and associated herbicides were detected in the samples. NOTE: "Associated Samples" refers to all samples analyzed since the last acceptable CCAL.	Report exceedances in case narrative.
Method Blanks	Laboratory Method Sensitivity (contamination evaluation)	(1) Extracted with every batch or every 20 samples, whichever is more frequent. (2) Matrix-specific (e.g., water, soil). (3) Target analytes must be less than or equal to reporting limit.	Yes	Locate source of contamination; correct problem; re-extract associated samples if contaminants are present in the method blank.	(1) Report nonconformances in case narrative. (2) If contamination of method blanks is suspected or present, the laboratory, using a "B" flag or some other convention (such as the case narrative), should qualify the sample results. (3) If re-extraction is performed within holding time, the laboratory may report results of the re-extraction only. (4) If re-extraction is performed outside of holding time, the laboratory must report results of both the initial extraction and re-extraction.

Massachusetts Department of Environmental Protection Bureau of Waste Site Cleanup	BWSC-CAM	Table V C-1
Quality Assurance and Quality Control Requirements and Performance Standards for SW-846 Method 8151A, Chlorinated Herbicides by Gas Chromatography	28 May 2004	Revision No. 2
	Final	Page 12 of 20

Title: **Specific QA/QC Requirements and Performance Standards for SW-846 Method 8151A**

Required QA/QC Parameter	Data Quality Objective	Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
Laboratory Control Spikes (LCSs)	Laboratory Method Accuracy	(1) Extracted with every batch or every 20 samples, whichever is more frequent. (2) Prepared using standard source different than used for initial calibration. (3) Concentration level should be ≤ mid-level standard. (4) Must contain all target analytes. (5) Matrix-specific (e.g., soil, water). (6) Percent recoveries must be between 40-140. (7) Laboratories are expected to develop their own in-house control limits, which should fall within the limits listed above.	Yes	Recalculate the percent recoveries. Check MS/MSD; if recoveries are acceptable in MS/MSD, nonconformance may be isolated to LCS. If recoveries are outside criteria in MS/MSD, re-extract associated samples.	(1) Report exceedances in case narrative. (2) If re-extraction is performed within holding time, the laboratory may report results of the re-extraction only. (3) If re-extraction is performed outside of holding time, the laboratory must report results of both the initial extraction and re-extraction.
LCS Duplicate	Laboratory Method Precision	(1) Analyzed with every batch or every 20 samples, whichever is more frequent. (2) Prepared using same standard source and concentration as LCS. (3) Must contain all target analytes. (4) Analyze immediately after LCS. (5) Laboratory-determined percent recoveries must be between 70 – 130 for target compounds (6) Matrix-specific (e.g., soil, water); and (7) Laboratory-determined Relative Percent Difference (RPD) must be ≤ 25.	Yes	Recalculate RPD; Locate source of problem; Narrate non-conformances	(1) Locate and rectify source of non-conformance before proceeding with the analyses of subsequent sample batches. (2) Narrate non-conformances
MS/MSDs	Method Accuracy in Sample Matrix Method Precision in Sample Matrix	(1) Extracted with every 20 samples (BATCH QC). (2) Matrix-specific. (3) Prepared using standard source different than that used for initial calibration. (4) Concentration level should be between low and mid-level standard. (5) Must contain all target analytes. (6) Percent recoveries should be between 30-150. (7) RPDs should be ≤30 for single-component analytes and ≤50 for multi-component analytes.	Yes (Only when requested by the data-user)	Check LCS; if recoveries acceptable in LCS, evaluate potential cleanup techniques (Section 7.2.4 of SW-846 8151A) for samples associated with MS/MSD.	Report exceedances in case narrative.

Massachusetts Department of Environmental Protection Bureau of Waste Site Cleanup

Quality Assurance and Quality Control Requirements and Performance Standards for SW-846 Method 8151A, Chlorinated Herbicides by Gas Chromatography

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Title: **Specific QA/QC Requirements and Performance Standards for SW-846 Method 8151A**

Required QA/QC Parameter	Data Quality Objective	Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
Surrogates	Accuracy in Sample Matrix	(1) Minimum of 1, that encompasses range of temperature program used in method and does not interfere with the target analytes. Recommended surrogate: DCAA (2) Percent recoveries must be between 30-150 for surrogate on both columns. (3) Laboratories are expected to develop their own in-house control limits, which should fall within the limits listed above. (4) If the surrogate exceeds limits on one column and greater than RL concentrations on both columns are not comparable (RPD > 40), re-extract and re-analyze the sample	Yes (report surrogate recoveries from both columns)	(1) If the surrogate is outside limits on both columns, re-extract and re-analyze the sample. (2) If a surrogate is diluted to a concentration below that of the lowest calibration standard, no corrective action is necessary. (3) If the surrogate exceeds limits on one column only, and the results on both columns are below RLs, no corrective action is necessary. (4) If the surrogate exceeds limits on one column only, and results on both columns are comparable (RPD < 40), then no corrective action is necessary.	(1) Report exceedances in case narrative. (2) If re-extraction or reanalysis yields similar surrogate nonconformances, the laboratory should report results of both extractions or analyses. (3) If re-extraction or reanalysis is performed within holding time and yields acceptable surrogate recoveries, the laboratory may report results of the re-extraction or reanalysis only. (4) If re-extraction or reanalysis is performed outside of holding time and yields acceptable surrogate recoveries, the laboratory must report results of both the initial and re-extraction or reanalysis. (5) If sample is not re-extracted or reanalyzed due to obvious interference, the laboratory must provide the chromatogram in the data report.
Internal Standards *(Optional)*	Laboratory Analytical Accuracy and Method Accuracy in Sample Matrix	(1) Minimum of 1. Recommended Internal Standard: DBOB (2) Area counts in samples must be between 50 – 200% of the area counts in the associated continuing calibration standard. (3) Retention times of internal standards must be within calculated retention time windows.	No	If internal standard is outside limits, reanalyze sample unless obvious interference present.	(1) Report exceedances in case narrative. (2) If reanalysis yields similar internal standard nonconformance, the laboratory should report both results of both analyses. (3) If reanalysis is performed within holding time and yields acceptable internal standard recovery, the laboratory may report results of the reanalysis only. (4) If reanalysis is performed outside of holding time and yields acceptable internal standard recovery, the laboratory must report results of both analyses. (5) If sample is not reanalyzed due to obvious interference, the laboratory must provide the chromatogram in the data report.

Massachusetts Department of Environmental Protection Bureau of Waste Site Cleanup	BWSC-CAM	Table V C-1
Quality Assurance and Quality Control Requirements and Performance Standards for SW-846 Method 8151A, Chlorinated Herbicides by Gas Chromatography	28 May 2004	Revision No. 2
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Title: **Specific QA/QC Requirements and Performance Standards for SW-846 Method 8151A**

Required QA/QC Parameter	Data Quality Objective	Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
Identification and Quantitation	Inter-laboratory consistency	(1) Laboratory should use the average calibration factor of each analyte for quantitation. (2) Secondary column analysis: Laboratory must utilize a second dissimilar column to confirm positive herbicide results. The laboratory must report the higher of the two results unless obvious interference is present on one of the columns in which case the laboratory can report the lower result. All required QA/QC parameters (e.g., calibrations, LCSs, etc.) must be met on the secondary column as well. (3) If calibration standards are prepared using methyl esters, the calculation of concentration must include a correction for the molecular weight of the methyl ester versus the acid herbicide.	No	NA	If the RPD between the dual column results exceeds 40, the laboratory should qualify the sample results and/or note the exceedance in the case narrative. NOTE: If the high RPD can be definitively attributed to interference on one of the two columns, the laboratory should report the lower value and provide a discussion in the case narrative that this approach was employed.
General Reporting Issues	NA	(1) The laboratory must report values ≥ the sample-specific reporting limit; optionally, values below the sample-specific reporting limit can be reported as estimated, if requested. The laboratory must report results for samples and blanks in a consistent manner. (2) Dilutions: If diluted and undiluted analyses are performed, the laboratory should report results for the lowest dilution within the valid calibration range for each analyte. The associated QC (e.g., method blanks, surrogates, etc.) for each analysis must be reported.	Yes	NA	(1) Qualification of the data is required if reporting values below the sample-specific reporting limit. (2) Recommendation is that the reporting of diluted and undiluted analyses should be a must to be consistent with the inorganic reporting convention of reporting all dilutions and to ensure the lowest possible reporting limit can be achieved if the data is available.

GC = Gas Chromatography
MS/MSDs = Matrix Spikes/Matrix Spike Duplicates
%RSD = Percent Relative Standard Deviation
DCAA = 2,4-Dichlorophenylacetic Acid

"r" = Correlation Coefficient
RPDs = Relative Percent Differences
ICV = Initial Calibration Verification – separate source standard
DBOB = 4,4'-Dibromooctafluorobiphenyl

Pr... ...k, the published version of the document should be viewed online.

Ignitability of Solids

Reference Method: **Method 1030**, Rev. 0, December 1996

Reference: SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update III, 1997.

1. Scope and Application

Matrices: Solids including pastes, powders, granular material, soils, clay and solids that can be cut into strips.

Definitions: See Alpha Laboratories Quality Manual Appendix A

This method may be used to meet certain regulatory applications; with respect to the characteristic of ignitability in CFR 261.21, this method may be used, but is not required, to determine whether a solid waste "when ignited, burns so vigorously and persistently that is creates a hazard." If it is impractical to perform the test because the physical form of the sample, generator knowledge should be used to determine the ignitability hazard posed by the material.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Laboratory Services Manager, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method by completing the record of training.

2. Summary of Method

In a preliminary test, the test material is formed into an unbroken strip or powder train 250mm in length. An ignition source is applied to one end of the test material to determine whether combustion will propagate along 200mm of the strip within a specified time period. Materials that propagate burning along a 200mm strip within the specified time period are then subjected to a burning rate test. Materials that do not ignite or propagate combustion as described above do not require further testing.

In the burning rate test, the burning time is measured over a distance of 100mm and the rate of burning is determined. The test method described here is based on the test procedure adopted by the Department of Transportation from the United Nations regulations for the international transportation of dangerous goods and is contained in Appendix E to Part 173 of 49 CFR.

2.1 Method Modifications from Reference

A high temperature gas torch is utilized as the flame source.

3. Detection Limits

There is no reportable detection limit for this analysis. Results are reported as either Negative or Positive. Refer to Section 11.2 for data reporting.

4. Interferences

In Laboratory tests, the burning rate of duplicate runs is usually repeatable to within 10%. However, large differences in burning rates may occur if experimental conditions are not held constant. Variation in airflow rates, particle size and moisture content of the test material will affect test results. Therefore, at least triplicate determinations of the burning rate are conducted.

Particle size of test material can affect not only the burning rate, but also the ignition of the material. Therefore particle size of the test material should be the same for each test run. Report the particle size of the test material in a simple descriptive format (e.g. fine powder, sand, coarse granular).

Temperature of some test material such as sulfur powder affects the burning rate. For reproducible results, all tests must be performed at approximately the same initial temperature (ambient room or laboratory temperature).

All tests must be carried out inside a fume hood with the test apparatus situated perpendicular (90°) to the direction of the airflow. Airflow parallel (0°) to the test apparatus results in non-reproducible burning rates.

The rate of airflow through the fume hood affects the burning rate. Too high an airflow will distort the flame and retard its horizontal propagation. The optimum airflow is within the range of 0.7 – 1 meter per second.

Materials that are moisture sensitive (i.e. readily absorb moisture from the air) should be tested as quickly as possible after removal from the sample container. All materials should be tested as received by the laboratory.

5. Health and Safety

Each sample should be treated as a potential health hazard. From this viewpoint, exposure must be reduced to the lowest possible level by whatever means available. References to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents. These practices include, but are not limited to, the use of: laboratory coat, safety glasses, and latex or nitrile gloves, and/or heat resistant gloves. Use caution when operating the high temperature torch. Also use caution in handling the ceramic plate after heating the sample, as it remains hot.

Prior to starting the preliminary test, all sample materials must be tested to determine if that material is explosive or extremely flammable. Use a very small portion of material (1 gram or less). ***If the sample displays explosivity or extreme flammability, do not continue this test.***

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Sample containers should be completely filled and tightly sealed to preserve sample integrity.

6.2 Sample Preservation

No sample preservation is required.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

All samples are tested as received unless requested otherwise. Sample aliquots should be tested as soon as possible after removal from the sample container (i.e. samples must not be allowed to dry or absorb moisture for excessive periods or to lose volatiles). Allow samples to equilibrate to the ambient laboratory temperature in the sample container.

7. Equipment and Supplies

7.1 Ceramic Tile: Low heat conducting, non-combustible and impervious with marks indicating 80mm, 180mm, 200mm and 250mm along the test path. Any material capable to withstand high temperatures that have marks indicating 80mm, 180mm, 200mm and 250mm can be used.

7.2 Powder Train Mold: For molding powdered and granular materials for the burn rate test. The material of construction can be aluminum, brass, stainless steel or plastic. The mold is 250mm in length and has a triangular cross-section, with a width of 20mm, and a depth of 10mm as measured from the bottom of the triangular opening to where the sides meet. On both sides of the mold, in the longitudinal direction, two sheets are mounted as lateral limitations that extend 2mm beyond the upper edge of the triangular cross-section.

7.3 High temperature gas torch: A 6 to 7cm flame, with a minimum diameter of 5mm capable of attaining a temperature of at least 1000 °C. MAPP gas is its fuel source.

7.4 Stop watch.

7.5 Thermocouple: To measure the temperature of the gas flame

7.6 Thermometer: To measure initial temperature of material (i.e. room temperature).

7.7 Anemometer: To measure airflow in the fume hood.

7.8 Fume Hood.

8. Reagents and Standards

No special reagents are required to conduct this test.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

Not applicable.

9.2 Laboratory Control Sample (LCS)

Not applicable.

9.3 Initial Calibration Verification (ICV)

Not applicable.

9.4 Continuing Calibration Verification (CCV)

Not applicable.

9.5 Matrix Spike

Not applicable.

9.6 Laboratory Duplicate

Samples with a positive response for the Preliminary Screening Test (Section 10.3.1) must be subjected to the Burn Rate Test (Section 10.3.2) in ***triplicate***.

Any burn rate for non-metallic samples that exceeds 2.2mm/sec (or a burn time of less than 45 seconds for 100mm) is considered to have a positive result.

For metals, a burn rate of more than 0.17 mm/sec (or burn time of less than 10 minutes for 100mm) is considered to have a positive result.

- All replicate runs must be at the same initial temperature (ambient laboratory temperature).
- All replicate tests must be run at approximately the same airflow through the fume hood.
- All replicate tests must use the same sample particle size distribution.

All replicate tests must agree within 15%. If this criterion is not met, discard the outlier and run another replicate. Results must agree within 15%.

9.7 Method-specific Quality Control Samples

Not applicable.

9.8 Method Sequence

- Perform preliminary screening test:
  - Prepare test strip or powder train
  - Light flame and measure temperature
    - If it does not burn within specified time, sample is considered a negative, nonflammable solid. Test is complete.
    - If it does burn within specified time, perform the burn rate test in triplicate. Sample is considered Positive for the preliminary screening test.
- Perform burn rate test:
  - Prepare ceramic plate and determine physical characteristics of sample:
    - If powder or granular, pour waste into the mold and drop three times, invert on marked ceramic plate.
    - If pasty, spread waste on marked ceramic plate 250mm x 1 cm^2
  - Light flame and measure temperature

- Apply flame to end of strip to ignite
- Note time (seconds) needed for the flame to travel 100mm distance
- Calculate burn rate
- Determine if sample is Positive or Negative for the Burn Rate test

10.Procedure

10.1 Equipment Set-up

Safety Test: Prior to starting the preliminary test, all sample materials must be tested to determine if that material is explosive or extremely flammable. Use a very small portion of material (1 gram or less). ***If the sample displays explosivity or extreme flammability, do not continue this test.***

10.2 Initial Calibration

Not applicable.

10.3 Equipment Operation and Sample Processing

10.3.1 Preliminary Screening Test

10.3.1.1 The preliminary ignitability test is conducted on all waste materials.

10.3.1.2 Place a platform (brick, stone, or other flame-resistant tile) in a fume hood about 20cm (or 8 inches) from the front of the hood in an area of laminar airflow. Position the sample perpendicular to the airflow. The airflow across the perpendicular axis of the sample should be sufficient to prevent fumes from escaping into the laboratory and should not be varied during the test. The air velocity should be approximately 0.7 meters/second. Measure and record the air velocity by using the anemometer (Section 7.7).

10.3.1.3 On the platform, prepare the test material in its “as received” form by forming an unbroken strip or powder train of sample 250mm long by 20mm wide by 10mm high on the platform. Position the sample perpendicular to the airflow. Use the mold (Section 7.3) to form the material as in Section 10.3.2.1 if appropriate.

10.3.1.4 Light the gas torch. Measure and record the temperature of the flame (tip of the flame) by a thermocouple (Section 7.6). The temperature of the flame must be at least 1000 °C.

10.3.1.5 Apply the tip of the flame to one end of the sample strip. Measure the propagation of combustion with the ceramic tile (Section 7.1). The test period will depend on the sample matrix as follows:

10.3.1.5.1 **Non-Metallic Waste, Soils, Clays:** Hold the flame tip on the sample strip until the sample ignites or for a maximum of 2 minutes. If combustion occurs, begin timing with a stopwatch and note whether the combustion propagates up to the 200mm mark within the 2-minute test period.

10.3.1.5.2 **Metal or Alloy Powders:** Hold the flame tip on the sample strip until the sample ignites or for a maximum of 5 minutes. If combustion occurs, begin timing with a stopwatch and note whether the combustion propagates up to the 200mm mark within the 20-minute test period.

10.3.1.6 If waste does not ignite and propagate combustion by either burning with open flame or by smoldering along the 200mm of sample strip within the 2 minute test

period (or 20 minute test period for metal powders), the waste is not considered flammable and no further testing is required.

10.3.1.7 If the waste propagates burning of 200mm of the test strip within the 2-minute test period (20 minute test period for metals), the material must be evaluated by the burning rate test in Section 10.3.2.

10.3.2 Burning Rate Test

10.3.2.1 The preparation of the test sample for the burning rate test will depend on the physical characteristics of the waste. Wastes that exist in a powdered or granular state are molded in a powder train mold, shown in Figure 1. Pasty or clay materials are formed into a rope 250mm in length with a cross-section of 1 cm^2. All tests for the burn rate test are performed on clean, ambient temperature, ceramic plates.

10.3.2.2 By utilizing the impervious ceramic tile (Section 7.1), the distance between the 80 and 180mm marks (100mm) will be used to calculate the rate of burn in Section 11.1.

10.3.2.3 Measure and record the ambient laboratory temperature.

10.3.2.4 For powdered or granular materials, pour the material to fill the triangular cross section of the mold loosely.

10.3.2.5 Drop the unit from a height of 2cm onto a solid surface three times to settle the powder. Add additional sample material if necessary and drop again three times. Place ceramic plate on top of train mold. Hold tightly and invert the setup and remove the mold. If sample clings to the inside of the mold, gently tap it against the ceramic plate until the sample is released in a 250mm triangular strip.

10.3.2.6 Clay or pasty wastes are prepared by spreading the waste on a marked ceramic tile (Section 10.3.2.2) in the form of a rope 250mm in length with a cross-section of 1 cm^2.

10.3.2.7 Place the ceramic tile with the loaded sample prepared in Sections 10.3.2.3 or 9.2.2.5 in a fume hood about 20cm (8 inches) from the front of the hood and in an area of laminar airflow. Position the sample perpendicular to the airflow. The airflow across the perpendicular axis of the sample should be sufficient to prevent fumes from escaping into the laboratory and should not be varied during the test. The air velocity should be approximately 0.7 meters/second. Measure and record the air velocity using an anemometer (Section 7.8).

10.3.2.8 Light the gas torch. Measure and record the temperature of the flame (tip of the flame) by a thermocouple. The temperature of the flame must be at least 1000 °C.

10.3.2.9 Apply the tip of the flame to one end of the sample strip to ignite the test strip as described in Section 10.3.1.5.1 and 10.3.1.5.2.

10.3.2.10 When the test strip or powder train has burned up to the 80mm time marker, begin timing the rate of combustion with a stopwatch. Stop the timer when the burned strip reaches the 180mm time marker. Record the amount of time (in seconds) required to burn the 100mm test strip.

10.4 Continuing Calibration

Not applicable.

10.5 Preventive Maintenance

The ceramic plate and train mold must be cleaned after each sample.

11. Data Evaluation, Calculations and Reporting

11.1 Calculate the rate of burning by dividing the length of the burn test strip (100mm) by the total time (seconds). Results of the burn rate test should be reported in mm/sec.

Wastes that have a rate of burning of more than 2.2mm/sec (or burn time of less than 45 seconds for 100mm) are considered to have a positive result for ignitability according to DOT regulations. For metals, this time is 10 minutes or less for 100mm (or a burn rate of more than 0.17mm/sec).

11.2 Include in the data package the following information for each sample:

Test Material Information:

- Date of test:
- Description of material: e.g. powder or paste, metallic or non-metallic
- Particle size: e.g. fine powder, granular, sand, etc.
- Preliminary Burning Time:_____ Seconds (from Section 10.3.1.5 or 10.3.1.6)
- If the preliminary screening test (Section10.3.1) was negative, the result for the sample is Not Ignitable (reported as “NI”) and no further data is required.

If the Burning Rate Test was Performed:

- Temperature of test material (°C):
- Air velocity through fume hood (m/s):
- For each replicate:
    - Time (sec) elapsed between application of flame and start of ignition
    - Burning time over 100mm (sec), (Section 10.3.2.10)
    - Burning rate (mm/sec), (Section 11.1)
    - Comments

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedence and improper preservation are noted on the Sample Delivery Group Form and/or Nonconformance Report form.

Record any unusual performance on a nonconformance action form.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

This study is not appliable to this method.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1734 and 1739 for further information regarding IDC/DOC Generation.

These studies are not applicable to this method.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/1732 MDL/LOD/LOQ Generation

SOP/1734, 1739 IDC/DOC Generation

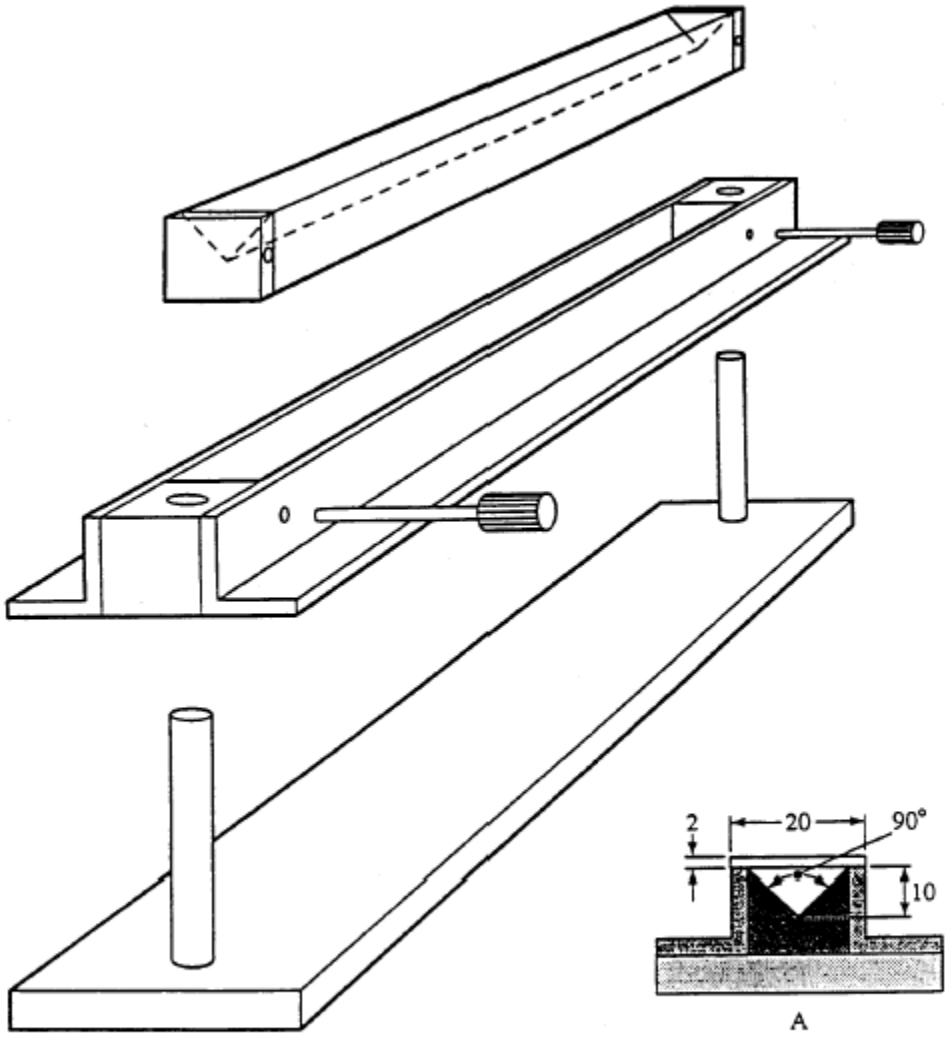
SOP/1728 Waste Management and Disposal SOP

16. Attachments

- FIGURE 1: Powder Train Mold
- Flowchart

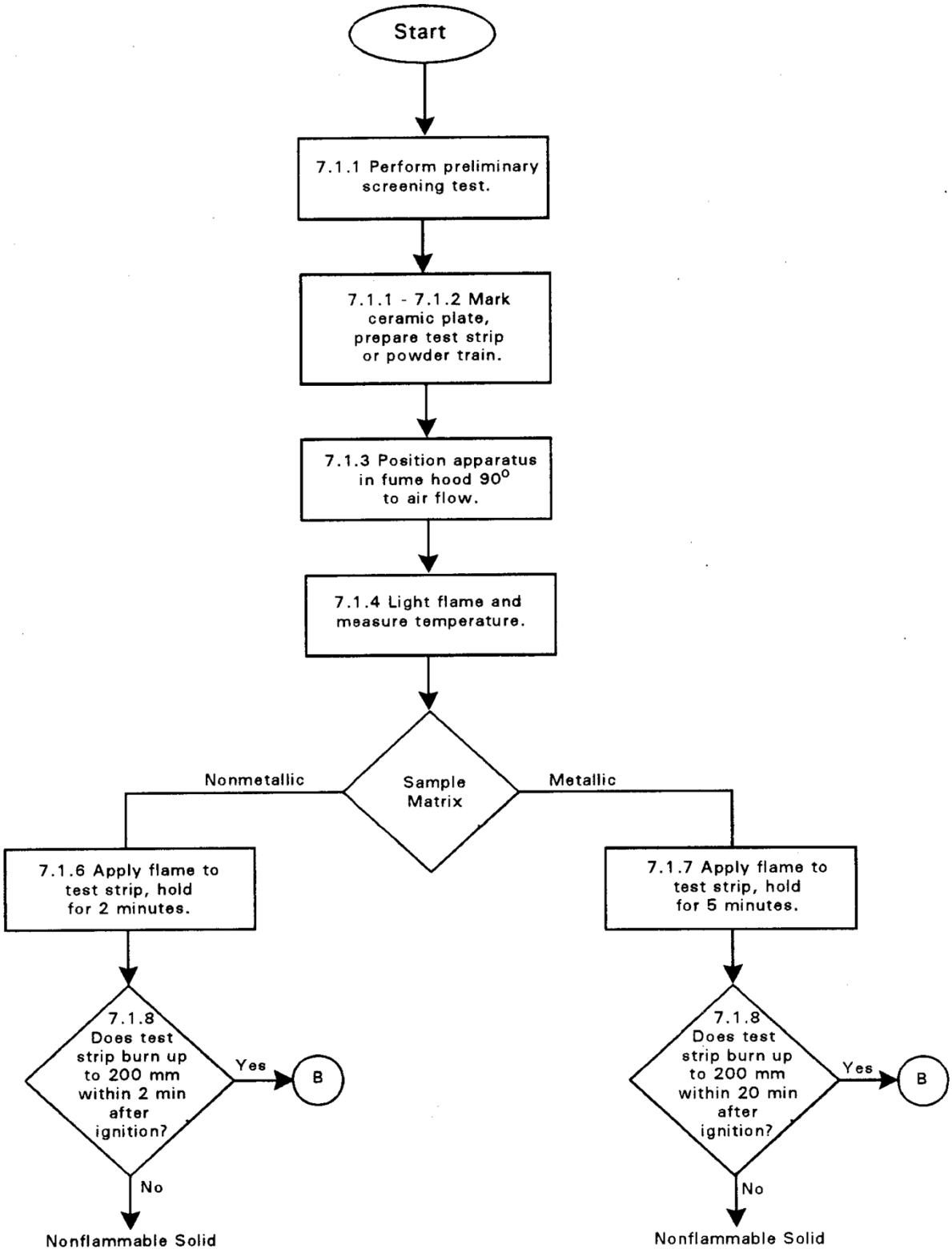
FIGURE 1

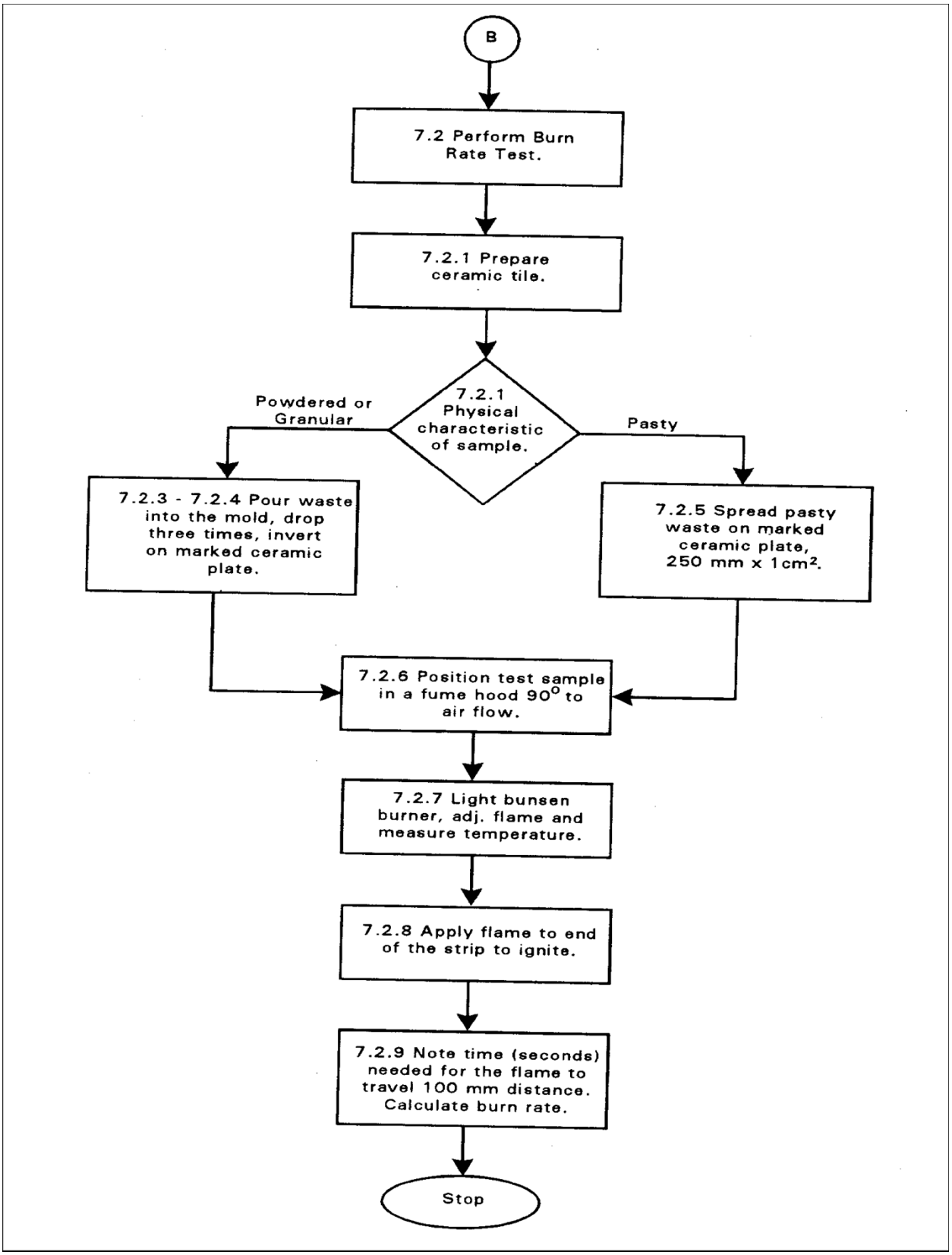
Powder Train Mold



(A) Cross-section of 250 mm long mould

Flowchart – Ignitability of Solids Method 1030


Start
7.1.1 Perform preliminary screening test.
7.1.1 - 7.1.2 Mark ceramic plate, prepare test strip or powder train.
7.1.3 Position apparatus in fume hood 90° to air flow.
7.1.4 Light flame and measure temperature.
Sample Matrix
Nonmetallic
Metallic
7.1.6 Apply flame to test strip, hold for 2 minutes.
7.1.7 Apply flame to test strip, hold for 5 minutes.
7.1.8 Does test strip burn up to 200 mm within 2 min after ignition?
Yes
B
No
Nonflammable Solid
7.1.8 Does test strip burn up to 200 mm within 20 min after ignition?
Yes
B
No
Nonflammable Solid


B
7.2 Perform Burn Rate Test.
7.2.1 Prepare ceramic tile.
7.2.1 Physical characteristic of sample.
Powdered or Granular
Pasty
7.2.3 - 7.2.4 Pour waste into the mold, drop three times, invert on marked ceramic plate.
7.2.5 Spread pasty waste on marked ceramic plate, 250 mm x 1cm².
7.2.6 Position test sample in a fume hood 90° to air flow.
7.2.7 Light bunsen burner, adj. flame and measure temperature.
7.2.8 Apply flame to end of the strip to ignite.
7.2.9 Note time (seconds) needed for the flame to travel 100 mm distance. Calculate burn rate.
Stop

VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE

FOR

ACID DIGESTION OF TOTAL RECOVERABLE OR

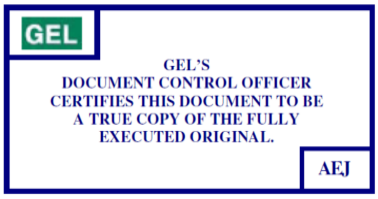
DISSOLVED METALS IN SURFACE AND GROUNDWATER

SAMPLES FOR ANALYSIS BY ICP OR ICP-MS

(GL-MA-E-006 REVISION 14)

PROPRIETARY INFORMATION

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1.0 STANDARD OPERATING PROCEDURE FOR ACID DIGESTION OF TOTAL RECOVERABLE OR DISSOLVED METALS IN SURFACE AND GROUNDWATER SAMPLES FOR ANALYSIS BY ICP OR ICP-MS

2.0 METHOD CODE

SW-846 3005A

3.0 PURPOSE

To describe the manner in which surface and groundwater samples for Inductively Coupled Plasma (ICP) and Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) analysis are digested by EPA SW-846 Method 3005A. Samples digested by this procedure are applicable for analysis by SW-846 methods.

4.0 DISCUSSION

This procedure deviates from Method 3005A as follows: sample and reagent volumes are half of the method recommendations. Samples prepared by this method maybe analyze for the following analytes:

Aluminum	Hafnium	Silica	Uranium-233
Antimony	Iron	Silicon	Uranium-234
Arsenic	Lead	Sulfur	Uranium-235
Barium	Lithium	Sodium	Uranium-236
Beryllium	Magnesium	Silver	Uranium-238
Bismuth	Manganese	Strontium	Vanadium
Boron	Molybdenum	Tantalum	Zinc
Cadmium	Potassium	Tin	Zirconium
Calcium	Nickel	Thorium	
Cesium	Phosphorus	Titanium	
Chromium	Rhenium	Thallium	
Cobalt	Rhodium	Tungsten	
Copper	Selenium	Uranium	

4.1 Total Recoverable Metals - The entire sample is acidified with nitric acid at the time of collection. This sample is then refluxed with nitric acid and hydrochloric acid until the volume is reduced. The sample is diluted to a final volume, and then is ready for analysis.

4.2 Dissolved Metals - The sample is filtered at the time of collection. The liquid phase is acidified at the time of collection with nitric acid. Samples for dissolved metals do not need to be digested, as long as the acid concentration in the

standards matches the acid concentration in the samples. Water samples that require lab filtration are filtered from non-preserved fractions and acidified to a pH <2 with HNO_3. A filter blank is filtered with the samples for contamination QC purposes. The acidified aliquots are held at least 24 hours before digestion.

4.3 The samples are tested using pH strips to verify the correct pH values of <2 before preparation. If samples do not have the correct pH, samples are acidified to a pH <2 with HNO_3. The acidified aliquots are held at least 24 hours before digestion.

5.0 APPLICABLE MATRICES

Groundwater and surface waters.

6.0 HOLDING TIME, SAMPLE PRESERVATION, COLLECTION, STORAGE

6.1 Samples requiring filtration should be received by the laboratory chemically unpreserved. Liquid samples must be preserved prior to aliquoting for digestion. The samples are typically received chemically preserved with nitric acid to pH <2. Preservation may be performed at the time of sample collection however samples may also be submitted to the laboratory without chemical preservation. If unpreserved at the time of collection, the samples should be preserved within two weeks of collection, upon receipt at the laboratory. Following acidification, the sample should be mixed by agitation and held for 24 hrs to equilibrate. The pH of all aqueous samples should be tested immediately prior to withdrawing an aliquot for preparation to assure proper preservation. If the sample pH is >2, additional acid must be added and the sample held for 24 hrs until pH is verified as <2. If properly preserved, the sample may be held up to 6 months before analysis.

6.2 Samples may be collected in polyethylene containers.

7.0 INTERFERENCES

There are rarely any interferences with this digestion. If any are encountered, consult the Group Leader, Team Leader or Quality Assurance Officer before continuing.

8.0 METHOD PERFORMANCE

Method Detection Limit (MDL) studies are performed annually and method detection limit verification (MDLV) studies are performed quarterly. The limits are stored in the laboratory AlphaLIMS.

9.0 DEFINITIONS

9.1 Blank: Type I water that has been taken through the digestion process. The blank is used to determine the amount of background contamination.

9.2 Laboratory Control Sample (LCS): A certified reference material that has been taken through the digestion process. The LCS is used to determine digestion accuracy and to determine if the digestion process is in control.

9.3 Laboratory Control Sample Duplicate (LCS DUP): A duplicate of the LCS. The LCS DUP is used to determine reproducibility and to indicate precision.

9.4 Matrix Spike (MS): A sample that has added to it a known amount of solution containing known concentrations of analytes. The MS is used to determine the presence or absence of interferences and matrix effects in the digested sample.

9.5 Matrix Spike Duplicate (MSD): A duplicate of the MS. The MSD indicates reproducibility.

9.6 Sample Duplicate (DUP): A duplicated sample. The DUP indicates reproducibility.

9.7 Type I water: Water that conforms to the performance specifications in SOP GL-LB-E-016. Type I water is dispensed within the metals prep lab by the "MilliQ" water system.

9.8 HNO_3: Concentrated reagent grade 70.9% nitric acid.

9.9 HCl: Concentrated reagent grade 37% hydrochloric acid.

9.10 Refer to GL-QS-B-001 the Quality Assurance Plan for additional lab-wide used definitions.

10.0 ANALYST VERIFICATION

Before a technician/analyst is allowed to analyze samples without supervision, he or she is trained by qualified personnel and is required to successfully analyze a proficiency sample. Training records are maintained as quality records (Refer to GL-QS-E-008).

11.0 DOCUMENTATION OF DATA

Sample preparation data are recorded in AlphaLIMS.

12.0 SAFETY, HEALTH, AND ENVIRONMENTAL HAZARDS

WARNING

CONCENTRATED HYDROCHLORIC ACID AND NITRIC ACID ARE EXTREMELY CORROSIVE AND CAN CAUSE SEVERE BURNS TO THE SKIN.

12.1 Wear eye protection with side shields while performing procedures in the lab.

12.2 Treat all chemicals and samples as potential health hazards, and reduce exposure to these chemicals to the lowest level possible. GEL maintains a current awareness file of OSHA regulations regarding the safe handling of the chemicals. A reference file of Material Safety Data Sheets (MSDS) and individual client sample MSDSs are also maintained.

12.3 Personal protective equipment

12.3.1 Disposable gloves are worn and changed frequently when working with acids, glassware, or samples. Dirty gloves pose a contamination hazard to the samples. Gloves that have holes can be dangerous to the wearer by allowing acids and toxic metals to come in contact with skin.

12.3.2 Hood doors are pulled down partially or enclosure doors are pulled together partially, while digesting samples. Acidified samples can splash and pop as they are being heated.

12.3.3 To protect clothes and skin from exposure to corrosive material, wear a lab jacket.

12.4 Prior to handling radioactive samples, analysts must have had radiation safety training and must understand their full responsibilities in radioactive sample handling. Some general guidelines follow:

12.4.1 Protect counter tops with counter paper, or work from radioactive sample handling trays.

12.4.2 Prohibit admittance to immediate work area.

12.4.3 Post signs indicating radioactive samples are in the area.

12.4.4 Take swipes of the counter tops upon completion of work. Deliver those swipes to the designated swipe count box.

12.4.5 Segregate radioactive wastes. Radioactive waste containers are obtained from the Waste Management.

12.5 All samples, chemicals, extracts, and extraction residues must be transferred, delivered, and disposed of safely according to all related SOPs.

12.5.1 Segregate solid wastes from liquid wastes in the satellite area containers.

12.5.2 Segregate oil wastes from water-soluble wastes in the satellite area containers.

12.6 In the event of an accident or medical emergency, call for help immediately. When time and safety permit, an accident report form should be completed and turned in to the safety committee.

12.7 Fire escape routes are posted in the lab, and all personnel should be familiar with them. In addition, fire safety equipment such as fire extinguishers is located in the lab. Training is available on the proper operation of this equipment.

13.0 SAMPLE RECEIPT FOR ANALYSIS

13.1 The analyst/technician submits the list of samples needed to the sample custodian group. The sample custodian removes the appropriate sample from the cooler and scans it using the barcode scanner to the appropriate area of the lab. The analyst then takes custody of the samples and scans them to the sample batch. The samples are now ready to be prepared or analyzed.

13.2 Analysts/technicians are responsible for retrieving their own samples when the sample custodian is unavailable.

14.0 INSTRUMENT/EQUIPMENT/GLASSWARE

14.1 Equipment

14.1.1 Air displacement pipettes

14.1.1.1 0.5-5 mL with disposable tips

14.1.1.2 100-1000 µL with disposable tips

14.1.1.3 10-100 µL with disposable tips

14.1.2 Environmental Express hot blocks or equivalent

14.1.3 Analytical balance capable of reading to three decimal places

14.1.4 Certified disposable 50 mL digestion tubes (polypropylene)

14.1.5 Ribbed disposable watch glasses (polypropylene)

14.1.6 Water resistant lab markers

14.1.7 Styrofoam trays to handle up to 25 digestion tubes

14.1.8 500 mL Nalgene squirt bottle

14.1.9 1-inch white laboratory tape

14.1.10 Borosilicate beakers (various sizes)

14.1.11 Borosilicate watch glasses (various sizes)

15.0 REAGENTS

15.1 Nitric acid (HNO_3), concentrated high purity grade 70% nitric acid

15.2 Hydrochloric acid (HCl), concentrated high purity grade 37% hydrochloric acid

15.3 Type I water, DI water (see GL-LB-E-016). Type I water is dispersed with the metals prep lab by the “Milli Q” water system.

15.4 Multi-element spiking solutions are purchased from NIST-traceable vendors.

16.0 PROCEDURES

A batch consists of samples of the same matrix and quality control (QC) samples that are digested together. Each of the quality control samples listed in section 19.2 can be included in each batch at the frequency listed or as per client request. The blank, LCS and/or LCS DUP are digested at a frequency of one in 20 or per batch, whichever is more frequent. The MS, MSD and/or DUP are digested at a frequency of one in 20 or per batch, whichever is more frequent, or per specific client/program requirements.

16.1 Glassware preparation:

16.1.1 Glassware that has been cleaned according to "Glassware Preparation" (GL-LB-E-003) is soaked in a water and acid mixture for at least 30 minutes.

16.1.2 After soaking, the glassware is rinsed with copious quantities of Type I water, then inverted over clean, absorbent paper or onto a rack for drying.

16.2 Label Teflon beakers or centrifuge tube with the sample numbers in the batch.

16.3 Mix the sample to achieve homogeneity. Transfer a 50 mL aliquot of sample to the appropriately labeled beaker or tube.

16.4 Quality control samples are prepared prior to digestion.

16.4.1 The beaker or tube to be used for the blank, MS, MSD and/or DUP, LCS and/or LCS DUP is labeled.

16.4.2 Unless otherwise noted, at least 50 mL of the sample is transferred to the MS, MSD and/or DUP beaker or tube.

16.4.3 Unless otherwise noted, 50 mL of Type I water is transferred to the LCS and/or LCS DUP beaker or tube.

16.4.4 The MS, MSD, LCS, and/or LCS DUP are spiked with known amounts of spiking solution.

16.4.5 The Teflon beaker or centrifuge tube to be used for the blank is labeled. 50 mL of Type I water is transferred to the beaker or tube. No spike or sample is added to the blank.

16.5 Sample digestion:

16.5.1 Add 1 mL of HNO_3 and 2.5 mL of HCl to the samples and quality control samples.

16.5.2 Cover the sample with a watchglass and heat the sample to 90 to 95° C until the sample volume is reduced to 7.5 to 10 mL or heat for 6 hours.

16.5.3 Do not allow the sample to go to dryness. Do not allow the sample to boil.

16.5.4 Remove the sample from the hot plate or block.

16.5.5 Allow the sample to cool.

16.5.6 If the sample contains particulate material that could clog the nebulizer, if necessary, you may filter or centrifuge the sample.

16.5.6.1 Be advised that filtration is a common cause of contamination. If a sample is filtered any QC associated with the sample must also be filtered. Additionally, if any sample in the batch is filtered the method blank and laboratory control sample must also be filtered.

16.5.7 Transfer the sample to a labeled centrifuge tube.

16.5.8 Dilute the sample to 50 mL with Type I water.

16.5.9 Cap the samples, organize them into a storage container, and label the storage container with the batch number of the sample group.

17.0 PREPARATION OF STANDARDS

Documentation of standards and their preparation is maintained in AlphaLIMS in accordance with GL-LB-E-007 for Laboratory Standards Documentation.

18.0 INSTRUMENT/EQUIPMENT START-UP PROCEDURE

Hot plates/blocks are allowed to come to the proper temperature before digestions are started. The temperatures are documented on the data entry screens.

19.0 QUALITY CONTROL (QC) REQUIREMENTS

19.1 Frequency of QC

19.1.1 A matrix spike (MS) and a matrix spike duplicate (MSD) or a sample duplicate (DUP) and a matrix spike are prepped for every batch of $\leq$ 20 samples.

19.1.2 A method blank (MB) and a laboratory control standard (LCS) are prepped for every batch of $\leq$ 20 samples. A laboratory control standard duplicate (LCSD) is prepared if matrix QC is unavailable or upon client request.

19.2 Makeup of QC Samples

19.2.1 Sample duplicate (DUP) is a separate aliquot taken through the prep process exactly the same as the original sample.

19.2.2 Matrix spike and/or matrix spike duplicate is a separate aliquot of the sample to which appropriate spike volumes and solutions are added. The ID numbers and volumes of the spikes are recorded in the prep logbook.

19.2.3 The method blank (MB) is a reagent blank taken through the same prep process as the samples.

19.2.4 The laboratory control standard (LCS) is a fortified reagent blank taken through the same prep process as the sample. A purchased spiking solution from a certified vendor is used to fortify the LCS. (The ID number and volumes of the spikes are recorded in the prep logbook).

19.3 Handling Out-Of-Control Situations

19.3.1 If sample reactions cause popping or splattering of the digestate, discontinue the prep and contact team leader or group leader.

20.0 RUN SEQUENCE

Not applicable

21.0 INSTRUMENT/EQUIPMENT SHUT-DOWN PROCEDURE

The hotplates/blocks are shut off at the end of day or when device is no longer in use that day.

22.0 METHOD VARIATION

22.1 This procedure deviates from method SW-846 3005A in that sample volumes are half the method recommendations.

23.0 DATA REVIEW, VALIDATION, AND APPROVAL PROCEDURE

23.1 Upon completion of batch preparation, digestion data shall be entered into the AlphaLIMS Prep Logbook following the guidelines in GL-LB-E-008 for Basic Requirements for the Use and Maintenance of Laboratory Notebooks, Logbooks, Forms, and Other Recordkeeping Devices.

23.2 Data to be entered into the electronic logbook include analyst name, prep data and time, initial volume or weight with units, and final volume with units.

23.3 Standards and reagents may also be entered into the logbook and fall under the guidelines of GL-LB-E-015 for Control of Laboratory Standards and GL-LB-E-007 for Laboratory Standards Documentation.

23.4 Upon entry of prep data, obtain a printout of the logbook. The logbook page is kept with the samples with which it is associated.

23.5 The entry of correct prep data is peer reviewed (correct dates, times, weights, volumes, SOP/revision, spikes, spike amounts, and reagent information, etc.) by the analyst(s) for correctness.

24.0 RECORDS MANAGEMENT

Records generated as a result of this procedure are maintained as quality documents in accordance with GL-QS-E-008 for Quality Records Management and Disposition.

25.0 LABORATORY WASTE

For the proper disposal of sample and reagent wastes from this procedure, refer to the Laboratory Waste Management Plan, GL-LB-G-001.

26.0 REFERENCES

26.1 Test Method for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 3005A, Revision 1, July 1992.

26.2 1992 Annual Book of ASTM Standards, D1193-91, “Standard Specification for Reagent Water”.

27.0 HISTORY

Revision 10: Updated to include list of analytes in discussion section. Type II water updated to type I. Format updated to reflect technical procedure.

Revision 11: Clarified wording in section 6.1, corrected reference in section 16.0, removed Appendix I.
Revision 12: Removed compounds from compound list.
Revision 13: Update several equipment changes as well as start up and shut down procedures for heating devices.
Revision 14: Extended equilibration time to 24 hrs after pH adjustment.

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VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE FOR ACID DIGESTION OF SEDIMENTS, SLUDGES, AND SOILS

(GL-MA-E-009 REVISION 29)

APPLICABLE TO METHODS:
EPA SW-846 3050B Modified

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1.0 STANDARD OPERATING PROCEDURE FOR ACID DIGESTION OF SEDIMENTS, SLUDGES, AND SOILS

2.0 METHOD CODE

2.1 EPA SW-846 3050B Modified

3.0 METHOD OBJECTIVE/PURPOSE

To describe the manner in which sediments, sludges, and soils for Inductively Coupled Plasma (ICP) and Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) analysis are digested by EPA SW-846 Method 3050B Modified. Samples digested by this procedure are applicable for analysis by SW-846 methods 6010 and 6020.

4.0 METHOD SUMMARY

A representative portion of sample is digested with nitric acid and hydrogen peroxide for ICP-MS analysis. A representative portion of sample is digested with nitric acid and hydrochloric acid for ICP analysis. Samples prepared by this method may be analyzed for all the listed metals. Other metals may be analyzed if they pass control standard criteria:

Aluminum	Copper	Rhodium	Thallium
Antimony	Hafnium	Selenium	Tungsten
Arsenic	Iron	Silica	Uranium
Barium	Lead	Silicon	Uranium-233
Beryllium	Lithium	Sulfur	Uranium-234
Bismuth	Magnesium	Sodium	Uranium-235
Boron	Manganese	Silver	Uranium-236
Cadmium	Molybdenum	Strontium	Uranium-238
Calcium	Potassium	Tantalum	Vanadium
Cesium	Nickel	Tin	Zinc
Chromium	Phosphorus	Thorium	Zirconium
Cobalt	Rhenium	Titanium	

This method is not a “total” digestion technique for most samples. It is a very strong acid digestion that will dissolve all elements that could become “environmentally available” by design; elements bound in silicate structures (boron, silicon, silica) are not normally dissolved by this procedure as they are not usually mobile in the environment.

5.0 APPLICABLE MATRICES

5.1 Soils

5.2 Sludges

5.3 Sediments

5.4 Solid debris/powders

5.5 Heavy oils

5.6 Filters

5.7 Paints

5.8 Ghost Wipes

6.0 HOLD TIME

Holding time is 180 days from the time and date of collection until the start of analysis unless otherwise specified by contract.

7.0 SAMPLE CONTAINER/PRESERVATION/COLLECTION/STORAGE REQUIREMENTS

Solid samples are not preserved but should be stored at 0°- 6° C.

8.0 INTERFERENCES

There are rarely any interferences with this digestion. If any are encountered, consult the group leader or quality officer before continuing.

9.0 PERFORMANCE CHARACTERISTICS

Method detection limits (MDLs) and method detection limit verification (MDLVs) are performed in accordance with SOP GL-LB-E-001 the determination of method limits and a method quantitation limits.

10.0 DEFINITIONS

10.1 Blank: Type I water that has been taken through the digestion process. The blank is used to determine the amount of background contamination.

10.2 Laboratory Control Sample (LCS): A certified reference material that has been taken through the digestion process. The LCS is used to determine digestion accuracy and to determine if the digestion process is in control.

10.3 Laboratory Control Sample Duplicate (LCS DUP): A duplicate of the LCS. The LCS DUP is used to determine reproducibility and to indicate precision.

10.4 Matrix Spike (MS): A sample that has added to it a known amount of solution containing known concentrations of analytes. The MS is used to determine the presence or absence of interferences and matrix effects in the digested sample.

10.5 Matrix Spike Duplicate (MSD): A duplicate of the MS. The MSD indicates reproducibility.

10.6 Sample Duplicate (DUP): A duplicate of a sample. The DUP indicates reproducibility.

10.7 AlphaLIMS: The Laboratory Information Management System used at GEL.

10.8 National Institute of Standards and Technology (NIST): For the purpose of this method, the national scientific body responsible for the standardization and acceptability of analyte solutions.

10.9 Refer to GL-QS-B-001 the Quality Assurance Plan for additional lab-wide used definitions.

11.0 ANALYST VERIFICATION

Before a technician/analyst is allowed to analyze samples without supervision, he or she is trained by qualified personnel and is required to successfully perform an Initial Demonstration of Capability (IDOC) as discussed in GL-QS-E-011. Training records are maintained as quality records (Refer to GL-QS-E-008).

12.0 DOCUMENTATION OF DATA

Sample preparation data are recorded in AlphaLIMS.

13.0 SAFETY, HEALTH, AND ENVIRONMENTAL HAZARDS

WARNING

CONCENTRATED HYDROCHLORIC ACID AND NITRIC ACID ARE EXTREMELY CORROSIVE AND CAN CAUSE SEVERE BURNS TO THE SKIN.

CONCENTRATED 30% HYDROGEN PEROXIDE IS A VIOLENT OXIDIZER. KEEP AWAY FROM OPEN FLAMES, AND RINSE WITH WATER IF SKIN CONTACT OCCURS.

13.1 Wear eye protection with side shields while performing procedures in the lab.

13.2 Treat all chemicals and samples as potential health hazards, and reduce exposure to these chemicals to the lowest level possible. GEL maintains a current awareness file of OSHA regulations regarding the safe handling of the chemicals. A reference file of Material Safety Data Sheets (MSDS) and individual client sample MSDSs are also maintained.

13.3 Personal protective equipment

13.3.1 Disposable gloves are worn and changed frequently when working with acids, glassware, or samples. Dirty gloves pose a contamination hazard to the samples. Gloves that have holes can be dangerous to the wearer by allowing acids and toxic metals to come in contact with skin.

13.3.2 Hood doors are pulled down partially or enclosure doors are pulled together partially while digesting samples. Acidified samples can splash and pop as they are being heated.

13.3.3 To protect clothes and skin from exposure to corrosive material, wear a lab jacket.

13.4 Prior to handling radioactive samples, analysts must have had radiation safety training and must understand their full responsibilities in radioactive sample handling. Some general guidelines follow:

13.4.1 Protect counter tops with counter paper, or work from radioactive sample handling trays.

13.4.2 Prohibit admittance to immediate work area.

13.4.3 Post signs indicating radioactive samples are in the area.

13.4.4 Take swipes of the counter tops upon completion of work. Deliver those swipes to the designated swipe count box.

13.4.5 Segregate radioactive wastes. Radioactive waste containers are obtained from the Waste Management.

13.5 All samples, chemicals, extracts, and extraction residues must be transferred, delivered, and disposed of safely according to all related SOPs.

13.5.1 Segregate solid wastes from liquid wastes in the satellite area containers.

13.5.2 Segregate oil wastes from water-soluble wastes in the satellite area containers.

13.6 In the event of an accident or medical emergency, call for help immediately. When time and safety permit, an accident report form should be completed and turned in to the safety committee.

13.7 Fire escape routes are posted in the lab, and all personnel should be familiar with them. In addition, fire safety equipment such as fire extinguishers is located in the lab. Training is available on the proper operation of this equipment.

14.0 SAMPLE RECEIPT FOR ANALYSIS

14.1 The analyst/technician submits the list of samples needed to the sample custodian group. The sample custodian removes the appropriate sample from the cooler and scans it using the barcode scanner to the appropriate area of the lab. The analyst then takes custody of the samples and scans them to the sample batch. The samples are now ready to be prepared or analyzed.

14.2 Analysts/technicians are responsible for retrieving their own samples when the sample custodian is unavailable.

15.0 INSTRUMENT/EQUIPMENT/GLASSWARE

15.1 Equipment

15.1.1 Air displacement pipettes

15.1.1.1 0.5-5 mL with disposable tips

15.1.1.2 100-1000 µL with disposable tips

15.1.1.3 10-100 µL with disposable tips

15.1.2 Environmental Express hot blocks or equivalent

15.1.3 Analytical balance capable of reading to three decimal places

15.1.4 Certified disposable 50 mL digestion tubes (polypropylene)

15.1.5 Ribbed disposable watch glasses (polypropylene)

15.1.6 Water resistant lab markers

15.1.7 Styrofoam trays to handle up to 25 digestion tubes

15.1.8 500 mL Nalgene squirt bottle

15.1.9 Teflon chips

15.1.10 1-inch white laboratory tape

15.1.11 Borosilicate beakers (various sizes)

15.1.12 Borosilicate watch glasses (various sizes)

16.0 REAGENTS

16.1 Nitric acid (HNO_3), concentrated high purity grade 70% nitric acid

16.2 Hydrochloric acid (HCl), concentrated high purity grade 37% hydrochloric acid

16.3 Hydrogen peroxide (H_2O_2), concentrated 30% hydrogen peroxide

16.4 Type I deionized (DI) water.

16.5 Multi-element spiking solutions are purchased from NIST-traceable vendors.

17.0 PREPARATION OF SAMPLES

A batch consists of samples of the same matrix and quality control (QC) samples that are digested together. Each of the quality control samples listed in steps 20.2 must be included in each batch at the frequency listed or as per client request. The blank, LCS, and/or LCS DUP are digested at a frequency of one in 20 or per batch, whichever is more frequent. The MS, MSD, and/or DUP are digested at a frequency of one in 20 or per batch, whichever is more frequent, or per specified client/program requirements.

17.1 Glassware preparation:

17.1.1 Glassware that has been cleaned according to GL-LB-E-003 for Glassware Preparation is soaked in a water and acid mixture for at least 30 minutes.

17.1.2 After soaking, the glassware is rinsed with copious quantities of Type I water and then inverted over clean, absorbent paper or onto a rack for drying.

17.2 Label the digestion tube with the sample numbers in the batch. If digestion tube is to be used for measuring initial and final volumes it either must be calibrated before usage or must be certified by the vendor. Refer to GL-LB-E-026 for digestion tube testing procedure.

17.3 Refer to GL-LB-E-029 for subsampling instructions. Mix the sample to achieve homogeneity. Weigh approximately 0.5 g of sample. Transfer the weighed sample to the appropriately labeled digestion tube.

17.3.1 Sample aliquots should not be taken from the top of an unmixed sample because large particles tend to rise in solid matrixes and heavy materials tend to sink in liquid matrixes.

17.3.2 Powdered samples may be homogenized by gently rocking the sample side to side. Then a representative aliquot may be taken from the center of the powder.

17.3.3 Other matrixes must be stirred, turned or mixed before sampling.

17.4 Quality control samples are prepared prior to digestion.

17.4.1 The tube to be used for the blank, MS, MSD, and/or DUP, LCS, and/or LCS DUP is labeled.

17.4.2 Weigh approximately 0.5 g of sample and transfer to the MS, MSD, and/or DUP beaker or tube.

The MS, MSD, LCS, and/or LCS DUP are spiked with known amounts of spiking solution.

17.4.3 Select a LCS. The LCS is purchased from an outside vendor and comes with a certificate of certified values and recovery ranges. The LCS is logged into the AlphaLIMS system for traceability and for the use of nominal calculations. Mix the LCS to achieve homogeneity. Weigh approximately 0.5 g of the sample and transfer to the LCS and/or LCS DUP digestion tube. For non-soil solid samples, a liquid LCS is used in combination with approximately 0.5 g of Teflon chips.

17.4.4 The blank tube is labeled and no water, spike, or sample is added to it. Approximately 0.5 g of Teflon chips is used.

17.5 If the samples are being prepared for ICP-MS analysis:

17.5.1 Add 2.5 mL nitric acid and Type I DI water to the samples and quality control samples.

17.5.2 Gently swirl the sample and acid mixture.

17.5.3 Cover the sample with a watch glass and heat the sample on a hot plate/block to 95° ± 5° C. Reflux the sample for 10 to 15 minutes.

17.5.4 Remove the sample from the hot plate or block and allow the sample to cool.

17.5.5 Add 2.5 mL of concentrated nitric acid, replace the watch glass, and reflux for 30 minutes. If brown fumes are generated indicating oxidation of the sample by nitric acid, repeat step 17.5.5 over and over until no brown fumes are given off by the sample.

17.5.6 Using a ribbed watch glass or vapor recovery system, allow the solution to evaporate to approximately 2.5 mL without boiling, or heat for 2 hours.

17.5.6.1 Remove the sample from the hot plate or block and allow the sample to cool.

17.5.6.2 Add 1.5 mL of hydrogen peroxide and 1.0 mL of Type I water. Return the sample to the hot plate or block and allow the peroxide reaction to occur. Continue to add hydrogen peroxide to the sample until the effervescence subsides. Do not add more than 5 mL hydrogen peroxide.

17.5.6.3 Cover the sample with a ribbed watchglass, heating the acid-peroxide digestate until the volume is reduced to approximately 2.5 mL, or heat at 95° ± 5° C without boiling for 2 hours.

17.5.6.4 Do not allow the sample to evaporate to dryness.

17.5.6.5 Remove the sample from step 17.5.6.3 from the hot plate or block.

17.5.6.6 Allow the sample to cool.

17.5.6.7 Dilute the sample to 50 mL with Type I water.

17.5.6.8 Cap and shake the sample.

17.5.6.9 Filter each sample with a 2.0 µm pore size plunger type filter (PTF grade) or allow to settle overnight.

17.5.6.10 Organize the samples in a storage container, and label the container with the batch number of the sample group.

17.6 If the samples are being prepared for ICP analysis:

17.6.1 Add 1.25 mL nitric acid and 10 mL hydrochloric acid to the samples and quality control samples.

17.6.2 Gently swirl to mix.

17.6.3 Cover the sample with a watch glass and heat the sample on a hotplate/block to 95° ± 5° C. Reflux the sample for 30 minutes.

17.6.4 Remove the sample from the hotplate/block and allow to cool.

17.6.5 Dilute the sample to 50 mL with Type I water.

17.6.6 Cap and shake the sample.

17.6.7 Filter each sample with 2.0 µm pore size plunger type filter (PTF grade) or allow to sit overnight.

17.6.8 Organize the samples in a storage container, and label the container with the batch number of the sample group.

17.7 If ghost wipes are being prepared for ICP or ICPMS analysis:

17.7.1 Remove ghost wipe from package and add to digestion tube.

17.7.2 Add 20 mL DI water, 10 mL Nitric Acid, and 1 mL of hydrochloric acid. Allow to sit for 30 minutes.

17.7.3 Gently swirl to mix.

17.7.4 Cover the sample with a watch glass and heat the sample on a hotblock/plate to 95 °C ± 5 °C. Reflux the sample for 30 minutes.

17.7.5 Dilute the sample to 50 mL with Type I water.

17.7.6 Cap and shake the sample.

17.7.7 Filter each sample with 2.0 µm pore size plunger type filter (PTF grade) or allow to sit overnight.

17.7.8 Organize the samples in a storage container, and label the container with the batch number of the sample group.

17.8 If the sample contains particulate material that could clog the nebulizer, you may filter or centrifuge the sample if necessary.

17.9 Be advised that filtration is a common cause of contamination. If a sample is filtered, any QC associated with the sample must also be filtered. Additionally, if any sample in the batch is filtered the method blank and laboratory control sample must also be filtered.

17.10 Filters may be prepared via this method. If the filters are small enough to fit inside the 50 mL digestion tubes, they can be treated as any solid prep materials. If the filters are too big to undergo adequate digestion using the 50 mL digestion tube, a borosilicate beaker will need to be used. All reagents and standards will need to be adjusted for any extra volumes needed. All filter analyses should be discussed and the process verified with the group/team leader prior to digestion. The group leader or project manager may have to contact the client to get the full description of what is required.

18.0 PREPARATION OF STANDARDS

Documentation of standards and their preparation is maintained in AlphaLIMS in accordance with GL-LB-E-007 for Laboratory Standards Documentation.

19.0 INSTRUMENT/EQUIPMENT START-UP PROCEDURE

Hot plates/blocks are allowed to come to the proper temperature before digestions are started. The temperatures are documented on the data entry screens.

20.0 QUALITY CONTROL (QC) REQUIREMENTS

20.1 Frequency of QC

20.1.1 A matrix spike (MS) and a matrix spike duplicate (MSD) or a sample duplicate (DUP) and a matrix spike are prepped for every batch of $\leq$ 20 samples

20.1.2 A method blank (MB) and a laboratory control standard (LCS) are prepped for every batch of $\leq$ 20 samples. A laboratory control standard duplicate (LCSD) is prepared if matrix QC is unavailable or upon client request.

20.2 Makeup of QC Samples

20.2.1 Sample duplicate (DUP) is a separate aliquot taken through the prep process exactly the same as the original sample.

20.2.2 Matrix spike and/or matrix spike duplicate is a separate aliquot of the sample to which appropriate spike volumes and solutions are added. The ID numbers and volumes of the spikes are recorded in the prep logbook.

20.2.3 The method blank (MB) is a reagent blank taken through the same prep process as the samples. Teflon chips are used to approximate matrix weights of 0.5 g.

20.2.4 The laboratory control standard (LCS) is a standard performed two different ways. For DOE-ALB clients, a purchased SRM is used at approximately 0.5 g and is taken through the same process as the samples. For all other clients, Teflon chips weighted to approximately 0.5 g are used. The chips and acid solution is spiked with the appropriate spike volumes and solutions. The ID number and volumes of the spikes are recorded in the prep logbook.

20.3 Handling Out-Of-Control Situations

If sample reactions cause popping or splattering of the digestate, discontinue the prep and contact team leader or group leader.

21.0 RUN SEQUENCE

Not applicable

22.0 PROCEDURE

Refer to section 17.0, Preparation of Samples

23.0 INSTRUMENT/EQUIPMENT SHUT-DOWN PROCEDURE

The hotplate/blocks are shut off at the end of each day.

24.0 METHOD VARIATION

24.1 This procedure deviates from method 3050B in that sample volumes are half the method recommendations.

24.2 The ICP procedure references a modified 3050B section 7.5 procedure. The modification eliminates the use of the Whatman 41 filters, thus eliminating contamination of with some common minerals.

25.0 DATA REVIEW, VALIDATION, AND APPROVAL PROCEDURE

25.1 Upon completion of batch preparation, digestion data shall be entered into the AlphaLIMS Prep Logbook following the guidelines in GL-LB-E-008 for Basic Requirements for the Use and Maintenance of Laboratory Notebooks, Logbooks, Forms, and Other Recordkeeping Devices.

25.2 Data to be entered into the electronic logbook include analyst name, prep data and time, initial volume or weight with units, and final volume with units.

25.3 Standards and reagents may also be entered into the logbook and fall under the guidelines of GL-LB-E-015 for Control of Laboratory Standards and GL-LB-E-007 for Laboratory Standards Documentation.

25.4 Upon entry of prep data, obtain a printout of the logbook. The logbook page is kept with the samples with which it is associated.

25.5 The entry of correct prep data is peer reviewed (correct dates, times, weights, volumes, SOP/revision, spikes, spike amounts, and reagent information, etc.) by the analyst(s) for corrections.

26.0 RECORDS MANAGEMENT

Records generated as a result of this procedure are maintained as quality documents in accordance with GL-QS-E-008 for Quality Records Management and Disposition.

27.0 LABORATORY WASTE

For the proper disposal of sample and reagent wastes from this procedure, refer to the Laboratory Waste Management Plan, GL-LB-G-001.

28.0 REFERENCES

28.1 Test Method for Evaluating Solid Waste; Laboratory Manual Physical/ Chemical Methods, Method 3050B, "Acid Digestion of Sediments, Sludges, and Soils," Revision 2, December 1996.

28.2 1992 Annual Book of ASTM Standards, Standard D1193-91, "Standard Specification for Reagent Water."

28.3 16 CFR Part 1303

29.0 HISTORY

Revision 24: Corrected section 17.0, and removed appendix 1 (out of date and difficult to read)

Revision 25: Removed compounds from compound list.

Revision 26: Updated to include the recording of temperatures for hotplates/blocks and shut-down processes for these devices. Updated the Analyst Verification Section.

Revision 27: Updated the reference SOP for the MDL and MDLVs.

Revision 28: Remove reference to testing of metal jewelry as a matrix.

Revision 29: Added Ghost wipe preparation to process.

VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE
FOR
MERCURY ANALYSIS USING THE PERKIN ELMER

AUTOMATED MERCURY ANALYZER

(GL-MA-E-010 REVISION 38)

APPLICABLE TO METHODS:
EPA SW-846 Methods: 7470 and, 7471A, and 7471B
EPA Methods: 245.1 and 245.2

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1.0 STANDARD OPERATING PROCEDURE FOR MERCURY ANALYSIS USING THE PERKIN ELMER AUTOMATED MERCURY ANALYZER

2.0 METHOD OBJECTIVE AND SUMMARY

This standard operating procedure (SOP) describes the digestion and analysis procedures for the determination of mercury (Hg) in surface and saline waters, wastewaters, industrial effluents, sanitary sewage, soils, sediments, bottom deposits, sludges, oils, and TCLP extracts. This is a cold-vapor atomic absorption technique based on the absorption of radiation at 253.7 nm by mercury vapor. Samples are treated with nitric acid and sulfuric acid or hydrochloric acid in the presence of potassium permanganate and potassium persulfate to oxidize all mercury to the mercuric (Hg^{++}) form. The mercury is then reduced to its elemental state and evacuated from the sample. The mercury vapor passes through an optical cell positioned in the light source of an atomic absorption spectrophotometer. Absorbance is measured as a function of mercury concentration. This SOP is applicable for the following methods:

2.1 EPA SW-846 Methods 7470A, 7471A, and 7471B

2.2 EPA Methods 245.1 and 245.2.

3.0 METHOD APPLICABILITY

3.1 Calibration Range: The typical calibration range used for analysis of mercury is 0.2 µg/L to 10.0 µg/L.

3.2 Tested Concentration Range: The range for the method is limited by calibration and can be extended by diluting the sample.

3.3 Method Detection Limit (MDL): The MDL is calculated using the procedure described in GL-LB-E-001 for The Determination of Method Detection Limits. The MDL is verified quarterly, or whenever there is a significant change in the instrument response.

3.4 Instrument Detection Limits (IDLs) are useful means to evaluate the instrument noise level and response changes over time for each analyte from a series of reagent blank analyses to obtain a calculated concentration. IDLs are determined by the mean of the blank results plus three times the standard deviation of 10 replicate analyses of the reagent blank solution. (Use zero for the mean if the mean is negative). Each measurement should be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure normally performed between the analysis of separate samples). IDLs should be determined at least once using a new equipment, after major instrument maintenance such as changing the detector, and/or at a frequency designated by the project.

3.5 Method Precision: Analytical precision can be determined by calculating the Relative Percent Difference (RPD) between a Laboratory Control Sample (LCS) and a Laboratory Control Sample Duplicate (LCSD) or a Matrix Spike (MS) and a Matrix Spike Duplicate (MSD).

3.6 Method Bias (Accuracy): Accuracy can be determined by calculating recoveries of a LCS and LCSD or an MS and MSD of a similar matrix.

4.0 DEFINITIONS

4.1 Continuing Calibration Blank (CCB): An aliquot of reagent water or other blank matrix that is analyzed after each CCV. The CCB is used to determine whether the analytical sequence is in control during sample analysis.

4.2 Continuing Calibration Verification (CCV): A solution of the method analytes used to evaluate the performance of the instrument with respect to a defined set of criteria. The CCV concentration is varied throughout the calibration range.

4.3 Initial Calibration Blank (ICB): An aliquot of reagent water or other blank matrix that is analyzed after each ICV. The ICB is used to determine whether there is carryover contamination after injection of the mid-level ICV.

4.4 Initial Calibration Verification (ICV): A solution of method analytes of known concentrations that is used to fortify an aliquot of Blank or sample matrix. The ICV is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check laboratory performance with externally prepared test materials.

4.5 Method Detection Limit (MDL): The minimum concentration of an analyte that can be measured and reported with 99% confidence that the concentration is greater than zero. The MDL is determined from analysis of a sample in a given matrix type containing the analyte.

4.6 National Institute of Standards and Technology (NIST): For the purpose of this method, the national scientific body responsible for the standardization and acceptability of analyte solutions.

4.7 Tumble Blank (TB): TCLP extraction fluid Type 1 or 2 that has been taken through the TCLP tumbling process. The tumble blank is used to determine the amount of background contamination resulting from the tumbling process.

4.8 Contract Required Detection Limit (CRDL): The lowest level in the calibration curve.

4.9 Serial Dilution (SDILT): A 1 to 5 sample dilution used to assess matrix suppression.

4.10 Limit of Detection (LOD): An analyte, method and matrix specific estimate of the minimum amount of a substance that can be reliably detected. GEL has established LOD = 2 x MDL.

4.11 Limit of Quantitation (LOQ): An analyte, method and matrix specific estimate of the minimum amount of a substance that can be reported with a specific level of confidence. The LOQ is set at or above the concentration of the lowest initial calibration standard. The laboratory must empirically demonstrate precision and bias at the LOQ. The LOQ and associated precision and bias must meet client

requirements and must be reported. GEL uses the following guidance (LOD < LOQ):

When LOD < PQL, PQL = LOQ

When LOD > PQL, LOQ is raised to next lowest calibration standard.

4.12 Refer to GL-QS-B-001 the Quality Assurance Plan for additional lab-wide used definitions.

5.0 METHOD VARIATIONS

This SOP varies from the referenced methods by utilizing a smaller sample aliquot. Reagent amounts are adjusted accordingly.

6.0 SAFETY PRECAUTIONS AND WARNINGS

WARNING

HYDROXYLAMINE HYDROCHLORIDE MAY EXPLODE WHEN HEATED TO 140º C.

MERCURY COMPOUNDS ARE TOXIC IF INHALED OR ABSORBED THROUGH THE SKIN.

ADDITION OF HYDROCHLORIC ACID AND NITRIC ACID IN THE PROPORTIONS 3 TO 1 FORMS AQUA REGIA.

ADDITION OF HYDROXYLAMINE HYDROCHLORIDE /SODIUM CHLORIDE TO AQUA REGIA CAN CAUSE THE SPONTANEOUS RELEASE OF CHLORINE GAS. PERFORM DIGESTIONS USING AQUA REGIA UNDER A HOOD.

NITRIC ACID AND SULFURIC ACID ARE HIGHLY CORROSIVE.

CONTACT WITH OXIDIZERS MAY GENERATE EXPLOSIVE MIXTURES.

NITRIC ACID IS CORROSIVE AND A POISON.

MERCURY VAPORS ARE TOXIC. DIGEST SAMPLES AND STANDARDS UNDER A FUME HOOD.

WHEN DIGESTING SOIL, OIL, AND SEDIMENT, BE CAREFUL TO AVOID INHALING TOXIC NITRATES.

WARNING

PREVENT SKIN AND EYE CONTACT BY USING SPECIFIED PERSONAL PROTECTIVE EQUIPMENT WHEN MAKING STOCK REAGENTS.

WORK UNDER A HOOD TO PREVENT INHALATION WHEN MAKING STOCK REAGENTS.

6.1 Wear eye protection with side shields while performing procedures in the lab.

6.2 Treat all chemicals as potential health hazards, and limit exposure to these chemicals to the lowest level possible. GEL maintains a current awareness file of OSHA regulations regarding the safe handling of the chemicals in the laboratory as well as a reference file of Material Safety Data Sheets (MSDS). These documents and individual sample MSDS forms are maintained in the laboratory.

6.3 Personal protective equipment

6.3.1 Gloves are required when handling the chemicals in this procedure.

6.3.2 Work under a hood when using concentrated acids and bases.

6.4 Prior to handling radioactive samples analysts must have had radiation safety training and understand their full responsibilities in radioactive sample handling. Some general guidelines follow:

6.4.1 Protect counter tops with counter paper or work from radioactive sample handling trays.

6.4.2 Prohibit admittance to immediate work area.

6.4.3 Post signs indicating radioactive samples are in the area.

6.4.4 Take swipes of the counter tops upon completion of work. Deliver those swipes to the appropriate swipe count box.

6.4.5 Segregate radioactive wastes. Radioactive waste containers are obtained from Waste Management.

6.5 All samples, chemicals, extracts, and extraction residues must be transferred, delivered, and disposed of safely according to all related SOPs.

6.6 Never leave gas cylinders unchained or untied, including when they are on the moving carts.

6.7 In the event of an accident or medical emergency, call for help immediately. When time and safety permit, an accident report form should be completed and turned in to the safety committee.

6.8 Fire escape routes are posted in the lab and all personnel should be familiar with them. In addition, fire safety equipment such as fire extinguishers is located in the lab. Training is available on the proper operation of this equipment.

7.0 INTERFERENCES TO THE METHOD

7.1 Potassium permanganate is added to eliminate possible interference from sulfide. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of added inorganic mercury from reagent water.

7.2 Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on recovery of mercury from spiked samples.

7.3 Seawaters, brines, and industrial effluents high in chlorides may require additional permanganate (as much as 25 mL) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs at 253.7 nm.

7.4 Certain volatile organic materials that absorb at 253.7 nm may also cause interference.

8.0 APPARATUS AND MATERIALS

8.1 50 mL digestion tubes

8.2 Flask, Volumetric, (100 mL and 250 mL)

8.3 Variable and single volume pipette and micropipettes

8.4 Hot Block (Temperature controlled at 95° ± 3° for solids and 90°- 95° for liquids)

8.5 Non-mercury thermometers

8.6 Culture tubes

9.0 REAGENTS

9.1 Type I deionized water

9.2 Nitric Acid (HNO_3), Concentrated, ACS grade certified for AA use.

9.3 Sulfuric Acid (H_2SO_4), Concentrated, ACS grade certified for AA use.

9.4 Hydrochloric Acid (HCl), Concentrated, ACS grade certified for AA use.

9.5 3% HCl: Dissolve 30 mL concentrated HCl in Type I water and dilute to 1000 mL. Volume may be scaled proportionately.

9.6 Potassium Permanganate Solution ($KMnO_4$), 5%: Dissolve 5 g potassium permanganate in Type I water and dilute to 100 mL. Volume and weight may be scaled up proportionally.

9.7 Potassium Persulfate Solution ($K_2S_2O_8$), 5%: Dissolve 5 g potassium persulfate in Type I water and dilute to 100 mL. Volume and weight may be scaled up proportionally.

9.8 Sodium Chloride-Hydroxylamine Hydrochloride Solution ($NaCl\text{-}NH_2OH\bullet HCl$): Dissolve 12 g of sodium chloride and 12 g of hydroxylamine hydrochloride in Type I water and dilute to 100 mL. (Hydroxylamine sulfate may be used in place of hydroxylamine hydrochloride.) Volume and weight may be scaled up proportionally.

9.9 Stannous Chloride Solution ($SnCl_2$), 1.1%: Dissolve 11 g stannous chloride in 3% hydrochloric acid and dilute to1 L. Volume and weight may be scaled proportionately.

9.10 Aqua Regia: Combine 3 parts of concentrated HCl and 1 part concentrated HNO_3. Prepare fresh before use.

NOTE: Due to the inherent dangers of working with full strength aqua regia, a 1+1 solution is prepared for use with the sample preparation.

9.11 Mercury Source Standard (Hg), 1000 ppm, from an Approved Vendor. Standards are received, labeled, prepared and stored according to GL-LB-E-007 for Laboratory Standards Documentation.

9.12 Liquid Nitrogen or Compressed Nitrogen at pressures 75 to 100 psi.

10.0 INSTRUMENTATION

10.1 Perkin Elmer FIMS100 Automated Mercury Analyzers

10.2 Balance

11.0 SAMPLE HANDLING AND PRESERVATION REQUIREMENTS

11.1 Aqueous samples must be acidified to a pH < 2 with nitric acid. The samples are tested using pH strips to verify the correct pH values of < 2 before preparation. If samples do not have the correct pH, samples are acidified to a pH of < 2 with nitric acid. The acidified aliquots are held at least 24 hours before preparation.

The suggested maximum holding time for these samples is 28 days. When performing CLP analyses, the holding time is 26 days from verified time of sample receipt.

11.2 Non-aqueous samples shall be refrigerated and analyzed as soon as possible. The preservation required for soil/sediment samples is maintenance at 0° ≤ 6°C until analysis. Water samples that require lab filtration are filtered from non-preserved fractions and acidified to a pH < 2 with nitric acid. A filter blank is filtered with the samples for contamination QC purposes. The acidified aliquots are held at least 24 hours before preparation.

11.3 TCLP extracts have a maximum holding time of 28 days before extraction and 28 days after extraction for a total of 56 days.

12.0 SAMPLE PREPARATION TECHNIQUES

12.1 Prior to determining the concentration of mercury, all samples, calibration standards, check standards, laboratory control samples, and blanks must be digested using the following procedures:

12.1.1 For standards preparation, see the mercury recipe section of the AlphaLIMS Standards Logbook.

12.1.2 Label each digestion tube with a sample number or lab Quality Control (QC) number.

12.2 Aqueous Sample Preparation by Methods EPA SW-846 7470A, EPA 245.1 and EPA 245.2.

12.2.1 Transfer 20 mL of Type I water, 20 mL of a well mixed representative sample, or an aliquot of sample diluted to 20 mL with Type I water, to a labeled digestion tube. Record the volume of sample dispensed into the digestion tube in the Mercury Prep logbook.

12.2.2 For TCLP and MISC-L matrices, transfer 18 mL of Type I water, 2 mL of a well-mixed representative sample, or an aliquot of sample diluted to 20 mL final volume, to a labeled digestion tube. Record the volume of sample dispensed into the digestion tube in the Mercury Prep logbook.

12.2.3 The following calibration standards are required: 0.2, 0.5, 2.0, 5.0, 10.0 µg/L, ICV/CCV, ICB/CCB, and CRDL (Contract Required Detection Limit).

12.2.4 Add 0.5 mL concentrated nitric acid. Add the appropriate amount of spiking solution to each appropriate QC sample tube. Then add 1.0 mL concentrated sulfuric acid to each digestion tube and mix.

12.2.5 Add 3 mL of 5% potassium permanganate solution to each digestion tube and mix. Let sample stand for approximately 15 minutes after addition of potassium permanganate. If sample does not maintain its brown/purple color, add an additional 3 mL aliquot of the 5% potassium permanganate

to the sample and all associated QC. Record this additional aliquot on the comment section of the Mercury Prep logbook. If after the second aliquot is added, the sample is still not able to maintain its purple/brown color throughout the digestion process, then redigest the sample using an appropriate dilution.

12.2.6 Add 1.5 mL of 5% potassium persulfate solution to each digestion tube and mix. Cap each digestion tube and place in a hot block for two hours at 90° - 95°C.

12.2.7 After two hours, remove digestion tubes from the hot block and allow to cool. Cooling can be facilitated by moving samples to the walk-in cooler until they reach approximate room temperature. When the samples have been cooled to room temperature, add 2 mL of sodium chloride-hydroxylamine hydrochloride solution to each digestion tube to reduce any excess potassium permanganate. Cap tubes and shake until the samples are fully reduced. The samples are now ready for analysis.

12.3 Sediment, Oil and Soil Sample preparation by Methods EPA SW-846 7471A, 7471B.

12.3.1 Weigh 0.25 to 0.3g of a representative sample and place the aliquot into a digestion tube. The (MB) method blank and (LCS) laboratory control standard uses Teflon chips or glass beads as a solid mass. The LCS uses a liquid spike on the weighed mass. If required by client, a solid reference material (SRM) is used for the LCS. Record the weight in the Mercury Prep logbook. Refer to GL-LB-E-029 for specifics on subsampling.

12.3.2 The following calibration standards are required: 0.2, 0.5, 2.0, 5.0, 10.0 µg/L, ICV/CCV, ICB/CCB, and CRDL.

12.3.3 Add the appropriate amount of spiking solution to each QC sample tube. Add 5 mL of 1+1 aqua regia to each sample and associated QC and mix.

12.3.4 Place each digestion tube in a hot block at 95° ± 3° C for two minutes.

12.3.5 Remove digestion tubes from the hot block. Cool for at least 15 minutes. Then add 25 mL of DI water and 7.5 mL of 5% potassium permanganate solution and mix thoroughly.

12.3.6 Cap each digestion tube and again place each digestion tube in a hot block 95° ± 3° C for 30 minutes.

12.3.7 Remove tubes from the hot block and allow to cool to room temperature for at least 60 minutes.

CAUTION: This next step can cause chlorine gas to be generated. Perform the following step under a hood.

12.3.8 Uncap the digestion tubes under a hood. Add 3 mL of sodium chloride-hydroxylamine hydrochloride solution to each tube. Cap and shake until samples are fully reduced. Uncap and allow time for chlorine gas to evolve and vent from the digestates. The samples and standards are now ready for analysis.

13.0 PREPARATION OF STANDARD SOLUTIONS AND QUALITY CONTROL SAMPLES

13.1 Source standard solutions

Source standard solutions are purchased from a certified vendor. These standards are traceable to National Institute of Standards and Technology (NIST) standards. The source standards are received from Purchasing. Two source standards of different lot numbers or vendors are used to prepare the curve and ICV working standards.

13.2 Intermediate and working standards must be PREPARED FRESH DAILY.

13.3 Intermediate #1 (first source) - 50µl of 1000 mg/L Hg stock #1 and 5 mL nitric acid to 250 mL with DI water.

Intermediate #2 (second source) - 50µl of 1000 mg/L Hg stock #2 and 5 mL nitric acid to 250 mL with DI water

	0.2µg/L	**0.5 µg/L**	**2.0 µg/L**	**5.0 µg/L**	**10.0µg/L**	**CCV 5.0 µg/L**	**ICV 5.0 µg/L**
Liquid Curve	20µl Int #1 to 20mL with DI Water	50µl Int #1 to 20mL with DI Water	200µl Int #1 to 20mL with DI Water	500µl Int #1 to 20mL with DI Water	1000µl Int #1 to 20mL with DI Water	500µl Int #1 to 20mL with DI Water	500µl Int #2 to 20mL with DI Water
Solid Curve	30µl Int #1 to 5mL with DI Water	75µl Int #1 to 5mL with DI Water	300µl Int #1 to 5mL with DI Water	750µl Int #1 to 5mL with DI Water	1500µl Int #1 to 5mL with DI Water	750µl Int #1 to 5mL with DI Water	750 µl Int #2 to 5mL with DI Water

13.4 For guidance on standard documentation, refer to GL-LB-E-007 for Laboratory Standards Documentation.

13.5 For Kerr-McGee Samples, reprep and reanalyze the samples if MB or CCB is greater than the reporting limit (RL) and samples are between IDL and RL. If samples are less than IDL or greater than 10 times the RL they can be reported.

14.0 INSTRUMENT CALIBRATION AND PERFORMANCE

14.1 Instrument calibration is performed using the following prepared mercury standards: 0.0, 0.2, 0.5, 2.0, 5.0 and 10.0µg/L.

14.2 Report data to the appropriate number of significant figures.

14.3 Data are reported in µg/L, mg/L, µg/kg or mg/kg Hg depending on client preference. Sample units are defined before data are input into AlphaLIMS by the analyst.

14.4 Any sample concentration less than 100% of the highest recorded calibration standard is acceptable. If sample concentrations are over 100% of the high standard, then these samples must be re-analyzed at a dilution that will bring the sample concentration within the range. Dilutions are made with digested blanks created during batch preparation.

14.5 A CRDL standard for CVAA is required to be run following calibration. The result of CRDL should be within 70%-130% of the accepted value. For DOD QSM, the CRDL should fall within 80%-120% of the accepted value or per client request.

14.6 The acceptable performance requirements for the mercury instrument are a correlation coefficient of 0.995 or better and ICV and CCV standards within the acceptance range prescribed in Section 18.2.

14.7 Proof of instrument performance during actual sample analysis is provided by records of the calibration curve, ICV, CCV, and CCB.

15.0 ANALYSIS AND INSTRUMENT OPERATION

15.1 Before actual operation, all samples, including standards and QC samples, are prepared and digested as described in Section 12. Instrument operation includes a calibration phase and a sample analysis phase. The two phases are usually run as one continuous procedure, but the instrument may be calibrated prior to loading and running samples. Also note that the analyst needs to monitor the process to ensure that the calibration is satisfactory prior to analyzing samples. If the calibration process is unsatisfactory it will need to be terminated, problems corrected, and the calibration rerun.

15.2 Turn on computer.

15.3 Input computer passwords.

15.4 Turn on Hg FIMS instrument and note that the autosampler initializes. Let it warm up for at least 30 minutes.

15.5 Prepare daily 1.1% stannous chloride reducing solution in 3% hydrochloric acid in a 2 L plastic bottle. Shake well and put in the appropriately labeled reductant reservoir bottle that feeds the instrument.

15.6 Fill other instrument reservoir bottle with 3% hydrochloric acid for instrument rinse and carrier acid.

15.7 Double-click AA WinLab Analysis icon.

15.8 Click on the applicable workspace button.

15.9 Click [Sample Info] button.

15.10 Load samples beginning in position #12. Note that samples go in the 15 mL digestion tubes. A CCV and CCB must separate each group of 10 samples. All samples must be bracketed by a satisfactory CCV and CCB.

15.11 Install dry membrane filter, rough side up (in final assembly position). Screw cap on tightly.

15.12 Hook up and clamp pump tubing. Check all pump tubing daily for proper flow and wear.

15.13 Click [FIAS] button icon.

15.14 Adjust gas flow to 40 to 70 cm^3/min.

15.15 Verify flow rates of reductant (5 to 7 mL/min, red/red tubing) and carrier (9 to 11 mL/min, blue/yellow tubing) with DI water using graduated cylinders.

15.16 Cycle [valve fill/inject] to further flush things out. Give it a minute or so in each position. Unclick all three buttons and close the window.

15.17 In the "Automated Analysis" window click on "Analyze Tab" then click on [Analyze All].

16.0 EQUIPMENT AND INSTRUMENTATION MAINTENANCE

16.1 If there are problems: During the analysis the foreground window is usually the "Results" text window that is also automatically printed, and the background window is the "Automated Analysis" window. If you need to abort a run, you must wait for the hourglass mouse cursor to change to an arrow, then click on the "Automated Analysis" window to bring it to the foreground. Then unclick the [Analyze All] button. A dialog box will come up giving you a couple of options.

16.2 For non-routine maintenance procedures, refer to Perkin Elmer manual for troubleshooting.

16.3 Whenever instrument is serviced or adjusted, an entry needs to be made in the maintenance log. The entry includes the analyst's initials, date, nature of the problem, and actions taken to correct it.

17.0 DATA RECORDING, CALCULATIONS AND DATA REDUCTION

17.1 The concentration of mercury can be determined by comparing the response obtained from analyzing the sample digestate to the calibration curve. The sample concentration of mercury is matrix specific and is calculated as follows:

Aqueous Sample

17.1.1 Concentration (µg/L) = $\frac{(C)(D)(V_t)}{V_i}$

Where:

C = Concentration (µg/L) digestate

D = Dilution Factor

V_t = Total Volume of sample digestate

V_i = Initial Volume of sample in L

17.1.2 Non-aqueous Samples

$$\text{Concentration (µg/kg)} = \frac{(C)(D)(V_t)}{W(\%S)}$$

Where:

C = Concentration (µg/mL) digestate

D = Dilution Factor

V_t = Total Volume of the sample digestate

W = Weight of the sample digested

%S = Percent Solids

18.0 QUALITY CONTROL REQUIREMENTS

18.1 Frequency of Quality Control Activities

18.1.1 ICV is performed immediately following each calibration and CCV is performed after every 10 analysis.

18.1.2 ICB is performed immediately following the ICV and CCB must follow each CCV.

18.1.3 A method blank is analyzed for each batch of 20 or less samples.

18.1.4 A matrix spike and matrix spike duplicate or matrix spike and a sample duplicate are analyzed for every 20 or fewer samples in a batch, or per client requirements. When performing EPA analyses, the requirement for matrix spike analysis is one per every 10 or fewer samples in a batch.

18.1.5 A LCS is analyzed with each batch of 20 or less samples. Additionally, a LCS duplicate also may be analyzed due to client specific requirements.

18.1.6 A SDILT is required with each batch of 20 or less samples, preferably the QC designated sample.

18.1.7 Linear Calibration Range (LCR) checks are performed on a 6 month basis. The high standard of the curve is read back against the calibration and must recover ± 10%. If this procedure fails, the problem must be investigated and rectified.

18.1.8 A post spike is required if the percent recovery of an MS or MSD is outside the acceptance limits or the MSD %RPD is greater than 20% RPD for all SW-846 Methods.

18.2 Acceptance Limits

18.2.1 Acceptance limits for the following methods are outlined below:

Method/ Acceptance Criteria	**SW-846 7471A SW-846 7471B**	**SW-846 7470A**	**EPA 245.1 EPA 245.2**	**DOD QSM Version 5.3**
ICV	90% - 110%	90% - 110%	95% - 105%	90-110%
ICB	-0.2 µg/L to 0.2 µg/L	-0.2 µg/L to 0.2 µg/L	-0.2 µg/L to 0.2 µg/L	± ½ LOQ
CRDL	70% - 130%	70% - 130%	70% - 130% advisory limits only	80% - 120% to investigate and recalibrate
CCV	80% - 120%	80% - 120%	90% - 110%	90% - 110%
CCB*	-0.2 µg/L to 0.2 µg/L	-0.2 µg/L to 0.2 µg/L	-0.2 µg/L to 0.2 µg/L	± ½ LOQ
Method Blank*	-0.2 µg/L to 0.2 µg/L	-0.2 µg/L to 0.2 µg/L	-0.2 µg/L to 0.2 µg/L	± ½ LOQ
LCS	80% - 120% (for non-SRM sources) certified SPC limits for SRM	80% - 120%	85% - 115%	Use QSM specified limits
Matrix Spikes	80% - 120%	75% - 125%	75% - 125%	Use QSM specified limits
Sample Duplicates	0 - 20%, when > 5 x RL, ± RL when < 5 x RL	0 - 20%, when > 5 x RL, ± RL when < 5 x RL	0 - 20%, when > 5 x RL, ± RL when < 5 x RL	0 - 20%, when > 5 x LOQ, ± LOQ < 5 x LOQ
Serial Dilution	<10% if > 25x IDL or MDL	<10% if > 25x IDL or MDL	< 10% if > 25x IDL or MDL	< 10% if > 50x LOQ
Matrix Spike Dup	0 - 20%, when > 5 x RL, ± RL when < 5 x RL	0 - 20%, when > 5 x RL, ± RL when < 5 x RL	0 - 20%, when > 5 x RL, ± RL when < 5 x RL	0 - 20%, when > 5 x LOQ, ± LOQ < 5 x LOQ
Post Spikes	80% - 120%	80% - 120%	75% - 125%	80% - 120%
Linear Calibration Range	90% - 110%	90% - 110%	90% - 110%	90% - 110%

* North Carolina sample batches require ± ½ LOQ for blanks.

18.2.2 All reported sample concentrations must be less than the highest calibration standard. Samples greater than the highest calibration standard must be diluted to bring the concentration within range.

18.2.3 The ICV is prepared from a second standard source other than the source employed for initial calibration. Generally, the concentration of the ICV is 5.0 µg/L.

18.2.4 The method blank must have an absolute value below the CRDL for CLP analyses and have an absolute value below ½ LOQ for DOD QSM analyses.

18.2.5 All LCS recoveries must fall within the stated acceptance limits. Statistical process control limits are established for LCS soils.

18.2.6 Matrix spikes, matrix spike duplicates, sample duplicates, and post spikes are indicators of method performance for a specific sample matrix. Acceptance limits are based on guidelines outlined in the specific methods and are listed in the table above.

18.2.7 The serial dilution must be < 10% difference for sample concentrations that are > 25x the IDL or the MDL or >50x the LOQ for DOD QSM, depending on client specifications.

19.0 CORRECTIVE ACTION FOR OUT OF CONTROL OR UNACCEPTABLE DATA

19.1 ICV and/or CCV failure requires recalibration of the instrument and/or preparation of new standard solutions. Samples analyzed between calibration verification that fail must be reanalyzed. An ICV or CCV that has failed may be rerun provided there is an attributable cause known to have affected the check standard only and not the previous samples. Examples of an acceptable cause may be a sample tip out of solution during analysis, no solution in the sample cup or obvious carryover in the CCV from a very high sample immediately prior to the CCV. If the CCV is reanalyzed, the data must be lined through, initialed and dated, and the reason for the rerun must be documented on the raw data. In addition, corrective action should be taken to eliminate the cause of the initial CCV failure to prevent future occurrence.

19.2 ICB or CCB failure requires recalibration of the instrument and/or repreparation of the calibration blank. When analyzing under SW-846, a CCB may be accepted above the RDL level under two conditions: 1) the CCB is acceptable if the sample value is ± RDL and 2) the CCB is acceptable if the level of analyte in the CCB is less than 1/10 the analyte value in the lowest reported sample. For DOD QSM, acceptance criteria must be the < ½ LOQ or less than 10 x the lowest value of the samples bracketed by the blank.

19.3 Method blank results higher than the RDL or ½ LOQ and greater than 10% of any sample value in that batch which has concentrations above the RDL requires that batch be redigested, reanalyzed, and/or reported with the appropriate qualifier. If the method blank results are less than -1x RDL or – ½ LOQ, there may be significant interference, calibration or contamination problems with the sample, or

instrument or calibration standards that must be resolved before the batch can be reported.

19.4 LCS results outside of the upper and lower control limits can be reanalyzed for verification. If reanalysis is still outside the limits, the batch representing the failing LCS must be reprepped for reanalysis.

20.0 DATA REVIEW, APPROVAL, AND TRANSMITTAL

20.1 The printout from the instrument is attached to the Laboratory Information Management System (AlphaLIMS) data report, original batch sheet, and the Mercury Prep Logbook.

20.2 The metals department analyst reviews mercury data by verifying that the values from the printout agree with values entered into the mercury data run logbook, and with the values that appear on computer printed data report sheets. The calibration standards, the correlation coefficient, CCV, LCS, duplicates, and spikes are also reviewed to make sure values are within the limits.

20.3 Levels of review responsibility are described below:

20.3.1 Analyst Review: Mercury data are reviewed by the analyst after the run is complete and checked once more after data are entered in AlphaLIMS.

20.3.2 Peer Analyst Review: Another analyst familiar with mercury and its data reviews all data in detail.

20.4 Review of each level

20.4.1 Analyst Review: Analyst checks his/her own data and makes necessary corrections on any errors (i.e., writing wrong numbers, checks correlation coefficient again, dilution factors, and continuous calibration standards).

20.4.2 Peer Analyst Review: Reviews all data on the printout, data reported in the Mercury Data Run Logbook, Mercury Prep Logbook, and information that are on the computer printed data report. It is important at this level to check what preparation date was entered, dilution factors, concentrations of spikes, and CCVs. The Case Narrative Report is used to standardize items checked by peer analysts.

20.5 The complete data review process requires the use of the Mercury Prep Logbook, Prep data report, batch sheet, AlphaLIMS data report, raw instrument data, auto-run sequence record if the samples were analyzed using an autosampler, and Case Narrative Report

21.0 RECORDS

21.1 Prep data are recorded in accordance with Section 12 of this SOP.

21.2 Data from an analysis are recorded on a printout from the instrument and stored in digital format on the instrument computer or on the network server.

21.3 Reported values come from one sample run if the instrument calibration and other required instrument specifications are met. The data are reported as a raw value along with any dilution factors used.

22.0 RECORDS MANAGEMENT

All data associated with the performance of this procedure, including relevant logbooks, are maintained as quality records in accordance with GL-QS-E-008 for Quality Records Management and Disposition.

23.0 LABORATORY AND WASTE HANDLING AND DISPOSAL

23.1 Standard solutions that must be disposed of are transferred by the Waste Management Technician to be disposed of in accordance with the Laboratory Waste Management Plan, GL-LB-G-001.

23.2 Special caution must be taken when samples are radioactive. Handling, delivery, transfer, and disposal of radioactive samples and residues must follow the related SOPs. Persons handling radioactive materials must first pass the training course.

24.0 REFERENCES

24.1 Methods for the Determination of Metals in Environmental Samples, Supplement 1, EPA 600/R-94-111. Method 245.1, Revision 3.0, May 1994.

24.2 Perkin Elmer FIMS400/FIMS100 Flow Injection Mercury System.

24.3 Test Methods for Evaluating Solid Waste: Laboratory Manual Physical/ Chemical Methods, Volume 1A, EPA SW-846, Final Update III, June 1997. USEPA Office of Solid Waste and Emergency Response, Washington, D.C. 20460.

24.4 Method 7470A, "Mercury in Liquid Waste (Manual Cold-Vapor Technique)," Revision 1, September 1994.

24.5 Method 7471A, “Mercury in Solid or Semisolid Waste (Manual Cold-Vapor Technique),” Revision 1, September 1994.

24.6 Method 7471B, "Mercury in Solid or Semisolid Waste (Manual Cold-Vapor Technique)," Revision 2, February 2002.

24.7 Methods for Chemical Analysis of Water and Wastes, 1983, (EPA-600/4-79/020-PB84-128677).

24.8 Method for the Determination of Metals in Environmental Samples, Method 245.2, 1974.

24.9 Dept. of Defense (DOD), Dept. of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories DOD QSM, Version 5.3, July 2019.

25.0 HISTORY

Revision 34: Added statement to clarify IEC verifications.

Revision 35: Clarification of DOD QSM criteria and method acceptance criteria.

Revision 36: Revised equilibration time to 24 after pH adjustment. Added acceptance criteria for blanks in NC sample batches.

Revision 37: Updated to volume of aqua regia used for samples and QC. Updated DoD QSM Version 5.3, July 2019. Deleted redundant sections.

Revision 38: Updated Sediment, oil and soil prep section to reflect current practices.

VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE

FOR

DETERMINATION OF METALS BY ICP-MS

APPLICABLE TO METHODS:
EPA Method 200.8
EPA SW-846 Method 6020
EPA SW-846 Method 6020A
EPA SW-846 Method 6020B

(GL-MA-E-014 REVISION 33)

PROPRIETARY INFORMATION

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1.0 STANDARD OPERATING PROCEDURE FOR DETERMINATION OF METALS BY ICP-MS

2.0 METHOD CODES

2.1 EPA Method 200.8

2.2 EPA SW-846 Method 6020

2.3 EPA SW-846 Method 6020A

2.4 EPA SW-846 Method 6020B

3.0 METHOD OBJECTIVE AND PURPOSE

This standard operating procedure (SOP) describes the determination of metals using a Perkin Elmer ICP-MS Model ELAN 9000 Spectrometer and Perkin Elmer ICP-MS NexION 300x and 350x. Prior to analysis, samples must be digested using appropriate sample preparation methods (such as Methods 3005, 3010, 3050, or 200.2) and other applicable requests.

4.0 METHOD APPLICABILITY AND METHOD SUMMARY

4.1 Refer to Appendix 3 for analyte lists and masses.

4.2 Applicable Matrices: These methods are applicable to the determinations of any of the analytes listed above for various matrices including waters, oils, soils, sludges, biological tissues, Toxicity Characteristic Leaching Procedure (TCLP) extracts and other more unusual types of sample which are generally classified as a miscellaneous matrix.

4.3 General Method Summary: After the samples are prepared in accordance with the sample preparation SOP, they are analyzed by Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) as follows:

4.3.1 The instrument is calibrated with a minimum of two calibration points for each element to be analyzed. The points consist of a calibration blank solution to define the lower calibration point and at least one standard calibration solution at the analyte concentrations to define the higher calibration point(s). A correlation coefficient of 0.995 or better (0.998 or better for SW-846 Method 6020A) is required for each analyte if multiple standards are used or the instrument is recalibrated for the analyte of interest.

4.3.2 Prepared client samples and numerous check standards and quality control samples, identified in Section 22.1 are then analyzed. The check standards and quality control samples are used to determine the quality and acceptability of the analytical data.

4.3.3 Continuing Calibration Verification standards (CCV) and Continuing Calibration Blanks (CCB) are analyzed a minimum of every 10 samples to ensure that the instrument is continuing to perform correctly. For 6020A, low level continuing calibration verification standards are analyzed in conjunction with the CCVs and CCBs.

4.4 Method Codes: Analyses must conform to EPA Method 200.8, SW-846 Method 6020, SW-846 Method 6020A, SW-846 6020B and/or customer contract specifications.

4.5 Radiochemistry conversion calculations for the uranium isotopes are included in Appendix 4.

5.0 METHOD SCOPE AND PERFORMANCE CHARACTERISTICS

5.1 Calibration Range: The range of concentrations between the calibration blank, typically 0, and that of the highest calibration standard for each analyte. Calibration standards vary according to method and equipment. A minimum of two, a blank and value standard, are required.

5.2 Linear Dynamic Range standards (LRS) are analyzed with each calibration. The linear calibration range that may be used for the analysis of samples should be judged by the analyst from the resulting data. The instrument is calibrated. The target linear range should be prepared and analyzed. The LRS results must fall within ± 10% of the target value. This LRS value is entered into the instrument's software. Any hits below this value will be valid. Hits at or above this value will be flagged by the system and must be diluted to fall within the linear dynamic range. If a linear range standard is not used for a specific calibration, the highest calibration standard becomes the upper limit of reporting.

5.3 Instrument Detection Limits (IDLs) are useful means to evaluate the instrument noise level and response changes over time for each analyte from a series of reagent blank analyses to obtain a calculated concentration. IDLs are determined by the mean of blank results plus three times the standard deviation of 10 replicate analyses of the reagent blank solution. (Use zero for the mean if the mean is negative). Each measurement should be performed as though it were a separate analytical sample (i.e., each measurement should be performed between the analysis of separate samples). IDLs should be determined at least once using new equipment, after major instrument maintenance such as changing the detector, and/or at a frequency designated by the project.

5.4 Method Detection Limit (MDL) studies for each analyte are performed and/or verified at least annually. These studies are conducted and calculated in accordance with SW-846, Chapter 1, paragraph 5.0, and GL-LB-E-001 for the Determination of Method Detection Limits. The relevant quantitation limits are established based on the most current MDL study. The current MDLs are maintained and can be found in the AlphaLIMS database. For 6020B mean recovery for the seven replicates is ± 35% of the known value with RSD of ≤ 20%.

5.5 Method Precision: To assure analytical precision of methods used, Laboratory Control Samples (LCS) are analyzed with each batch. LCS duplicates are analyzed with each batch when requested.

5.6 Method Bias (Accuracy) is determined by calculating recoveries of LCS of a similar matrix.

5.7 If uncertainty and total propagated uncertainty measurements are needed, they may be determined using GL-QS-E-014 for Quality Assurance Measurement Calculations and Processes.

6.0 DEFINITIONS

6.1 Continuing Calibration Blank (CCB): An aliquot of reagent water or other blank matrix that is analyzed after each CCV. The CCB is used to determine whether the analytical sequence is in control during sample analysis.

6.2 Continuing Calibration Verification (CCV) Standard: An aliquot of reagent water or other blank matrix to which known quantities of the method analytes are added. The CCV is analyzed exactly like a sample, periodically throughout the run sequence. Its purpose is to determine whether the analytical sequence is in control during the sample analysis. It may be prepared from the same source as the calibration standards, and is usually of varied concentrations.

6.3 Contract Required Detection Limit (CRDL): Minimum level of detection acceptable under the client project requirements.

6.4 Correlation Coefficient: A number (r) that indicates the degree of dependence between two variables (concentration-absorbance). The more dependent they are, the closer the value to one. Determined on the basis of the least squares line.

6.5 Data Qualifiers: The following qualifiers should be used in order to identify analytical situations that might need additional information stated in narrative before the release of the data.

U - Non-Detect. Below the Instrument or Method Detection Limit (depending upon specific project requirements)

B - Sample concentration value is between the MDL (or IDL) and the CRDL or analyte was detected in the Method Blank (Client Specific)

J - Sample concentration is between the MDL (or IDL) and the CRDL-client specific qualifier.

Blank - Concentration value is above the CRDL

* - An RPD value in the duplicate sample is out of criteria

N - A percent recovery value in the spike sample is out of criteria

E - A percent difference in the serial dilution sample is out of criteria because of the presence interference.

6.6 Initial Calibration Blank (ICB): An aliquot of reagent water or other blank matrix that is analyzed after each ICV. The ICB is used to determine whether there is carryover contamination.

6.7 Initial Calibration Verification (ICV): A solution of method analytes of known concentrations. The ICV is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check laboratory performance with externally prepared test materials.

6.8 Instrument Performance Check Solution (IPC): A solution of one or more method analytes, surrogates, internal standards, or other test substances used to evaluate the performance of the instrument system with respect to a defined set of criteria.

6.9 Laboratory Control Standard (LCS): An aliquot of reagent water or other blank matrix to which known quantities of the method analytes are added in the laboratory. The LCS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements.

6.10 Linear Calibration Range (LCR): The concentration range over which the instrument response is linear.

6.11 Method Blank (MB): An aliquot of reagent water or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The MB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

6.12 Method Detection Limit (MDL): The minimum concentration of an analyte that can be identified measured and reported with 99% confidence that the analyte concentration is greater than zero.

6.13 Spike (Matrix Spike or Post Spike): An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS or PS is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS or PS corrected for background concentrations.

6.14 Limit of Detection (LOD): An analyte, method and matrix specific estimate of the minimum amount of a substance that can be reliably detected. GEL has established LOD = 2 x MDL.

6.15 Limit of Quantitation (LOQ): An analyte, method and matrix specific estimate of the minimum amount of a substance that can be reported with a specific level of confidence. The LOQ is set at or above the concentration of the lowest initial calibration standard. The laboratory must empirically demonstrate precision and bias at the LOQ. The LOQ and associated precision and bias must meet client requirements and must be reported. GEL uses the following guidance (LOD < LOQ):

When LOD < PQL, PQL = LOQ

When LOD > PQL, LOQ is raised to next lowest calibration standard.

6.16 Practical Quantitation Limit (PQL): The PQL is typically at or above the lowest point on an acceptable initial calibration curve. It may also be determined by multiplying the MDL by approximately 2 to 10. Concentrations of a target analyte

determined to be greater than its PQL are defined as quantitative results. This limit is not used in DoD ELAP reporting.

6.17 10% Frequency: A frequency specification during an analytical sequence allowing for not more than 10 analytical samples between required calibration verification measurement, as specified by the EPA methodology.

6.18 Refer to GL-QS-B-001 the Quality Assurance Plan for additional lab-wide used definitions.

7.0 INTERFERENCES TO THE METHOD

7.1 Chemical interferences are minimal in ICP-MS Spectroscopy because the extremely high energy of the plasma breaks nearly all the chemical bonds. However, ICP-MS analysis is subject to the following three types of interferences:

7.1.1 Physical interferences are those physical properties of a sample solution that prevent their introduction to the plasma with efficiency equal to that of the calibration standards. This type of interference can be corrected via the bias correction calculation in SW-846, Chapter 1, paragraph 5.0, through the use of an internal standard in accordance with the instrument operating manual or by diluting the sample in reagent blank solution until the percent recovery falls with method guidelines.

7.1.2 Isobaric elemental interferences are caused by isotopes of different elements that form singly or doubly charged ions of the same nominal mass-to-charge ratio and that cannot be resolved by the mass spectrometer. If analytical isotopes are selected that may have an isobaric interference, then all data obtained under such conditions must be corrected by measuring the signal from another isotope of the interfering element and subtracting the appropriate signal ratio from the isotope of interest.

7.1.3 Isobaric polyatomic ion interferences are caused by ions consisting of more than one atom that have the same nominal mass-to-charge ratio as the isotope of interest, and that cannot be resolved by the mass spectrometer in use. These ions are commonly formed in the plasma or interface system from support gases or sample components. Most of the common interferences have been identified and are listed in Method 200.8, Table 2 together with the method elements affected. Such interferences must be recognized, and when they cannot be avoided by the selection of alternative analytical isotopes or sample prep procedures, appropriate corrections must be made to the data.

8.0 SAFETY PRECAUTIONS AND HAZARD WARNINGS

8.1 PREVENT SKIN AND EYE CONTACT BY USING SPECIFIED PERSONAL PROTECTIVE EQUIPMENT WHEN MAKING STOCK REAGENTS.

8.2 WORK UNDER A HOOD TO PREVENT INHALATION WHEN MAKING STOCK REAGENTS.

8.3 Sample digestates are not extremely volatile or spontaneously combustible, but they are normally acidic and should be handled with care. Small spills may generally be wiped up with paper towels that can be disposed of in the trash. Larger spills may require the use of a mop, and the mop head may have to be disposed of as potentially hazardous waste in accordance with the Laboratory Waste Management Plan (GL-LB-G-001). If the spilled digestates begin any obvious fuming or reacting, pour a generous amount of the acid neutralizer, which is located in each lab, onto the spill before attempting to clean it up.

8.3.1 Gloves should be worn to avoid skin contact with digestate during clean-up.

8.3.2 Eye protection is required when handling samples and an eyewash station is located in each analysis lab.

8.3.3 Do not persist in cleaning up a spill in the presence of strong fumes. Move out of the area, try to isolate the area and notify your supervisor immediately.

8.4 Handling radioactive samples requires the use of gloves, a lab coat or an apron in addition to eye protection. Refer to GL-RAD-S-004 for Radioactive Material Handling

8.5 These instruments use high voltage electricity and therefore, should be shut completely down any time electronic components may be exposed to personnel or any liquids.

8.6 Wear eye protection with side shields while performing procedures in the lab.

8.7 All chemicals and samples should be treated as potential health hazards, and exposure to these chemicals must be reduced to the lowest level possible. GEL maintains a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals in the laboratory as well as a reference file of Material Safety Data Sheets (MSDS). These documents are maintained in the laboratory. Individual sample MSDS forms provided by the clients are kept in Login.

8.8 Personal protective equipment

8.8.1 Gloves are required when handling the chemicals in this procedure.

8.8.2 Work under a hood when using concentrated acids.

8.9 Prior to handling radioactive samples, analysts must have had radiation safety training and must understand their full responsibilities in radioactive sample handling. Some general guidelines follow:

8.9.1 Wear a lab coat when working with radioactive samples.

8.9.2 Prohibit admittance to immediate work area.

8.9.3 Protect counter tops with counter paper or work from radioactive sample handling trays.

8.9.4 Post signs indicating radioactive samples are in the area.

8.9.5 Take swipes of the counter tops upon completion of work. Deliver those swipes to the nearest swipe count box.

8.9.6 Segregate radioactive wastes. Radioactive waste containers are obtained from Waste Management.

8.10 All samples, chemicals, extracts, and extraction residues must be transferred, delivered, and disposed of safely according to all related SOPs.

8.10.1 Segregate solid wastes from liquid wastes in the satellite area containers.

8.10.2 Segregate oil wastes from water-soluble wastes in the satellite area containers

8.11 Never leave gas cylinders unchained or untied, including when they are on the moving carts.

8.12 In the event of an accident or medical emergency, call for help immediately. When time and safety permit, an accident report form should be completed and turned in to the safety committee.

8.13 Fire escape routes are posted in the lab; all personnel should be familiar with them. In addition, fire safety equipment such as fire extinguishers is located in the lab. Training is available on the proper operation of this equipment.

9.0 CAUTION WARNINGS

9.1 Because they can be health hazards, the exhaust gases from the plasma and vacuum systems must be eliminated through the laboratory's ventilation duct, which is attached to the instrument's exhaust vent. If inadequate ventilation occurs, pump-fluid vapor, ozone, and other toxic products of combustion can accumulate in the laboratory and cause bodily harm. Hydrofluoric acid (HF) fumes, if inhaled, extensively burn lung tissue. Ensure that the exhaust system established at installation continues to operate effectively.

9.2 Prepare sample and transfer acids using a hood to avoid fumes.

9.3 Store and prepare sample away from the instrument to minimize corrosion.

9.4 Clean up any spills quickly.

9.5 The drain vessel contains the spray chamber's effluent, which can be toxic. Corrosion of the vessel and connecting tube can result in leaks that damage the instrument or cause bodily harm.

9.5.1 Use the capped plastic drain vessel that was provided with the instrument. Never use glass.

9.5.2 Place the drain vessel on the instrument table below the peristaltic pump where the container is easy to check.

9.5.3 Check the drain vessel frequently. Empty it before it is three-fourths full.

9.5.4 Check the tubing and vessel for deterioration. If the tubing becomes brittle or cracked, replace it. Organic solvents cause more rapid deterioration than aqueous solutions.

9.6 The torch and interface remain hot after the plasma is turned off. Do not touch the torch box or interface cones for 10 minutes after the plasma has been shut off.

9.7 High voltages and radio frequencies are potential hazards of the ICP-MS. Shut the instrument down completely before removing any of the outside panels (to clean air filters, replace a fuse, etc.).

9.8 When changing the rotary pump oil, remember that the pump oil may be hot. The oil can cause a burn if allowed to contact the skin.

10.0 APPARATUS, MATERIALS, REAGENTS, EQUIPMENT, AND INSTRUMENTS

10.1 Apparatus and Equipment

10.1.1 Replacement special glass parts for the ICP-MS such as quartz torch bodies, injector tips and spray chambers may be ordered from a qualified vendor through the GEL Purchasing Agent.

10.1.2 Replacement ICP-MS interface parts such as sampling cones, skimmer cones and ion-optics can be purchased from a qualified vendor through the GEL Purchasing Agent.

10.1.3 Consumable materials such as tubing are often attainable from various scientific product companies. The GEL Purchasing Agent can help to find the best prices. These items may also be ordered from the instrument manufacturer if necessary. All orders must be placed through the GEL Purchasing Agent.

10.2 Reagents, Chemicals, and Standards

10.2.1 Reagents: Refer to Reagent Logbook

10.2.2 Standards: Refer to GL-LB-E-007 for Laboratory Standards

10.2.3 Other Chemicals: Additional compounds, surfactants, oils, cleaning agents, etc., may be routinely ordered through the GEL Purchasing Agent.

10.3 Instrumentation

10.3.1 Perkin Elmer ICP-MS ELAN Model 9000 with PC monitor and printer.

10.3.2 Perkin Elmer ICP-MS NexION 300x and 350x with PC, monitor and printer

10.3.3 ESI SC4 DX

10.3.4 CETAC Model ASX-510 Autosampler with accessory autodiluter (PE 9000).

10.3.5 Neslab CFT75 recirculating bath or equivalent provides cooling to the ICP-MS.

11.0 SAMPLE HANDLING AND PRESERVATION REQUIREMENTS

11.1 Aqueous samples should be preserved with nitric acid to a pH of < 2 prior to receipt by the analyst. Solid samples should be kept at 0° ≤ 6° C prior to digestion.

11.2 Refer to GL-SR-E-001 for Sample Receipt, Login and Storage.

12.0 SAMPLE PREPARATION TECHNIQUES

12.1 All samples except drinking water with Turbidity < 1 NTU and samples specifically exempted by contract, are prepared in accordance with the following SOPs:

12.1.1 GL-MA-E-016 for Sample Preparation for Total Recoverable Elements by EPA Method 200.2 (USEPA Method 200.2)

12.1.2 GL-MA-E-006 for Acid Digestion of Total Recoverable or Dissolved Metals in Surface and Groundwater Samples for Analysis by ICP or ICP-MS (USEPA SW-846 Method 3005A)

12.1.3 GL-MA-E-008 for Acid Digestion of Total Metals in Aqueous Samples and Extracts for Analysis by ICP or ICP-MS (USEPA SW-846 Method 3010A)

12.1.4 GL-MA-E-009 for Acid Digestion of Sediments, Sludges and Soils (USEPA SW-846 Method 3050B)

12.1.5 Dept. of Defense (DOD), Dept. of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories DOD QSM, Version 5.1, January 2017 and DOE QSAS Version 3.1 January 2017.

12.2 Additional filtration may be required to prevent clogging of sample introduction system.

12.3 All sample preparation records are stored in AlphaLIMS.

12.4 Sample spills should be handled as stated in Section 8.3.

13.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

13.1 Routine Preventative and Special Operational (Failure)

13.2 Routine Preventative Maintenance (PM) Procedures are done as follows:

Frequency	Procedure
When Needed	Clean nebulizer tip after use. Replace peripump sample introduction tubing. Change pump hoses on drain systems. Check drain waste collection containers, and empty as necessary. Check Neslab water level and add water if required. Clean/replace interface cones. Clean/replace nebulizer. Clean/replace torch. Check/replace water filter.
Quarterly	Change oil in interface rotary pump (or as needed). Clean ion lenses 4-6 months (or as needed).
6 Months	Clean air filters.
12 months	Change pump oil in backing rotary pump. Evaluate/replace EM (electron multiplier)

13.3 Non-Routine Maintenance Procedures (Special, Operational or Failure Mode Maintenance)

13.3.1 If the instrument will not function properly see the trouble shooting section in the appropriate Maintenance Manual.

13.3.2 If the analyst is unable to determine cause/fix instrument, the GEL Service Engineer is called, and if needed, the manufacturer's customer support number may be found in the owner's maintenance manuals.

13.4 Refer to ICP-MS Maintenance Logbook for routine records. Service call records are also available.

14.0 PREPARATION OF STANDARD SOLUTION AND QUALITY CONTROL SAMPLES

14.1 Source standards records are recorded in AlphaLIMS.

14.2 Recommended Suppliers: Refer to source log and use Approved Vendors List maintained in Procurement.

14.3 Standards are receipted, labeled, prepared and stored in accordance with the GL-LB-E-007 for Laboratory Standards Documentation.

14.4 Acid Screens and ICSA Standard Screens are performed prior to use.

14.4.1 Prior to use, new lots of nitric acid, hydrochloric acid, and 30% hydrogen peroxide are screened to test for contamination. If an analyte is detected above ½ RL, the acid is re-screened. If the re-screen is also ½ RL, the resulting lot is transferred to another area of the lab where this low-level contamination doesn't affect the analysis. New lots are then screened until a suitable lot is obtained. Copies of the acid screens by lot number are kep on file. The following concentrations are submitted for analysis.

14.4.1.1 3% solution of 30% hydrogen peroxide (per SW-846 3050B prep volumes)

14.4.1.2 10% solution of nitric acid (per SW-846 3050B prep volumes)

14.4.1.3 5% solution of hydrochloric acid (per SW-846 3010A prep volumes)

14.4.1.4 20% solution of hydrochloric acid is analyzed via ICP for Sulfur determination only (per SW-846 3050B prep volumes)

14.4.2 Prior to use, new lots of ICSA standards are screened to document low-level inherent concentrations of non-target analytes. Typically 20 analyses of the ICSA are averaged and the average concentration is then used to assess the ICSA and ICSAB interferent and recovery data.

15.0 INSTRUMENT CALIBRATION

15.1 Samples may be analyzed manually or automatically.

15.1.1 Tuning for each instrument is performed daily according to the following directions and criteria.

15.1.2 Aspirate a tuning solution consisting of 10 µg/L each of ^{9}Be, ^{24}Mg, ^{59}Co, ^{115}In and ^{208}Pb. Perform 5 replicates. Manufacturer's recommended tune criteria are as follows:

Parameter	Starting Point
^{9}Be	± 0.10 amu
^{24}Mg	± 0.10 amu
^{59}Co	± 0.10 amu
^{115}In	± 0.10 amu
^{208}Pb	± 0.10 amu
Ba^{++} net intensity mean	< 0.05 or 5%
CeO net intensity mean	< 0.03 or 3%
Be, Mg, Co, In, Pb net intensity RSD	< 5%
Resolution at 10% peak height	< 0.9 amu

15.1.3 Conduct additional tuning procedures as specified by client contract or other methodology.

15.1.4 If any of the preceding tune criteria does not meet the recommended requirements, investigate the problem, correct the situation, and reanalyze the tune sequence.

15.1.5 Standardization: Standardization is required on a daily basis.

15.2 Internal standards are used as appropriate for the analytes of interest. The internal standards are made in the appropriate acid and contain varying concentrations of such elements as ^{45}Sc, ^{74}Ge, ^{115}In, ^{175}Lu, and/or ^{181}Ta. The internal standard solution is mixed in-line with the sample stream using a dedicated channel of the peristaltic pump. Internal standards may be added at the time of analysis as an alternative to in-line mixing. Alternate internal standards may be used to meet client needs.

15.3 Calculations are described in the instrument manual.

15.4 Calibration standards vary according to method and equipment. A minimum of two, a blank and value standard, is required.

15.5 For required quality control standards refer to Section 21.0.

15.6 For continuing calibration requirements refer to Section 21.0.

15.7 For what to do when initial or continuing calibrations fail to meet requirements, refer to Section 21.0.

16.0 INSTRUMENT PERFORMANCE REQUIREMENTS

16.1 Before samples may be analyzed to generate reportable data, the instrument must have been tuned and calibrated. Also, the Initial Calibration Verification (ICV), which is prepared from an independent source, Initial Calibration Blank (ICB), the Reportable Detection Limit (CRDL), the Interference Check Standards (ICS-A, ICS-AB), and the Linear Range Standards (LRS) must meet the requirements stated in Section 21.2 for each analyte being reported, unless otherwise required by methodology, clients or contracts.

16.2 The instrument calibration and all continuing verification data is maintained in the printed hard copy file. The printouts are kept in chronological order by instrument. Recent files are in the metals laboratory; older records are archived in short or long-term storage.

17.0 ANALYST AND METHOD VERIFICATION REQUIREMENTS

17.1 Analyst training is conducted and certified in accordance with the GL-HR-E-002 for Employee Training.

17.2 Method performance is verified by the conductance of MDL studies in accordance with GL-LB-E-001 for The Determination of Method Detection Limits, and by the evaluation of LCS and LCS duplicates for each batch of samples.

18.0 ANALYSIS PROCEDURES AND INSTRUMENTAL OPERATION

18.1 All samples are introduced in a set measured quantity, via a peristaltic pump, through a nebulizer into a spray chamber and carried with argon gas through the Radio Frequency (RF) field to generate analyte ions that are selected and measured by the quadruple mass spectrometer.

18.2 Run Sequence

18.2.1 Instrument Calibration executed in accordance with Section 16.

18.2.2 Initial Calibration Verification steps include:

18.2.2.1 ICV

18.2.2.2 ICB

18.2.2.3 CRDL (low level ICV for 6020A)

18.2.2.4 ICS-A

18.2.2.5 ICS-AB

18.2.2.6 CCV

18.2.2.7 CCB

18.2.2.8 LRS

18.2.2.9 CCV

18.2.2.10 CCB

18.2.3 Sample Run (10 samples or less)

18.2.4 Continuing Calibration Verification:

18.2.4.1 CCV

18.2.4.2 CRDL (for SW-846 Method 6020A only and analyzed at end of batch).

18.2.4.3 CCB

18.2.5 Repeat steps 18.2.3 and 18.2.4 until end of run or verification is out of specification. When the latter occurs, repeat step 18.2.1 and continue with steps 18.2.2 and 18.2.3.

18.2.6 Repeat steps 18.2.3 through 18.2.5 until the end of the run or verification is out of specification. When latter condition occurs, repeat 18.2.1 and continue sequentially through 18.2.7.

18.2.7 Final Verification Steps

18.2.7.1 ICS-A (if required)

18.2.7.2 ICS-AB (if required)

18.2.7.3 CCV

18.2.7.4 CRDL (for SW-846 Method 6020A only)

18.2.7.5 CCB

NOTE: Method 6020A only requires there to be a closing low level continuing calibration standard (CRDL). For ease of analysis, the CRDL standard can be analyzed every 10 samples.

18.3 For data storage refer to Section 24.1.

18.4 General operation of the instrument.

18.4.1 The Perkin Elmer ICP-MSs are inductively-coupled argon plasma mass spectrometers. The ICP-MS is capable of determining analytes from m/z =

6 (Li) through m/z = 238 (U). The operating software of the system is based on Microsoft Windows.

18.4.2 Set-up

18.4.2.1 Attach the nebulizer argon line to the quick-connect on the nebulizer's argon tube and ensure that it is tightly in place.

18.4.2.2 Attach the nebulizer and endcap to the spray chamber.

18.4.2.3 Attach sample pump tubing to the front peristaltic pump (various sizes may be used depending on need), and attach feed end to endcap.

18.4.2.4 If running manually, place the suction end of the sample tubing in acidified rinse water; otherwise attach it to the autosampler.

18.4.2.5 Ensure that drain hose is connected to the spray chamber and properly plumbed through the drain peristaltic pump.

18.4.3 Run Procedure

18.4.3.1 Refer to the respective owner's manuals for specific instructions for tuning, sequence loading, and method development on each instrument; ELAN 9000 software guide, NexION 300x/350x software guide.

18.4.3.2 The owner's manual for each instrument is located in the ICP-MS laboratory.

18.4.4 Typical Analysis Problems

18.4.4.1 Sample Overrange: If a requested element is overrange for a sample, it is necessary to dilute the sample and rerun. Dilution factors must be taken into account when reporting final values.

18.4.4.2 Interferences: Although the ICP-MS can compensate for many interferences with appropriate correction factors, unpredicted interferences may still occur. If the analyst suspects this, then the sample should be diluted and rerun. Dilution factors must be taken into account when reporting final values.

18.4.4.3 Torch/Sample Introduction Drift: Various changes in torch plasma conditions or sample introduction can have a great effect on the detector counts. They must be corrected or the instrument must be re-standardized.

18.4.4.3.1 If oils or solid samples have been run, and the CCV is not acceptable, allow the instrument to rinse 15 to 20 minutes. If CCV is then acceptable, reanalyze

the samples and continue. If the CCV again fails its requirements, re-standardize as necessary.

18.4.4.3.2 If sample introduction drift is suspected, check peristaltic pump tubing for collapse and replace as needed.

18.4.4.3.3 Tubing connections may become loose allowing air to bubble into the sample path. Tighten or replace tubing. If necessary, seal with Parafilm.

18.4.4.4 Serial dilution: If the analyte concentration is sufficiently high (minimally, a factor of 100 times above the MDL, 25x PQL for 6020B and 50 x the LOQ for DoD QSM in the original sample), an analysis of a five fold (1 + 4) dilution should agree within 10% (20% for 6020B) of the original determination. If not, a chemical or physical interference effect should be suspected.

18.5 Power switches and auxiliaries

18.5.1 Each instrument has one main power switch. Refer to the respective maintenance manuals for the location. This switch remains on except while servicing the instrument.

18.5.2 The computer system has 3 power cords all connected to a switchable power strip: the computer main, the monitor and the printer power cords. The power to these is left on while the instrument is not in use for short times (e.g. overnight).

18.5.2.1 The main computer switch is on the front of the CPU.

18.5.2.2 The ***monitor switch*** is on the front of the monitor itself.

NOTE: If the computer is left in use without an analyst present for an extended period of time, turn the monitor off to prevent screen burn-in.

18.5.2.3 The ***printer switch*** is on the lower front of the printer. Refer to printer manual for operating instructions.

18.5.3 Argon gas is provided through a manifold from the storage tank. Under normal conditions a constant argon flow is available.

18.5.4 Start-up

18.5.4.1 Before starting, ensure that the power is on and the vacuum system is switched on.

18.5.4.2 The owner's manual for each instrument provides specific instructions for ignition of the plasma (refer to Section 18.5.1 for software manuals).

18.5.4.3 The plasma should be steady and should not flicker.

18.5.4.4 Once ignition has occurred, levels may be adjusted; it is best to set levels appropriate to the method by loading or editing an appropriate tune file and allow 30 minutes for warm-up.

18.5.5 Shutdown

18.5.5.1 When the plasma is off, the instrument is either in Standby or Shutdown mode. The ICP-MS should be completely shut down only in case of major maintenance, relocation of the instrument, or when the lab is closed for an extended time.

18.5.5.2 Daily shutdown (if required): Refer to the respective owner's manual shutdown procedures (Refer to Section 18.5.1 for software manuals.)

18.6 Sample quantity requirements are approximately 5 mL for each run with the nebulizer. If out of specification or range, further sample may be necessary for reruns.

18.7 The autosampler can be used by attaching introduction tube to sample introduction system, and rinsing tubing to peristaltic pump and following autosampler table set-up and procedures. To define a sequence, refer to the appropriate software manual (refer to Section 18.5.1).

19.0 CALCULATIONS AND DATA REDUCTION METHODS

19.1 Any dilutions, concentrations, or preparation factors must be taken into account prior to reporting data to a client.

$$\text{Relative Percent Difference} = \frac{100 * \left|\text{Sample 1 Value} - \text{Sample 2 Value}\right|}{\frac{(\text{Sample 1 Value} + \text{Sample 2 Value})}{2}}$$

$$\text{Matrix Spike Recovery} = \frac{100 * (\text{Spike Value} * \text{DF} * \text{PF} - \text{Sample Value} * \text{DF} * \text{PF})}{\text{Spike Nominal Concentration} * \text{DF} * \text{PF}}$$

$$\text{Post Spike Recovery} = \frac{100 * (\text{Spike Value} - \text{Sample Value})}{\text{Spike Nominal Concentration}}$$

$$\text{LCS Recovery} = \frac{100 * (\text{Sample Value} * \text{DF} * \text{PF})}{\text{Nominal Concentration} * \text{DF} * \text{PF}}$$

Where: Sample Value = instrument reading for the sample
Spike Value = instrument reading for the spiked sample

DF = Dilution Factor

PF = Preparation Factor

Relative Error Ratio (RER) 2 sigma equation:

$$RER = \frac{|\text{Sample Activity} - \text{Duplicate Activity}|}{\sqrt{(\text{Sample 2 sigma TPU}/1.96)^2 + (\text{Duplicate 2 sigma TPU}/1.96)^2)}} =\leq 3$$

NOTE: Activity calculations can be found in Appendix 4 of this SOP. Two sigma TPU calculations can be found in SOP GL-QS-E-014.

19.2 Care must be taken that the correct units are being employed.

20.0 DATA RECORDING

20.1 ICP-MS data are generally stored to the hard disk drive of the ICP-MS computer system and printed at the instrument as each sample is analyzed.

20.2 ICP-MS samples are analyzed in three replicates and the reported value is the average of the replicates. The data for individual replicates is stored in the computer.

20.3 Data are processed locally by manual or programmable procedures to eliminate unused data, to enter dilution factors, and to enter relevant conversion factors prior to uploading to AlphaLIMS.

21.0 QUALITY CONTROL REQUIREMENTS

21.1 Frequency of Quality Control Activities (also refer to Appendix 1)

21.1.1 Initial Calibration Verification (ICV) is performed immediately following each calibration and Continuing Calibration Verification (CCV) is performed after at least every 10 samples.

21.1.2 Initial Calibration Blank (ICB) is performed immediately following the ICV and Continuing Calibration Blanks (CCB) must run with each CCV.

21.1.3 An Interference Check Standard (ICS) is analyzed at the beginning of each analytical run and at least once every twelve hours (if required). Additional requirements may be specified by client contract or methodology.

21.1.4 The PQL standard is analyzed after each calibration and recommended at least every 10 samples between the CCV and CCB analyses for SW-846 Method 6020A. Method 6020A requires analysis at the end of every batch and only recommends analysis every 10 samples. This standard may also be labeled as a CRDL standard.

21.1.5 A method blank (MB) is performed for each batch of 20 or fewer samples or per client requirement.

21.1.6 A matrix spike (MS) and a duplicate (DUP), or matrix spike (MS) and matrix spike duplicate (MSD) are analyzed for each batch of 20 or fewer

samples or per client requirements (5% frequency). For EPA 200.8, the same QC are analyzed for each batch of 10 or fewer samples (10% frequency).

21.1.7 A laboratory control sample (LCS) is analyzed with each batch of 20 or fewer samples. An LCS duplicate (LCS DUP) may be added if required by the client.

21.1.8 Serial dilutions or analytical spikes are analyzed to confirm the presence or absence of interferences when analyzing a new matrix type. The serial dilution is generally performed at a 5x of the test sample.

21.1.9 A post spike (PS) is required for DoD-QSM, SW-846 6020A, or SW-846 6020B if the matrix spike (MS) or matrix spike duplicate (MSD) recoveries fall outside of the limits in section 21.2.8. Post spikes can also be performed at client request.

21.2 Acceptance Limits (also refer to Appendix 2)

21.2.1 ICV results must be between 90% and 110% of the true values for all methods.

NOTE: The ICV is the second source standard and may be used as the CCV as it also will show calibration verification.

21.2.2 ICB and CCB results must have an absolute value less than PQL for 200.8, SW-846 6020 and SW-846 6020A . For DoD QSM analysis, the absolute value must be less than the ½ LOQ. The ICB results must have an absolute value less than or equal to ½ PQL for 6020B and less than or equal to the PQL for the CCB for 6020B. If this is not the case, the reason for the out-of-control condition must be found and corrected, or any data reported must be 10 times greater than the absolute value for the element or less than the PQL.

21.2.3 Interference Check Sample results must be monitored at the beginning of an analytical run or once every 12 hours, whichever is more frequent, for work under SW-846. The ISCA and ICSAB must recover 80-120% the reporting level for the spiked analysis and must have an absolute value less that 2x the reporting level for the non-spiked analyte. For DoD QSM, the ICSA must have an absolute value of less than ½ LOQ for the non-spiked analytes.

21.2.4 The Linear Range Standard (LRS) is analyzed within the calibration verification read back and must fall between 90% and 110% of the true values. Meeting these criteria allows target analyte concentration to be reported up to the LRS concentration thus extending the calibration range of the instrument. Any sample concentrations above the LRS concentration will be diluted to fall below the concentration of the linear calibration range standard.

21.2.5 The intensities of all internal standards must be monitored for every analysis. When the intensity of any internal standard fails to fall between 30% and 120% (or 70% and 130% for SW-846 Method 6020A and 6020B) of the intensity of that internal standard in the initial calibration then the sample must be diluted five fold (1 + 4) and reanalyzed with the addition of appropriate amounts of internal standards. The intensity levels of the internal standards for the calibration blanks (ICB and CCB) and instrument check standards (ICV and CCV) must agree within $\pm$ 20% of the intensity level of the internal standard of the original calibration solution. For work done under EPA Method 200.8, the internal standard responses of any one internal standard must not deviate more than 60% to 125% of the original response in the calibration blank. Five internal standards (^{45}Sc, ^{74}Ge, ^{115}In, ^{175}Lu, and/or ^{181}Ta) are used to cover the mass ranges reported. Refer to Appendix 3 for list of internal standard/ analyte associations. Other exotic analytes may be used as needed. Refer to Section 15.2.

21.2.6 Method blank results must be lower than the PQL (SW-846 6020 and 6020A), lower than ½ the PQL (SW-846 6020B), or less than 10% of the determined value of all samples in the batch. When performing work under EPA Method 200.8, if LRB (laboratory reagent blank) values are 10% or more of the analyte level determined for a sample or are 2.2 times the analyte MDL, then fresh aliquots of the samples must be prepared and analyzed again for the affected analytes after the source of contamination has been corrected and acceptable LRB values have been obtained. For DoD QSM work, the absolute value must be less than ½ LOQ or less than 10% of the determined value of all samples in the batch. Al, Fe, Mg, Ca, Na, and K must be less than the PQL.

21.2.7 LCS results, and LCS duplicate (if performed), must be within process control limits as established by Statistical Process Control, manufacturer's certification, or method requirements.

21.2.8 Matrix spikes with recoveries between 75% and 125% suggest the absence of interference for work under EPA Method 200.8. Matrix spikes with recoveries between 75% and 125% suggest the absence of interference for work under SW-846 Method 6020 and SW-846 Method 6020A. Matrix spikes with recoveries between 80% and 120% suggest the absence of interferences for work under DoD QSM.

21.2.9 The relative percent difference (RPD) between a sample and a sample duplicate must be within ± 20% if the analyte concentration in the sample or duplicate is greater than 5 times the PQL. If either the sample or duplicate concentration is less than 5 times the PQL, the results should agree within the absolute value of the PQL. Results less than the MDL or IDL are not evaluated. The relative error ratio (RER) between a sample and a sample duplicate should be $\leq$ 3%.

21.2.10 Serial dilution results that agree within 10% of the original analytical results, if the original results are greater than 100 times the instrument detection limit or greater than 50 times the LOQ, suggest the absence of interference. For SW-846 6020B, results that agree within 20% of the original analytical results are greater than 25 x the PQL, suggest the absence of interference.

21.2.11 Post spikes with recoveries between 75% and 125% under EPA Method 200.8 and SW-846 Method 6020 and 6020B, and between 80% and 120% under SW-846 6020A and DOD QSM suggest the absence of interference.

21.3 Out-of-Control Situations

21.3.1 ICV and/or CCV failure requires recalibration of the instrument and/or preparation of new standard solutions. Samples analyzed prior to or after calibration verifications that are not acceptable for required analytes must be reanalyzed. An ICV or CCV that has failed may be rerun once only if there is an attributable cause known to have affected the CCV only and not the previous samples. Examples of an acceptable cause may be a sample tip out of solution during analysis, an incorrectly prepared CCV, or obvious carryover in the CCV from a very high sample immediately prior to the CCV. If a CCV is reanalyzed, the data must be lined through, initialed and dated, and the reason for the rerun must be documented on the raw data. In addition, corrective action should be taken to eliminate the cause of the initial CCV failure to prevent future occurrence.

21.3.2 ICB and CCB failure requires recalibration of the instrument and/or calibration blank solution to be remade. The CCB is acceptable if the level of analyte in the corresponding sample(s) is 10 times greater or less than the PQL for the failing element. For DoD QSM work, the absolute values must be less than the ½ the LOQ or less than 10% of the determined value of all samples in the batch.

21.3.3 ICS failure requires that the instrument be re-calibrated or the interferences be corrected, via recalculation, of Interelement Correction Factors so that the ICS can be read within the required limits before samples are analyzed. ICS failure at the end of an analysis period will require that the samples' ICSA run for the affected analyte(s) during that period to be reanalyzed. The ICSA and ICSAB must recover 80-120% for the spiked analytes and must have an absolute value less than 2x the reporting level for the non-spiked analytes. For DoD QSM, the ICSA must have an absolute value of less than ½ LOQ for the non-spiked analytes.

21.3.4 LRS failures limit the reportable calibration range to the high standard in the calibration curve. Any sample concentration that falls above the high calibration standard will be diluted to fall within the calibration range.

21.3.5 Internal Standard failure requires one or more of the following: five-fold dilution of the sample, correction of the problem, termination of analysis, recalibration of the instrument, and/or reanalysis of the affected samples depending on whether the failure is due to the samples or the instrumental drift.

21.3.6 Method blank results higher than the limits in 21.2.6 and greater than 10% of any sample value in that batch that has concentrations above the PQL require that batch be redigested and reanalyzed. For DoD QSM work, the absolute value must be less than ½ LOQ or less than 10% of the determined value of all samples in the batch. Al, Fe, Ca, Mg, Na, and K must be less than PQL.

21.3.7 Matrix spikes, duplicates and spike duplicates are used only as indicators of method effectiveness on that sample and will not be used as acceptability criteria for the process, unless a special requirement of the client.

21.3.8 LCS and/or LCS duplicate results outside of established acceptance limits require the batch to be redigested and reanalyzed.

21.3.9 When analytical results suggest the presence of interference, one of the methods listed in Section 18.4.4.2 should be employed.

21.3.10 The CRDL standard should be evaluated, but no action is required if the results fall outside of the 70-130% advisory window for SW-846 6020 and EPA 200.8. For DoD QSM and SW-846 6020B, the CRDL standard must be 80-120% of the true value or recalibration is required. For SW-846 Method 6020A, the CRDL standard must be 70-130% of the true value or recalibration is required. This also includes the low level continuing calibration verification standard analyzed every 10 samples. Sample results can be evaluated for reporting if they are at least 2x the PQL for a given analyte.

21.4 Corrective actions taken for data not conforming to the requirements in Section 21.2 are stated in Section 21.3. If these corrective actions can be taken by the analyst prior to the acceptance of the data, then no nonconformance documentation is required. However, if these corrective actions include redigestion of the batch or sample, if the data have already been accepted, or if the corrective action requires an instrument service call, then a nonconformance and/or corrective action report should be completed. This report includes the date, person requesting the action, sample(s) or batch(s) affected, and action requested, all provided by the requester. The person taking the action will provide any pertinent comments, their signature, and the date the action is completed. The disposition of the nonconformance will then be verified by the Quality Systems specialist. These reports will be kept on file. Refer to GL-QS-E-002 for Documentation of Nonconformance Reporting and Dispositioning and Control of Nonconforming Items.

21.5 Analytical data are evaluated for conformance with the requirements stated in Section 21.2 by the analyst during and/or after the analysis, but before the data are entered into AlphaLIMS. Data may be accepted or rejected by the analyst at this point or by the data reviewer(s) as stated in Section 22.0.

22.0 DATA REVIEW, VALIDATION, AND APPROVAL PROCEDURES

22.1 After samples are analyzed, the data must go through the review process before it can be reported out of the lab. The analyst who performed the analysis will review the raw data prior to uploading it into AlphaLIMS. The upload process may be handled by the analyst or by a data entry clerk.

22.2 After the upload is complete, an AlphaLIMS data report is generated that will be passed (along with the batch sheet, data review checklist, and the raw data) to another analyst for a peer review. Discrepancies found in this review will be resolved before the batch data are passed and the status updated.

22.3 When this peer review is completed and all of the data are found to be acceptable, the reviewer signs and dates the batch data report and updates the status of the batch to done. If the reviewer determines that the reviewed data are not acceptable and requires additional work or correction, data are returned to the analyst or representative with an appropriate explanation.

22.4 Listed below are the data review responsibilities of the Analyst and Peer Reviewer:

22.4.1 Analyst Review is performed by the analyst who generated the data before the data are submitted for entry into AlphaLIMS. Analyst completes and attaches the run data cover sheet to the printout.

22.4.2 Peer Analyst Review is performed by an individual who did not perform the analysis but is familiar with the analytical method used and the reporting requirements. This person will review the complete data report after all data are entered, will ensure that any data entry corrections are made before data are approved, and will complete the reviewer portion of the data review check list. If the data do not meet the necessary quality requirements and need further analysis, they are returned to the analyst or representative.

22.5 A Third Review is performed for Data Packages (level 3 to level 6 CLP-Like) and/or by client requirement. This review is by the Metals Team Validator or other qualified person.

22.6 Specific items that are reviewed at each level include the following:

22.6.1 Analyst Review: Before data is submitted for entry into AlphaLIMS.

22.6.1.1 All analyses in the batch are completed.

22.6.1.2 Any corrections and comments on the data are properly initialed and dated.

22.6.1.3 Proper standard identification numbers appear on the runlog to ensure traceability from the data to original source standards.

22.6.1.4 All data are complete and accurate in the AlphaLIMS data report.

22.6.1.5 Data acceptance limit criteria identified in Section 21.2 are met or an explanation given.

22.6.2 Peer Analyst Review: Before data are submitted for further review or update:

22.6.2.1 All data are complete and accurate in the AlphaLIMS Data Report.

22.6.2.2 Any exceptions or shortcomings have been sufficiently explained or corrected.

22.6.2.3 Data are reported in the proper units or an explanation is given.

22.6.2.4 Prep factor and dilution calculations by AlphaLIMS are present in the data report and AlphaLIMS calculations are correct.

22.6.2.5 LCS and Spike Recoveries and RPD calculations by AlphaLIMS are correct, and the values are within control limits.

22.7 When the data review is completed and the data have been reported out of the lab, they are bound with the batching sheet and kept on file in the lab.

22.8 The complete data review process requires the use of the Prep Log Book, Prep data report, batch sheet, AlphaLIMS data report, raw instrument data, and runlog.

23.0 DATA REPORTING

23.1 To report data after the review process has been completed:

23.1.1 Enter the AlphaLIMS program through an available terminal and select DATA MENU, BATCH ITEMS, CHANGE BATCH STATUS.

23.1.2 Enter the Batch Number and "Submit."

23.1.3 Use the down arrow key to move the cursor down the new status column to the end. Change status from REVW to DONE and "Save."

24.0 RECORDS MANAGEMENT

All data associated with the performance of this procedure, including relevant logbooks, are maintained as quality records in accordance with GL-QS-E-008 for Quality Records Management and Disposition.

25.0 LABORATORY WASTE HANDLING AND DISPOSAL: SAMPLES, EXTRACTS, DIGESTATES, AND REAGENTS

25.1 Standard solutions that must be disposed are taken to the Waste Disposal coordinator for disposal in accordance with Laboratory Waste Management Plan, GL-LB-G-001.

25.2 Sample digestates are stored in the lab for a specified period of time following analysis. At this time, they are composited into a waste container that is picked up by the Waste Management Technician for proper disposal.

25.3 Radioactive Waste:

25.3.1 Samples returned to sample storage

25.3.2 Drain waste collected in the radioactive waste carboy is dumped when full into the appropriate 55 gallon drum sitting outside the ICPMS laboratory. Ultimate disposal of liquid radioactive waste done by waste management department.

25.3.3 Implements, vials, gloves, etc., are wrapped and labeled with radioactive tape and placed in the radioactive waste container in high bay area.

25.3.4 Expired Standard Solutions: Refer to Section 25.1.

26.0 REFERENCES

26.1 Perkin Elmer ELAN 9000 Hardware Guide

26.2 Perkin Elmer ELAN 9000 Software Guide

26.3 Perkin Elmer NexION 300x/350x Hardware

26.4 Perkin Elmer NexION 300x/350 Software Guide.

26.5 Test Methods for Evaluating Solid Waste: Laboratory Manual Physical/Chemical Methods. Volume 1A, USEPA SW-846, Third Edition, Revision 2, September 1994.

26.5.1 Method 6020A, “Inductively Coupled Plasma – Mass Spectrometry,” Revision 1, February 2007.

26.5.2 Method 6020, “Inductively Coupled Plasma – Mass Spectrometry,” Revision 0, September 1994.

26.5.3 Method 6020B, “Inductively Coupled Plasma – Mass Spectrometry,” Revision 2, July 2014.

26.5.4 Method 3005A, “Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA or ICP Spectroscopy”, Revision 1, July 1992.

26.5.5 Method 3010A, “Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA or ICP Spectroscopy,” Revision 1, July 1992.

26.5.6 Method 3050B, “Acid Digestion of Sediments, Sludges, and Soils”, Revision 2, December 1996.

26.6 USEPA Method 200.8, “Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma – Mass Spectrometry,” Revision 5.4, May 1994.

26.7 Conversion Constants for Uranium Isotopes, Dr. Robert Litman, April 2009.

26.8 Department of Defense (DoD), Department of Energy (DoE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, DoD QSM Version 5.1, DoE QSAS Version 3.1, January 2017.

27.0 HISTORY

Revision 33: Replace “IDL for non DOE Clients, 100 x the MDL for DOE Alb Clients” with MDL

Revision 32: Updated for clarification for ICP-MS replicates. Updated the Appendices 1 and 2 to remove readback requirements no longer needed.

Revision 31: Clarification of DOD QSM criteria and method acceptance criteria.

Revision 30: Removed reference to obsolete SOP. Updated reference to current DOD/DOE QSM version 5.1 and 3.1. Added section 14.4 for clarification on Acid Screens.

Revision 29: Added statement to clarify the IEC verifications

APPENDIX 1: FREQUENCY OF QUALITY CONTROL ACTIVITIES

(For illustrative purposes only)

Frequency of Quality Control Activities

Method/ Frequency	**SW-846 6020 and 6020B**	**EPA 200.8**	**SW-846 6020A**
ICV	Per calibration	Per calibration	Per calibration
ICB	Per calibration	Per calibration	Per calibration
PQL	Per calibration	Per calibration	Per calibration and at end of each analytical batch.
ICSA	Per calibration	Per calibration	Per calibration
ICSAB	Per calibration; every 12 hours after	Per calibration	Per calibration; every 12 hours after
Linear Range Standard (LRS)	Per calibration, if applicable	Per calibration, if applicable	Per calibration, if applicable
CCV	Every 10 instrument runs	Every 10 instrument runs	Every 10 instrument runs
CCB	Every 10 instrument runs	Every 10 instrument runs	Every 10 instrument runs
Method Blank	5% or per batch	5% or per batch	5% or per batch
LCS – liquid LCS – soil	5% or per batch	5% or per batch	5% or per batch
Matrix Spikes	5% or per request	10% or per request	5% or per request
Sample Duplicates	5% or per request	5% or per request	5% or per request
Serial Dilutions	5% or per request	5% or per request	5% or per request
Matrix Spike Duplicates	5% or per request	10% or per request	5% or per request
Post-Digestion Spikes	5% or per request	10% or per request	5% or per request

APPENDIX 2: ACCEPTANCE LIMITS

Method/ Acceptance Criteria	SW-846 6020	EPA 200.8	DoD QSM Version 5.1	SW-846 6020A	SW-846 6020B
ICV	90% - 110%	90% - 110%	90%-110%	90% - 110%	90% - 110%
ICB	< absolute value of PQL	< absolute value of PQL *	< ± ½ LOQ	< absolute value of PQL	< absolute value of ½ PQL
PQL/CRI (CLP)	70% - 130% advisory limits only	70% - 130% advisory limits only	80%-120% or investigate and recalibrate	70% - 130% or investigate and recalibrate	80%-120% or investigate and recalibrate
ICSA	80-120% for major components; ± 2x PQL for non-spiked	80-120% for major components; ± 2x PQL for non-spiked	80%-120% for major compounds; < ± ½ LOQ for non-spiked	80-120% for major components; ± 2x PQL for non-spiked	80-120% for major components; ± 2x PQL for non-spiked
ICSAB	80%-120%	80%-120% (may be requested)	80%-120%	80%-120%	80%-120%
Linear Range Standard (LRS)	90%-110%, or up to the high calibration standard	90%-110%, or up to the high calibration standard	90%-110%, or up to the high calibration standard	90%-110%, or up to the high calibration standard	90%-110%, or up to the high calibration standard
CCV	90% - 110%	90% - 110%	90%-110%	90% - 110%	90% - 110%
CCB	± PQL	± PQL *	± ½ LOQ	± PQL	± PQL
Method Blank	± PQL	± PQL *	± ½ LOQ, except for Al, Fe, Mg, Ca, Na, and K	± PQL	± ½ PQL
LCS - liquid LCS - soil	80% - 120% current SPC limits	85% - 115% current SPC limits	Use QSM specified limits	80% - 120% current SPC limits	80% - 120% current SPC limits
Matrix Spikes	75% - 125%, when applicable	75% - 125%, when applicable	Use QSM specified limits	75% - 125%, when applicable	75% - 125%, when applicable
Sample Duplicates	0% - 20% when greater than 5X PQL, ± PQL when less than 5X PQL	0% - 20% when greater than 5X PQL, ± PQL when less than 5X PQL	0% - 20% when greater than 5X PQL, ± PQL when less than 5X PQL	0% - 20% when greater than 5X PQL, ± PQL when less than 5X PQL	0% - 20% when greater than 5X PQL, ± PQL when less than 5X PQL
Serial Dilutions	0% - 10% of initial raw value, when applicable	0% - 10% of initial raw value, when applicable	0% - 10% of initial raw value, when applicable (> 50x LOQ)	0% - 10% of initial raw value, when applicable	0%-20% of initial raw value, when applicable
Post-digestion spikes	75%-125%, when applicable	75%-125%, when applicable	80%-120%	80%-120%, when applicable	75%-125%, when applicable
Internal Standards	30%-120%, samples 80%-120% for ICB, ICV, CCV, CCB	60%-125% for all	30%-120%	70%-130%, for all	70%-130%, for all

APPENDIX 2: CONT'D

Method/ Acceptance Criteria	SW-846 6020	EPA 200.8	DoD QSM Version 5.1	SW-846 6020A	SW-846 6020B
Matrix Spike Duplicate	0% - 20%	0% - 20%	0% - 20%	0% - 20%	0% - 20%
Sample Duplicates RER activity	≤ 3%	≤ 3%	≤ 3%	≤ 3%	≤ 3%

* North Carolina requires ½ the PQL

APPENDIX 3: INTERNAL STANDARDS WITH ASSOCIATED ANALYTES/ISOTOPES

IS ^{45}Sc	IS ^{74}Ge	IS ^{115}In	IS ^{175}Lu ^{181}Ta
^{7}Li	^{66}Zn	^{88}Sr	^{133}Cs
^{9}Be	^{67}Zn (calc)	^{90}Zr	^{135}Ba (calc)
^{11}B	^{68}Zn (calc)	^{98}Mo	^{137}Ba
^{23}Na	^{75}As	^{103} Rh	^{178}Hf
^{24}Mg	^{77}Se (calc)	^{107}Ag	^{181} Ta
^{27}Al	^{82}Se	^{111}Cd	^{184}W
^{31}P	^{83}Kr (calc)	^{114}Cd (calc)	^{187}Re
^{39}K		^{120}Sn	^{205}Tl
^{43}Ca		^{121}Sb	^{208}Pb
^{47}Ti		^{123}Sb (calc)	^{209}Bi
^{51}V			^{232}Th
^{52}Cr			^{233}U
^{53}Cr(calc)			^{234}U
^{55}Mn			^{235}U
^{57}Fe			^{236}U
^{59}Co			^{238}U
^{60}Ni			
^{63}Cu(calc)			
^{65}Cu			

*(calc) – isotope used in calculations

APPENDIX 4: RADIOCHEMISTRY CONVERSION CALCULATIONS FOR URANIUM ISOTOPES

Conversion for liquids (µg/L x CF = pCi/L)
^{233}U (µg/L to pCi/L) = 9640.6
^{234}U (µg/L to pCi/L) = 6224.9
^{235}U (µg/L to pCi/L) = 2.1615
^{236}U (µg/L to pCi/L) = 64.698
^{238}U (µg/L to pCi/L) = 0.33627

Conversion for solids (mg/kg x CF = pCi/g)
^{233}U (mg/kg to pCi/g) = 9640.6
^{234}U (mg/kg to pCi/g) = 6224.9
^{235}U (mg/kg to pCi/g) = 2.1615
^{236}U (mg/kg to pCi/g) = 64.698
^{238}U (mg/kg to pCi/g) = 0.33627

The Commonwealth of Massachusetts

Department of Environmental Protection

Division of Environmental Laboratory Sciences
Senator William X. Wall Experiment Station

certifies

M- MA086

ALPHA ANALYTICAL
8 WALKUP DR
WESTBOROUGH, MA 01581-1019

Laboratory Director: **JOSEPH WATKINS**

for the analysis of

POTABLE WATER (MICROBIOLOGY)
POTABLE WATER (CHEMISTRY)
NON POTABLE WATER (CHEMISTRY)
NON POTABLE WATER (MICROBIOLOGY)

pursuant to 310 CMR 42.00

This certificate supersedes all previous Massachusetts certificates issued to this laboratory. The laboratory is regulated by and shall be responsible for being in compliance with Massachusetts regulations at 310 CMR 42.00.

This certificate is valid only when accompanied by the latest dated Certified Parameter List as issued by the Massachusetts D.E.P. Contact the Division of Environmental Laboratory Sciences to verify the current certification status of the laboratory.

Certification is no guarantee of the validity of the data. This certification is subject to unannounced laboratory inspections.

Issued: **01 JUL 2020**

Expires: **30 JUN 2021**

Oscar C. Jacobs

Director, Division of Environmental Laboratory Sciences

COMMONWEALTH OF MASSACHUSETTS
DEPARTMENT OF ENVIRONMENTAL PROTECTION

Certified Parameter List as of: 01 JUL 2020

M-MA086 ALPHA ANALYTICAL
WESTBOROUGH MA

NON POTABLE WATER (MICROBIOLOGY)

Effective Date 30 APR 2020 **Expiration Date** 30 JUN 2021

Analytes		Methods
E. COLI	AMBIENT WATER	SM 9223B-COLILERT
E. COLI	AMBIENT WATER	EPA 1603
ENTEROCOCCI	AMBIENT WATER	ENTEROLERT
ENTEROCOCCI	AMBIENT WATER	EPA 1600
FECAL COLIFORM	WASTEWATER	MPN-SM9221E
FECAL COLIFORM	WASTEWATER	MF-SM9222D
FECAL COLIFORM	WASTEWATER	SM9223B-COLILERT-18
E. COLI	WASTEWATER	SM 9223B-COLILERT
E. COLI	WASTEWATER	EPA 1603
ENTEROCOCCI	WASTEWATER	ENTEROLERT
ENTEROCOCCI	WASTEWATER	EPA 1600

POTABLE WATER (MICROBIOLOGY)

Effective Date 01 JUL 2015 **Expiration Date** 30 JUN 2021

Analytes		Methods
HETEROTROPHIC PLATE COUNT		SM9215B
TOTAL COLIFORM	WATER TREATMENT AND DISTRIBUTION (P/A)	ENZ. SUB. SM9223
FECAL COLIFORM	SOURCE WATER (ENUMERATION)	MF-SM9222D
E. COLI	WATER TREATMENT AND DISTRIBUTION (P/A)	ENZ. SUB. SM9223
TOTAL COLIFORM	SOURCE WATER (ENUMERATION)	SM 9223B-COLILERT
E. COLI	SOURCE WATER (ENUMERATION)	SM 9223B-COLILERT

COMMONWEALTH OF MASSACHUSETTS
DEPARTMENT OF ENVIRONMENTAL PROTECTION

Certified Parameter List as of: 01 JUL 2020

M-MA086 ALPHA ANALYTICAL
WESTBOROUGH MA

NON POTABLE WATER (CHEMISTRY) **Effective Date** **09 AUG 2018** **Expiration Date** **30 JUN 2021**

Analytes	Methods
PH	SM 4500-H-B
SPECIFIC CONDUCTIVITY	EPA 120.1
SPECIFIC CONDUCTIVITY	SM 2510B
TOTAL DISSOLVED SOLIDS	SM 2540C
ALKALINITY, TOTAL	SM 2320B
CHLORIDE	SM 4500-CL-E
CHLORIDE	EPA 300.0
FLUORIDE	SM 4500-F-B,C
SULFATE	SM 4500-SO4-E
SULFATE	EPA 300.0
AMMONIA-N	SM 4500-NH3-B, H
AMMONIA-N	EPA 350.1
AMMONIA-N	LACHAT 10-107-06-1-B
NITRATE-N	SM 4500-NO3-F
NITRATE-N	EPA 300.0
NITRATE-N	EPA 353.2
KJELDAHL-N	SM 4500-NH3-B, H
KJELDAHL-N	EPA 351.1
ORTHOPHOSPHATE	SM 4500-P-E
PHOSPHORUS, TOTAL	SM 4500-P-B,E
CHEMICAL OXYGEN DEMAND	SM 5220D
CHEMICAL OXYGEN DEMAND	EPA 410.4
BIOCHEMICAL OXYGEN DEMAND	SM 5210B
TOTAL ORGANIC CARBON	SM 5310C
CYANIDE, TOTAL	SM 4500-CN-C,E
NON-FILTERABLE RESIDUE	SM 2540D
CHLORINE, TOTAL RESIDUAL	SM 4500-CL-D
OIL AND GREASE	EPA 1664
PHENOLICS, TOTAL	EPA 420.1
VOLATILE HALOCARBONS	EPA 624.1
VOLATILE AROMATICS	EPA 624.1
CHLORDANE	EPA 608.3
TOXAPHENE	EPA 608.3
ALDRIN	EPA 608.3
ALPHA-BHC	EPA 608.3
BETA-BHC	EPA 608.3
GAMMA-BHC	EPA 608.3
DELTA-BHC	EPA 608.3
DIELDRIN	EPA 608.3
DDD	EPA 608.3
DDE	EPA 608.3
DDT	EPA 608.3
ENDOSULFAN I	EPA 608.3

COMMONWEALTH OF MASSACHUSETTS
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M-MA086 ALPHA ANALYTICAL
WESTBOROUGH MA

NON POTABLE WATER (CHEMISTRY) **Effective Date** **09 AUG 2018** **Expiration Date** **30 JUN 2021**

Analytes	Methods
ENDOSULFAN II	EPA 608.3
ENDOSULFAN SULFATE	EPA 608.3
ENDRIN	EPA 608.3
ENDRIN ALDEHYDE	EPA 608.3
HEPTACHLOR	EPA 608.3
HEPTACHLOR EPOXIDE	EPA 608.3
SVOC-ACID EXTRACTABLES	EPA 625.1
SVOC-BASE/NEUTRAL EXTRACTABLES	EPA 625.1
POLYCHLORINATED BIPHENYLS (WATER)	EPA 608.3
POLYCHLORINATED BIPHENYLS (OIL)	EPA 600/4-81-045

POTABLE WATER (CHEMISTRY) **Effective Date** **10 MAY 2019** **Expiration Date** **30 JUN 2021**

Analytes	Methods
NITRATE-N	EPA 300.0
NITRATE-N	EPA 353.2
NITRATE-N	SM 4500-NO3-F
NITRITE-N	EPA 353.2
NITRITE-N	SM 4500-NO3-F
NITRITE-N	SM 4500-NO2-B
FLUORIDE	EPA 300.0
FLUORIDE	SM 4500-F-C
CHLORIDE	EPA 300.0
SULFATE	EPA 300.0
CYANIDE, TOTAL	SM 4500-CN-C,E
TURBIDITY	EPA 180.1
TURBIDITY	SM 2130B
CHLORINE, RESIDUAL FREE	SM 4500-CL-D
ALKALINITY, TOTAL	SM 2320B
TOTAL DISSOLVED SOLIDS	SM 2540C
PH	SM 4500-H-B
TRIHALOMETHANES	EPA 524.2
VOLATILE ORGANIC COMPOUNDS	EPA 524.2
1,2-DIBROMOETHANE	EPA 504.1
1,2-DIBROMO-3-CHLOROPROPANE	EPA 504.1
PERCHLORATE	EPA 332.0

The Commonwealth of Massachusetts

Department of Environmental Protection

Division of Environmental Laboratory Sciences
Senator William X. Wall Experiment Station

certifies

M- MA030

ALPHA ANALYTICAL
320 FORBES BLVD
MANSFIELD, MA 02048-1806

Laboratory Director: **JOHN TRIMBLE**

for the analysis of **POTABLE WATER (CHEMISTRY)**
NON POTABLE WATER (CHEMISTRY)

pursuant to 310 CMR 42.00

This certificate supersedes all previous Massachusetts certificates issued to this laboratory. The laboratory is regulated by and shall be responsible for being in compliance with Massachusetts regulations at 310 CMR 42.00.

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Issued: **01 JUL 2020**

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Oscar C. Jacobs

Director, Division of Environmental Laboratory Sciences

COMMONWEALTH OF MASSACHUSETTS
DEPARTMENT OF ENVIRONMENTAL PROTECTION

Certified Parameter List as of: 01 JUL 2020

M-MA030 ALPHA ANALYTICAL
MANSFIELD MA

NON POTABLE WATER (CHEMISTRY) **Effective Date** **15 AUG 2019** **Expiration Date** **30 JUN 2021**

Analytes	Methods
ALUMINUM	EPA 200.7
ALUMINUM	EPA 200.8
ANTIMONY	EPA 200.7
ANTIMONY	EPA 200.8
ARSENIC	EPA 200.7
ARSENIC	EPA 200.8
BERYLLIUM	EPA 200.7
BERYLLIUM	EPA 200.8
CADMIUM	EPA 200.7
CADMIUM	EPA 200.8
CHROMIUM	EPA 200.7
CHROMIUM	EPA 200.8
COBALT	EPA 200.7
COPPER	EPA 200.7
COPPER	EPA 200.8
IRON	EPA 200.7
IRON	EPA 200.8
LEAD	EPA 200.7
LEAD	EPA 200.8
MANGANESE	EPA 200.7
MANGANESE	EPA 200.8
MERCURY	EPA 245.1
MOLYBDENUM	EPA 200.7
NICKEL	EPA 200.7
NICKEL	EPA 200.8
SELENIUM	EPA 200.7
SELENIUM	EPA 200.8
SILVER	EPA 200.7
SILVER	EPA 200.8
STRONTIUM	EPA 200.7
THALLIUM	EPA 200.7
THALLIUM	EPA 200.8
TITANIUM	EPA 200.7
VANADIUM	EPA 200.7
ZINC	EPA 200.7
ZINC	EPA 200.8
HARDNESS (CACO3), TOTAL	SM 2340B
CALCIUM	EPA 200.7
MAGNESIUM	EPA 200.7
SODIUM	EPA 200.7
SODIUM	EPA 200.8
POTASSIUM	EPA 200.7
POTASSIUM	EPA 200.8

COMMONWEALTH OF MASSACHUSETTS
DEPARTMENT OF ENVIRONMENTAL PROTECTION

Certified Parameter List as of: 01 JUL 2020

M-MA030 ALPHA ANALYTICAL
MANSFIELD MA

POTABLE WATER (CHEMISTRY) **Effective Date** **02 JUL 2018** **Expiration Date** **30 JUN 2021**

Analytes	Methods
ALUMINUM	EPA 200.7
ALUMINUM	EPA 200.8
ANTIMONY	EPA 200.8
ARSENIC	EPA 200.8
BARIUM	EPA 200.7
BARIUM	EPA 200.8
BERYLLIUM	EPA 200.8
CADMIUM	EPA 200.7
CADMIUM	EPA 200.8
CHROMIUM	EPA 200.7
CHROMIUM	EPA 200.8
COPPER	EPA 200.7
COPPER	EPA 200.8
IRON	EPA 200.7
LEAD	EPA 200.8
MANGANESE	EPA 200.7
MANGANESE	EPA 200.8
MERCURY	EPA 245.1
NICKEL	EPA 200.7
NICKEL	EPA 200.8
SELENIUM	EPA 200.8
SILVER	EPA 200.7
SILVER	EPA 200.8
THALLIUM	EPA 200.8
ZINC	EPA 200.7
ZINC	EPA 200.8
SODIUM	EPA 200.7
CALCIUM	EPA 200.7
1,4-DIOXANE	EPA 522

Inductively Coupled Plasma - Atomic Emission Spectrometry

Reference Method No.: **Method 6010D** SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update V, July, 2014.

SM 2340B, Hardness by Calculation, Standard Methods for the Examination of Water and Wastewater, APHA-AWWA-WPCF, 21st Edition, 1997.

1. Scope and Application

Matrices: Digestates from all matrices.

Definitions: See Alpha Laboratories Quality Manual Appendix A

Inductively coupled plasma-atomic emission spectrometry (ICP-AES) determines trace elements, including metals, in solution. The method is applicable to all of the elements listed in Table 1. All matrices, excluding filtered groundwater samples but including ground water, aqueous samples, TCLP and EP extracts, industrial and organic wastes, soils, sludge, sediments, and other solid wastes, require digestion prior to analysis. Groundwater samples that have been prefiltered and acidified will not need acid digestion unless chemical interferences are suspected. Samples which are not digested are matrix matched with the standards. Refer to Metals Preparation SOPs for the appropriate digestion procedures.

Table 1 lists the elements for which this method is applicable. Detection limits, sensitivity, and the optimum and linear concentration ranges of the elements can vary with the wavelength, spectrometer, matrix and operating conditions. Table 1 lists the recommended analytical wavelengths for the elements in clean aqueous matrices. Table 3 lists the Reported Detection Limits. The reported detection limit data may be used to estimate instrument and method performance for other sample matrices. Elements other than those listed in Table 1 may be analyzed by this method if performance at the concentration levels of interest (see Section 9) is demonstrated.

Users of the method should state the data quality objectives prior to analysis and must document and have on file the required initial demonstration performance data described in the following sections prior to using the method for analysis.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is made by one of the following laboratory personnel before performing the modification: Area Supervisor, Metals Manager, Laboratory Services Manager, Laboratory Director, or Quality Assurance Officer.

Use of this method is restricted to spectroscopists who are knowledgeable in the correction of spectral, chemical, and physical interferences described in this method. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

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2. Summary of Method

Prior to analysis, samples must be solubilized or digested using appropriate Sample Preparation Methods. When analyzing groundwater samples for dissolved constituents, acid digestion is not necessary if the samples are filtered and acid preserved prior to analysis.

This method describes multielemental determinations by ICP-AES using sequential or simultaneous optical systems and axial or radial viewing of the plasma. The instrument measures characteristic emission spectra by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific emission spectra are produced by a radio-frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the emission lines are monitored by photosensitive devices. Background correction is required for trace element determination. Background must be measured adjacent to analyte lines on samples during analysis. The position selected for the background-intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. In one mode of analysis the position used must be as free as possible from spectral interference and must reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. The possibility of additional interferences named in Section 4.0 must also be recognized and appropriate corrections made; tests for their presence are described in Section 9.7. Alternatively, users may choose multivariate calibration methods. In this case, point selections for background correction are superfluous since whole spectral regions are processed.

This SOP includes the manual calculations for Total Hardness and Calcium Hardness, according to SM 2340B.

2.1 Method Modifications from Reference

None.

3. Reporting Limits

Refer to Table 3 for method Reporting Limits.

4. Interferences

4.1 Spectral

Spectral interferences are caused by background emission from continuous or recombination phenomena, stray light from the line emission of high concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra.

4.1.1 Background emission and stray light can usually be compensated for by subtracting the background emission determined by measurements adjacent to the analyte wavelength peak. Spectral scans of samples or single element solutions in the analyte regions may indicate when alternate wavelengths are desirable because of severe spectral interference. These scans will also show whether the most appropriate estimate of the background emission is provided by an interpolation from measurements on both sides of the wavelength peak or by measured emission on only one side. The locations selected for the measurement of background intensity will be determined by the complexity of the spectrum adjacent to the wavelength peak. The locations used for

routine measurement must be free of off-line spectral interference (interelement or molecular) or adequately corrected to reflect the same change in background intensity as occurs at the wavelength peak. For multivariate methods using whole spectral regions, background scans must be included in the correction algorithm. Off-line spectral interferences are handled by including spectra on interfering species in the algorithm.

4.1.2 To determine the appropriate location for off-line background correction, the user must scan the area on either side adjacent to the wavelength and record the apparent emission intensity from all other method analytes. This spectral information must be documented and kept on file. The location selected for background correction must be either free of off-line interelement spectral interference or a computer routine must be used for automatic correction on all determinations. If a wavelength other than the recommended wavelength is used, the analyst must determine and document both the overlapping and nearby spectral interference effects from all method analytes and common elements and provide for their automatic correction on all analyses. Tests to determine spectral interference must be done using analyte concentrations that will adequately describe the interference. Normally, 100 mg/L single element solutions are sufficient; however, for analytes such as iron that may be found at high concentration, a more appropriate test would be to use a concentration near the upper analytical range limit.

4.1.3 Spectral overlaps may be avoided by using an alternate wavelength or can be compensated by equations that correct for interelement contributions. Instruments that use equations for interelement correction require the interfering elements be analyzed at the same time as the element of interest. When operative and uncorrected, interferences will produce false positive determinations and be reported as analyte concentrations. More extensive information on interferant effects at various wavelengths and resolutions is available in reference wavelength tables and books. Users may apply interelement correction equations determined on their instruments with tested concentration ranges to compensate (off line or on line) for the effects of interfering elements. For multivariate methods using whole spectral regions, spectral interferences are handled by including spectra of the interfering elements in the algorithm. The interferences listed are only those that occur between method analytes. Only interferences of a direct overlap nature are listed. These overlaps were observed with a single instrument having a working resolution of 0.035 nm.

4.1.4 When using inter-element correction equations, the interference may be expressed as analyte concentration equivalents (i.e. false analyte concentrations) arising from 100 mg/L of the interference element. For example, assume that As is to be determined (at 193.696 nm) in a sample containing approximately 10 mg/L of Al. 100 mg/L of Al would yield a false signal for As equivalent to approximately 1.3 mg/L. Therefore, the presence of 10 mg/L of Al would result in a false signal for As equivalent to approximately 0.13 mg/L. The user is cautioned that each instrument may exhibit somewhat different levels of interference. The interference effects must be evaluated for each individual instrument since the intensities will vary.

Major known interferences are Fe, Al, Ca, Mg, V, Ni, Cu, and Cr. To minimize any of these interferences, every analyte is analyzed on each instrument at or near its linear range and corrected for these interferences. This is done on an annual basis, and data is kept on file.

4.1.5 Inter-element corrections will vary for the same emission line among instruments because of differences in resolution, as determined by the grating, the entrance and exit slit widths, and by the order of dispersion. Inter-element corrections will also vary depending upon the choice of background correction points. Selecting a background correction point where an interfering emission line may appear must be avoided when practical. Inter-element corrections that constitute a major portion of an emission signal may not yield accurate data. Users must not forget that some samples may contain uncommon elements that could contribute spectral interferences.

4.1.6 The interference effects must be evaluated for each individual instrument whether configured as a sequential or simultaneous instrument. For each instrument, intensities will vary not only with optical resolution but also with operating conditions (such as power, viewing height and argon flow rate). When using the recommended wavelengths, the analyst is required to determine and document for each wavelength the effect from referenced interferences as well as any other suspected interferences that may be specific to the instrument or matrix. The analyst is encouraged to utilize a computer routine for automatic correction on all analyses.

4.1.7 The primary wavelength for each analyte is based upon the instrument manufacturer's recommendations. An alternate wavelength is chosen if there is an indication of elevated background or overlap of another spectral wavelength. The wavelength for each analyte must be as free from interferences as possible.

4.1.8 If the correction routine is operating properly, the determined apparent analyte(s) concentration from analysis of each interference solution must fall within a specific concentration range around the calibration blank. The concentration range is calculated by multiplying the concentration of the interfering element by the value of the correction factor being tested and divided by 10. If after the subtraction of the calibration blank the apparent analyte concentration falls outside of this range in either a positive or negative direction, a change in the correction factor of more than 10% should be suspected. The cause of the change must be determined and corrected and the correction factor updated. The interference check solutions must be analyzed more than once to confirm a change has occurred. Adequate rinse time between solutions and before analysis of the calibration blank will assist in the confirmation.

4.1.9 When inter-element corrections are applied, their accuracy must be verified, daily, by analyzing the spectral interference check solution. The correction factor or multivariate correction matrices tested on a daily basis. All inter-element spectral correction factors or multivariate correction matrices are verified and updated when an instrumentation change, such as in the torch, nebulizer, injector, or plasma conditions occurs. The standard solution must be inspected to ensure that there is no contamination that may be perceived as a spectral interference.

4.1.10 When inter-element corrections are <u>not</u> used, verification of absence of interferences is required.

4.1.10.1 One method is to use a computer software routine for comparing the determinative data to limits, files for notifying the analyst when an interfering element is detected in the sample at a concentration that will produce either an apparent false positive concentration, (i.e., greater than) the analyte

instrument detection limit, or false negative analyte concentration, (i.e., less than the lower control limit of the calibration blank defined for a 99% confidence interval).

4.1.10.2 Another method is to analyze an Interference Check Solution(s) which contains similar concentrations of the major components of the samples (>10 mg/L) on a continuing basis to verify the absence of effects at the wavelengths selected. These data must be kept on file with the sample analysis data. If the check solution confirms an operative interference that is >20% of the analyte concentration, the analyte must be determined using (1) analytical and background correction wavelengths (or spectral regions) free of the interference, (2) by an alternative wavelength, or (3) by another documented test procedure.

4.2 Physical

Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by diluting the sample, using a peristaltic pump, use of an internal standard or by using a high solids nebulizer. Another problem that can occur with high dissolved solids is salt buildup at the tip of the nebulizer, affecting aerosol flow rate and causing instrumental drift. The problem can be controlled by wetting the argon prior to nebulization, using a tip washer, using a high solids nebulizer or diluting the sample. Also, it has been reported that better control of the argon flow rate, especially to the nebulizer, improves instrument performance: this may be accomplished with the use of mass flow controllers. The test described in Section 10.3.4.1 will help determine if a physical interference is present.

4.3 Chemical

Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Normally, these effects are not significant with the ICP technique, but if observed, can be minimized by careful selection of operating conditions (incident power, observation position, and so forth), by buffering of the sample, by matrix matching, and by standard addition procedures. Additionally, if filtered samples are found to have an organic or sulfur like odor they are processed by heating after the addition of the acids to matrix match. Chemical interferences are highly dependent on matrix type and the specific analyte element.

4.4 Memory

Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer and from the build-up of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the element and can be minimized by flushing the system with a rinse blank between samples. The possibility of memory interferences must be recognized within an analytical run and suitable rinse times must be used to reduce them. The rinse times necessary for a particular element must be estimated prior to analysis. This may be achieved by aspirating a standard containing elements at a concentration ten times the usual amount or at the top of the linear dynamic range. The aspiration time for this sample must be the same as a normal sample analysis period, followed by analysis of the rinse blank at designated intervals. The length of time required to reduce analyte signals to within a factor of two of the method detection limit must be noted. Until the required rinse time is established, this method suggests a rinse period of at least 60 seconds between samples and standards. If memory interference is suspected, the sample must be reanalyzed after a rinse

period of sufficient length. Alternate rinse times may be established by the analyst based upon their DQOs.

4.5 Other Interferences

4.5.1 Users are advised that high salt concentrations can cause analyte signal suppressions and confuse interference tests. If the instrument does not display negative values, fortify the interference check solution with the elements of interest at 0.5 to 1 mg/L and measure the added standard concentration accordingly. Concentrations must be within 20% of the true spiked concentration or dilution of the samples will be necessary. In the absence of measurable analyte, overcorrection could go undetected if a negative value is reported as zero.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound must be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples are collected in plastic bottles.

6.2 Sample Preservation

If samples are for soluble metals analysis, filtration must take place prior to preservation with 1:1 HNO3 to a pH < 2. Soluble samples must be held at pH < 2 for at least 24 hours prior to digestion if not preserved at the time of filtration. Samples for total metals analysis are preserved with 1:1 HNO3 to a pH < 2. Samples must be pH <2 for at least 24 hours prior to digestion if not preserved at the time of collection.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

Samples to be analyzed for soluble metals, that have not been filtered, must be filtered and preserved within 24 hours of sample collection.

Preserved samples have a hold time of 6 months, and are stored at ambient temperature.

7. Equipment and Supplies

7.1 Inductively coupled argon plasma emission spectrometer:

- **Thermo Scientific ICAP Duo 6500 (Trace7)**

7.1.1 Computer-controlled emission spectrometer with background correction.

7.1.2 Radio-frequency generator compliant with FCC regulations.

7.1.3 Optional mass flow controller for argon nebulizer gas supply.

7.1.4 Optional peristaltic pump.

7.1.5 Optional Autosampler.

7.1.6 Argon gas supply - high purity.

7.2 Volumetric flasks of suitable precision and accuracy.

7.3 Volumetric pipets of suitable precision and accuracy.

8. Standards and Reagents

Reagent semiconductor and/or trace grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. If the purity of a reagent is in question, analyze for contamination. If the concentration of the contamination is less than the MDL then the reagent is acceptable.

8.1 Hydrochloric acid (conc), HCl. Stored at room temperature in acid resistant cabinet. Expiration date if defined by vendor.

8.2 Hydrochloric acid (1:1), HCl. Add 500 mL concentrated HCl to 400 mL DI water and dilute to 1 liter in an appropriately sized beaker. Stored at room temperature in polypropylene bottle, expiration date if defined by vendor..

8.3 Nitric acid (conc), HNO_3. Stored at room temperature in acid resistant cabinet. Expiration date if defined by vendor.

8.4 Nitric acid (1:1), HNO_3. Add 500 mL concentrated HNO_3 to 400 mL DI water and dilute to 1 liter in an appropriately sized beaker. Stored at room temperature in polypropylene bottle, expiration date if defined by vendor..

8.5 Reagent Water. All references to water in the method refer to reagent water unless otherwise specified. Reagent water will be interference free. Refer to Chapter One for a definition of reagent water.

8.6 Standard stock solutions may be purchased or prepared from ultra- high purity grade chemicals or metals (99.99% pure or greater). All stock standards are ordered through ISO and American Association for Lab Accreditation vendors. All standards are in aqueous solutions and are generally at concentrations of 1000ppm and 10,000ppm.

8.7 Mixed calibration standard solutions

Prepare mixed calibration standard solutions by combining appropriate volumes of the stock solutions in volumetric flasks. Add the appropriate types and volumes of acids so that the standards are matrix matched with the sample digestates. Care must be taken when preparing the mixed standards to ensure that the elements are compatible and stable together. Transfer the mixed standard solutions to HDPE or polypropylene bottles for storage. Fresh mixed standards must be prepared, as needed, with the realization that concentration can change on aging as evidenced by failures in the ICV.

NOTE: If the addition of silver to the recommended acid combination results in an initial precipitation, add 15 mL of water and warm the flask until the solution clears. Cool and dilute to 100 mL with water. For this acid combination, the silver concentration must be limited to 2 mg/L. Silver under these conditions is stable in a tap-water matrix for 30 days. Higher concentrations of silver require additional HCl.
Additionally, sulfur standards are stand-alone single element standards and therefore are not to be combined in a mixed calibration standard solution.

8.8 Blanks

Three types of blanks are required for the analysis for samples. The calibration blank is used in establishing the analytical curve, and the method blank is used to identify possible contamination resulting from varying amounts of the acids used in the sample processing. The rinse blank is used to flush the system between all samples and standards.

8.8.1 The calibration blank is prepared by acidifying reagent water to the same concentrations of the acids found in the standards. Prepare a sufficient quantity to flush the system between standards and samples. The calibration blank will also be used for all initial (ICB) and continuing calibration blank (CCB) determinations (see Sections 10.2 and 10.4). Refer to Section 10.4.1.2 for acceptance criteria and/or corrective actions.

8.8.2 The method blank must contain all of the reagents in the same volumes as used in the processing of the samples. The method blank must be carried through the complete procedure and contain the same acid concentration in the final solution as the sample solution used for analysis. Refer to Section 9.1 for acceptance criteria and/or corrective actions.

8.8.3 The rinse blank consists of HNO_3 (1% or 2%) (v/v) in reagent water. Prepare a sufficient quantity to flush the system between standards and samples.

8.9 The Initial Calibration Verification Standard (ICV) and the Continuing Calibration Verification Standard (CCV)

These ICV is prepared by the analyst by combining compatible elements from a standard source different than that of the calibration standard at a concentration at or near the mid-point of the calibration curve. The CCV is prepared from the same source as the calibration standards and must be at a concentration near the mid-point of the calibration curve.

8.10 Low Level of Quantification, (LLOQ)

The LLOQ is initially verified by the analysis of at least 7 replicate samples, spiked at the LLOQ and processed through all preparation and analysis steps of the method at or below the lowest calibration point. The mean recovery and relative standard deviation of these samples provide an initial statement of precision and accuracy at the LLOQ. In most cases the mean recovery should be +/- 35% of the true value and RSD should be < 20%. In-house limits may be calculated when sufficient data points exist. Monitoring recovery of LLOQ over time is useful for assessing precision and bias.

Ongoing LLOQ verification, at a minimum, is on a quarterly basis to validate quantitation capability at low analyte concentration levels. This verification may be accomplished either with clean control material (e.g., reagent water, method blanks. Optimally, the LLOQ should be less than the desired regulatory action levels based on the stated project-specific requirements.

8.11 Spectral Interference Check Solution

These solutions are prepared to contain known concentrations of interfering elements that will provide an adequate test of the correction factors. Analysts are advised that high salt concentrations can cause analyte signal suppressions and confuse interference tests.

Single element interference checks - At a minimum, single element SIC checks must be performed for the following elements: Aluminum 500mg/L; Boron 50mg/L, Barium, 50mg/L, Calcium 500mg/L; Copper 50mg/L; Iron 200mg/L; Magnesium 500mg/L; Manganese 50mg/L; Molybdenum 20mg/L; Sodium 1000mg/L; Nickel 20mg/L; Selenium 20mg/L; Silicon 200mg/L; Tin 20mg/L; Vanadium 20mg/L; Zinc 20mg/L The absolute value of the concentration observed for any unspiked analyte in the single element SIC checks must be less than two times the analytes' LLOQ.

The concentration of the SIC checks are suggested, but become the highest concentration allowed in a sample analysis, and cannot be higher than the highest established linear range. Samples with concentrations of elements higher than the SIC check must be diluted until the concentration is less than the SIC check solution. Note that reanalysis of a diluted sample is required even if the high concentration element is not required to be reported for the specific sample, since the function of the SIC check is to evaluate spectral interferences on other elements. The single element SIC checks are performed when the instrument is setup and periodically (at least once every 6 months) thereafter.

Mixed element interference check - The mixed element SIC solution is analyzed at least once per day, immediately after the initial calibration. The concentration measured for any target analytes must be less than +/- the LLOQ. If this criterion is not met then sample analysis may not proceed until the problem is corrected, or alternatively the LLOQ may be raised to twice the concentration observed in the SIC solution. The only exceptions are those elements that have been demonstrated to be contaminants in the SIC solutions These may be present up to the concentration documented plus the LLOQ.

Mixed element SIC solution: Aluminum, 500mg/L; Calcium, 500mg/L; Iron, 200mg/L; Magnesium, 500mg/L

8.12 Ongoing Low Level of Quantification, (LLOQ)

Ongoing LLOQ verification, at a minimum, is on a quarterly basis to validate quantitation capability at low analyte concentration levels. This verification may be accomplished either with clean control material (e.g., reagent water, method blanks. Optimally, the LLOQ should be less than the desired regulatory action levels based on the stated project-specific requirements.

8.13 Internal Standard

The internal standard consists of a multi-element solution; each internal standard covers a range of the spectrum (low, middle, or high wavelengths) and the elements within that range.

100 mg/L Ce

20 mg/L Cs

2.0 mg/L Lu

Note: The standard is used to monitor instrument fluctuations including but not limited to nebulization efficiency, plasma variations, environmental temperature changes, peristaltic pump pulsations, etc. Therefore, the solution used to start an analysis calibration cannot be added to or changed out during analysis without requiring subsequent full recalibration.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

Employ a minimum of one method blank per sample batch to determine if contamination or any memory effects are occurring. A method blank is a volume of reagent water carried through the same preparation process as a sample.

The method blank results must be less than ½ of the LLOQ for all analytes of concern. If the results of the method blank exceed the RDL for any analyte, perform re-analysis of a new aliquot of the method blank.

If the results continue to exceed the RDL, proceed as follows:

If all of the samples for the analyte are non-detected, and the method blank is at or above the RDL, no action is required.

If one or more associated samples for that analyte have positive results at or above the RDL, those samples must be considered to be out of control, and are re-digested and reanalyzed.

9.2 Laboratory Control Sample (LCS)

Analyze one LCSW/SRM per sample batch. A LCS/SRM sample is a spiked volume of reagent water that is brought through the entire preparation and analytical process. The LCSW must have a % Recovery of ± 20% within the actual value or within vendor control limits (95% confidence limits) for the solid SRM.

If the LCSW or SRM % Recovery is outside the acceptable limits as stated in Table 2, or outside any vendor control limits, the LCS is rerun once. If upon reanalysis the LCS is still out

of control, the failed analytes are re-prepped and re-analyzed. Otherwise, a nonconformance report form is raised to document the exact problem and this form is then authorized by the QA/QC Director and/or the Laboratory Manager(s).

9.3 Initial Calibration Verification (ICV)

For all analytes and determinations, the laboratory must analyze an ICV (Section 8.9), and a calibration blank (ICB, Section 8.8.1), immediately following daily calibration. The results of the ICV are to agree within 10% of the expected value; if not, re-analyze once, if still failing terminate the analysis, correct the problem, and recalibrate the instrument.

9.4 Continuing Calibration Verification (CCV)

A calibration blank (CCB, Section 8.8.1) and a calibration verification standard (CCV, Section 8.9) must be analyzed after every tenth sample and at the end of the sample run. Analysis of the calibration verification (CCV) must verify that the instrument is within 10% of the calibration with the relative standard deviation < 5% from replicate (minimum of two) integrations.

Immediate corrective action for a failing CCV/CCB includes reanalyzing the failing standard. If the standard passes the second time then the analysis may be continued. The batch sheet is noted. If the standard fails again, instrument maintenance must be performed and the CCV/CCB standard is reanalyzed. If the standard passes, then all samples run after the last passing CCV/CCB pair must be re-analyzed.

If the standard fails after instrument maintenance, the instrument is recalibrated. A new ICV/ICB is performed, and all previous data after the last passing CCV/CCB is reanalyzed.

9.5 Matrix Spike

Analyze matrix spike samples at a frequency of one per matrix batch. The matrix spike is the same solution as used for the LCS (Table 4). A matrix spike sample is a sample brought through the entire sample preparation and analytical process.

9.5.1 The percent recovery is to be calculated as follows:

$$\% \text{ Recovery} = \frac{MS - S}{C} \times 100$$

where:

MS = Matrix Spike value
S = Sample value.
C = Concentration of the Spiking solution.

9.5.2 If the Matrix Spike falls outside of the limits as stated in Table 2, or outside any historical documentation for analytes of interest a post analytical spike is performed for the failed analytes. The same sample from which the MS/MSD aliquots were prepared should be spiked with a post digestion spike at a minimum level of 10 times and a maximum of 100 times the lower limit of quantitation. The acceptable % Recovery of the post analytical spike is 80-120%. A nonconformance is noted in the LIMS and approved in secondary peer review and/or by the Metals Manager.

9.5.3 If the Post Spike fails the dilution test should be performed**.** If the analyte concentration is sufficiently high (minimally, a factor of 25 above the lower limit of quantitation after dilution), an analysis of a 1:5 dilution should agree within ± 20% of the original determination. If not, then a chemical or physical interference effect should be suspected.

9.6 Laboratory Duplicate

A duplicate sample is analyzed once per matrix batch. This sample is brought through the entire sample preparation and analytical process.

9.6.1 The relative percent difference between duplicate determinations is to be calculated as follows:

$$RPD = \frac{|D_1 - D_2|}{(|D_1 + D_2|)/2} \times 100$$

where:

RPD = relative percent difference.
D_1 = first sample value.
D_2 = second sample value (replicate).

9.6.2 If the Duplicate falls outside of the limits as stated in Table 2, or outside any historical documentation and the concentrations of the failing analytes are less than 5x the RL or a matrix interference is found a nonconformance is noted in the LIMS and approved in secondary peer review and/or by the Metals Manager.

9.7 Method-specific Quality Control Samples

9.7.1 Spectra Interference Check Standard

A mixed check solution is analyzed once daily (section 8.11). One solution (SIC) has only elevated concentrations of Fe, Al, Ca, Mg to ensure no interferences occur. The concentrations of the analytes of interest must have an absolute value of the LLOQ. This solution is analyzed at the beginning of the first analytical run of the day.

The high level interferences are not evaluated for recovery. If the SIC fails take corrective action which may include re-evaluation of the inter-element correction values (IECs) after running single element SIC. The instrument calibration routine must then be performed and confirmed by the ICV/ICB pair and the SIC re-analyzed before proceeding with analysis. Otherwise, the nonconformance issue is raised to the Department Supervisor and/or the QA Department.

9.7.2 Internal Standard

The internal standards are added prior to the nebulizer and corrects for intensity differences in the instrument response between the standard's and sample's matrix. They are monitored for any variation in response during the sample analyses and used to ratio the sample response to the internal standard response of the calibration blank. The ratio is applied to compensate for instrument conditions in the plasma or nebulization caused by the matrix. The internal standard is monitored for 50-150% recovery or laboratory generated control ranges difference from the calibration blank IS response to ensure the proper functioning of the internal standard introduction system and matrix interferences. If an injection falls outside of this acceptance range the sample or QC check is rerun once to check for an introduction error.

If a sample continues to fail it's to be run on successive increasing dilutions until the internal standards associated with the elements of interest are within range. If a

QC check fails on the single rerun the analysis is stopped, the root cause investigated, corrected and the instrument re-calibrated/verified. The analysis begins again with all samples that were run after the last acceptable CCV/CCB pair.

9.7.3 LDR Check Solution: A multiple element or single element solution run at a point above the highest calibration standard under the same calibration used to quantify the associated sample data. The LDR check must be within +/-10% of the true value of each element of interest to be considered valid.

9.8 Method Sequence

- Calibration of instrument
- Initial Calibration Verification Standard
- Initial Calibration Blank
- Mixed SIC Solution
- Continuing Calibration Verification Standard
- Continuing Calibration Blank
- 10 samples
- Continuing Calibration Verification Standard
- Continuing Calibration Blank
- 10 Samples
- Continuing Calibration Verification Standard
- Continuing Calibration Blank

10. Procedure

10.1 Equipment Set-up

10.1.1 Sample Preparation

Preliminary treatment of most matrices is necessary because of the complexity and variability of sample matrices. Groundwater samples which have been prefiltered and acidified will not need acid digestion. Samples which are not digested must be matrix matched with the standards.

10.1.2 Instrument Set-Up

Set up the instrument with proper operating parameters established as detailed below. The instrument must be allowed to become thermally stable before beginning (usually requiring at least 30 minutes of operation prior to calibration).

Startup Procedures

For iCAP Duo 6500

- Turn on power to the chiller
- Click on ThermoSpec Icon; enter analyst initials in login screen
- Click on Plasma icon to start instrument
- Allow to warm up for 30 minutes
- Enter analytical workgroup number (obtained from LIMS) globally under the Instrument menu by selecting Tools, then Options, then Analyst.

- Click on the Sequence tab and enter the sequence by selecting New Autosampler Table, Add Sequence, Add # of spaces.
- Enter the sample locations and IDs
- Press Run Auto-Session button (►) in menu bar.

10.1.2.1 Specific wavelengths are listed in Table 1. Other wavelengths may be substituted if they can provide the needed sensitivity and are corrected for spectral interference. The instrument and operating conditions utilized for determination must be capable of providing data of acceptable quality to the program and data user.

Operating conditions for axial plasma will vary from 1100 – 1500 watts forward power, 15-19 Liters/min argon coolant flow, 0.5 – 0.7 L/min argon nebulizer flow, 140 – 200 rpm pump rate and a default 1 minute preflush time and 10 second measurement time is recommended for all simultaneous instruments.

10.1.2.2 The plasma operating conditions need to be optimized prior to use of the instrument. This routine is not required on a daily basis, but only when first setting up a new instrument or following a change in operating conditions. The following procedure is recommended or follow manufacturer's recommendations. The purpose of plasma optimization is to provide a maximum signal to background ratio for some of the least sensitive elements in the analytical array. The use of a mass flow controller to regulate the nebulizer gas flow or source optimization software greatly facilitates the procedure.

10.1.2.2.1 The Thermo ICP's typically use a Meinhard Nebulizer. The nebulizer flow for each instrument is 1.0 +/- 0.2 mL/min.

10.1.2.2.2 The 6500 Duo instruments automatically perform a wavelength check at start up without user interaction.

10.1.2.2.3 The instrument operating condition finally selected as being optimum must provide the lowest reliable instrument detection limits and method detection limits.

10.1.2.2.4 If either the instrument operating conditions, such as incident power or nebulizer gas flow rate are changed, or a new torch injector tube with a different orifice internal diameter is installed, the plasma and argon pressures must be reoptimized.

10.1.2.2.5 After completing the initial optimization of operating conditions, but before analyzing samples, the laboratory must establish and initially verify an interelement spectral interference correction routine to be used during sample analysis. A general description concerning spectral interference and the analytical requirements for background correction in particular are discussed in the section on interferences. Criteria for determining an interelement spectral interference is an apparent positive or negative concentration for the analyte that falls within ± ½ LLOQ. The upper control limit is the analyte instrument detection limit. Once established, the entire routine is periodically verified every six months. In between that time, IEC's are done on a need be basis per analyte. Only a portion of the

correction routine must be verified more frequently or on a daily basis. Initial and periodic verification of the routine must be kept on file. Special cases where continual verification is required are described elsewhere.

10.1.2.3 Sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects must be established for each individual analyte line on each particular instrument. All measurements must be within the instrument linear range where the correction equations are valid.

10.1.2.3.1 Method detection limits must be established for all wavelengths utilized for each type of matrix commonly analyzed. The matrix used for the MDL calculation must contain analytes of known concentrations within 3-5 times the anticipated detection limit.

10.1.2.3.2 Determination of limits using reagent water MDLs represent a best case situation and do not represent possible matrix effects of real world samples.

10.1.2.3.3 If additional confirmation is desired, reanalyze the seven replicate aliquots on two more non-consecutive days and again calculate the method detection limit values for each day. An average of the three values for each analyte may provide for a more appropriate estimate.

10.1.2.3.4 The upper limit of the linear dynamic range must be established for each wavelength utilized by determining the signal responses with 10% of the true value of each element from a concentration standard at the upper limit of the range run on the same calibration as required by the sample responses above the calibration range. The range which may be used for the analysis of samples must be no more than 90% of the resulting data. Determined analyte concentrations that are above the upper range limit must be diluted and reanalyzed. The analyst must also be aware that if an inter-element correction from an analyte above the linear range exists, a second analyte where the inter-element correction has been applied may be inaccurately reported.

NOTE: Many of the alkali and alkaline earth metals have non-linear response curves due to ionization and self-absorption effects. These curves may be used if the instrument allows; however the effective range must be checked and the second order curve fit must have a correlation coefficient of 0.995 or better. Third order fits are not acceptable. These curves are much more sensitive to changes in operating conditions than the linear lines and must be checked whenever there have been moderate equipment changes.

10.1.2.4 The analyst must (1) verify that the instrument configuration and operating conditions satisfy the analytical requirements and (2) maintain quality control data confirming instrument performance and analytical results.

10.2 Initial Calibration

Calibrate the instrument according to the instrument manufacturer's recommended procedures, using the typical mixed calibration standard solutions described in Section 8.7. Flush the system with the calibration blank (Section 8.8.1) between each standard or as the manufacturer

recommends. (Use the average intensity of multiple exposures for both standardization and sample analysis to reduce random error.) The calibration curve consists of a calibration blank, RL standard and a high level standard. Calibration curve verification is accomplished through the analysis of the ICV, ICB and SIC standards.

10.3 Equipment Operation and Sample Processing

10.3.1 For all analytes and determinations, the laboratory must analyze an ICV (Section 8.9), and a calibration blank (ICB, Section 8.8.1), immediately following daily calibration.

A calibration blank (CCB, Section 8.8.1) and a calibration verification standard (CCV, Section 8.9) must be analyzed after every tenth sample and at the end of the sample run. Analysis of the calibration verification (CCV) must verify that the instrument is within 10% of the calibration with the relative standard deviation < 5% from replicate (minimum of three) integrations.

If the calibration cannot be verified within the specified limits, the sample analysis must be discontinued, the cause determined and the instrument recalibrated. All samples following the last acceptable ICV/ICB, or CCV/CCB must be reanalyzed. The analysis data for the calibration blank, check standard, and ICV or CCV must be kept on file with the sample analysis data.

10.3.2 Rinse the system with the rinse blank solution (Section 8.8.3) before the analysis of each sample. The suggested default rinse time is one minute. Each ICP instrument may establish a reduction in this rinse time through a suitable demonstration.

10.3.3 Dilute and reanalyze samples that exceed the linear range or use a calibrated alternate, less sensitive line for which quality control data is already established.

10.3.4 If less than acceptable accuracy and precision data are generated a series of tests are performed prior to reporting concentration data for analyte elements. At a minimum, these tests should be performed with each batch of samples prepared/analyzed with corresponding unacceptable data quality results. These tests, as outlined in Sections 10.3.5 and 10.3.6, will ensure that neither positive nor negative interferences are operating on any of the analyte elements to distort the accuracy of the reported values.

10.3.5 **Post Digestion Spike Addition:** If the sample concentrations are insufficient to perform a dilution test a post digestion spike added to a portion of a prepared sample, or its dilution for the elements failing the matrix spike recoveries must be run, recovery limits equal to 75% to 125% of the known spike value. If the spike is not recovered within the specified limits If the post-digestion recovery fails to meet the acceptance criteria, the sample results must be reported as estimated values

10.3.6 **Dilution Test:** If the analyte concentration is sufficiently high (minimally, a factor of 25 above the lower limit of quantitation after dilution), an analysis of a 1:5 dilution must agree within ± 20% of the original determination. Elements that fail the dilution test are reported as estimated values.

10.3.7 **CAUTION:** If spectral overlap is suspected, use of computerized compensation, an alternate wavelength, or comparison with an alternate method is recommended.

10.4 Continuing Calibration

10.4.1 Check calibration with an ICV following the initial calibration (Section 8.9). Verify calibration with the Continuing Calibration Verification (CCV) Standard (Section 8.9) at the end of the initial calibration sequence (ICV, ICB), after every ten samples, and at the

end of an analytical run. Use a calibration blank (Section 8.8.1) immediately following daily calibration, after every 10 samples and at the end of the analytical run.

10.4.1.1 The results of the ICV are to agree within 10% of the expected value, and CCVs are to agree within 10% of the expected value; if not, terminate the analysis, correct the problem, and recalibrate the instrument. Each may be rerun once to confirm or cure the initial failure.

10.4.1.2 The results of the calibration blank should be below ½ of LLOQ or RL (whichever is lower). If not, repeat the analysis and if the failure is repeated terminate the analysis, correct the problem, recalibrate, and reanalyze the previous 10 samples. If the blank is less than 1/10 the concentration of the action level of interest, and no sample is within ten percent of the action limit, analyses need not be rerun and recalibration need not be performed before continuation of the run.

10.4.2 Verify the inter-element and background correction factors at the beginning of each analytical run. Do this by analyzing the SIC (Section 8.10). Results must be less than +/- LLOQ for all non-spiked elements.

10.4.3 When low-level sensitivity is required, a check standard at the requested limit of quantitation is analyzed to confirm the reported detection limit (RDL). This is performed on a project-by-project basis.

10.5 Preventive Maintenance

Whenever instrument maintenance is performed, it is noted in the instrument's Maintenance Logbook.

10.5.1 Daily

Inspect the nebulizer pump tubing from the Autosampler to the Nebulizer. Replace if necessary.

10.5.2 Monthly or as needed

Remove the torch, "shot glass", nebulizer and spray chamber. Clean each with 10% Nitric Acid and rinse with tap water. Coat the inside of the spray chamber and shot glass with concentrated Sulfuric Acid and soak for one hour, then rinse well with DI water. Soak the torch and nebulizer in aqua regia overnight, then rinse with DI water.

10.5.3 Every 6 months

Preventive Maintenance is performed by the Vendor or in-house personnel as follows:

- check the cooling system
- flush/refill the chiller with distilled water and antibacterial conditioner
- clean the instrument to regain intensity
- clean/replace air filters.

11. Data Evaluation, Calculations and Reporting

11.1 If dilutions were performed, the appropriate factors must be applied to sample values. All results must be reported with up to three significant figures.

11.2 Soil samples

Soil samples are calculated as follows:

$$\mathbf{A} = \frac{\text{Sample weight (grams)}}{\text{Final Volume (mL)}}$$

$$\mathbf{B}\ (\text{concentration in mg/Kg}) = \frac{\text{Concentration of analyte (mg/L)}}{\mathbf{A}}$$

11.2.1 Dry weight correction

The LIMS calculates the dry weight correction, however it is calculated as follows:

$$\text{Final concentration in mg/Kg dry weight} = \frac{\mathbf{B}}{\%\ \text{Solids}}$$

11.3 Liquid samples

Liquid samples are calculated as follows:

$$\text{Dilution Factor} = \frac{\text{Final Volume (mL)}}{\text{Sample Volume (mL)}}$$

$$\text{Final concentration in mg/L} = \text{Concentration of analyte (mg/L)} \times \text{Dilution Factor}$$

11.4 Calculations for Hardness

The method for determining hardness is to compute it from the results of separate determinations of Calcium and Magnesium on aqueous samples.

11.4.1 Total Hardness

Total Hardness, mg equivalent $CaCO_3$/L = [2.497 (Ca, mg/L)] + [4.118 (Mg, mg/L)]

11.4.2 Calcium Hardness

Calcium Hardness, mg equivalent $CaCO_3$/L = [2.497 (Ca, mg/L)]

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Also refer to Section 9 for Quality Control and acceptance criteria.

If the SIC is outside of the recovery window, then the standard is reanalyzed. If the standard failure continues, the IECs for the element/elements in question are reviewed and recalculated if necessary.

Immediate corrective action for a failing CCV/CCB includes reanalyzing the failing standard. If the standard passes the second time then the analysis may be continued. The raw data is noted. If the standard fails again, the problem must be found and corrected and the instrument is recalibrated. The ICV/ICB standard is reanalyzed and all previous data that had failed back to the previous passing CCV/CCB is reanalyzed.

The reanalysis procedure outline above is also conducted for a failing LCS or Method Blank; they may be rerun alone on the new or any subsequent passing bracket. The LCS or Method Blank do not qualify a bracket of samples but the batch run itself.

If the Matrix Spike does not meet acceptance criteria, a dilution test is performed. If the levels of the native sample is inadequate (see section 10.3.6) The RPD must be within 20% of the true value of the native sample. If the dilution test fails or the concentrations in the native sample are inadequate, the post spike is analyzed and evaluated (section 10.3.5). If these criteria are met, then the Matrix Spike data is reported, with the post spike narrated on the final report. If the post spike fails the data is reported as estimated.

If sample Duplicates are outside of the acceptance criteria, the analyst examines the sample for homogeneity. If the sample is not homogenous, this is narrated on the final report. Clean, homogenous samples are reanalyzed and if still outside of the acceptance limits, redigested and reanalyzed.

Sample nonconformance regarding a Matrix Spike recovery or a duplicate %RSD is narrated on the final report along with the corrective action(s) taken.

The mixed element SIC solution is analyzed at least once per day, immediately after the initial calibration. The concentration measured for any target analytes must be less than +/- the LLOQ. If this criterion is not met then sample analysis may not proceed until the problem is corrected, instrument is recalibrated, verified with the ICV/ICB and the SIC is then re-analyzed. Alternatively, the LLOQ may be raised to twice the concentration observed in the SIC solution if approved by the Department Manger or QA Department and the level is below the regulatory action limit or project specific requirements. The only exceptions are those elements that have been demonstrated to be contaminants in the SIC solutions These may be present up to the concentration documented plus the LLOQ. If failure continues notify the Department Supervisor or Manager.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/08-05 unless supersede within this SOP. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/08-12 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP #1732 MDL/LOD/LOQ Generation

SOP# 1739 IDC/DOC Generation

SOP# 1728 Waste Management and Disposal

16. Attachments

TABLE 1: Element Wavelengths

TABLE 2: Precision and Accuracy Acceptance Criteria

TABLE 3: Reporting Limits

TABLE 1
ELEMENT WAVELENGTHS

Element	6500 Duo wavelength (nm)
Pb	220.3
Se	196.0
Sb	206.8
As	189.0
Ba	455.4
Be	313.0
Cd	214.4
Co	228.6
Cu	324.7
Cr	267.7
Fe	259.9
Mn	257.6
Mo	202.0
Ni	231.6
Ag	328.0
Tl	190.8
V	292.4
Zn	206.2
Al	396.1
Ca	315.8
Mg	279.0
B	208.9
Si	212.9
Sn	189.9
Sr	421.5
Ti	334.9
Bi	223.0
Na	589.5
K	766.4
S	180.7

TABLE 2
PRECISION AND ACCURACY ACCEPTANCE CRITERIA

	% Recovery LCS		Aqueous % Recovery MS		Soil % Recovery SRM *		Duplicate	
Element	**Lower Control Limit**	**Upper Control Limit**	**Lower Control Limit**	**Upper Control Limit**	**Lower Control Limit**	**Upper Control Limit**	**Aqueous %RPD**	**Soil %RPD**
Aluminum	80	120	75	125	29	171	20	20
Antimony	80	120	75	125	4	196	20	20
Arsenic	80	120	75	125	81	119	20	20
Barium	80	120	75	125	83	118	20	20
Beryllium	80	120	75	125	83	117	20	20
Boron	80	120	75	125	70	129	20	20
Cadmium	80	120	75	125	82	117	20	20
Calcium	80	120	75	125	83	117	20	20
Chromium	80	120	75	125	80	119	20	20
Cobalt	80	120	75	125	83	117	20	20
Copper	80	120	75	125	83	117	20	20
Iron	80	120	75	125	51	150	20	20
Lead	80	120	75	125	80	120	20	20
Magnesium	80	120	75	125	74	126	20	20
Manganese	80	120	75	125	83	117	20	20
Molybdenum	80	120	75	125	81	119	20	20
Nickel	80	120	75	125	82	117	20	20
Potassium	80	120	75	125	74	126	20	20
Sulfur	80	120	75	125	NA	NA	20	20
Selenium	80	120	75	125	80	120	20	20
Silica (SiO_2)	80	120	75	125	NA	NA	20	20
Silver	80	120	75	125	66	134	20	20
Sodium	80	120	75	125	74	127	20	20
Strontium	80	120	75	125	80	120	20	20
Thallium	80	120	75	125	79	120	20	20
Tin	80	120	75	125	69	131	20	20
Titanium	80	120	75	125	82	118	20	20
Vanadium	80	120	75	125	79	121	20	20
Zinc	80	120	75	125	82	119	20	20

** Ranges of the SRM are presented as an example of a typical SRM; actual limits may vary by lot provided by the vendor.

TABLE 3
REPORTING LIMITS

Element	Aqueous (mg/L)	Soil (mg/Kg)
ALUMINUM	0.10	4.0
ANTIMONY	0.05	2.0
ARSENIC	0.005	0.40
BARIUM	0.01	0.40
BERYLLIUM	0.005	0.20
BORON	0.03	1.2
CADMIUM	0.005	0.40
CALCIUM	0.10	4.0
CHROMIUM	0.01	0.40
COBALT	0.02	0.80
COPPER	0.01	0.40
IRON	0.05	2.0
LEAD	0.01	2.0
MAGNESIUM	0.10	4.0
MANGANESE	0.01	0.40
MOLYBDENUM	0.05	2.0
NICKEL	0.025	1.0
POTASSIUM	2.5	100
SULFUR	0.25	10
SELENIUM	0.01	0.80
SILICA	0.50	20
SILVER	0.007	0.40
SODIUM	2.0	80
STRONTIUM	0.01	2.0
THALLIUM	0.02	0.80
TIN	0.05	4.0
TITANIUM	0.01	0.40
VANADIUM	0.01	0.40
ZINC	0.05	2.0

TABLE 4
LCS and Matrix Spike

Analyte	Liquid Concentration (mg/L)	Soil Concentration * (MS spike only) (mg/Kg)
Antimony	0.5	160
Arsenic	0.12	160
Barium	2.00	160
Beryllium	0.05	80
Cadmium	0.051	80
Chromium	0.20	160
Copper	0.25	160
Lead	0.51	160
Nickel	0.50	160
Selenium	0.12	160
Silver	0.05	40
Thallium	0.12	160
Zinc	0.50	160
Iron	1.00	800
Manganese	0.50	160
Calcium	10.0	800
Magnesium	10.0	800
Potassium	10.0	800
Sodium	10.0	800
Silica	1.0	800
Aluminum	2.00	800
Cobalt	0.50	160
Vanadium	0.50	160
Boron	1.0	NA
Molybdenum	1.0	NA
Titanium	1.0	NA

*MS spike of a solid based on 1.25g and a final volume of 50 mL.

Note: Solids LCS is an SRM with certified value provided by the vendor on a lot basis.

Microwave Extraction

Reference Methods: **EPA 3546**, SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update IV, February 2007.

1. Scope and Application

Matrices: Soil, solid, clay, sediment, sludge, solid waste

Definitions: Refer to Alpha Analytical Quality Manual.

Method 3546 is a procedure for extracting water insoluble or slightly water soluble organic compounds from soils, clays, sediments, sludges, and solid wastes. The procedure uses microwave energy to produce elevated temperature and pressure conditions (i.e., 75°C and 50 - 175 psi) in a closed vessel containing the sample and organic solvent(s) to achieve analyte recoveries equivalent to those from Soxhlet extraction (Method 3540C), using less solvent and taking significantly less time than the Soxhlet procedure.

This method is applicable to the extraction of a variety of semivolatile organic compounds, some of which are: substituted phenols, PCBs, and PCDDs/PCDFs. The extracts are analyzed by the appropriate chromatographic procedure(s).

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the Microwave. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

Samples are prepared for extraction following the LEAN one-piece flow methodology and loaded into the extraction vessel. The appropriate solvent (See WI/14825 Microwave Extraction Guide) is added to the vessel and sealed. The extraction vessel containing the sample and solvent is heated to the extraction temperature and extracted for 20- 40 minutes. The extraction mixture is allowed to cool. The vessel is opened and the contents are filtered.

The extract is then concentrated and (as needed) exchanged into a solvent compatible with the cleanup or determinative procedure being employed. Extracts are then vialed and transferred to the Analytical Department.

2.1 Method Modifications from Reference

None.

3. Reporting Limits

Reporting Limit information may be found in the various determinative method SOPs.

4. Interferences

4.1 The most common cause of contamination is from improperly cleaned glassware and lab supplies. All glassware and re-useable extraction equipment (i.e. spatulas, extraction vessels) must be scrupulously cleaned, following the Organic Extraction Glassware Cleaning and Handling SOP and Work instruction 10995, Solvent rinsing/filtering guide.

4.2 Impurities in solvents and reagents may also yield artifacts and/or interferences that may compromise the results of sample analysis. All of these materials must be demonstrated to be free from interferences under the conditions of extract preparation and analysis by preparing method blanks with each extraction batch. The same solvents and reagents are used for the method blank and the associated samples.

4.3 Phthalate esters contaminate many types of products used in the laboratory. Plastic materials must not contact the samples or extracts, as phthalates could be easily leached from the plastic. The exception is in the use of various pre-packed reagent cartridges (Florisil, Silica gel) used in the extract cleanup steps. Each new lot of cartridges is checked for contamination, and is monitored on an on-going basis through the analysis of method blanks.

4.4 Additional specific interference or contamination concerns are addressed in the various analytical SOPs. If necessary, Florisil, Sulfuric Acid, Silica Gel and/or Sulfur cleanup procedures may be employed.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

5.1 The extraction vessels are at elevated temperatures and pressure after the extraction stage. Allow the vessels to cool at room temperature or in the refrigerator before opening.

5.2 During the heating step, some solvent vapors may escape through the vessel liner/seal cover. Follow the manufacturer's directions regarding the vessel assembly and instrument setup to prevent release of solvent vapors to the laboratory atmosphere.

5.3 The instrument contains flammable vapor sensors and should be operated with all covers in place and doors closed to ensure proper operation of the sensors. Follow the manufacturer's directions regarding replacement of extraction vessel seals when frequent vapor leaks are detected.

5.4 Lab coats, safety glasses, and gloves must be worn when handling samples, extracts, standards or solvents and when washing glassware.

5.5 All extract concentration steps must be performed in the extraction hoods. All solvent and extract transfers must also be handled in the hood.

5.6 All expired stock standards, working standards, and spent sample extracts must be placed into the waste bucket in the lab, for future disposal by the Hazardous Waste Manager. The container must be properly labeled with hazard warning labels indicating the container contents.

5.7 Bottles containing flammable solvents must be stored in the flammables cabinet or in the vented cabinets found under the hoods.

5.8 All waste solvents must be transferred to the satellite waste storage containers located in the extraction lab. Separate containers are provided for chlorinated and non-chlorinated solvents and must be used accordingly. Under no circumstances are solvents to be poured down the sink drains.

5.9 Inspect all glassware prior to use. Do not use any glassware that is chipped, cracked or etched if it could present a safety hazard. Damaged glassware is put aside for repair, otherwise discard the piece.

5.10 All Field Samples must be opened and handled in a hood.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Sample collection and preservation requirements are described the in the various analytical method SOPs.

6.2 Sample Preservation

None.

6.3 Sample Shipping

See applicable Sample Custody SOP.

6.4 Sample Handling

All soil samples are stored, refrigerated, in the Custody sample refrigerators. Samples are removed by the analyst immediately prior to sample extraction. The chemist must take custody of the samples by signing them out utilizing the LIMS.

When possible, samples must be homogenized prior to taking the sample aliquot, as described in Section 10.1. After the sample aliquot is removed, the samples are returned to the Sample Bank and placed in the appropriate sample refrigerator. Custody of the samples is transferred utilizing the LIMS.

7. Equipment and Supplies

7.1 Spatulas: Stainless steel.

7.2 Beakers: Stainless Steel 250mL.

7.3 Mortar and Pestle: Capable of reducing particle size to <1mm.

7.4 Kuderna-Danish (K-D) apparatus: Assemble by attaching the Concentrator Tube to the Evaporation Flask using the Plastic Kek clip. Add the Macro Snyder column to the Evaporation Flask. The Micro Snyder Column is attached directly to the Concentrator Tube using the Plastic Kek Clip.

7.4.1 Concentrator tube: 25mL, graduated and calibrated. A ground-glass stopper is used to prevent evaporation of extracts.

7.4.2 Evaporation flask: 500mL. Attach to concentrator tube with plastic kek clips.

7.4.3 Snyder column: Three-ball macro.

7.4.4 Plastic Kek Clips.

7.5 S-EVAP Water Bath with Solvent Collection Capability: Heated. Capable of temperature control (0.1C). Baths are located in a hood. Baths are equipped with chilled water condensers for solvent collection.

7.6 Buchi Concentration System: Base Unit, Chiller, Pump, Block, Controller and 180mL Glass Vessels.

7.7 Boiling Chips: Solvent-extracted, approximately 10/40 mesh (silicon carbide or equivalent).

7.8 Syringe: 1.0mL, 250µL, 25uL, Various sizes for measuring surrogates and spikes.

7.9 Disposable Borosilicate Transfer Pipets.

7.10 Brady labeling system: Thermal label generator.

7.11 Sodium Sulfate Stainless Steel filtering funnels. Add a plug of glass wool to the base of the 104mm stainless steel funnel. Add approximately 20grams of baked sodium sulfate.

7.12 Glass wool: SUPELCO, silane treated.

7.13 Whatman Filter Paper: used for filtering all Pesticide/8081.(Whatman no.1 or equivalent)

7.14 Graduated Cylinder: 25 and 50mL. Class A.

7.15 N-EVAP: Organomation; Various sizes utilized for micro blowdown.

7.16 Aluminum weighing dishes: Electron Microscopy Sciences Cat#70051-01.

7.17 Solvent pump dispenser: Dispensette Organic 5-100ml, 1-5mL.

7.18 Analytical Balance: Capable of weighing to 0.01g

7.19 Multi-position Stirring Plates

7.20 Magnetic Stirring Bars

7.21 250mL Erlenmeyer Flask

7.22 Microwave Accelerated Reaction System (MARS): CEM Corporation. The temperature performance requirements necessitate that the microwave extraction system be capable of sensing the temperature to within ± 2.0°C and automatically adjusting the microwave field output power within 2 seconds of sensing. Temperature sensor is accurate to ± 2°C and adjustable microwave wattage to 1600W. Temperature feedback control provides the primary performance mechanism for the method.

7.22.1 Microwave extraction vessels: 75ml and 100mL (Plus). With plugs and caps. Capable of accommodating 1g to 30g samples. Vessels are transparent to microwave energy, relatively inert to reagents and sample components, and capable of withstanding the temperature and pressure requirements (minimum conditions of 75°C and 200psi) necessary to perform this procedure. Follow the manufacturer's instructions regarding cleaning, handling, and sealing the vessels.

7.22.2 Kevlar sleeves.

7.22.3 Extraction Vessel Turntable: Used to hold and rotate the extraction vessels during extraction.

7.23 Tongue Depressors: Premiere, Wooden, senior size, No. 95-8801.

8. Reagents and Standards

Reagent grade chemicals are used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.1 Reagent Water: All references to water in this method refer to reagent water from Alpha's RO water treatment system.

8.2 Sodium Sulfate (Na_2SO_4): Granular anhydrous; purified by baking at 400ºC for 4 hours in a glass or stainless steel beaker. Store in closed glass containers. All references to sodium sulfate in this method refer to this prepared reagent. Sodium sulfate is also used for filtering.

8.3 Ottwa Sand: VWR catalog #JT3382-5. Purified by baking at 400C for 4 hours in a shallow tray or stainless steel beaker.

8.4 Hexane: Pesticide quality.

8.5 Acetone: Pesticide quality.

8.6 Dichloromethane: Pesticide quality.

8.7 Nitrogen Gas: Reagent grade, used to purge and pressurize the extraction cell and as the concentration gas in the Turbovap II auto-concentrator units and the N-EVAP.

8.8 Spiking Solutions: Commonly used surrogate and LCS/MS spiking solutions used in the extraction steps are listed in WI/14825 Microwave Extraction Guide. Additionally, the WI/14825 Microwave Extraction Guide has a complete listing of all surrogate and LCS/MS spiking solutions. The preparation and expiration dates of these solutions are described in the analytical SOPs.

8.9 Extraction Solvents: This method has been validated using a 1:1 mixture of hexane and acetone, 1:1 mixture of methylene chloride and acetone, or 100% Methylene Chloride for matrices such as soil, glass-fibers, and sand. Other solvent systems may have applicability in microwave extraction, provided that at least one component absorbs microwave energy. The choice of extraction solvent will depend on the analytes of interest and no single solvent is universally applicable to all analyte groups. Whatever solvent system is employed, *including* those specifically listed in this method, the analyst *must* demonstrate adequate performance for the analytes of interest, at the levels of interest. Method 8000 describes procedures that may be used to develop performance criteria for such demonstrations as well as for matrix spike and laboratory control sample results.

Hexane is a water-immiscible solvent and acetone is a water-miscible solvent. The purpose of the water-miscible solvent is to facilitate the extraction of wet solids by allowing the mixed solvent to penetrate the layer of water of the surface of the solid particles. The water immiscible solvent extracts organic compounds with similar polarities. The polarity of acetone may also help extract polar analytes in mixed solvent systems. When 100% Methylene Chloride is used, water is added as a catalyst to absorb microwave energy for method 8270.

8.10 Silica Gel: VWR, Cat# TX4694MAAA. 60 - 200 mesh, chromatography grade. Activated by baking at 140 °C for a minimum of 16 hours in a shallow tray. The silica gel is stored in the oven or desiccator until ready for use. All references to silica gel in this method refer to this prepared reagent.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

Each extraction batch contains various QC samples used to ensure the validity of the sample results. The particular QC elements performed for a given extraction batch are determined by the requirements of the determinative method. The purpose and definition of the QC samples performed are listed below. Specific QC requirements of the analytical methods are listed in WI/14825 Microwave Extraction Guide.

9.1 Blank

Blanks, or method blanks, are measured aliquots of clean matrix (typically sodium sulfate or sand for soil extractions) that are treated identically to the associated samples. Surrogates are added, and the blanks are carried through all stages of the sample extraction, concentration, and cleanup procedures. Blanks serve to ensure that no systematic contamination exists. A blank is extracted with each batch or 20 or less samples.

9.2 Laboratory Control Sample / Laboratory Control Sample Duplicate (LCS/LCSD)

LCS samples are measured aliquots of clean matrix (typically sodium sulfate for soil extractions) that are spiked with a solution containing known amounts of target compounds, in addition to the surrogate solution. The LCS is carried through all stages of the sample extraction, concentration, and cleanup procedures. LCS samples serve as batch specific quantitative checks of the extraction. An LCS is extracted with each batch of 20 or less samples.

An LCSD is performed in addition to an LCS for all Massachusetts Contingency Plan (MCP) methods, as well as in lieu of the MS/MSD or Duplicate when there is insufficient sample volume available. The required solutions are listed in WI/14825 Microwave Extraction Guide.

9.3 Initial Calibration Verification (ICV)

Not Applicable.

9.4 Continuing Calibration Verification (CCV)

Not Applicable.

9.5 Matrix Spike / Matrix Spike Duplicate (MS/MSD)

MS and MSDs are field samples spiked with a known quantity of the target analyte(s). They are prepared by taking additional sample aliquots, and adding the appropriate amounts of surrogate and spiking solutions. The MS/MSD is carried through all stages of the sample extraction, concentration, and cleanup procedures. MS samples serve as a measure of extraction accuracy, by allowing the comparison of the found amount(s) of target analyte(s) with the spiked amount(s). An MS/MSD set also allows for the calculation of the extraction precision, by comparing the results of the two samples. Requirements for MS and MSD are listed in WI/14825 Microwave Extraction Guide.

For samples with a state of origin of New Jersey, a MS and MSD/DUP must be extracted for every twenty samples within a 24hr period.

9.6 Laboratory Duplicate

Duplicates are laboratory selected replicate samples, prepared by taking an additional sample aliquot of a sample. The duplicate is carried through all stages of the sample extraction, concentration, and cleanup procedures. Duplicates serve as a measure of the extraction precision, by comparing the results of the sample and duplicate. Requirements for Duplicates are listed in WI/14825 Microwave Extraction Guide.

9.7 Method-specific Quality Control Samples

9.7.1 Surrogates

Surrogates are compounds specified by the analytical method that are added to all samples and QC samples prior to beginning the extraction process. Surrogate recoveries are calculated and serve as a sample specific quantitative check of the extraction. The various spiking solutions are prepared according to the directions found in the analytical SOPs. The required solutions and volumes used are listed in WI/14825 Microwave Extraction Guide.

9.8 Method Sequence

Refer to Section 10.

10. Procedure

All soil microwave soil extracts follow the LEAN "one-piece flow". All extraction information is recorded by the chemist performing the work in the ELN (Electronic Lab Notebook) see WI/2517. In addition to recording the extraction, concentration, clean-up and vialing information, the analyst must note the matrix "type" along with any observations, deviations from the procedure, or difficulties encountered with the samples in the comment section of the logbook.

10.1 Sample Preparation and Extraction

10.1.1 Soil Samples are scanned and removed from Sample Login Custody to Oprep Custody. Immediately after scanning, the samples batched into the ELN to create the Work Group. Labels are printed and placed on the cap of the soil container. See Work Instruction 2421, Labeling and Generating Work Groups and Batches.

10.1.2 All Glassware is cleaned prior to the Extraction following SOP 1953, Organic Extraction Glassware Cleaning and Handling.

10.1.3 During the extraction process, each soil or sediment sample is visually inspected. If a sample contains a significant amount of free water, the chemist must contact their supervisor or manager to determine if the water is to be considered part of the sample. If the water is not to be homogenized with the solid material, decant and discard the water layer. Record this information in the comments section of the ELN.

Any artifacts (rocks, leaves, sticks, or similar materials) are not typically considered part of the soil sample and are not to be included. If necessary, transfer these artifacts to another container prior to homogenizing the sample. Note the presence of sample artifacts in the ELN. Gummy, fibrous, or oily materials not amenable to grinding must be cut, shredded, or otherwise reduced in size to allow mixing and maximum exposure of the sample surfaces for the extraction.

10.1.4 When possible, homogenize the sample well using a spatula by mixing the contents of the sample container. If this is difficult due to sample matrix, describe the non-homogeneity in the ELN.

10.1.5 The chemist must demonstrate that all equipment used during the extraction process interference-free. This is accomplished through the analysis of a solid matrix (Sodium Sulfate or Ottawa Sand) Method Blank (SB). A Method Blank is extracted with each batch of 20 or less samples.

10.1.6 **MARS Microwave System Operation:** Rinse the reaction vessels, caps, and plugs with 1:1 Acetone/DCM.

10.1.6.1 See Work Instruction WI/2421 for proper labeling procedures and The Microwave Extraction Guide (ID/14825) for one-piece flow operation and appropriate surrogate/spike and solvent to be used. Typically 15-30 grams of the sample and 15grams of sodium sulfate is extracted. Transfer the sample into the reaction vessel from the beaker or weighing tray. Add the appropriate surrogate and spiking solution. Sodium Sulfate is used for the QC substrate for all methods except 8270 and 8270 SIMTECH where Ottawa Sand is used for 8270 QC.

10.1.6.1.1 Note: Samples that are commented to isolate the glassware must be weighed with disposable aluminum weigh dishes (instead of beakers) and wooden tongue depressors (instead of stainless steel spatulas).

10.1.6.2 **For all Methods except 8270:** Add 35mL of 1:1 Hexane:Acetone, 1:1 DCM:Acetone or 100% DCM (or the appropriate extraction solvent) to each

reaction vessel. (Refer to Table 1 or ID#14825). Assure the sample matrix is covered with the extraction solvent prior to microwave extraction.

10.1.6.2.1 Place a plug and a cap on each reaction vessel.

10.1.6.2.2 Place all labeled vessels onto the carousal and place the carousal into the microwave.

10.1.6.2.3 On the front panel of the microwave, select “Use This Method” or UTM from the User Directory. NOTE: UTM-75 is used for the smaller 75mL vessels while UTM-Plus is for the larger 100mL “Plus” vessels.

The following parameters are loaded with “Use this Method” program:

- Stage 1: 1600W @ 100% Power.
- Ramp to 75°C for 10 minutes and hold for 30 minutes.
- This stage is followed by a 5 minute Cool Down step.

10.1.6.2.4 Remove each reaction vessel from the carousel for cooling.

10.1.6.2.5 Once samples have cooled to room temperature, twist the cap off the reaction vessel and filter the sample using The Sample Filtering Guide (ID#25052). The sodium sulfate funnel contains glass wool and approximately 15-30 grams of sodium sulfate. The sample is filtered with the solvent that was used to extract the sample. Due to contamination issues, pesticide samples require filter paper instead of the glass wool. Note- When opening the cap on the Teflon reaction vessel, point away from your body and perform this task in a fume hood.

10.1.6.3 **For 8270 and 8270 SIM:** See Work Instruction WI/2421 for proper labeling procedures and The Microwave Extraction Guide (ID/14825) for one-piece flow operation and appropriate surrogate/spike and solvent to be used. Transfer 30 grams of the sample (a requirement) into the reaction vessel from the beaker or weighing tray. Note: sodium sulfate is **not** mixed with the sample. For QC samples, Ottawa sand is used. Add the appropriate surrogate and spiking solution. After adding the sample to the reaction vessel, using a 1.0mL syringe or 1-5mL pump bottle, add 1.0mL of DI water to the sample.

10.1.6.3.1 Add 40mL of 100% DCM to each reaction vessel. (Refer to Table 1). Additional solvent may be necessary in some cases depending on sample matrix. Assure the sample matrix is covered with the extraction solvent prior to microwave extraction.

10.1.6.3.2 Place a plug and a cap on each reaction vessel.

10.1.6.3.3 Place all labeled vessels onto the carousal.

10.1.6.3.4 On the front panel of the microwave, select “8270” from the User Directory. NOTE: 8270-75 is used for the smaller 75mL vessels while 8270-Plus is for the larger 100mL “Plus” vessels.

The following parameters are loaded with 8270 method:

- Stage 1: 1600W @ 100% Power.
- Ramp to 75°C for 10 minutes and hold for 10 minutes.
- This stage is followed by a 5 minute Cool Down step.

10.1.6.4 Once samples have cooled to room temperature, filter through a sodium sulfate funnel (using DCM only) and into labeled Buchi vessels for DRO, ETPH, and all ABN samples (See Sample Filtering Guide ID#25052). Note- When opening the cap on the Teflon reaction vessel, point away from your body and perform this task in a fume hood.

10.1.6.5 ETPH Analysis: Filter the sample extract through a 20gram sodium sulfate funnel containing glasswool into a 250mL Erlenmeyer flask. Add 3 grams of Deactivated Silica Gel and a stir bar to the extract. Place the sample on a stirring plate and stir for 5 minutes @ 650rpm. Filter the extract through a funnel containing filter paper and collect into a Bucchi vessel for concentration, see Section 10.2.2.

10.1.6.6 Proceed to sample concentration. Note all DRO, ETPH, and ABN products are concentrated using the Buchi Concentration System.

10.2 Sample Concentration Techniques

10.2.1 KD Technique

10.2.1.1 Attach a three-ball Snyder column to the top of the flask. Place the KD apparatus on a hot water bath(SEVAP), (heated to approximately 75°C so that the concentrator tube is partially immersed in the hot water, and so that the entire lower rounded surface of the flask is bathed in hot water vapor. Attach the chilled water condenser to the top of the Synder Column. Adjust the position of the apparatus as required. At the proper rate of distillation, the balls in the column will actively chatter, but the chambers will not flood with solvent.

10.2.1.2 If a Hexane exchange is required (see Table 1), when the sample volume reaches 5-15mL, remove the condenser from the Synder Column and add 20mL of hexane using a graduated cylinder. Add the hexane to the top of the Snyder Column. Allow sample to continue to concentrate to 15-20mL and exchange with another 20mL of hexane. Allow the sample to boil until the intensity decreases (little to no chatter in the Snyder column). Remove the KD concentration setup and move to the 95C bath. Re-attach the condenser and continue with the concentration until the extract volume is reduced to 15mL.

10.2.1.3 Remove the KD apparatus from the water bath and remove the plastic Kek clip. Wipe the joint of the flask and the concentration tube with a dry paper towel to remove any moisture from the outside of the glassware. Allow to cool for 5minutes. Disassemble the KD apparatus. Move the label from the K-D Flask to the concentrator tube (See WI 2421).

10.2.1.4 Place the Concentrator tube on the N-EVAP. Using a disposable pipet, direct the nitrogen over the sample. The N-EVAP is set at 65 °C for samples extracted in Hexane with the nitrogen flow at 5 - 7. Samples remain on N-EVAP until they are reduced to the appropriate volume (see S-Evap/N-Evap Concentration Standard Process WI#18528 for listing of appropriate volumes).

10.2.1.5 The extract is now ready for sample cleanup or vialing (See Table 1). Refer to the relevant Clean-up SOP or proceed with extract vialing (See WI 3827 Extract Vialing Procedure, WI 2426 GC Extract Vialing Procedure and WI 2423 GC/MS Extract Vialing Procedure).

10.2.2 Alternative Concentration Technique: Bucchi

The Bucchi is a self-contained sample concentration and solvent recovery system that utilizes vacuum, heat and oscillation to concentrate samples. The Bucchi will recover >95% of solvent emissions. Refer to Alpha SOP/12838 for Buchi concentration set-up and procedure.

10.3 Preventive Maintenance

10.3.1 Microwave System (MARS):

10.3.1.1 The instrument must be kept clean. Wipe the inside of microwave with soap and water and dry with a cloth as needed.

10.3.1.2 All microwave cells, caps and plugs are to be dish washed and rinsed with solvent prior to use. Additionally, the sleeves must be handled with care. If the edge of the sleeve is dented or chipped it will not hold pressure.

10.3.2 Analytical Balance

10.3.2.1 All balances are checked for accuracy daily and calibrated/serviced every six months by an instrument service company. All service records are kept on file.

10.3.2.2 Keep balances clean. Brush of any sample spills immediately.

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11. Data Evaluation, Calculations and Reporting

None.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

12.1 All Holding time exceedences, improper preservation and Extraction Anomalies are to be reported to a Supervisor or Manager. Non Conformance Reports may need to be issued through the Qualtrax System.

12.2 If the KD Concentrator tube is allowed to run dry, the extract volume is spilled, etc. the sample must be re-extracted.

12.3 Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

12.4 Refer to determinative method SOPs for additional Corrective Action information.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

SOP/2121 Chemical Hygiene Plan

SOP/1732 DL/LOD/LOQ Generation

SOP/1739 DOC Generation

SOP/1728 Waste Management and Disposal SOP

SOP/1953 Organic Extraction Glassware Cleaning and Handling

WI/2421 Labeling and Generating Work Groups and Batches

WI/2517 LIMS Electronic Laboratory Notebook Procedure

WI/3827 Extract Vialing Procedure

WI/10995 Solvent Rinsing/Filtering

WI/14825 Microwave Extraction Guide

SOP/12838 Buchi Concentration

WI/25052 Sample Filtering Guide

WI/18528 S-Evap/N-Evap Concentration Standard Process

16. Attachments

Table 1 – Specific Extraction Conditions for Various Determinative Methods

Table 1

Specific Extraction Conditions for Various Determinative Methods

LIMS Product Code	Extraction Solvent	Exchange Solvent Required	Typical Final Volume	Appropriate Cleanup Technique
8082	1:1 Hexane/Acetone	hexane	5-10 mL	Sulfuric acid/ Sulfur
8081	1:1 DCM/Acetone	hexane	10 mL	Florisil
8270 SIM	DCM	---	1mL	---
8270/8270SIM	DCM	---	1 mL	---
TPH *	DCM	---	1 mL	---
EPH	DCM	hexane	1 mL	Silica gel Fractionation
EPH-TPH**	DCM	hexane	1 mL	---
ETPH	DCM	---	1 mL	Silica gel

***TPH includes the following LIMS Products: TPH-DRO and TPH-DRO-D**

****EPH-TPH includes the following LIMS Products: NJEPH-TPH-CAT1, NJEPH-TPH-CAT2**

(For a full list of products and final volumes see WI/18528 S-Evap/N-Evap Concentration Standard Process or SOP/12838 Buchi Concentration.)

State of New Hampshire

Environmental Laboratory Accreditation Program

Awards

PRIMARY NH ELAP ACCREDITATION

to

ALPHA ANALYTICAL (MANSFIELD) [#2062]

of

MANSFIELD, MA

For the matrix, method and analytes listed on the latest Analyte List in accordance with the provisions on the 2009 TNI Standards and Env-C 300.

Certificate Number: 206219

Effective Date: 9/6/2019

Expiration Date: 9/5/2020

Laboratory ID: 2062

ALPHA AN9/3/2019

Bill Hall
NH ELAP Program Manager

Method accreditation does not imply acceptance for NHDES compliance testing. Laboratory is required to use EPA-approved methods required by regulation. Continuing accreditation status is dependent on successful ongoing participation in the program. Customers may verify the laboratory's current accreditation status by calling (603) 271-2998 or by visiting the NH ELAP website (https://www.des.nh.gov/organization/divisions/water/dwgb/nhelap/index.htm).

NEW HAMPSHIRE ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM

29 Hazen Drive, PO Box 95, Concord, NH 03302 (603) 271-2998

PRIMARY ACCREDITATION ANALYTE LIST

ANALYTE LIST NUMBER: 206219-F

ALPHA ANALYTICAL (MANSFIELD) [#2062]
320 FORBES BLVD

MANSFIELD MA 02048
508-822-9300
Lab ID: 2062

Analyte Code	Analyte Name	Effective Date	Expiration Date	Matrix	Category	Accr. Type
Method Code: 10248405	**Method Ref: EPA TO-13A**		**Revision:**		**Date:**	
5005	NAPHTHALENE	02/21/2017	09/05/2020	AI	VOC	NE
Method Code: 10248449	**Method Ref: EPA TO-13A SIM**		**Revision:**		**Date:**	
5005	NAPHTHALENE	02/21/2017	09/05/2020	AI	VOC	NE
Method Code: 10248803	**Method Ref: EPA TO-15**		**Revision:**		**Date:**	
5160	1,1,1-TRICHLOROETHANE	02/21/2017	09/05/2020	AI	VOC	NE
5110	1,1,2,2-TETRACHLOROETHANE	02/21/2017	09/05/2020	AI	VOC	NE
5185	1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE (FREON 113)	02/21/2017	09/05/2020	AI	VOC	NE
5165	1,1,2-TRICHLOROETHANE	02/21/2017	09/05/2020	AI	VOC	NE
4630	1,1-DICHLOROETHANE	02/21/2017	09/05/2020	AI	VOC	NE
4640	1,1-DICHLOROETHENE	02/21/2017	09/05/2020	AI	VOC	NE
5155	1,2,4-TRICHLOROBENZENE	02/21/2017	09/05/2020	AI	VOC	NE
5210	1,2,4-TRIMETHYLBENZENE	02/21/2017	09/05/2020	AI	VOC	NE
4570	1,2-DIBROMO-3-CHLOROPROPANE (DBCP)	02/21/2017	09/05/2020	AI	VOC	NE
4585	1,2-DIBROMOETHANE (EDB)	02/21/2017	09/05/2020	AI	VOC	NE
4695	1,2-DICHLORO-1,1,2,2-TETRAFLUOROETHANE (FREON 114)	02/05/2020	09/05/2020	AI	VOC	NE
4610	1,2-DICHLOROBENZENE	02/21/2017	09/05/2020	AI	VOC	NE
4635	1,2-DICHLOROETHANE	02/21/2017	09/05/2020	AI	VOC	NE
4655	1,2-DICHLOROPROPANE	02/21/2017	09/05/2020	AI	VOC	NE
5215	1,3,5-TRIMETHYLBENZENE	02/21/2017	09/05/2020	AI	VOC	NE
4615	1,3-DICHLOROBENZENE	02/21/2017	09/05/2020	AI	VOC	NE
4620	1,4-DICHLOROBENZENE	02/21/2017	09/05/2020	AI	VOC	NE
4735	1,4-DIOXANE (1 4-DIETHYLENEOXIDE)	02/21/2017	09/05/2020	AI	VOC	NE
5220	2,2,4-TRIMETHYLPENTANE	02/21/2017	09/05/2020	AI	VOC	NE
4410	2-BUTANONE (METHYL ETHYL KETONE MEK)	02/21/2017	09/05/2020	AI	VOC	NE
4535	2-CHLOROTOLUENE	02/21/2017	09/05/2020	AI	VOC	NE
4860	2-HEXANONE	02/21/2017	09/05/2020	AI	VOC	NE
4542	4-ETHYLTOLUENE	02/21/2017	09/05/2020	AI	VOC	NE
4995	4-METHYL-2-PENTANONE (MIBK)	02/21/2017	09/05/2020	AI	VOC	NE
4300	ACETALDEHYDE	02/21/2017	09/05/2020	AI	VOC	NE
4315	ACETONE	02/21/2017	09/05/2020	AI	VOC	NE
4320	ACETONITRILE	02/21/2017	09/05/2020	AI	VOC	NE
4325	ACROLEIN (PROPENAL)	02/21/2017	09/05/2020	AI	VOC	NE
4340	ACRYLONITRILE	02/21/2017	09/05/2020	AI	VOC	NE
4355	ALLYL CHLORIDE (3-CHLOROPROPENE)	02/21/2017	09/05/2020	AI	VOC	NE

This analyte list supersedes all previously issued analyte lists. Method accreditation does not imply acceptance for NHDES compliance testing. Laboratory is required to use EPA-approved methods required by regulation. Continuing accreditation status is dependent on successful ongoing participation in the program. Customers may verify the laboratory's current accreditation status by calling (603) 271-2998 or by visiting the NH ELAP website (https://www.des.nh.gov/organization/divisions/water/dwgb/nhelap/index.htm).

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4375	BENZENE	02/21/2017	09/05/2020	AI	VOC	NE
5635	BENZYL CHLORIDE	04/18/2017	09/05/2020	AI	VOC	NE
4395	BROMODICHLOROMETHANE	02/21/2017	09/05/2020	AI	VOC	NE
4400	BROMOFORM	02/21/2017	09/05/2020	AI	VOC	NE
4950	BROMOMETHANE (METHYL BROMIDE)	02/21/2017	09/05/2020	AI	VOC	NE
4450	CARBON DISULFIDE	02/21/2017	09/05/2020	AI	VOC	NE
4455	CARBON TETRACHLORIDE	02/21/2017	09/05/2020	AI	VOC	NE
4475	CHLOROBENZENE	02/21/2017	09/05/2020	AI	VOC	NE
4485	CHLOROETHANE	02/21/2017	09/05/2020	AI	VOC	NE
4505	CHLOROFORM	02/21/2017	09/05/2020	AI	VOC	NE
4960	CHLOROMETHANE (METHYL CHLORIDE)	02/21/2017	09/05/2020	AI	VOC	NE
4645	CIS-1 2-DICHLOROETHYLENE	02/21/2017	09/05/2020	AI	VOC	NE
4680	CIS-1,3-DICHLOROPROPENE	02/21/2017	09/05/2020	AI	VOC	NE
4555	CYCLOHEXANE	02/21/2017	09/05/2020	AI	VOC	NE
4575	DIBROMOCHLOROMETHANE	02/21/2017	09/05/2020	AI	VOC	NE
4625	DICHLORODIFLUOROMETHANE	02/21/2017	09/05/2020	AI	VOC	NE
4627	DICHLOROFLUOROMETHANE (FREON 21)	02/21/2017	09/05/2020	AI	VOC	NE
4975	DICHLOROMETHANE (METHYLENE CHLORIDE)	02/21/2017	09/05/2020	AI	VOC	NE
4750	ETHANOL	02/21/2017	09/05/2020	AI	VOC	NE
4755	ETHYL ACETATE	02/21/2017	09/05/2020	AI	VOC	NE
4765	ETHYLBENZENE	02/21/2017	09/05/2020	AI	VOC	NE
4835	HEXACHLOROBUTADIENE	02/21/2017	09/05/2020	AI	VOC	NE
4895	ISOPROPYL ALCOHOL (2-PROPANOL)	02/21/2017	09/05/2020	AI	VOC	NE
4900	ISOPROPYLBENZENE	02/21/2017	09/05/2020	AI	VOC	NE
5240	M/P-XYLENE	02/05/2020	09/05/2020	AI	VOC	NE
4930	METHANOL	02/21/2017	09/05/2020	AI	VOC	NE
4990	METHYL METHACRYLATE	02/21/2017	09/05/2020	AI	VOC	NE
5000	METHYL TERT-BUTYL ETHER (MTBE)	02/21/2017	09/05/2020	AI	VOC	NE
4435	N-BUTYLBENZENE	02/21/2017	09/05/2020	AI	VOC	NE
4825	N-HEPTANE	02/21/2017	09/05/2020	AI	VOC	NE
4855	N-HEXANE (HEXANE)	02/21/2017	09/05/2020	AI	VOC	NE
5090	N-PROPYLBENZENE	02/21/2017	09/05/2020	AI	VOC	NE
5005	NAPHTHALENE	02/21/2017	09/05/2020	AI	VOC	NE
5250	O-XYLENE	02/21/2017	09/05/2020	AI	VOC	NE
4440	SEC-BUTYLBENZENE	02/21/2017	09/05/2020	AI	VOC	NE
5100	STYRENE	02/21/2017	09/05/2020	AI	VOC	NE

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4420	TERT-BUTYL ALCOHOL (TBA)	02/21/2017	09/05/2020	AI	VOC	NE
4445	TERT-BUTYLBENZENE	02/21/2017	09/05/2020	AI	VOC	NE
5115	TETRACHLOROETHENE (PERCHLOROETHYLENE)	02/21/2017	09/05/2020	AI	VOC	NE
5120	TETRAHYDROFURAN (THF)	02/21/2017	09/05/2020	AI	VOC	NE
5140	TOLUENE	02/21/2017	09/05/2020	AI	VOC	NE
4700	TRANS-1,2-DICLOROETHYLENE	02/21/2017	09/05/2020	AI	VOC	NE
4685	TRANS-1,3-DICHLOROPROPENE	02/21/2017	09/05/2020	AI	VOC	NE
5170	TRICHLOROETHENE (TRICHLOROETHYLENE)	02/21/2017	09/05/2020	AI	VOC	NE
5175	TRICHLOROFLUOROMETHANE	02/21/2017	09/05/2020	AI	VOC	NE
5225	VINYL ACETATE	02/21/2017	09/05/2020	AI	VOC	NE
5230	VINYL BROMIDE	02/21/2017	09/05/2020	AI	VOC	NE
5235	VINYL CHLORIDE	02/21/2017	09/05/2020	AI	VOC	NE
Method Code: 10248858	**Method Ref: EPA TO-15 (GC-MS) SIM**		**Revision: 2**		**Date: 1999**	
5160	1,1,1-TRICHLOROETHANE	07/13/2020	09/05/2020	AI	VOC	NE
5110	1,1,2,2-TETRACHLOROETHANE	07/13/2020	09/05/2020	AI	VOC	NE
5185	1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE (FREON 113)	07/13/2020	09/05/2020	AI	VOC	NE
5165	1,1,2-TRICHLOROETHANE	07/13/2020	09/05/2020	AI	VOC	NE
4630	1,1-DICHLOROETHANE	07/13/2020	09/05/2020	AI	VOC	NE
4640	1,1-DICHLOROETHENE	07/13/2020	09/05/2020	AI	VOC	NE
5155	1,2,4-TRICHLOROBENZENE	07/13/2020	09/05/2020	AI	VOC	NE
5210	1,2,4-TRIMETHYLBENZENE	07/13/2020	09/05/2020	AI	VOC	NE
4585	1,2-DIBROMOETHANE (EDB)	07/13/2020	09/05/2020	AI	VOC	NE
4610	1,2-DICHLOROBENZENE	07/13/2020	09/05/2020	AI	VOC	NE
4635	1,2-DICHLOROETHANE	07/13/2020	09/05/2020	AI	VOC	NE
4655	1,2-DICHLOROPROPANE	07/13/2020	09/05/2020	AI	VOC	NE
5215	1,3,5-TRIMETHYLBENZENE	07/13/2020	09/05/2020	AI	VOC	NE
4620	1,4-DICHLOROBENZENE	07/13/2020	09/05/2020	AI	VOC	NE
4735	1,4-DIOXANE (1 4-DIETHYLENEOXIDE)	07/13/2020	09/05/2020	AI	VOC	NE
4410	2-BUTANONE (METHYL ETHYL KETONE MEK)	07/13/2020	09/05/2020	AI	VOC	NE
4995	4-METHYL-2-PENTANONE (MIBK)	07/13/2020	09/05/2020	AI	VOC	NE
4315	ACETONE	07/13/2020	09/05/2020	AI	VOC	NE
4340	ACRYLONITRILE	07/13/2020	09/05/2020	AI	VOC	NE
4375	BENZENE	07/13/2020	09/05/2020	AI	VOC	NE
4395	BROMODICHLOROMETHANE	07/13/2020	09/05/2020	AI	VOC	NE
4400	BROMOFORM	07/13/2020	09/05/2020	AI	VOC	NE
4950	BROMOMETHANE (METHYL BROMIDE)	07/13/2020	09/05/2020	AI	VOC	NE

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4455	CARBON TETRACHLORIDE	07/13/2020	09/05/2020	AI	VOC	NE
4475	CHLOROBENZENE	07/13/2020	09/05/2020	AI	VOC	NE
4485	CHLOROETHANE	07/13/2020	09/05/2020	AI	VOC	NE
4505	CHLOROFORM	07/13/2020	09/05/2020	AI	VOC	NE
4960	CHLOROMETHANE (METHYL CHLORIDE)	07/13/2020	09/05/2020	AI	VOC	NE
4645	CIS-1 2-DICHLOROETHYLENE	07/13/2020	09/05/2020	AI	VOC	NE
4680	CIS-1,3-DICHLOROPROPENE	07/13/2020	09/05/2020	AI	VOC	NE
4575	DIBROMOCHLOROMETHANE	07/13/2020	09/05/2020	AI	VOC	NE
4625	DICHLORODIFLUOROMETHANE	07/13/2020	09/05/2020	AI	VOC	NE
4975	DICHLOROMETHANE (METHYLENE CHLORIDE)	07/13/2020	09/05/2020	AI	VOC	NE
4652	DICHLOROTETRAFLUOROETHANE	07/13/2020	09/05/2020	AI	VOC	NE
4765	ETHYLBENZENE	07/13/2020	09/05/2020	AI	VOC	NE
4835	HEXACHLOROBUTADIENE	07/13/2020	09/05/2020	AI	VOC	NE
4900	ISOPROPYLBENZENE	07/13/2020	09/05/2020	AI	VOC	NE
5000	METHYL TERT-BUTYL ETHER (MTBE)	07/13/2020	09/05/2020	AI	VOC	NE
4435	N-BUTYLBENZENE	07/13/2020	09/05/2020	AI	VOC	NE
5005	NAPHTHALENE	07/13/2020	09/05/2020	AI	VOC	NE
5250	O-XYLENE	07/13/2020	09/05/2020	AI	VOC	NE
5255	P-XYLENE	07/13/2020	09/05/2020	AI	VOC	NE
4440	SEC-BUTYLBENZENE	07/13/2020	09/05/2020	AI	VOC	NE
5100	STYRENE	07/13/2020	09/05/2020	AI	VOC	NE
5115	TETRACHLOROETHENE (PERCHLOROETHYLENE)	07/13/2020	09/05/2020	AI	VOC	NE
5140	TOLUENE	07/13/2020	09/05/2020	AI	VOC	NE
4700	TRANS-1,2-DICLOROETHYLENE	07/13/2020	09/05/2020	AI	VOC	NE
4685	TRANS-1,3-DICHLOROPROPENE	07/13/2020	09/05/2020	AI	VOC	NE
5170	TRICHLOROETHENE (TRICHLOROETHYLENE)	07/13/2020	09/05/2020	AI	VOC	NE
5235	VINYL CHLORIDE	07/13/2020	09/05/2020	AI	VOC	NE
Method Code: 10248405	**Method Ref: EPA TO-13A**		**Revision:**		**Date:**	
5500	ACENAPHTHENE	02/21/2017	09/05/2020	AI	SBN	NE
5505	ACENAPHTHYLENE	02/21/2017	09/05/2020	AI	SBN	NE
5555	ANTHRACENE	02/21/2017	09/05/2020	AI	SBN	NE
5575	BENZO(A)ANTHRACENE	02/21/2017	09/05/2020	AI	SBN	NE
5580	BENZO(A)PYRENE	02/21/2017	09/05/2020	AI	SBN	NE
5585	BENZO(B)FLUORANTHENE	02/21/2017	09/05/2020	AI	SBN	NE
5605	BENZO(E)PYRENE	02/21/2017	09/05/2020	AI	SBN	NE
5590	BENZO(G,H,I)PERYLENE	02/21/2017	09/05/2020	AI	SBN	NE

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5600	BENZO(K)FLUORANTHENE	02/21/2017	09/05/2020	AI	SBN	NE
5855	CHRYSENE	02/21/2017	09/05/2020	AI	SBN	NE
5895	DIBENZO(A,H)ANTHRACENE	02/21/2017	09/05/2020	AI	SBN	NE
6265	FLUORANTHENE	02/21/2017	09/05/2020	AI	SBN	NE
6270	FLUORENE	02/21/2017	09/05/2020	AI	SBN	NE
6315	INDENO(1,2,3-CD)PYRENE	02/21/2017	09/05/2020	AI	SBN	NE
6608	PERYLENE	02/21/2017	09/05/2020	AI	SBN	NE
6615	PHENANTHRENE	02/21/2017	09/05/2020	AI	SBN	NE
6665	PYRENE	02/21/2017	09/05/2020	AI	SBN	NE
Method Code: 10248449	**Method Ref: EPA TO-13A SIM**		**Revision:**		**Date:**	
6380	1-METHYLNAPHTHALENE	02/21/2017	09/05/2020	AI	SBN	NE
6385	2-METHYLNAPHTHALENE	02/21/2017	09/05/2020	AI	SBN	NE
5500	ACENAPHTHENE	02/21/2017	09/05/2020	AI	SBN	NE
5505	ACENAPHTHYLENE	02/21/2017	09/05/2020	AI	SBN	NE
5555	ANTHRACENE	02/21/2017	09/05/2020	AI	SBN	NE
5575	BENZO(A)ANTHRACENE	02/21/2017	09/05/2020	AI	SBN	NE
5580	BENZO(A)PYRENE	02/21/2017	09/05/2020	AI	SBN	NE
5585	BENZO(B)FLUORANTHENE	02/21/2017	09/05/2020	AI	SBN	NE
5605	BENZO(E)PYRENE	02/21/2017	09/05/2020	AI	SBN	NE
5590	BENZO(G,H,I)PERYLENE	02/21/2017	09/05/2020	AI	SBN	NE
5600	BENZO(K)FLUORANTHENE	02/21/2017	09/05/2020	AI	SBN	NE
5855	CHRYSENE	02/21/2017	09/05/2020	AI	SBN	NE
5895	DIBENZO(A,H)ANTHRACENE	02/21/2017	09/05/2020	AI	SBN	NE
6265	FLUORANTHENE	02/21/2017	09/05/2020	AI	SBN	NE
6270	FLUORENE	02/21/2017	09/05/2020	AI	SBN	NE
6315	INDENO(1,2,3-CD)PYRENE	02/21/2017	09/05/2020	AI	SBN	NE
6608	PERYLENE	02/21/2017	09/05/2020	AI	SBN	NE
6615	PHENANTHRENE	02/21/2017	09/05/2020	AI	SBN	NE
6665	PYRENE	02/21/2017	09/05/2020	AI	SBN	NE
Method Code: 10248803	**Method Ref: EPA TO-15**		**Revision:**		**Date:**	
9318	1,3-BUTADIENE	02/21/2017	09/05/2020	AI	SNO	NE
Method Code: 10248858	**Method Ref: EPA TO-15 (GC-MS) SIM**		**Revision: 2**		**Date: 1999**	
9318	1,3-BUTADIENE	07/13/2020	09/05/2020	AI	SNO	NE
Method Code: 10247504	**Method Ref: EPA TO-10A (GC/ECD)**		**Revision:**		**Date:**	
8880	AROCLOR-1016 (PCB-1016)	02/21/2017	09/05/2020	AI	SPC	NE
8885	AROCLOR-1221 (PCB-1221)	02/21/2017	09/05/2020	AI	SPC	NE
8890	AROCLOR-1232 (PCB-1232)	02/21/2017	09/05/2020	AI	SPC	NE

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8895	AROCLOR-1242 (PCB-1242)	02/21/2017	09/05/2020	AI	SPC	NE
8900	AROCLOR-1248 (PCB-1248)	02/21/2017	09/05/2020	AI	SPC	NE
8905	AROCLOR-1254 (PCB-1254)	02/21/2017	09/05/2020	AI	SPC	NE
8910	AROCLOR-1260 (PCB-1260)	02/21/2017	09/05/2020	AI	SPC	NE
8912	AROCLOR-1262 (PCB-1262)	02/21/2017	09/05/2020	AI	SPC	NE
8913	AROCLOR-1268 (PCB-1268)	02/21/2017	09/05/2020	AI	SPC	NE
Method Code: 10249204	**Method Ref: EPA TO-4A**		**Revision: 2**		**Date: 1999**	
8880	AROCLOR-1016 (PCB-1016)	05/13/2019	09/05/2020	AI	SPC	NE
8885	AROCLOR-1221 (PCB-1221)	05/13/2019	09/05/2020	AI	SPC	NE
8890	AROCLOR-1232 (PCB-1232)	05/13/2019	09/05/2020	AI	SPC	NE
8895	AROCLOR-1242 (PCB-1242)	05/13/2019	09/05/2020	AI	SPC	NE
8900	AROCLOR-1248 (PCB-1248)	05/13/2019	09/05/2020	AI	SPC	NE
8905	AROCLOR-1254 (PCB-1254)	05/13/2019	09/05/2020	AI	SPC	NE
8910	AROCLOR-1260 (PCB-1260)	05/13/2019	09/05/2020	AI	SPC	NE
8912	AROCLOR-1262 (PCB-1262)	05/13/2019	09/05/2020	AI	SPC	NE
8913	AROCLOR-1268 (PCB-1268)	05/13/2019	09/05/2020	AI	SPC	NE
Method Code: 10000405	**Method Ref: 40 CFR PART 50 APPENDIX G (LEAD IN SPM)**		**Revision:**		**Date:**	
3907	PARTICULATE LEAD	02/21/2017	09/05/2020	AI	AIR	NE
Method Code: 10248803	**Method Ref: EPA TO-15**		**Revision:**		**Date:**	
4836	PROPENE	02/21/2017	09/05/2020	AI	DG	NE
Method Code: 10156419	**Method Ref: SW-846 6020A**		**Revision:**		**Date: 2007**	
1000	ALUMINUM, TOTAL	02/21/2017	09/05/2020	BT	MET	NE
1005	ANTIMONY, TOTAL	02/21/2017	09/05/2020	BT	MET	NE
1010	ARSENIC, TOTAL	02/21/2017	09/05/2020	BT	MET	NE
1015	BARIUM, TOTAL	02/21/2017	09/05/2020	BT	MET	NE
1020	BERYLLIUM, TOTAL	02/21/2017	09/05/2020	BT	MET	NE
1030	CADMIUM, TOTAL	02/21/2017	09/05/2020	BT	MET	NE
1035	CALCIUM, TOTAL	02/21/2017	09/05/2020	BT	MET	NE
1040	CHROMIUM, TOTAL	02/21/2017	09/05/2020	BT	MET	NE
1050	COBALT, TOTAL	02/21/2017	09/05/2020	BT	MET	NE
1055	COPPER, TOTAL	02/21/2017	09/05/2020	BT	MET	NE
1070	IRON, TOTAL	02/21/2017	09/05/2020	BT	MET	NE
1075	LEAD, TOTAL	02/21/2017	09/05/2020	BT	MET	NE
1085	MAGNESIUM, TOTAL	02/21/2017	09/05/2020	BT	MET	NE
1090	MANGANESE, TOTAL	02/21/2017	09/05/2020	BT	MET	NE
1100	MOLYBDENUM, TOTAL	02/21/2017	09/05/2020	BT	MET	NE
1105	NICKEL, TOTAL	02/21/2017	09/05/2020	BT	MET	NE

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1125	POTASSIUM, TOTAL	02/21/2017	09/05/2020	BT	MET	NE
1140	SELENIUM, TOTAL	02/21/2017	09/05/2020	BT	MET	NE
1150	SILVER, TOTAL	02/21/2017	09/05/2020	BT	MET	NE
1160	STRONTIUM,TOTAL	02/21/2017	09/05/2020	BT	MET	NE
1165	THALLIUM, TOTAL	02/21/2017	09/05/2020	BT	MET	NE
1175	TIN, TOTAL	02/21/2017	09/05/2020	BT	MET	NE
1185	VANADIUM, TOTAL	02/21/2017	09/05/2020	BT	MET	NE
1190	ZINC, TOTAL	02/21/2017	09/05/2020	BT	MET	NE
Method Code: 10156420	**Method Ref: SW-846 6020B**		**Revision:**		**Date: 2014**	
1000	ALUMINUM, TOTAL	06/29/2018	09/05/2020	BT	MET	NE
1005	ANTIMONY, TOTAL	06/29/2018	09/05/2020	BT	MET	NE
1010	ARSENIC, TOTAL	06/29/2018	09/05/2020	BT	MET	NE
1015	BARIUM, TOTAL	06/29/2018	09/05/2020	BT	MET	NE
1020	BERYLLIUM, TOTAL	06/29/2018	09/05/2020	BT	MET	NE
1030	CADMIUM, TOTAL	06/29/2018	09/05/2020	BT	MET	NE
1035	CALCIUM, TOTAL	06/29/2018	09/05/2020	BT	MET	NE
1040	CHROMIUM, TOTAL	06/29/2018	09/05/2020	BT	MET	NE
1050	COBALT, TOTAL	06/29/2018	09/05/2020	BT	MET	NE
1055	COPPER, TOTAL	06/29/2018	09/05/2020	BT	MET	NE
1070	IRON, TOTAL	06/29/2018	09/05/2020	BT	MET	NE
1075	LEAD, TOTAL	06/29/2018	09/05/2020	BT	MET	NE
1085	MAGNESIUM, TOTAL	06/29/2018	09/05/2020	BT	MET	NE
1090	MANGANESE, TOTAL	06/29/2018	09/05/2020	BT	MET	NE
1100	MOLYBDENUM, TOTAL	06/29/2018	09/05/2020	BT	MET	NE
1105	NICKEL, TOTAL	06/29/2018	09/05/2020	BT	MET	NE
1125	POTASSIUM, TOTAL	06/29/2018	09/05/2020	BT	MET	NE
1140	SELENIUM, TOTAL	06/29/2018	09/05/2020	BT	MET	NE
1150	SILVER, TOTAL	06/29/2018	09/05/2020	BT	MET	NE
1160	STRONTIUM,TOTAL	06/29/2018	09/05/2020	BT	MET	NE
1165	THALLIUM, TOTAL	06/29/2018	09/05/2020	BT	MET	NE
1175	TIN, TOTAL	06/29/2018	09/05/2020	BT	MET	NE
1185	VANADIUM, TOTAL	06/29/2018	09/05/2020	BT	MET	NE
1190	ZINC, TOTAL	06/29/2018	09/05/2020	BT	MET	NE
Method Code: 10166457	**Method Ref: SW-846 7471B**		**Revision:**		**Date: 2007**	
1095	MERCURY, TOTAL	12/29/2017	09/05/2020	BT	MET	NE
Method Code: 10167007	**Method Ref: SW-846 7474**		**Revision:**		**Date: 2007**	
1095	MERCURY, TOTAL	02/21/2017	09/05/2020	BT	MET	NE

NEW HAMPSHIRE ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM

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PRIMARY ACCREDITATION ANALYTE LIST

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Method Code: 10136002	**Method Ref: SW-846 3051A**		**Revision:**		**Date: 1998**	
1426	MICROWAVE DIGESTION OF SOLIDS	02/21/2017	09/05/2020	BT	PRE	NE
Method Code: 10138202	**Method Ref: SW-846 3510C**		**Revision: 3**		**Date: 1996**	
1444	SEPERATORY FUNNEL LIQUID-LIQUID EXTRACTION	02/21/2017	09/05/2020	BT	PRE	NE
Method Code: 10142708	**Method Ref: SW-846 3570**		**Revision:**		**Date: 2002**	
1429	MICROSCALE SOLVENT EXTRACTION	02/21/2017	09/05/2020	BT	PRE	NE
Method Code: 10144602	**Method Ref: SW-846 3610B**		**Revision:**		**Date: 1996**	
1411	ALUMINA CLEANUP	02/21/2017	09/05/2020	BT	PRE	NE
Method Code: 10146802	**Method Ref: SW-846 3630C**		**Revision: 3**		**Date: 1996**	
1446	SILICA GEL CLEAN-UP	02/21/2017	09/05/2020	BT	PRE	NE
Method Code: 10147203	**Method Ref: SW-846 3640A**		**Revision: 1**		**Date: 1994**	
1418	GEL-PERMEATION CLEAN-UP	02/21/2017	09/05/2020	BT	PRE	NE
Method Code: 10186002	**Method Ref: SW-846 8270D**		**Revision:**		**Date: 2007**	
5005	NAPHTHALENE	02/21/2017	09/05/2020	BT	VOC	NE
Method Code: 10242509	**Method Ref: SW-846 8270D SIM**		**Revision:**		**Date: 2007**	
5005	NAPHTHALENE	02/21/2017	09/05/2020	BT	VOC	NE
Method Code: 10242543	**Method Ref: SW-846 8270E**		**Revision:**		**Date: 2017**	
5005	NAPHTHALENE	07/13/2020	09/05/2020	BT	VOC	NE
Method Code: 10242565	**Method Ref: SW-846 8270E SIM**		**Revision:**		**Date: 2017**	
5005	NAPHTHALENE	07/13/2020	09/05/2020	BT	VOC	NE
Method Code: 10186002	**Method Ref: SW-846 8270D**		**Revision:**		**Date: 2007**	
6703	1,1'-BIPHENYL (BZ-0)	02/21/2017	09/05/2020	BT	SBN	NE
6380	1-METHYLNAPHTHALENE	02/21/2017	09/05/2020	BT	SBN	NE
5795	2-CHLORONAPHTHALENE	02/21/2017	09/05/2020	BT	SBN	NE
6385	2-METHYLNAPHTHALENE	02/21/2017	09/05/2020	BT	SBN	NE
5500	ACENAPHTHENE	02/21/2017	09/05/2020	BT	SBN	NE
5505	ACENAPHTHYLENE	02/21/2017	09/05/2020	BT	SBN	NE
5555	ANTHRACENE	02/21/2017	09/05/2020	BT	SBN	NE
5570	BENZALDEHYDE	02/21/2017	09/05/2020	BT	SBN	NE
5575	BENZO(A)ANTHRACENE	02/21/2017	09/05/2020	BT	SBN	NE
5580	BENZO(A)PYRENE	02/21/2017	09/05/2020	BT	SBN	NE
5585	BENZO(B)FLUORANTHENE	02/21/2017	09/05/2020	BT	SBN	NE
5590	BENZO(G,H,I)PERYLENE	02/21/2017	09/05/2020	BT	SBN	NE
5600	BENZO(K)FLUORANTHENE	02/21/2017	09/05/2020	BT	SBN	NE
5670	BENZYL BUTYL PHTHALATE	02/21/2017	09/05/2020	BT	SBN	NE
6065	BIS(2-ETHYLHEXYL) PHTHALATE	02/21/2017	09/05/2020	BT	SBN	NE
5855	CHRYSENE	02/21/2017	09/05/2020	BT	SBN	NE
5925	DI-N-BUTYL PHTHALATE	02/21/2017	09/05/2020	BT	SBN	NE

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6200	DI-N-OCTYL PHTHALATE	02/21/2017	09/05/2020	BT	SBN	NE
5895	DIBENZO(A,H)ANTHRACENE	02/21/2017	09/05/2020	BT	SBN	NE
5905	DIBENZOFURAN	02/21/2017	09/05/2020	BT	SBN	NE
6070	DIETHYL PHTHALATE	02/21/2017	09/05/2020	BT	SBN	NE
6135	DIMETHYL PHTHALATE	02/21/2017	09/05/2020	BT	SBN	NE
6265	FLUORANTHENE	02/21/2017	09/05/2020	BT	SBN	NE
6270	FLUORENE	02/21/2017	09/05/2020	BT	SBN	NE
6315	INDENO(1,2,3-CD)PYRENE	02/21/2017	09/05/2020	BT	SBN	NE
6605	PENTACHLOROPHENOL	02/21/2017	09/05/2020	BT	SBN	NE
6615	PHENANTHRENE	02/21/2017	09/05/2020	BT	SBN	NE
6665	PYRENE	02/21/2017	09/05/2020	BT	SBN	NE
Method Code: 10242509	**Method Ref: SW-846 8270D SIM**		**Revision:**		**Date: 2007**	
6385	2-METHYLNAPHTHALENE	02/21/2017	09/05/2020	BT	SBN	NE
5500	ACENAPHTHENE	02/21/2017	09/05/2020	BT	SBN	NE
5505	ACENAPHTHYLENE	02/21/2017	09/05/2020	BT	SBN	NE
5555	ANTHRACENE	02/21/2017	09/05/2020	BT	SBN	NE
5575	BENZO(A)ANTHRACENE	02/21/2017	09/05/2020	BT	SBN	NE
5580	BENZO(A)PYRENE	02/21/2017	09/05/2020	BT	SBN	NE
5585	BENZO(B)FLUORANTHENE	02/21/2017	09/05/2020	BT	SBN	NE
5590	BENZO(G,H,I)PERYLENE	02/21/2017	09/05/2020	BT	SBN	NE
5600	BENZO(K)FLUORANTHENE	02/21/2017	09/05/2020	BT	SBN	NE
5895	DIBENZO(A,H)ANTHRACENE	02/21/2017	09/05/2020	BT	SBN	NE
6265	FLUORANTHENE	02/21/2017	09/05/2020	BT	SBN	NE
6270	FLUORENE	02/21/2017	09/05/2020	BT	SBN	NE
6315	INDENO(1,2,3-CD)PYRENE	02/21/2017	09/05/2020	BT	SBN	NE
6605	PENTACHLOROPHENOL	02/21/2017	09/05/2020	BT	SBN	NE
6615	PHENANTHRENE	02/21/2017	09/05/2020	BT	SBN	NE
6665	PYRENE	02/21/2017	09/05/2020	BT	SBN	NE
Method Code: 10242543	**Method Ref: SW-846 8270E**		**Revision:**		**Date: 2017**	
6703	1,1'-BIPHENYL (BZ-0)	07/13/2020	09/05/2020	BT	SBN	NE
6380	1-METHYLNAPHTHALENE	07/13/2020	09/05/2020	BT	SBN	NE
5795	2-CHLORONAPHTHALENE	07/13/2020	09/05/2020	BT	SBN	NE
6385	2-METHYLNAPHTHALENE	07/13/2020	09/05/2020	BT	SBN	NE
5500	ACENAPHTHENE	07/13/2020	09/05/2020	BT	SBN	NE
5505	ACENAPHTHYLENE	07/13/2020	09/05/2020	BT	SBN	NE
5555	ANTHRACENE	07/13/2020	09/05/2020	BT	SBN	NE

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5570	BENZALDEHYDE	07/13/2020	09/05/2020	BT	SBN	NE
5575	BENZO(A)ANTHRACENE	07/13/2020	09/05/2020	BT	SBN	NE
5580	BENZO(A)PYRENE	07/13/2020	09/05/2020	BT	SBN	NE
5585	BENZO(B)FLUORANTHENE	07/13/2020	09/05/2020	BT	SBN	NE
5590	BENZO(G,H,I)PERYLENE	07/13/2020	09/05/2020	BT	SBN	NE
5600	BENZO(K)FLUORANTHENE	07/13/2020	09/05/2020	BT	SBN	NE
5670	BENZYL BUTYL PHTHALATE	07/13/2020	09/05/2020	BT	SBN	NE
6065	BIS(2-ETHYLHEXYL) PHTHALATE	07/13/2020	09/05/2020	BT	SBN	NE
5855	CHRYSENE	07/13/2020	09/05/2020	BT	SBN	NE
5925	DI-N-BUTYL PHTHALATE	07/13/2020	09/05/2020	BT	SBN	NE
6200	DI-N-OCTYL PHTHALATE	07/13/2020	09/05/2020	BT	SBN	NE
5895	DIBENZO(A,H)ANTHRACENE	07/13/2020	09/05/2020	BT	SBN	NE
5905	DIBENZOFURAN	07/13/2020	09/05/2020	BT	SBN	NE
6070	DIETHYL PHTHALATE	07/13/2020	09/05/2020	BT	SBN	NE
6135	DIMETHYL PHTHALATE	07/13/2020	09/05/2020	BT	SBN	NE
6265	FLUORANTHENE	07/13/2020	09/05/2020	BT	SBN	NE
6270	FLUORENE	07/13/2020	09/05/2020	BT	SBN	NE
6315	INDENO(1,2,3-CD)PYRENE	07/13/2020	09/05/2020	BT	SBN	NE
6605	PENTACHLOROPHENOL	07/13/2020	09/05/2020	BT	SBN	NE
6615	PHENANTHRENE	07/13/2020	09/05/2020	BT	SBN	NE
6665	PYRENE	07/13/2020	09/05/2020	BT	SBN	NE

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6385	2-METHYLNAPHTHALENE	07/13/2020	09/05/2020	BT	SBN	NE
5500	ACENAPHTHENE	07/13/2020	09/05/2020	BT	SBN	NE
5505	ACENAPHTHYLENE	07/13/2020	09/05/2020	BT	SBN	NE
5555	ANTHRACENE	07/13/2020	09/05/2020	BT	SBN	NE
5575	BENZO(A)ANTHRACENE	07/13/2020	09/05/2020	BT	SBN	NE
5580	BENZO(A)PYRENE	07/13/2020	09/05/2020	BT	SBN	NE
5585	BENZO(B)FLUORANTHENE	07/13/2020	09/05/2020	BT	SBN	NE
5590	BENZO(G,H,I)PERYLENE	07/13/2020	09/05/2020	BT	SBN	NE
5600	BENZO(K)FLUORANTHENE	07/13/2020	09/05/2020	BT	SBN	NE
5895	DIBENZO(A,H)ANTHRACENE	07/13/2020	09/05/2020	BT	SBN	NE
6265	FLUORANTHENE	07/13/2020	09/05/2020	BT	SBN	NE
6270	FLUORENE	07/13/2020	09/05/2020	BT	SBN	NE
6315	INDENO(1,2,3-CD)PYRENE	07/13/2020	09/05/2020	BT	SBN	NE
6605	PENTACHLOROPHENOL	07/13/2020	09/05/2020	BT	SBN	NE

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6615	PHENANTHRENE	07/13/2020	09/05/2020	BT	SBN	NE
6665	PYRENE	07/13/2020	09/05/2020	BT	SBN	NE
Method Code: 10187403	**Method Ref: EPA 8290A**		**Revision:**		**Date: 2007**	
9519	1,2,3,4,6,7,8,9-OCTACHLORODIBENZO-P-DIOXIN (OCDD)	05/13/2019	09/05/2020	BT	SNO	NE
9516	1,2,3,4,6,7,8,9-OCTACHLORODIBENZOFURAN (OCDF)	05/13/2019	09/05/2020	BT	SNO	NE
9426	1,2,3,4,6,7,8-HEPTACHLORODIBENZO-P-DIOXIN (1,2,3,4,6,7,8-HPCDD)	05/13/2019	09/05/2020	BT	SNO	NE
9420	1,2,3,4,6,7,8-HEPTACHLORODIBENZOFURAN (1,2,3,4,6,7,8-HPCDF)	05/13/2019	09/05/2020	BT	SNO	NE
9423	1,2,3,4,7,8,9-HEPTACHLORODIBENZOFURAN (1,2,3,4,7,8,9-HPCDF)	05/13/2019	09/05/2020	BT	SNO	NE
9453	1,2,3,4,7,8-HEXACHLORODIBENZO-P-DIOXIN (1,2,3,4,7,8-HXCDD)	05/13/2019	09/05/2020	BT	SNO	NE
9471	1,2,3,4,7,8-HEXACHLORODIBENZOFURAN (1,2,3,4,7,8-HXCDF)	05/13/2019	09/05/2020	BT	SNO	NE
9456	1,2,3,6,7,8-HEXACHLORODIBENZO-P-DIOXIN(1,2,3,6,7,8-HXCDD)	05/13/2019	09/05/2020	BT	SNO	NE
9474	1,2,3,6,7,8-HEXACHLORODIBENZOFURAN (1,2,3,6,7,8-HXCDF)	05/13/2019	09/05/2020	BT	SNO	NE
9459	1,2,3,7,8,9-HEXACHLORODIBENZO-P-DIOXIN (1,2,3,7,8,9-HXCDD)	05/13/2019	09/05/2020	BT	SNO	NE
9477	1,2,3,7,8,9-HEXACHLORODIBENZOFURAN (1,2,3,7,8,9-HXCDF)	05/13/2019	09/05/2020	BT	SNO	NE
9540	1,2,3,7,8-PENTACHLORODIBENZO-P-DIOXIN (1,2,3,7,8-PECDD)	05/13/2019	09/05/2020	BT	SNO	NE
9543	1,2,3,7,8-PENTACHLORODIBENZOFURAN (1,2,3,7,8-PECDF)	05/13/2019	09/05/2020	BT	SNO	NE
9480	2,3,4,6,7,8-HEXACHLORODIBENZOFURAN	05/13/2019	09/05/2020	BT	SNO	NE
9549	2,3,4,7,8-PENTACHLORODIBENZOFURAN	05/13/2019	09/05/2020	BT	SNO	NE
9618	2,3,7,8-TETRACHLORODIBENZO- P-DIOXIN (2,3,7,8-TCDD)	05/13/2019	09/05/2020	BT	SNO	NE
9612	2,3,7,8-TETRACHLORODIBENZOFURAN (TCDF)	05/13/2019	09/05/2020	BT	SNO	NE
9438	TOTAL HEPTACHLORODIBENZO-P-DIOXIN (TOTAL HPCDD)	05/13/2019	09/05/2020	BT	SNO	NE
9444	TOTAL HEPTACHLORODIBENZOFURAN (TOTAL HPCDF)	05/13/2019	09/05/2020	BT	SNO	NE
9468	TOTAL HEXACHLORODIBENZO-P-DIOXIN (TOTAL HXCDD)	05/13/2019	09/05/2020	BT	SNO	NE
9483	TOTAL HEXACHLORODIBENZOFURAN (TOTAL HXCDF)	05/13/2019	09/05/2020	BT	SNO	NE
9555	TOTAL PENTACHLORODIBENZO-P-DIOXIN (TOTAL PECDD)	05/13/2019	09/05/2020	BT	SNO	NE
9552	TOTAL PENTACHLORODIBENZOFURAN (TOTAL PECDF)	05/13/2019	09/05/2020	BT	SNO	NE
9660	TOTAL POLYCHLORINATED DIBENZO-P-DIOXINS (TOTAL PCDDS)	05/13/2019	09/05/2020	BT	SNO	NE
9657	TOTAL POLYCHLORINATED DIBENZOFURANS (TOTAL PCDFS)	05/13/2019	09/05/2020	BT	SNO	NE
9609	TOTAL TETRACHLORODIBENZO-P-DIOXIN (TOTAL TCDD)	05/13/2019	09/05/2020	BT	SNO	NE
9615	TOTAL TETRACHLORODIBENZOFURAN (TOTAL TCDF)	05/13/2019	09/05/2020	BT	SNO	NE

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Method Code: 10179201	**Method Ref: SW-846 8082A**		**Revision:**		**Date: 2000**	
8880	AROCLOR-1016 (PCB-1016)	02/21/2017	09/05/2020	BT	SPC	NE
8885	AROCLOR-1221 (PCB-1221)	02/21/2017	09/05/2020	BT	SPC	NE
8890	AROCLOR-1232 (PCB-1232)	02/21/2017	09/05/2020	BT	SPC	NE
8895	AROCLOR-1242 (PCB-1242)	02/21/2017	09/05/2020	BT	SPC	NE
8900	AROCLOR-1248 (PCB-1248)	02/21/2017	09/05/2020	BT	SPC	NE
8905	AROCLOR-1254 (PCB-1254)	02/21/2017	09/05/2020	BT	SPC	NE
8910	AROCLOR-1260 (PCB-1260)	02/21/2017	09/05/2020	BT	SPC	NE
Method Code: 10178800	**Method Ref: SW-846 8081B**		**Revision:**		**Date: 2000**	
7355	4,4-DDD	02/21/2017	09/05/2020	BT	SPE	NE
7360	4,4-DDE	02/21/2017	09/05/2020	BT	SPE	NE
7365	4,4-DDT	02/21/2017	09/05/2020	BT	SPE	NE
7025	ALDRIN	02/21/2017	09/05/2020	BT	SPE	NE
7110	ALPHA-BHC (ALPHA-HEXACHLOROCYCLOHEXANE)	02/21/2017	09/05/2020	BT	SPE	NE
7240	ALPHA-CHLORDANE	02/21/2017	09/05/2020	BT	SPE	NE
7115	BETA-BHC (BETA-HEXACHLOROCYCLOHEXANE)	02/21/2017	09/05/2020	BT	SPE	NE
7250	CHLORDANE (TECH.)	02/21/2017	09/05/2020	BT	SPE	NE
7105	DELTA-BHC	02/21/2017	09/05/2020	BT	SPE	NE
7470	DIELDRIN	02/21/2017	09/05/2020	BT	SPE	NE
7510	ENDOSULFAN I	02/21/2017	09/05/2020	BT	SPE	NE
7515	ENDOSULFAN II	02/21/2017	09/05/2020	BT	SPE	NE
7520	ENDOSULFAN SULFATE	02/21/2017	09/05/2020	BT	SPE	NE
7540	ENDRIN	02/21/2017	09/05/2020	BT	SPE	NE
7530	ENDRIN ALDEHYDE	02/21/2017	09/05/2020	BT	SPE	NE
7535	ENDRIN KETONE	02/21/2017	09/05/2020	BT	SPE	NE
7120	GAMMA-BHC (LINDANE)	02/21/2017	09/05/2020	BT	SPE	NE
7245	GAMMA-CHLORDANE	02/21/2017	09/05/2020	BT	SPE	NE
7685	HEPTACHLOR	02/21/2017	09/05/2020	BT	SPE	NE
7690	HEPTACHLOR EPOXIDE	02/21/2017	09/05/2020	BT	SPE	NE
7810	METHOXYCHLOR	02/21/2017	09/05/2020	BT	SPE	NE
8250	TOXAPHENE (CHLORINATED CAMPHENE)	02/21/2017	09/05/2020	BT	SPE	NE
Method Code: 10013806	**Method Ref: EPA 200.7**		**Revision: 4.4**		**Date: 1994**	
1000	ALUMINUM, TOTAL	02/21/2017	09/05/2020	D	MET	NE
1015	BARIUM, TOTAL	02/21/2017	09/05/2020	D	MET	NE
1025	BORON, TOTAL	02/21/2017	09/05/2020	D	MET	NE
1030	CADMIUM, TOTAL	02/21/2017	09/05/2020	D	MET	NE
1035	CALCIUM, TOTAL	02/21/2017	09/05/2020	D	MET	NE

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1040	CHROMIUM, TOTAL	02/21/2017	09/05/2020	D	MET	NE
1050	COBALT, TOTAL	02/21/2017	09/05/2020	D	MET	NE
1055	COPPER, TOTAL	02/21/2017	09/05/2020	D	MET	NE
1070	IRON, TOTAL	02/21/2017	09/05/2020	D	MET	NE
1085	MAGNESIUM, TOTAL	02/21/2017	09/05/2020	D	MET	NE
1090	MANGANESE, TOTAL	02/21/2017	09/05/2020	D	MET	NE
1105	NICKEL, TOTAL	02/21/2017	09/05/2020	D	MET	NE
1125	POTASSIUM, TOTAL	02/21/2017	09/05/2020	D	MET	NE
1150	SILVER, TOTAL	02/21/2017	09/05/2020	D	MET	NE
1155	SODIUM, TOTAL	02/21/2017	09/05/2020	D	MET	NE
1160	STRONTIUM,TOTAL	02/21/2017	09/05/2020	D	MET	NE
1175	TIN, TOTAL	02/21/2017	09/05/2020	D	MET	NE
1180	TITANIUM, TOTAL	02/21/2017	09/05/2020	D	MET	NE
1185	VANADIUM, TOTAL	02/21/2017	09/05/2020	D	MET	NE
1190	ZINC, TOTAL	02/21/2017	09/05/2020	D	MET	NE

Method Code: 10014605 **Method Ref: EPA 200.8** **Revision: 5.4** **Date: 1994**

1000	ALUMINUM, TOTAL	02/21/2017	09/05/2020	D	MET	NE
1005	ANTIMONY, TOTAL	02/21/2017	09/05/2020	D	MET	NE
1010	ARSENIC, TOTAL	02/21/2017	09/05/2020	D	MET	NE
1015	BARIUM, TOTAL	02/21/2017	09/05/2020	D	MET	NE
1020	BERYLLIUM, TOTAL	02/21/2017	09/05/2020	D	MET	NE
1030	CADMIUM, TOTAL	02/21/2017	09/05/2020	D	MET	NE
1040	CHROMIUM, TOTAL	02/21/2017	09/05/2020	D	MET	NE
1050	COBALT, TOTAL	02/21/2017	09/05/2020	D	MET	NE
1055	COPPER, TOTAL	02/21/2017	09/05/2020	D	MET	NE
1070	IRON, TOTAL	05/10/2018	09/05/2020	D	MET	NE
1075	LEAD, TOTAL	02/21/2017	09/05/2020	D	MET	NE
1090	MANGANESE, TOTAL	02/21/2017	09/05/2020	D	MET	NE
1105	NICKEL, TOTAL	02/21/2017	09/05/2020	D	MET	NE
1125	POTASSIUM, TOTAL	05/10/2018	09/05/2020	D	MET	NE
1140	SELENIUM, TOTAL	02/21/2017	09/05/2020	D	MET	NE
1150	SILVER, TOTAL	02/21/2017	09/05/2020	D	MET	NE
1155	SODIUM, TOTAL	05/10/2018	09/05/2020	D	MET	NE
1165	THALLIUM, TOTAL	02/21/2017	09/05/2020	D	MET	NE
1185	VANADIUM, TOTAL	02/21/2017	09/05/2020	D	MET	NE
1190	ZINC, TOTAL	02/21/2017	09/05/2020	D	MET	NE

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Method Code: 10036609	**Method Ref: EPA 245.1**		**Revision: 3**		**Date: 1994**	
1095	MERCURY, TOTAL	02/21/2017	09/05/2020	D	MET	NE
Method Code: 10013806	**Method Ref: EPA 200.7**		**Revision: 4.4**		**Date: 1994**	
1550	CALCIUM HARDNESS AS CACO3	02/21/2017	09/05/2020	D	NMI	NE
1755	HARDNESS AS CACO3	02/21/2017	09/05/2020	D	NMI	NE
Method Code: 10088570	**Method Ref: EPA 522**		**Revision: 1**		**Date: 2008**	
4735	1,4-DIOXANE (1 4-DIETHYLENEOXIDE)	12/12/2017	09/05/2020	D	VOC	NE
Method Code: 10091675	**Method Ref: EPA 537**		**Revision: 1.1**		**Date: 2009**	
6905	PERFLUORODECANOIC ACID (PFDA) 335-76-2	06/30/2017	09/05/2020	D	SBN	NE
6903	PERFLUORODODECANOIC ACID (PFDOA) 307-55-1	06/30/2017	09/05/2020	D	SBN	NE
6908	PERFLUOROHEPTANOIC ACID (PFHPA) 375-85-9	06/30/2017	09/05/2020	D	SBN	NE
6913	PERFLUOROHEXANOIC ACID (PFHXA) 307-24-4	06/30/2017	09/05/2020	D	SBN	NE
6906	PERFLUORONONANOIC ACID (PFNA) 375-95-1	06/30/2017	09/05/2020	D	SBN	NE
6912	PERFLUOROOCTANOIC ACID (PFOA) 335-67-1	06/30/2017	09/05/2020	D	SBN	NE
6902	PERFLUOROTETRADECANOIC ACID (PFTDA) 376-06-7	06/30/2017	09/05/2020	D	SBN	NE
6904	PERFLUOROUNDECANOIC ACID (PFUDA) 2058-94-8	06/30/2017	09/05/2020	D	SBN	NE
Method Code: NH0274	**Method Ref: PFAS BY LC/MS/MS ISOTOPE DILUTION**		**Revision:**		**Date:**	
6915	PERFLUOROBUTANOIC ACID (PFBA) 375-22-4	06/30/2017	09/05/2020	D	SBN	NE
6905	PERFLUORODECANOIC ACID (PFDA) 335-76-2	06/30/2017	09/05/2020	D	SBN	NE
6903	PERFLUORODODECANOIC ACID (PFDOA) 307-55-1	06/30/2017	09/05/2020	D	SBN	NE
6908	PERFLUOROHEPTANOIC ACID (PFHPA) 375-85-9	06/30/2017	09/05/2020	D	SBN	NE
6913	PERFLUOROHEXANOIC ACID (PFHXA) 307-24-4	06/30/2017	09/05/2020	D	SBN	NE
6906	PERFLUORONONANOIC ACID (PFNA) 375-95-1	06/30/2017	09/05/2020	D	SBN	NE
6917	PERFLUOROOCTANE SULFONAMIDE (PFOSAM)	06/30/2017	09/05/2020	D	SBN	NE
6912	PERFLUOROOCTANOIC ACID (PFOA) 335-67-1	06/30/2017	09/05/2020	D	SBN	NE
6914	PERFLUOROPENTANOIC ACID (PFPEA) 2706-90-3	06/30/2017	09/05/2020	D	SBN	NE
6902	PERFLUOROTETRADECANOIC ACID (PFTDA) 376-06-7	06/30/2017	09/05/2020	D	SBN	NE
6904	PERFLUOROUNDECANOIC ACID (PFUDA) 2058-94-8	06/30/2017	09/05/2020	D	SBN	NE
Method Code: 10091642	**Method Ref: EPA 537.1**		**Revision: 1**		**Date: 2018**	
4847	N-METHYLPERFLUOROOCTANE SULFONAMIDO ACETIC ACID (NMEFOSAA)	05/10/2019	09/05/2020	D	SNO	NE
6902	PERFLUOROTETRADECANOIC ACID (PFTDA) 376-06-7	05/10/2019	09/05/2020	D	SNO	NE
Method Code: 10091675	**Method Ref: EPA 537**		**Revision: 1.1**		**Date: 2009**	
4846	N-ETHYLPERFLUOROOCTANE SUFONAMIDO ACETIC ACID (NETFOSAA) 2991-50-6	06/30/2017	09/05/2020	D	SNO	NE
4847	N-METHYLPERFLUOROOCTANE SULFONAMIDO ACETIC ACID (NMEFOSAA)	06/30/2017	09/05/2020	D	SNO	NE
9563	PERFLUOROTRIDECANOIC ACID (PFTRDA) 72629-94-8	06/30/2017	09/05/2020	D	SNO	NE

This analyte list supersedes all previously issued analyte lists. Method accreditation does not imply acceptance for NHDES compliance testing. Laboratory is required to use EPA-approved methods required by regulation. Continuing accreditation status is dependent on successful ongoing participation in the program. Customers may verify the laboratory's current accreditation status by calling (603) 271-2998 or by visiting the NH ELAP website (https://www.des.nh.gov/organization/divisions/water/dwgb/nhelap/index.htm).

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Method Code: 10120602	**Method Ref: EPA 1613B**		**Revision:**		**Date: 1994**	
9618	2,3,7,8-TETRACHLORODIBENZO- P-DIOXIN (2,3,7,8-TCDD)	05/10/2019	09/05/2020	D	SNO	NE
Method Code: NH0274	**Method Ref: PFAS BY LC/MS/MS ISOTOPE DILUTION**		**Revision:**		**Date:**	
0136NH	1H,1H,2H,2H-PERFLUORODECANE SULFONATE (8:2) 8:2FTS	06/30/2017	09/05/2020	D	SNO	NE
9562	PERFLUORODECANE SULFONATE (PFDS)	06/30/2017	09/05/2020	D	SNO	NE
9470	PERFLUOROHEPTANE SULFONIC ACID (PFHPS) 375-92-8	06/30/2017	09/05/2020	D	SNO	NE
9563	PERFLUOROTRIDECANOIC ACID (PFTRDA) 72629-94-8	06/30/2017	09/05/2020	D	SNO	NE
Method Code: NH0274	**Method Ref: PFAS BY LC/MS/MS ISOTOPE DILUTION**		**Revision:**		**Date:**	
0137NH	1H,1H,2H,2H-PERFLUOROHEXANE SULFONATE (4:2) 4:2FTS	06/30/2017	09/05/2020	D	OTH	NE
0138NH	1H,1H,2H,2H-PERFLUOROOCTANE SULFONATE (6:2) 6:2FTS	06/30/2017	09/05/2020	D	OTH	NE
0139NH	N-ETHYLPERFLUORO-1-OCTANESULFONAMIDOACETIC ACID (N-ETFOSAA)	06/30/2017	09/05/2020	D	OTH	NE
6955	N-METHYLPERFLUOROOCTANE SULFONAMIDO ACETIC ACID (NMEFOSAA)	06/30/2017	09/05/2020	D	OTH	NE
9464	PERFLUORONONANE SULFONATE (474511-07-4)	06/30/2017	09/05/2020	D	OTH	NE
9467	PERFLUOROPENTANESULFONATE (PFPES 2706-91-4)	06/30/2017	09/05/2020	D	OTH	NE
Method Code: 10091642	**Method Ref: EPA 537.1**		**Revision: 1**		**Date: 2018**	
9490	11-CHLOROEICOSAFLUORO-3-OXAUNDECANE-1-SULFONIC ACID (11-CL-PF3OUDS)	05/10/2019	09/05/2020	D	PFC	NE
6951	4,8-DIOXA-3H-PERFLUORONONANOIC ACID (ADONA) 919005-14-4	05/10/2019	09/05/2020	D	PFC	NE
6952	9-CHLOROHEXADECAFLUORO-3-OXANONANE-1-SULFONIC ACID (9-CL-PF3ONS)	05/10/2019	09/05/2020	D	PFC	NE
9460	HEXAFLUOROPROPYLENEOXIDE DIMER ACID (HFPO-DA)	05/10/2019	09/05/2020	D	PFC	NE
4846	N-ETHYLPERFLUOROOCTANE SUFONAMIDO ACETIC ACID (NETFOSAA) 2991-50-6	05/10/2019	09/05/2020	D	PFC	NE
6918	PERFLUOROBUTANE SULFONIC ACID (PFBS) 375-73-5	05/10/2019	09/05/2020	D	PFC	NE
6905	PERFLUORODECANOIC ACID (PFDA) 335-76-2	05/10/2019	09/05/2020	D	PFC	NE
6903	PERFLUORODODECANOIC ACID (PFDOA) 307-55-1	05/10/2019	09/05/2020	D	PFC	NE
6908	PERFLUOROHEPTANOIC ACID (PFHPA) 375-85-9	05/10/2019	09/05/2020	D	PFC	NE
6927	PERFLUOROHEXANE SULFONIC ACID (PFHXS) 355-46-4	05/10/2019	09/05/2020	D	PFC	NE
6913	PERFLUOROHEXANOIC ACID (PFHXA) 307-24-4	05/10/2019	09/05/2020	D	PFC	NE
6906	PERFLUORONONANOIC ACID (PFNA) 375-95-1	05/10/2019	09/05/2020	D	PFC	NE
6931	PERFLUOROOCTANE SULFONIC ACID (PFOS) 1763-23-1	05/10/2019	09/05/2020	D	PFC	NE
6912	PERFLUOROOCTANOIC ACID (PFOA) 335-67-1	05/10/2019	09/05/2020	D	PFC	NE
9563	PERFLUOROTRIDECANOIC ACID (PFTRDA) 72629-94-8	05/10/2019	09/05/2020	D	PFC	NE
6904	PERFLUOROUNDECANOIC ACID (PFUDA) 2058-94-8	05/10/2019	09/05/2020	D	PFC	NE
Method Code: 10091675	**Method Ref: EPA 537**		**Revision: 1.1**		**Date: 2009**	
6918	PERFLUOROBUTANE SULFONIC ACID (PFBS) 375-73-5	03/25/2019	09/05/2020	D	PFC	NE

This analyte list supersedes all previously issued analyte lists. Method accreditation does not imply acceptance for NHDES compliance testing. Laboratory is required to use EPA-approved methods required by regulation. Continuing accreditation status is dependent on successful ongoing participation in the program. Customers may verify the laboratory's current accreditation status by calling (603) 271-2998 or by visiting the NH ELAP website (https://www.des.nh.gov/organization/divisions/water/dwgb/nhelap/index.htm).

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6927	PERFLUOROHEXANE SULFONIC ACID (PFHXS) 355-46-4	03/25/2019	09/05/2020	D	PFC	NE
6931	PERFLUOROOCTANE SULFONIC ACID (PFOS) 1763-23-1	03/25/2019	09/05/2020	D	PFC	NE
Method Code: NH0274	**Method Ref: PFAS BY LC/MS/MS ISOTOPE DILUTION**		**Revision:**		**Date:**	
6918	PERFLUOROBUTANE SULFONIC ACID (PFBS) 375-73-5	03/25/2019	09/05/2020	D	PFC	NE
6927	PERFLUOROHEXANE SULFONIC ACID (PFHXS) 355-46-4	03/25/2019	09/05/2020	D	PFC	NE
6931	PERFLUOROOCTANE SULFONIC ACID (PFOS) 1763-23-1	03/25/2019	09/05/2020	D	PFC	NE
Method Code: NH0307	**Method Ref: EPA 533**		**Revision:**		**Date:**	
9490	11-CHLOROEICOSAFLUORO-3-OXAUNDECANE-1-SULFONIC ACID (11-CL-PF3OUDS)	03/20/2020	09/05/2020	D	PFC	NE
6948	1H, 1H, 2H, 2H-PERFLUORODECANESULFONIC ACID (8:2 FTS)	03/20/2020	09/05/2020	D	PFC	NE
6946	1H, 1H, 2H, 2H-PERFLUOROHEXANESULFONIC ACID (4:2 FTS)	03/20/2020	09/05/2020	D	PFC	NE
6947	1H, 1H, 2H, 2H-PERFLUOROOCTANESULFONIC ACID (6:2 FTS)	03/20/2020	09/05/2020	D	PFC	NE
6951	4,8-DIOXA-3H-PERFLUORONONANOIC ACID (ADONA) 919005-14-4	03/20/2020	09/05/2020	D	PFC	NE
6952	9-CHLOROHEXADECAFLUORO-3-OXANONANE-1-SULFONIC ACID (9-CL-PF3ONS)	03/20/2020	09/05/2020	D	PFC	NE
9460	HEXAFLUOROPROPYLENEOXIDE DIMER ACID (HFPO-DA)	03/20/2020	09/05/2020	D	PFC	NE
6956	NONFLUORO-3,6-DIOXAHEPTANOIC ACID (NFDHA)	03/20/2020	09/05/2020	D	PFC	NE
6957	PERFLUORO(2-ETHOXYETHANE)SULFONIC ACID (PFEESA)	03/20/2020	09/05/2020	D	PFC	NE
6965	PERFLUORO-3-METHOXYPROPANOIC ACID (PFMPA)	03/20/2020	09/05/2020	D	PFC	NE
6966	PERFLUORO-4-METHOXYBUTANOIC ACID (PFMBA)	03/20/2020	09/05/2020	D	PFC	NE
6918	PERFLUOROBUTANE SULFONIC ACID (PFBS) 375-73-5	03/20/2020	09/05/2020	D	PFC	NE
6915	PERFLUOROBUTANOIC ACID (PFBA) 375-22-4	03/20/2020	09/05/2020	D	PFC	NE
6905	PERFLUORODECANOIC ACID (PFDA) 335-76-2	03/20/2020	09/05/2020	D	PFC	NE
6903	PERFLUORODODECANOIC ACID (PFDOA) 307-55-1	03/20/2020	09/05/2020	D	PFC	NE
9470	PERFLUOROHEPTANE SULFONIC ACID (PFHPS) 375-92-8	03/20/2020	09/05/2020	D	PFC	NE
6908	PERFLUOROHEPTANOIC ACID (PFHPA) 375-85-9	03/20/2020	09/05/2020	D	PFC	NE
6927	PERFLUOROHEXANE SULFONIC ACID (PFHXS) 355-46-4	03/20/2020	09/05/2020	D	PFC	NE
6913	PERFLUOROHEXANOIC ACID (PFHXA) 307-24-4	03/20/2020	09/05/2020	D	PFC	NE
6906	PERFLUORONONANOIC ACID (PFNA) 375-95-1	03/20/2020	09/05/2020	D	PFC	NE
6931	PERFLUOROOCTANE SULFONIC ACID (PFOS) 1763-23-1	03/20/2020	09/05/2020	D	PFC	NE
6912	PERFLUOROOCTANOIC ACID (PFOA) 335-67-1	03/20/2020	09/05/2020	D	PFC	NE
6934	PERFLUOROPENTANE SULFONIC ACID (PFPES) 2706-91-4	03/20/2020	09/05/2020	D	PFC	NE
6914	PERFLUOROPENTANOIC ACID (PFPEA) 2706-90-3	03/20/2020	09/05/2020	D	PFC	NE
6904	PERFLUOROUNDECANOIC ACID (PFUDA) 2058-94-8	03/20/2020	09/05/2020	D	PFC	NE
Method Code: 10013806	**Method Ref: EPA 200.7**		**Revision: 4.4**		**Date: 1994**	
1000	ALUMINUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE

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1005	ANTIMONY, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1010	ARSENIC, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1015	BARIUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1020	BERYLLIUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1025	BORON, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1030	CADMIUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1035	CALCIUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1040	CHROMIUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1050	COBALT, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1055	COPPER, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1070	IRON, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1075	LEAD, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1085	MAGNESIUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1090	MANGANESE, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1100	MOLYBDENUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1105	NICKEL, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1125	POTASSIUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1140	SELENIUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1150	SILVER, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1155	SODIUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1160	STRONTIUM,TOTAL	02/21/2017	09/05/2020	N	MET	NE
1165	THALLIUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1175	TIN, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1180	TITANIUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1185	VANADIUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1190	ZINC, TOTAL	02/21/2017	09/05/2020	N	MET	NE

Method Code: 10014605 **Method Ref: EPA 200.8** **Revision: 5.4** **Date: 1994**

1000	ALUMINUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1005	ANTIMONY, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1010	ARSENIC, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1015	BARIUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1020	BERYLLIUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1030	CADMIUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1040	CHROMIUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1050	COBALT, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1055	COPPER, TOTAL	02/21/2017	09/05/2020	N	MET	NE

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1070	IRON, TOTAL	05/10/2018	09/05/2020	N	MET	NE
1075	LEAD, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1090	MANGANESE, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1100	MOLYBDENUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1105	NICKEL, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1125	POTASSIUM, TOTAL	05/10/2018	09/05/2020	N	MET	NE
1140	SELENIUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1150	SILVER, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1155	SODIUM, TOTAL	05/10/2018	09/05/2020	N	MET	NE
1165	THALLIUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1185	VANADIUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1190	ZINC, TOTAL	02/21/2017	09/05/2020	N	MET	NE
Method Code: 10036609	**Method Ref: EPA 245.1**		**Revision: 3**		**Date: 1994**	
1095	MERCURY, TOTAL	02/21/2017	09/05/2020	N	MET	NE
Method Code: 10155905	**Method Ref: SW-846 6010C**		**Revision: 3**		**Date: 2007**	
1000	ALUMINUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1005	ANTIMONY, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1010	ARSENIC, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1015	BARIUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1020	BERYLLIUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1025	BORON, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1030	CADMIUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1035	CALCIUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1040	CHROMIUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1050	COBALT, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1055	COPPER, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1070	IRON, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1075	LEAD, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1085	MAGNESIUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1090	MANGANESE, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1100	MOLYBDENUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1105	NICKEL, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1125	POTASSIUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1140	SELENIUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1150	SILVER, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1155	SODIUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE

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1160	STRONTIUM,TOTAL	02/21/2017	09/05/2020	N	MET	NE
1165	THALLIUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1175	TIN, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1180	TITANIUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1185	VANADIUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1190	ZINC, TOTAL	02/21/2017	09/05/2020	N	MET	NE
Method Code: 10155916	**Method Ref: SW-846 6010D**		**Revision:**		**Date: 2014**	
1000	ALUMINUM, TOTAL	06/29/2018	09/05/2020	N	MET	NE
1005	ANTIMONY, TOTAL	06/29/2018	09/05/2020	N	MET	NE
1010	ARSENIC, TOTAL	06/29/2018	09/05/2020	N	MET	NE
1015	BARIUM, TOTAL	06/29/2018	09/05/2020	N	MET	NE
1020	BERYLLIUM, TOTAL	06/29/2018	09/05/2020	N	MET	NE
1025	BORON, TOTAL	06/29/2018	09/05/2020	N	MET	NE
1030	CADMIUM, TOTAL	06/29/2018	09/05/2020	N	MET	NE
1035	CALCIUM, TOTAL	06/29/2018	09/05/2020	N	MET	NE
1040	CHROMIUM, TOTAL	06/29/2018	09/05/2020	N	MET	NE
1050	COBALT, TOTAL	06/29/2018	09/05/2020	N	MET	NE
1055	COPPER, TOTAL	06/29/2018	09/05/2020	N	MET	NE
1070	IRON, TOTAL	06/29/2018	09/05/2020	N	MET	NE
1075	LEAD, TOTAL	06/29/2018	09/05/2020	N	MET	NE
1085	MAGNESIUM, TOTAL	06/29/2018	09/05/2020	N	MET	NE
1090	MANGANESE, TOTAL	06/29/2018	09/05/2020	N	MET	NE
1100	MOLYBDENUM, TOTAL	06/29/2018	09/05/2020	N	MET	NE
1105	NICKEL, TOTAL	06/29/2018	09/05/2020	N	MET	NE
1125	POTASSIUM, TOTAL	06/29/2018	09/05/2020	N	MET	NE
1140	SELENIUM, TOTAL	06/29/2018	09/05/2020	N	MET	NE
1150	SILVER, TOTAL	06/29/2018	09/05/2020	N	MET	NE
1155	SODIUM, TOTAL	06/29/2018	09/05/2020	N	MET	NE
1160	STRONTIUM,TOTAL	06/29/2018	09/05/2020	N	MET	NE
1165	THALLIUM, TOTAL	06/29/2018	09/05/2020	N	MET	NE
1175	TIN, TOTAL	06/29/2018	09/05/2020	N	MET	NE
1180	TITANIUM, TOTAL	06/29/2018	09/05/2020	N	MET	NE
1185	VANADIUM, TOTAL	06/29/2018	09/05/2020	N	MET	NE
1190	ZINC, TOTAL	06/29/2018	09/05/2020	N	MET	NE
Method Code: 10156419	**Method Ref: SW-846 6020A**		**Revision:**		**Date: 2007**	
1000	ALUMINUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE

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1005	ANTIMONY, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1010	ARSENIC, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1015	BARIUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1020	BERYLLIUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1025	BORON, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1030	CADMIUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1035	CALCIUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1040	CHROMIUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1050	COBALT, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1055	COPPER, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1070	IRON, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1075	LEAD, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1085	MAGNESIUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1090	MANGANESE, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1100	MOLYBDENUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1105	NICKEL, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1125	POTASSIUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1140	SELENIUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1150	SILVER, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1155	SODIUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1160	STRONTIUM,TOTAL	02/21/2017	09/05/2020	N	MET	NE
1165	THALLIUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1175	TIN, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1180	TITANIUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1185	VANADIUM, TOTAL	02/21/2017	09/05/2020	N	MET	NE
1190	ZINC, TOTAL	02/21/2017	09/05/2020	N	MET	NE
Method Code: 10156420	**Method Ref: SW-846 6020B**		**Revision:**		**Date: 2014**	
1000	ALUMINUM, TOTAL	06/29/2018	09/05/2020	N	MET	NE
1005	ANTIMONY, TOTAL	06/29/2018	09/05/2020	N	MET	NE
1010	ARSENIC, TOTAL	06/29/2018	09/05/2020	N	MET	NE
1015	BARIUM, TOTAL	06/29/2018	09/05/2020	N	MET	NE
1020	BERYLLIUM, TOTAL	06/29/2018	09/05/2020	N	MET	NE
1025	BORON, TOTAL	06/29/2018	09/05/2020	N	MET	NE
1030	CADMIUM, TOTAL	06/29/2018	09/05/2020	N	MET	NE
1035	CALCIUM, TOTAL	06/29/2018	09/05/2020	N	MET	NE
1040	CHROMIUM, TOTAL	06/29/2018	09/05/2020	N	MET	NE

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1050	COBALT, TOTAL	06/29/2018	09/05/2020	N	MET	NE
1055	COPPER, TOTAL	06/29/2018	09/05/2020	N	MET	NE
1070	IRON, TOTAL	06/29/2018	09/05/2020	N	MET	NE
1075	LEAD, TOTAL	06/29/2018	09/05/2020	N	MET	NE
1085	MAGNESIUM, TOTAL	06/29/2018	09/05/2020	N	MET	NE
1090	MANGANESE, TOTAL	06/29/2018	09/05/2020	N	MET	NE
1100	MOLYBDENUM, TOTAL	06/29/2018	09/05/2020	N	MET	NE
1105	NICKEL, TOTAL	06/29/2018	09/05/2020	N	MET	NE
1125	POTASSIUM, TOTAL	06/29/2018	09/05/2020	N	MET	NE
1140	SELENIUM, TOTAL	06/29/2018	09/05/2020	N	MET	NE
1150	SILVER, TOTAL	06/29/2018	09/05/2020	N	MET	NE
1155	SODIUM, TOTAL	06/29/2018	09/05/2020	N	MET	NE
1160	STRONTIUM,TOTAL	06/29/2018	09/05/2020	N	MET	NE
1165	THALLIUM, TOTAL	06/29/2018	09/05/2020	N	MET	NE
1175	TIN, TOTAL	06/29/2018	09/05/2020	N	MET	NE
1180	TITANIUM, TOTAL	06/29/2018	09/05/2020	N	MET	NE
1185	VANADIUM, TOTAL	06/29/2018	09/05/2020	N	MET	NE
1190	ZINC, TOTAL	06/29/2018	09/05/2020	N	MET	NE
Method Code: 10165807	**Method Ref: SW-846 7470A**		**Revision: 1**		**Date: 1994**	
1095	MERCURY, TOTAL	02/21/2017	09/05/2020	N	MET	NE
Method Code: 10237204	**Method Ref: EPA 1631E**		**Revision:**		**Date: 2002**	
1095	MERCURY, TOTAL	02/21/2017	09/05/2020	N	MET	NE
Method Code: 10013806	**Method Ref: EPA 200.7**		**Revision: 4.4**		**Date: 1994**	
1755	HARDNESS AS CACO3	02/21/2017	09/05/2020	N	NMI	NE
1990	SILICA AS SIO2	02/21/2017	09/05/2020	N	NMI	NE
Method Code: 10155905	**Method Ref: SW-846 6010C**		**Revision: 3**		**Date: 2007**	
2017	SULFUR	02/21/2017	09/05/2020	N	NMI	NE
Method Code: 10155916	**Method Ref: SW-846 6010D**		**Revision:**		**Date: 2014**	
1755	HARDNESS AS CACO3	07/13/2020	09/05/2020	N	NMI	NE
1990	SILICA AS SIO2	07/13/2020	09/15/2020	N	NMI	NE
2017	SULFUR	06/29/2018	09/05/2020	N	NMI	NE
Method Code: 10156420	**Method Ref: SW-846 6020B**		**Revision:**		**Date: 2014**	
1755	HARDNESS AS CACO3	07/13/2020	09/05/2020	N	NMI	NE
Method Code: 20046611	**Method Ref: SM 2340 B-2011**		**Revision:**		**Date: 2011**	
1755	HARDNESS AS CACO3	02/21/2017	09/05/2020	N	NMI	NE
Method Code: 10133207	**Method Ref: SW-846 3005A**		**Revision: 1**		**Date: 1992**	
1438	PRECONCENTRATION UNDER ACID	11/22/2016	09/05/2020	N	PRE	NE

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Method Code: 10134006	**Method Ref: SW-846 3015A**		**Revision:**		**Date: 1998**	
1430	MICROWAVE-ASSISTED ACID DIGESTION OF TCLP EXTRACTS	02/21/2017	09/05/2020	N	PRE	NE
Method Code: 10138202	**Method Ref: SW-846 3510C**		**Revision: 3**		**Date: 1996**	
1444	SEPERATORY FUNNEL LIQUID-LIQUID EXTRACTION	02/21/2017	09/05/2020	N	PRE	NE
Method Code: 10143007	**Method Ref: SW-846 3580A**		**Revision: 1**		**Date: 1992**	
1470	WASTE DILUTION	02/21/2017	09/05/2020	N	PRE	NE
Method Code: 10146802	**Method Ref: SW-846 3630C**		**Revision: 3**		**Date: 1996**	
1446	SILICA GEL CLEAN-UP	02/21/2017	09/05/2020	N	PRE	NE
Method Code: 10147203	**Method Ref: SW-846 3640A**		**Revision: 1**		**Date: 1994**	
1418	GEL-PERMEATION CLEAN-UP	02/21/2017	09/05/2020	N	PRE	NE
Method Code: 10148400	**Method Ref: SW-846 3660B**		**Revision: 2**		**Date: 1996**	
1456	SULFUR CLEAN-UP	02/21/2017	09/05/2020	N	PRE	NE
Method Code: 10148808	**Method Ref: SW-846 3665A**		**Revision: 1**		**Date: 1996**	
1458	SULFURIC ACID / PERMANGANATE CLEAN-UP	02/21/2017	09/05/2020	N	PRE	NE
Method Code: 10088570	**Method Ref: EPA 522**		**Revision: 1**		**Date: 2008**	
4735	1,4-DIOXANE (1 4-DIETHYLENEOXIDE)	02/21/2017	09/05/2020	N	VOC	NE
Method Code: 10186002	**Method Ref: SW-846 8270D**		**Revision:**		**Date: 2007**	
5155	1,2,4-TRICHLOROBENZENE	02/21/2017	09/05/2020	N	VOC	NE
4610	1,2-DICHLOROBENZENE	02/21/2017	09/05/2020	N	VOC	NE
4615	1,3-DICHLOROBENZENE	02/21/2017	09/05/2020	N	VOC	NE
4620	1,4-DICHLOROBENZENE	02/21/2017	09/05/2020	N	VOC	NE
4835	HEXACHLOROBUTADIENE	02/21/2017	09/05/2020	N	VOC	NE
4840	HEXACHLOROETHANE	02/21/2017	09/05/2020	N	VOC	NE
5005	NAPHTHALENE	02/21/2017	09/05/2020	N	VOC	NE
5015	NITROBENZENE	02/21/2017	09/05/2020	N	VOC	NE
5095	PYRIDINE	02/21/2017	09/05/2020	N	VOC	NE
Method Code: 10212905	**Method Ref: RSK 175**		**Revision:**		**Date:**	
4747	ETHANE	02/21/2017	09/05/2020	N	VOC	NE
4752	ETHENE	02/21/2017	09/05/2020	N	VOC	NE
4926	METHANE	02/21/2017	09/05/2020	N	VOC	NE
Method Code: 10242509	**Method Ref: SW-846 8270D SIM**		**Revision:**		**Date: 2007**	
4735	1,4-DIOXANE (1 4-DIETHYLENEOXIDE)	02/21/2017	09/05/2020	N	VOC	NE
5005	NAPHTHALENE	02/21/2017	09/05/2020	N	VOC	NE
Method Code: 10242543	**Method Ref: SW-846 8270E**		**Revision:**		**Date: 2017**	
5155	1,2,4-TRICHLOROBENZENE	07/13/2020	09/05/2020	N	VOC	NE
4610	1,2-DICHLOROBENZENE	07/13/2020	09/05/2020	N	VOC	NE
4615	1,3-DICHLOROBENZENE	07/13/2020	09/05/2020	N	VOC	NE
4620	1,4-DICHLOROBENZENE	07/13/2020	09/05/2020	N	VOC	NE

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4835	HEXACHLOROBUTADIENE	07/13/2020	09/05/2020	N	VOC	NE
4840	HEXACHLOROETHANE	07/13/2020	09/05/2020	N	VOC	NE
5005	NAPHTHALENE	07/13/2020	09/05/2020	N	VOC	NE
5015	NITROBENZENE	07/13/2020	09/05/2020	N	VOC	NE
5095	PYRIDINE	07/13/2020	09/05/2020	N	VOC	NE
Method Code: 10242565	**Method Ref: SW-846 8270E SIM**		**Revision:**		**Date: 2017**	
4735	1,4-DIOXANE (1 4-DIETHYLENEOXIDE)	07/13/2020	09/05/2020	N	VOC	NE
5005	NAPHTHALENE	07/13/2020	09/05/2020	N	VOC	NE
Method Code: 10305609	**Method Ref: SW-846 8015D**		**Revision:**		**Date: 2003**	
4750	ETHANOL	02/21/2017	09/05/2020	N	VOC	NE
4785	ETHYLENE GLYCOL	02/21/2017	09/05/2020	N	VOC	NE
4875	ISOBUTYL ALCOHOL (2-METHYL-1-PROPANOL)	02/21/2017	09/05/2020	N	VOC	NE
4895	ISOPROPYL ALCOHOL (2-PROPANOL)	02/21/2017	09/05/2020	N	VOC	NE
4930	METHANOL	02/21/2017	09/05/2020	N	VOC	NE
5055	N-PROPANOL	02/21/2017	09/05/2020	N	VOC	NE
4368	T-AMYL ALCOHOL	02/21/2017	09/05/2020	N	VOC	NE
4420	TERT-BUTYL ALCOHOL (TBA)	02/21/2017	09/05/2020	N	VOC	NE
Method Code: 10178800	**Method Ref: SW-846 8081B**		**Revision:**		**Date: 2000**	
6275	HEXACHLOROBENZENE	02/14/2017	09/05/2020	N	SBN	NE
Method Code: 10186002	**Method Ref: SW-846 8270D**		**Revision:**		**Date: 2007**	
6703	1,1'-BIPHENYL (BZ-0)	02/21/2017	09/05/2020	N	SBN	NE
6710	1,2,3,5-TETRACHLOROBENZENE	02/21/2017	09/05/2020	N	SBN	NE
6715	1,2,4,5-TETRACHLOROBENZENE	02/21/2017	09/05/2020	N	SBN	NE
6220	1,2-DIPHENYLHYDRAZINE	02/21/2017	09/05/2020	N	SBN	NE
4659	2,2'-OXYBIS(1-CHLOROPROPANE)	02/21/2017	09/05/2020	N	SBN	NE
6735	2,3,4,6-TETRACHLOROPHENOL	02/21/2017	09/05/2020	N	SBN	NE
6835	2,4,5-TRICHLOROPHENOL	02/21/2017	09/05/2020	N	SBN	NE
6840	2,4,6-TRICHLOROPHENOL	02/21/2017	09/05/2020	N	SBN	NE
6000	2,4-DICHLOROPHENOL	02/21/2017	09/05/2020	N	SBN	NE
6130	2,4-DIMETHYLPHENOL	02/21/2017	09/05/2020	N	SBN	NE
6175	2,4-DINITROPHENOL	02/21/2017	09/05/2020	N	SBN	NE
6185	2,4-DINITROTOLUENE (2 4-DNT)	02/21/2017	09/05/2020	N	SBN	NE
6190	2,6-DINITROTOLUENE (2 6-DNT)	02/21/2017	09/05/2020	N	SBN	NE
5795	2-CHLORONAPHTHALENE	02/21/2017	09/05/2020	N	SBN	NE
5800	2-CHLOROPHENOL	02/21/2017	09/05/2020	N	SBN	NE
6360	2-METHYL-4,6-DINITROPHENOL	02/21/2017	09/05/2020	N	SBN	NE
6385	2-METHYLNAPHTHALENE	02/21/2017	09/05/2020	N	SBN	NE

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6400	2-METHYLPHENOL (O-CRESOL)	02/21/2017	09/05/2020	N	SBN	NE
6460	2-NITROANILINE	02/21/2017	09/05/2020	N	SBN	NE
6490	2-NITROPHENOL	02/21/2017	09/05/2020	N	SBN	NE
5945	3,3-DICHLOROBENZIDINE	02/21/2017	09/05/2020	N	SBN	NE
6405	3-METHYLPHENOL (M-CRESOL)	02/21/2017	09/05/2020	N	SBN	NE
6465	3-NITROANILINE	02/21/2017	09/05/2020	N	SBN	NE
5660	4-BROMOPHENYL PHENYL ETHER	02/21/2017	09/05/2020	N	SBN	NE
5700	4-CHLORO-3-METHYLPHENOL	02/21/2017	09/05/2020	N	SBN	NE
5745	4-CHLOROANILINE	02/21/2017	09/05/2020	N	SBN	NE
5825	4-CHLOROPHENYL PHENYL ETHER	02/21/2017	09/05/2020	N	SBN	NE
6410	4-METHYLPHENOL (P-CRESOL)	02/21/2017	09/05/2020	N	SBN	NE
6470	4-NITROANILINE	02/21/2017	09/05/2020	N	SBN	NE
6500	4-NITROPHENOL	02/21/2017	09/05/2020	N	SBN	NE
5500	ACENAPHTHENE	02/21/2017	09/05/2020	N	SBN	NE
5505	ACENAPHTHYLENE	02/21/2017	09/05/2020	N	SBN	NE
5510	ACETOPHENONE	02/21/2017	09/05/2020	N	SBN	NE
5545	ANILINE	02/21/2017	09/05/2020	N	SBN	NE
5555	ANTHRACENE	02/21/2017	09/05/2020	N	SBN	NE
5570	BENZALDEHYDE	02/21/2017	09/05/2020	N	SBN	NE
5595	BENZIDINE	02/21/2017	09/05/2020	N	SBN	NE
5575	BENZO(A)ANTHRACENE	02/21/2017	09/05/2020	N	SBN	NE
5580	BENZO(A)PYRENE	02/21/2017	09/05/2020	N	SBN	NE
5585	BENZO(B)FLUORANTHENE	02/21/2017	09/05/2020	N	SBN	NE
5590	BENZO(G,H,I)PERYLENE	02/21/2017	09/05/2020	N	SBN	NE
5600	BENZO(K)FLUORANTHENE	02/21/2017	09/05/2020	N	SBN	NE
5610	BENZOIC ACID	02/21/2017	09/05/2020	N	SBN	NE
5630	BENZYL ALCOHOL	02/21/2017	09/05/2020	N	SBN	NE
5670	BENZYL BUTYL PHTHALATE	02/21/2017	09/05/2020	N	SBN	NE
5760	BIS(2-CHLOROETHOXY) METHANE	02/21/2017	09/05/2020	N	SBN	NE
5765	BIS(2-CHLOROETHYL) ETHER	02/21/2017	09/05/2020	N	SBN	NE
6065	BIS(2-ETHYLHEXYL) PHTHALATE	02/21/2017	09/05/2020	N	SBN	NE
5680	CARBAZOLE	02/21/2017	09/05/2020	N	SBN	NE
5855	CHRYSENE	02/21/2017	09/05/2020	N	SBN	NE
5925	DI-N-BUTYL PHTHALATE	02/21/2017	09/05/2020	N	SBN	NE
6200	DI-N-OCTYL PHTHALATE	02/21/2017	09/05/2020	N	SBN	NE
5895	DIBENZO(A,H)ANTHRACENE	02/21/2017	09/05/2020	N	SBN	NE

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5905	DIBENZOFURAN	02/21/2017	09/05/2020	N	SBN	NE
6070	DIETHYL PHTHALATE	02/21/2017	09/05/2020	N	SBN	NE
6135	DIMETHYL PHTHALATE	02/21/2017	09/05/2020	N	SBN	NE
6265	FLUORANTHENE	02/21/2017	09/05/2020	N	SBN	NE
6270	FLUORENE	02/21/2017	09/05/2020	N	SBN	NE
6275	HEXACHLOROBENZENE	02/21/2017	09/05/2020	N	SBN	NE
6315	INDENO(1,2,3-CD)PYRENE	02/21/2017	09/05/2020	N	SBN	NE
6320	ISOPHORONE	02/21/2017	09/05/2020	N	SBN	NE
6545	N-NITROSODI-N-PROPYLAMINE	02/21/2017	09/05/2020	N	SBN	NE
6530	N-NITROSODIMETHYLAMINE	02/21/2017	09/05/2020	N	SBN	NE
6535	N-NITROSODIPHENYLAMINE	02/21/2017	09/05/2020	N	SBN	NE
6600	PCNB (PENTACHLORONITROBENZENE)	02/21/2017	09/05/2020	N	SBN	NE
6605	PENTACHLOROPHENOL	02/21/2017	09/05/2020	N	SBN	NE
6615	PHENANTHRENE	02/21/2017	09/05/2020	N	SBN	NE
6625	PHENOL	02/21/2017	09/05/2020	N	SBN	NE
6665	PYRENE	02/21/2017	09/05/2020	N	SBN	NE

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6380	1-METHYLNAPHTHALENE	02/21/2017	09/05/2020	N	SBN	NE
6385	2-METHYLNAPHTHALENE	02/21/2017	09/05/2020	N	SBN	NE
5500	ACENAPHTHENE	02/21/2017	09/05/2020	N	SBN	NE
5505	ACENAPHTHYLENE	02/21/2017	09/05/2020	N	SBN	NE
9297	ALKALATED PAHS	02/21/2017	09/05/2020	N	SBN	NE
5555	ANTHRACENE	02/21/2017	09/05/2020	N	SBN	NE
5575	BENZO(A)ANTHRACENE	02/21/2017	09/05/2020	N	SBN	NE
5580	BENZO(A)PYRENE	02/21/2017	09/05/2020	N	SBN	NE
5585	BENZO(B)FLUORANTHENE	02/21/2017	09/05/2020	N	SBN	NE
5590	BENZO(G,H,I)PERYLENE	02/21/2017	09/05/2020	N	SBN	NE
5600	BENZO(K)FLUORANTHENE	02/21/2017	09/05/2020	N	SBN	NE
5855	CHRYSENE	02/21/2017	09/05/2020	N	SBN	NE
5895	DIBENZO(A,H)ANTHRACENE	02/21/2017	09/05/2020	N	SBN	NE
6265	FLUORANTHENE	02/21/2017	09/05/2020	N	SBN	NE
6270	FLUORENE	02/21/2017	09/05/2020	N	SBN	NE
6315	INDENO(1,2,3-CD)PYRENE	02/21/2017	09/05/2020	N	SBN	NE
6605	PENTACHLOROPHENOL	02/21/2017	09/05/2020	N	SBN	NE
6615	PHENANTHRENE	02/21/2017	09/05/2020	N	SBN	NE
6665	PYRENE	02/21/2017	09/05/2020	N	SBN	NE

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6703	1,1'-BIPHENYL (BZ-0)	07/13/2020	09/05/2020	N	SBN	NE
6710	1,2,3,5-TETRACHLOROBENZENE	07/13/2020	09/05/2020	N	SBN	NE
6715	1,2,4,5-TETRACHLOROBENZENE	07/13/2020	09/05/2020	N	SBN	NE
6220	1,2-DIPHENYLHYDRAZINE	07/13/2020	09/05/2020	N	SBN	NE
4659	2,2'-OXYBIS(1-CHLOROPROPANE)	07/13/2020	09/05/2020	N	SBN	NE
6735	2,3,4,6-TETRACHLOROPHENOL	07/13/2020	09/05/2020	N	SBN	NE
6835	2,4,5-TRICHLOROPHENOL	07/13/2020	09/05/2020	N	SBN	NE
6840	2,4,6-TRICHLOROPHENOL	07/13/2020	09/05/2020	N	SBN	NE
6000	2,4-DICHLOROPHENOL	07/13/2020	09/05/2020	N	SBN	NE
6130	2,4-DIMETHYLPHENOL	07/13/2020	09/05/2020	N	SBN	NE
6175	2,4-DINITROPHENOL	07/13/2020	09/05/2020	N	SBN	NE
6185	2,4-DINITROTOLUENE (2 4-DNT)	07/13/2020	09/05/2020	N	SBN	NE
6190	2,6-DINITROTOLUENE (2 6-DNT)	07/13/2020	09/05/2020	N	SBN	NE
5795	2-CHLORONAPHTHALENE	07/13/2020	09/05/2020	N	SBN	NE
5800	2-CHLOROPHENOL	07/13/2020	09/05/2020	N	SBN	NE
6360	2-METHYL-4,6-DINITROPHENOL	07/13/2020	09/05/2020	N	SBN	NE
6385	2-METHYLNAPHTHALENE	07/13/2020	09/05/2020	N	SBN	NE
6400	2-METHYLPHENOL (O-CRESOL)	07/13/2020	09/05/2020	N	SBN	NE
6460	2-NITROANILINE	07/13/2020	09/05/2020	N	SBN	NE
6490	2-NITROPHENOL	07/13/2020	09/05/2020	N	SBN	NE
5945	3,3-DICHLOROBENZIDINE	07/13/2020	09/05/2020	N	SBN	NE
6405	3-METHYLPHENOL (M-CRESOL)	07/13/2020	09/05/2020	N	SBN	NE
6465	3-NITROANILINE	07/13/2020	09/05/2020	N	SBN	NE
5660	4-BROMOPHENYL PHENYL ETHER	07/13/2020	09/05/2020	N	SBN	NE
5700	4-CHLORO-3-METHYLPHENOL	07/13/2020	09/05/2020	N	SBN	NE
5745	4-CHLOROANILINE	07/13/2020	09/05/2020	N	SBN	NE
5825	4-CHLOROPHENYL PHENYL ETHER	07/13/2020	09/05/2020	N	SBN	NE
6410	4-METHYLPHENOL (P-CRESOL)	07/13/2020	09/05/2020	N	SBN	NE
6470	4-NITROANILINE	07/13/2020	09/05/2020	N	SBN	NE
6500	4-NITROPHENOL	07/13/2020	09/05/2020	N	SBN	NE
5500	ACENAPHTHENE	07/13/2020	09/05/2020	N	SBN	NE
5505	ACENAPHTHYLENE	07/13/2020	09/05/2020	N	SBN	NE
5510	ACETOPHENONE	07/13/2020	09/05/2020	N	SBN	NE
5545	ANILINE	07/13/2020	09/05/2020	N	SBN	NE
5555	ANTHRACENE	07/13/2020	09/05/2020	N	SBN	NE

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5570	BENZALDEHYDE	07/13/2020	09/05/2020	N	SBN	NE
5595	BENZIDINE	07/13/2020	09/05/2020	N	SBN	NE
5575	BENZO(A)ANTHRACENE	07/13/2020	09/05/2020	N	SBN	NE
5580	BENZO(A)PYRENE	07/13/2020	09/05/2020	N	SBN	NE
5585	BENZO(B)FLUORANTHENE	07/13/2020	09/05/2020	N	SBN	NE
5590	BENZO(G,H,I)PERYLENE	07/13/2020	09/05/2020	N	SBN	NE
5600	BENZO(K)FLUORANTHENE	07/13/2020	09/05/2020	N	SBN	NE
5610	BENZOIC ACID	07/13/2020	09/05/2020	N	SBN	NE
5630	BENZYL ALCOHOL	07/13/2020	09/05/2020	N	SBN	NE
5670	BENZYL BUTYL PHTHALATE	07/13/2020	09/05/2020	N	SBN	NE
5760	BIS(2-CHLOROETHOXY) METHANE	07/13/2020	09/05/2020	N	SBN	NE
5765	BIS(2-CHLOROETHYL) ETHER	07/13/2020	09/05/2020	N	SBN	NE
6065	BIS(2-ETHYLHEXYL) PHTHALATE	07/13/2020	09/05/2020	N	SBN	NE
5680	CARBAZOLE	07/13/2020	09/05/2020	N	SBN	NE
5855	CHRYSENE	07/13/2020	09/05/2020	N	SBN	NE
5925	DI-N-BUTYL PHTHALATE	07/13/2020	09/05/2020	N	SBN	NE
6200	DI-N-OCTYL PHTHALATE	07/13/2020	09/05/2020	N	SBN	NE
5895	DIBENZO(A,H)ANTHRACENE	07/13/2020	09/05/2020	N	SBN	NE
5905	DIBENZOFURAN	07/13/2020	09/05/2020	N	SBN	NE
6070	DIETHYL PHTHALATE	07/13/2020	09/05/2020	N	SBN	NE
6135	DIMETHYL PHTHALATE	07/13/2020	09/05/2020	N	SBN	NE
6265	FLUORANTHENE	07/13/2020	09/05/2020	N	SBN	NE
6270	FLUORENE	07/13/2020	09/05/2020	N	SBN	NE
6275	HEXACHLOROBENZENE	07/13/2020	09/05/2020	N	SBN	NE
6315	INDENO(1,2,3-CD)PYRENE	07/13/2020	09/05/2020	N	SBN	NE
6320	ISOPHORONE	07/13/2020	09/05/2020	N	SBN	NE
6545	N-NITROSODI-N-PROPYLAMINE	07/13/2020	09/05/2020	N	SBN	NE
6530	N-NITROSODIMETHYLAMINE	07/13/2020	09/05/2020	N	SBN	NE
6535	N-NITROSODIPHENYLAMINE	07/13/2020	09/05/2020	N	SBN	NE
6600	PCNB (PENTACHLORONITROBENZENE)	07/13/2020	09/05/2020	N	SBN	NE
6605	PENTACHLOROPHENOL	07/13/2020	09/05/2020	N	SBN	NE
6615	PHENANTHRENE	07/13/2020	09/05/2020	N	SBN	NE
6625	PHENOL	07/13/2020	09/05/2020	N	SBN	NE
6665	PYRENE	07/13/2020	09/05/2020	N	SBN	NE
Method Code: 10242565	**Method Ref: SW-846 8270E SIM**		**Revision:**		**Date: 2017**	
6380	1-METHYLNAPHTHALENE	07/13/2020	09/05/2020	N	SBN	NE

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6385	2-METHYLNAPHTHALENE	07/13/2020	09/05/2020	N	SBN	NE
5500	ACENAPHTHENE	07/13/2020	09/05/2020	N	SBN	NE
5505	ACENAPHTHYLENE	07/13/2020	09/05/2020	N	SBN	NE
9297	ALKALATED PAHS	07/13/2020	09/05/2020	N	SBN	NE
5555	ANTHRACENE	07/13/2020	09/05/2020	N	SBN	NE
5575	BENZO(A)ANTHRACENE	07/13/2020	09/05/2020	N	SBN	NE
5580	BENZO(A)PYRENE	07/13/2020	09/05/2020	N	SBN	NE
5585	BENZO(B)FLUORANTHENE	07/13/2020	09/05/2020	N	SBN	NE
5590	BENZO(G,H,I)PERYLENE	07/13/2020	09/05/2020	N	SBN	NE
5600	BENZO(K)FLUORANTHENE	07/13/2020	09/05/2020	N	SBN	NE
5855	CHRYSENE	07/13/2020	09/05/2020	N	SBN	NE
5895	DIBENZO(A,H)ANTHRACENE	07/13/2020	09/05/2020	N	SBN	NE
6265	FLUORANTHENE	07/13/2020	09/05/2020	N	SBN	NE
6270	FLUORENE	07/13/2020	09/05/2020	N	SBN	NE
6315	INDENO(1,2,3-CD)PYRENE	07/13/2020	09/05/2020	N	SBN	NE
6605	PENTACHLOROPHENOL	07/13/2020	09/05/2020	N	SBN	NE
6615	PHENANTHRENE	07/13/2020	09/05/2020	N	SBN	NE
6665	PYRENE	07/13/2020	09/05/2020	N	SBN	NE
Method Code: 10305609	**Method Ref: SW-846 8015D**		**Revision:**		**Date: 2003**	
6657	PROPYLENE GLYCOL	02/21/2017	09/05/2020	N	SBN	NE
Method Code: NH0274	**Method Ref: PFAS BY LC/MS/MS ISOTOPE DILUTION**		**Revision:**		**Date:**	
6915	PERFLUOROBUTANOIC ACID (PFBA) 375-22-4	06/30/2017	09/05/2020	N	SBN	NE
6905	PERFLUORODECANOIC ACID (PFDA) 335-76-2	06/30/2017	09/05/2020	N	SBN	NE
6903	PERFLUORODODECANOIC ACID (PFDOA) 307-55-1	06/30/2017	09/05/2020	N	SBN	NE
6908	PERFLUOROHEPTANOIC ACID (PFHPA) 375-85-9	06/30/2017	09/05/2020	N	SBN	NE
6913	PERFLUOROHEXANOIC ACID (PFHXA) 307-24-4	06/30/2017	09/05/2020	N	SBN	NE
6906	PERFLUORONONANOIC ACID (PFNA) 375-95-1	06/30/2017	09/05/2020	N	SBN	NE
6917	PERFLUOROOCTANE SULFONAMIDE (PFOSAM)	06/30/2017	09/05/2020	N	SBN	NE
6912	PERFLUOROOCTANOIC ACID (PFOA) 335-67-1	06/30/2017	09/05/2020	N	SBN	NE
6914	PERFLUOROPENTANOIC ACID (PFPEA) 2706-90-3	06/30/2017	09/05/2020	N	SBN	NE
6902	PERFLUOROTETRADECANOIC ACID (PFTDA) 376-06-7	06/30/2017	09/05/2020	N	SBN	NE
6904	PERFLUOROUNDECANOIC ACID (PFUDA) 2058-94-8	06/30/2017	09/05/2020	N	SBN	NE
Method Code: NH0275	**Method Ref: PFAS BY LC/MS/MS**		**Revision:**		**Date:**	
6905	PERFLUORODECANOIC ACID (PFDA) 335-76-2	06/30/2017	09/05/2020	N	SBN	NE
6903	PERFLUORODODECANOIC ACID (PFDOA) 307-55-1	06/30/2017	09/05/2020	N	SBN	NE
6908	PERFLUOROHEPTANOIC ACID (PFHPA) 375-85-9	06/30/2017	09/05/2020	N	SBN	NE
6913	PERFLUOROHEXANOIC ACID (PFHXA) 307-24-4	06/30/2017	09/05/2020	N	SBN	NE

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6906	PERFLURONONANOIC ACID (PFNA) 375-95-1	06/30/2017	09/05/2020	N	SBN	NE
6912	PERFLUOROOCTANOIC ACID (PFOA) 335-67-1	06/30/2017	09/05/2020	N	SBN	NE
6902	PERFLUOROTETRADECANOIC ACID (PFTDA) 376-06-7	06/30/2017	09/05/2020	N	SBN	NE
6904	PERFLUOROUNDECANOIC ACID (PFUDA) 2058-94-8	06/30/2017	09/05/2020	N	SBN	NE
Method Code: 10120602	**Method Ref: EPA 1613B**		**Revision:**		**Date: 1994**	
9519	1,2,3,4,6,7,8,9-OCTACHLORODIBENZO-P-DIOXIN (OCDD)	11/01/2018	09/05/2020	N	SNO	NE
9516	1,2,3,4,6,7,8,9-OCTACHLORODIBENZOFURAN (OCDF)	11/01/2018	09/05/2020	N	SNO	NE
9426	1,2,3,4,6,7,8-HEPTACHLORODIBENZO-P-DIOXIN (1,2,3,4,6,7,8-HPCDD)	11/01/2018	09/05/2020	N	SNO	NE
9420	1,2,3,4,6,7,8-HEPTACHLORODIBENZOFURAN (1,2,3,4,6,7,8-HPCDF)	11/01/2018	09/05/2020	N	SNO	NE
9423	1,2,3,4,7,8,9-HEPTACHLORODIBENZOFURAN (1,2,3,4,7,8,9-HPCDF)	11/01/2018	09/05/2020	N	SNO	NE
9453	1,2,3,4,7,8-HEXACHLORODIBENZO-P-DIOXIN (1,2,3,4,7,8-HXCDD)	11/01/2018	09/05/2020	N	SNO	NE
9471	1,2,3,4,7,8-HEXACHLORODIBENZOFURAN (1,2,3,4,7,8-HXCDF)	11/01/2018	09/05/2020	N	SNO	NE
9456	1,2,3,6,7,8-HEXACHLORODIBENZO-P-DIOXIN(1,2,3,6,7,8-HXCDD)	11/01/2018	09/05/2020	N	SNO	NE
9474	1,2,3,6,7,8-HEXACHLORODIBENZOFURAN (1,2,3,6,7,8-HXCDF)	11/01/2018	09/05/2020	N	SNO	NE
9459	1,2,3,7,8,9-HEXACHLORODIBENZO-P-DIOXIN (1,2,3,7,8,9-HXCDD)	11/01/2018	09/05/2020	N	SNO	NE
9477	1,2,3,7,8,9-HEXACHLORODIBENZOFURAN (1,2,3,7,8,9-HXCDF)	11/01/2018	09/05/2020	N	SNO	NE
9540	1,2,3,7,8-PENTACHLORODIBENZO-P-DIOXIN (1,2,3,7,8-PECDD)	11/01/2018	09/05/2020	N	SNO	NE
9543	1,2,3,7,8-PENTACHLORODIBENZOFURAN (1,2,3,7,8-PECDF)	11/01/2018	09/05/2020	N	SNO	NE
9480	2,3,4,6,7,8-HEXACHLORODIBENZOFURAN	11/01/2018	09/05/2020	N	SNO	NE
9549	2,3,4,7,8-PENTACHLORODIBENZOFURAN	11/01/2018	09/05/2020	N	SNO	NE
9618	2,3,7,8-TETRACHLORODIBENZO- P-DIOXIN (2,3,7,8-TCDD)	11/01/2018	09/05/2020	N	SNO	NE
9612	2,3,7,8-TETRACHLORODIBENZOFURAN (TCDF)	11/01/2018	09/05/2020	N	SNO	NE
9438	TOTAL HEPTACHLORODIBENZO-P-DIOXIN (TOTAL HPCDD)	11/01/2018	09/05/2020	N	SNO	NE
9444	TOTAL HEPTACHLORODIBENZOFURAN (TOTAL HPCDF)	11/01/2018	09/05/2020	N	SNO	NE
9468	TOTAL HEXACHLORODIBENZO-P-DIOXIN (TOTAL HXCDD)	11/01/2018	09/05/2020	N	SNO	NE
9483	TOTAL HEXACHLORODIBENZOFURAN (TOTAL HXCDF)	11/01/2018	09/05/2020	N	SNO	NE
9555	TOTAL PENTACHLORODIBENZO-P-DIOXIN (TOTAL PECDD)	11/01/2018	09/05/2020	N	SNO	NE
9552	TOTAL PENTACHLORODIBENZOFURAN (TOTAL PECDF)	11/01/2018	09/05/2020	N	SNO	NE
9660	TOTAL POLYCHLORINATED DIBENZO-P-DIOXINS (TOTAL PCDDS)	11/01/2018	09/05/2020	N	SNO	NE
9657	TOTAL POLYCHLORINATED DIBENZOFURANS (TOTAL PCDFS)	11/01/2018	09/05/2020	N	SNO	NE
9609	TOTAL TETRACHLORODIBENZO-P-DIOXIN (TOTAL TCDD)	11/01/2018	09/05/2020	N	SNO	NE

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9615	TOTAL TETRACHLORODIBENZOFURAN (TOTAL TCDF)	11/01/2018	09/05/2020	N	SNO	NE
Method Code: 10187403	**Method Ref: EPA 8290A**		**Revision:**		**Date: 2007**	
9519	1,2,3,4,6,7,8,9-OCTACHLORODIBENZO-P-DIOXIN (OCDD)	11/01/2018	09/05/2020	N	SNO	NE
9516	1,2,3,4,6,7,8,9-OCTACHLORODIBENZOFURAN (OCDF)	11/01/2018	09/05/2020	N	SNO	NE
9426	1,2,3,4,6,7,8-HEPTACHLORODIBENZO-P-DIOXIN (1,2,3,4,6,7,8-HPCDD)	11/01/2018	09/05/2020	N	SNO	NE
9420	1,2,3,4,6,7,8-HEPTACHLORODIBENZOFURAN (1,2,3,4,6,7,8-HPCDF)	11/01/2018	09/05/2020	N	SNO	NE
9423	1,2,3,4,7,8,9-HEPTACHLORODIBENZOFURAN (1,2,3,4,7,8,9-HPCDF)	11/01/2018	09/05/2020	N	SNO	NE
9453	1,2,3,4,7,8-HEXACHLORODIBENZO-P-DIOXIN (1,2,3,4,7,8-HXCDD)	11/01/2018	09/05/2020	N	SNO	NE
9471	1,2,3,4,7,8-HEXACHLORODIBENZOFURAN (1,2,3,4,7,8-HXCDF)	11/01/2018	09/05/2020	N	SNO	NE
9456	1,2,3,6,7,8-HEXACHLORODIBENZO-P-DIOXIN(1,2,3,6,7,8-HXCDD)	11/01/2018	09/05/2020	N	SNO	NE
9474	1,2,3,6,7,8-HEXACHLORODIBENZOFURAN (1,2,3,6,7,8-HXCDF)	11/01/2018	09/05/2020	N	SNO	NE
9459	1,2,3,7,8,9-HEXACHLORODIBENZO-P-DIOXIN (1,2,3,7,8,9-HXCDD)	11/01/2018	09/05/2020	N	SNO	NE
9477	1,2,3,7,8,9-HEXACHLORODIBENZOFURAN (1,2,3,7,8,9-HXCDF)	11/01/2018	09/05/2020	N	SNO	NE
9540	1,2,3,7,8-PENTACHLORODIBENZO-P-DIOXIN (1,2,3,7,8-PECDD)	11/01/2018	09/05/2020	N	SNO	NE
9543	1,2,3,7,8-PENTACHLORODIBENZOFURAN (1,2,3,7,8-PECDF)	11/01/2018	09/05/2020	N	SNO	NE
9480	2,3,4,6,7,8-HEXACHLORODIBENZOFURAN	11/01/2018	09/05/2020	N	SNO	NE
9549	2,3,4,7,8-PENTACHLORODIBENZOFURAN	11/01/2018	09/05/2020	N	SNO	NE
9618	2,3,7,8-TETRACHLORODIBENZO- P-DIOXIN (2,3,7,8-TCDD)	11/01/2018	09/05/2020	N	SNO	NE
9612	2,3,7,8-TETRACHLORODIBENZOFURAN (TCDF)	11/01/2018	09/05/2020	N	SNO	NE
9438	TOTAL HEPTACHLORODIBENZO-P-DIOXIN (TOTAL HPCDD)	11/01/2018	09/05/2020	N	SNO	NE
9444	TOTAL HEPTACHLORODIBENZOFURAN (TOTAL HPCDF)	11/01/2018	09/05/2020	N	SNO	NE
9468	TOTAL HEXACHLORODIBENZO-P-DIOXIN (TOTAL HXCDD)	11/01/2018	09/05/2020	N	SNO	NE
9483	TOTAL HEXACHLORODIBENZOFURAN (TOTAL HXCDF)	11/01/2018	09/05/2020	N	SNO	NE
9555	TOTAL PENTACHLORODIBENZO-P-DIOXIN (TOTAL PECDD)	11/01/2018	09/05/2020	N	SNO	NE
9552	TOTAL PENTACHLORODIBENZOFURAN (TOTAL PECDF)	11/01/2018	09/05/2020	N	SNO	NE
9660	TOTAL POLYCHLORINATED DIBENZO-P-DIOXINS (TOTAL PCDDS)	11/01/2018	09/05/2020	N	SNO	NE
9657	TOTAL POLYCHLORINATED DIBENZOFURANS (TOTAL PCDFS)	11/01/2018	09/05/2020	N	SNO	NE
9609	TOTAL TETRACHLORODIBENZO-P-DIOXIN (TOTAL TCDD)	11/01/2018	09/05/2020	N	SNO	NE
9615	TOTAL TETRACHLORODIBENZOFURAN (TOTAL TCDF)	11/01/2018	09/05/2020	N	SNO	NE
Method Code: 10305609	**Method Ref: SW-846 8015D**		**Revision:**		**Date: 2003**	
9369	DIESEL RANGE ORGANICS (DRO)	02/21/2017	09/05/2020	N	SNO	NE

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Method Code: NH0274	**Method Ref: PFAS BY LC/MS/MS ISOTOPE DILUTION**		**Revision:**		**Date:**	
0136NH	1H,1H,2H,2H-PERFLUORODECANE SULFONATE (8:2) 8:2FTS	06/30/2017	09/05/2020	N	SNO	NE
9562	PERFLUORODECANE SULFONATE (PFDS)	06/30/2017	09/05/2020	N	SNO	NE
9470	PERFLUOROHEPTANE SULFONIC ACID (PFHPS) 375-92-8	06/30/2017	09/05/2020	N	SNO	NE
9563	PERFLUOROTRIDECANOIC ACID (PFTRDA) 72629-94-8	06/30/2017	09/05/2020	N	SNO	NE
Method Code: NH0275	**Method Ref: PFAS BY LC/MS/MS**		**Revision:**		**Date:**	
4846	N-ETHYLPERFLUOROOCTANE SUFONAMIDO ACETIC ACID (NETFOSAA) 2991-50-6	06/30/2017	09/05/2020	N	SNO	NE
4847	N-METHYLPERFLUOROOCTANE SULFONAMIDO ACETIC ACID (NMEFOSAA)	06/30/2017	09/05/2020	N	SNO	NE
9563	PERFLUOROTRIDECANOIC ACID (PFTRDA) 72629-94-8	06/30/2017	09/05/2020	N	SNO	NE
Method Code: 10179201	**Method Ref: SW-846 8082A**		**Revision:**		**Date: 2000**	
9095	2,2',3,3',4,4',5,5',6-NONACHLOROBIPHENYL (BZ-206)	02/21/2017	09/05/2020	N	SPC	NE
9065	2,2',3,3',4,4',5-HEPTACHLOROBIPHENYL (BZ-170)	02/21/2017	09/05/2020	N	SPC	NE
9020	2,2',3,3',4,4'-HEXACHLOROBIPHENYL (BZ-128)	02/21/2017	09/05/2020	N	SPC	NE
9025	2,2',3,4,4', 5'-HEXACHLOROBIPHENYL (BZ-138)	02/21/2017	09/05/2020	N	SPC	NE
8945	2,2',3,5'-TETRACHLOROBIPHENYL (BZ-44)	02/21/2017	09/05/2020	N	SPC	NE
8955	2,2',5,5'-TETRACHLOROBIPHENYL (BZ-52)	02/21/2017	09/05/2020	N	SPC	NE
8930	2,2',5-TRICHLOROBIPHENYL (BZ-18)	02/21/2017	09/05/2020	N	SPC	NE
8995	2,3',4,4',5-PENTACHLOROBIPHENYL (BZ-118)	02/21/2017	09/05/2020	N	SPC	NE
8960	2,3',4,4'-TETRACHLOROBIPHENYL (BZ-66)	02/21/2017	09/05/2020	N	SPC	NE
8880	AROCLOR-1016 (PCB-1016)	02/21/2017	09/05/2020	N	SPC	NE
8885	AROCLOR-1221 (PCB-1221)	02/21/2017	09/05/2020	N	SPC	NE
8890	AROCLOR-1232 (PCB-1232)	02/21/2017	09/05/2020	N	SPC	NE
8895	AROCLOR-1242 (PCB-1242)	02/21/2017	09/05/2020	N	SPC	NE
8900	AROCLOR-1248 (PCB-1248)	02/21/2017	09/05/2020	N	SPC	NE
8905	AROCLOR-1254 (PCB-1254)	02/21/2017	09/05/2020	N	SPC	NE
8910	AROCLOR-1260 (PCB-1260)	02/21/2017	09/05/2020	N	SPC	NE
8912	AROCLOR-1262 (PCB-1262)	02/21/2017	09/05/2020	N	SPC	NE
8913	AROCLOR-1268 (PCB-1268)	02/21/2017	09/05/2020	N	SPC	NE
Method Code: 10178800	**Method Ref: SW-846 8081B**		**Revision:**		**Date: 2000**	
7355	4,4-DDD	02/14/2017	09/05/2020	N	SPE	NE
7360	4,4-DDE	02/14/2017	09/05/2020	N	SPE	NE
7365	4,4-DDT	02/14/2017	09/05/2020	N	SPE	NE
7025	ALDRIN	02/14/2017	09/05/2020	N	SPE	NE
7110	ALPHA-BHC (ALPHA-HEXACHLOROCYCLOHEXANE)	02/14/2017	09/05/2020	N	SPE	NE
7240	ALPHA-CHLORDANE	02/14/2017	09/05/2020	N	SPE	NE

This analyte list supersedes all previously issued analyte lists. Method accreditation does not imply acceptance for NHDES compliance testing. Laboratory is required to use EPA-approved methods required by regulation. Continuing accreditation status is dependent on successful ongoing participation in the program. Customers may verify the laboratory's current accreditation status by calling (603) 271-2998 or by visiting the NH ELAP website (https://www.des.nh.gov/organization/divisions/water/dwgb/nhelap/index.htm).

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7115	BETA-BHC (BETA-HEXACHLOROCYCLOHEXANE)	02/14/2017	09/05/2020	N	SPE	NE
7250	CHLORDANE (TECH.)	02/14/2017	09/05/2020	N	SPE	NE
7105	DELTA-BHC	02/14/2017	09/05/2020	N	SPE	NE
7470	DIELDRIN	02/14/2017	09/05/2020	N	SPE	NE
7510	ENDOSULFAN I	02/14/2017	09/05/2020	N	SPE	NE
7515	ENDOSULFAN II	02/14/2017	09/05/2020	N	SPE	NE
7520	ENDOSULFAN SULFATE	02/14/2017	09/05/2020	N	SPE	NE
7540	ENDRIN	02/14/2017	09/05/2020	N	SPE	NE
7530	ENDRIN ALDEHYDE	02/14/2017	09/05/2020	N	SPE	NE
7535	ENDRIN KETONE	02/14/2017	09/05/2020	N	SPE	NE
7120	GAMMA-BHC (LINDANE)	02/14/2017	09/05/2020	N	SPE	NE
7245	GAMMA-CHLORDANE	02/14/2017	09/05/2020	N	SPE	NE
7685	HEPTACHLOR	02/14/2017	09/05/2020	N	SPE	NE
7690	HEPTACHLOR EPOXIDE	02/14/2017	09/05/2020	N	SPE	NE
7810	METHOXYCHLOR	02/14/2017	09/05/2020	N	SPE	NE
7870	MIREX	02/14/2017	09/05/2020	N	SPE	NE
8250	TOXAPHENE (CHLORINATED CAMPHENE)	02/14/2017	09/05/2020	N	SPE	NE
Method Code: 10186002	**Method Ref: SW-846 8270D**		**Revision:**		**Date: 2007**	
7065	ATRAZINE	02/21/2017	09/05/2020	N	SPE	NE
7180	CAPROLACTAM	02/21/2017	09/05/2020	N	SPE	NE
6285	HEXACHLOROCYCLOPENTADIENE	02/21/2017	09/05/2020	N	SPE	NE
Method Code: 10242543	**Method Ref: SW-846 8270E**		**Revision:**		**Date: 2017**	
7065	ATRAZINE	07/13/2020	09/05/2020	N	SPE	NE
7180	CAPROLACTAM	07/13/2020	09/05/2020	N	SPE	NE
6285	HEXACHLOROCYCLOPENTADIENE	07/13/2020	09/05/2020	N	SPE	NE
Method Code: 10212905	**Method Ref: RSK 175**		**Revision:**		**Date:**	
5029	PROPANE	02/21/2017	09/05/2020	N	DG	NE
Method Code: NH0274	**Method Ref: PFAS BY LC/MS/MS ISOTOPE DILUTION**		**Revision:**		**Date:**	
0137NH	1H,1H,2H,2H-PERFLUOROHEXANE SULFONATE (4:2) 4:2FTS	06/30/2017	09/05/2020	N	OTH	NE
0138NH	1H,1H,2H,2H-PERFLUOROOCTANE SULFONATE (6:2) 6:2FTS	06/30/2017	09/05/2020	N	OTH	NE
0139NH	N-ETHYLPERFLUORO-1-OCTANESULFONAMIDOACETIC ACID (N-ETFOSAA)	06/30/2017	09/05/2020	N	OTH	NE
6955	N-METHYLPERFLUOROOCTANE SULFONAMIDO ACETIC ACID (NMEFOSAA)	06/30/2017	09/05/2020	N	OTH	NE
9464	PERFLUORONONANE SULFONATE (474511-07-4)	06/30/2017	09/05/2020	N	OTH	NE
9467	PERFLUOROPENTANESULFONATE (PFPES 2706-91-4)	06/30/2017	09/05/2020	N	OTH	NE

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Method Code: NH0274	**Method Ref: PFAS BY LC/MS/MS ISOTOPE DILUTION**		**Revision:**		**Date:**	
6918	PERFLUOROBUTANE SULFONIC ACID (PFBS) 375-73-5	03/25/2019	09/05/2020	N	PFC	NE
6927	PERFLUOROHEXANE SULFONIC ACID (PFHXS) 355-46-4	03/25/2019	09/05/2020	N	PFC	NE
6931	PERFLUOROOCTANE SULFONIC ACID (PFOS) 1763-23-1	03/25/2019	09/05/2020	N	PFC	NE
Method Code: NH0275	**Method Ref: PFAS BY LC/MS/MS**		**Revision:**		**Date:**	
6918	PERFLUOROBUTANE SULFONIC ACID (PFBS) 375-73-5	03/25/2019	09/05/2020	N	PFC	NE
6927	PERFLUOROHEXANE SULFONIC ACID (PFHXS) 355-46-4	03/25/2019	09/05/2020	N	PFC	NE
6931	PERFLUOROOCTANE SULFONIC ACID (PFOS) 1763-23-1	03/25/2019	09/05/2020	N	PFC	NE
Method Code: 10155905	**Method Ref: SW-846 6010C**		**Revision: 3**		**Date: 2007**	
1000	ALUMINUM, TOTAL	02/14/2017	09/05/2020	SC	MET	NE
1005	ANTIMONY, TOTAL	02/14/2017	09/05/2020	SC	MET	NE
1010	ARSENIC, TOTAL	02/14/2017	09/05/2020	SC	MET	NE
1015	BARIUM, TOTAL	02/14/2017	09/05/2020	SC	MET	NE
1020	BERYLLIUM, TOTAL	02/14/2017	09/05/2020	SC	MET	NE
1025	BORON, TOTAL	02/14/2017	09/05/2020	SC	MET	NE
1030	CADMIUM, TOTAL	02/14/2017	09/05/2020	SC	MET	NE
1035	CALCIUM, TOTAL	02/14/2017	09/05/2020	SC	MET	NE
1040	CHROMIUM, TOTAL	02/14/2017	09/05/2020	SC	MET	NE
1050	COBALT, TOTAL	02/14/2017	09/05/2020	SC	MET	NE
1055	COPPER, TOTAL	02/14/2017	09/05/2020	SC	MET	NE
1070	IRON, TOTAL	02/14/2017	09/05/2020	SC	MET	NE
1075	LEAD, TOTAL	02/14/2017	09/05/2020	SC	MET	NE
1085	MAGNESIUM, TOTAL	02/14/2017	09/05/2020	SC	MET	NE
1090	MANGANESE, TOTAL	02/14/2017	09/05/2020	SC	MET	NE
1100	MOLYBDENUM, TOTAL	02/14/2017	09/05/2020	SC	MET	NE
1105	NICKEL, TOTAL	02/14/2017	09/05/2020	SC	MET	NE
1125	POTASSIUM, TOTAL	02/14/2017	09/05/2020	SC	MET	NE
1140	SELENIUM, TOTAL	02/14/2017	09/05/2020	SC	MET	NE
1150	SILVER, TOTAL	02/14/2017	09/05/2020	SC	MET	NE
1155	SODIUM, TOTAL	02/14/2017	09/05/2020	SC	MET	NE
1160	STRONTIUM,TOTAL	02/14/2017	09/05/2020	SC	MET	NE
1165	THALLIUM, TOTAL	02/14/2017	09/05/2020	SC	MET	NE
1175	TIN, TOTAL	02/14/2017	09/05/2020	SC	MET	NE
1180	TITANIUM, TOTAL	02/14/2017	09/05/2020	SC	MET	NE
1185	VANADIUM, TOTAL	02/14/2017	09/05/2020	SC	MET	NE
1190	ZINC, TOTAL	02/14/2017	09/05/2020	SC	MET	NE

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Method Code: 10155916	**Method Ref: SW-846 6010D**		**Revision:**		**Date: 2014**	
1000	ALUMINUM, TOTAL	06/29/2018	09/05/2020	SC	MET	NE
1005	ANTIMONY, TOTAL	06/29/2018	09/05/2020	SC	MET	NE
1010	ARSENIC, TOTAL	06/29/2018	09/05/2020	SC	MET	NE
1015	BARIUM, TOTAL	06/29/2018	09/05/2020	SC	MET	NE
1020	BERYLLIUM, TOTAL	06/29/2018	09/05/2020	SC	MET	NE
1025	BORON, TOTAL	06/29/2018	09/05/2020	SC	MET	NE
1030	CADMIUM, TOTAL	06/29/2018	09/05/2020	SC	MET	NE
1035	CALCIUM, TOTAL	06/29/2018	09/05/2020	SC	MET	NE
1040	CHROMIUM, TOTAL	06/29/2018	09/05/2020	SC	MET	NE
1050	COBALT, TOTAL	06/29/2018	09/05/2020	SC	MET	NE
1055	COPPER, TOTAL	06/29/2018	09/05/2020	SC	MET	NE
1070	IRON, TOTAL	06/29/2018	09/05/2020	SC	MET	NE
1075	LEAD, TOTAL	06/29/2018	09/05/2020	SC	MET	NE
1085	MAGNESIUM, TOTAL	06/29/2018	09/05/2020	SC	MET	NE
1090	MANGANESE, TOTAL	06/29/2018	09/05/2020	SC	MET	NE
1100	MOLYBDENUM, TOTAL	06/29/2018	09/05/2020	SC	MET	NE
1105	NICKEL, TOTAL	06/29/2018	09/05/2020	SC	MET	NE
1125	POTASSIUM, TOTAL	06/29/2018	09/05/2020	SC	MET	NE
1140	SELENIUM, TOTAL	06/29/2018	09/05/2020	SC	MET	NE
1150	SILVER, TOTAL	06/29/2018	09/05/2020	SC	MET	NE
1155	SODIUM, TOTAL	06/29/2018	09/05/2020	SC	MET	NE
1160	STRONTIUM,TOTAL	06/29/2018	09/05/2020	SC	MET	NE
1165	THALLIUM, TOTAL	06/29/2018	09/05/2020	SC	MET	NE
1175	TIN, TOTAL	06/29/2018	09/05/2020	SC	MET	NE
1180	TITANIUM, TOTAL	06/29/2018	09/05/2020	SC	MET	NE
1185	VANADIUM, TOTAL	06/29/2018	09/05/2020	SC	MET	NE
1190	ZINC, TOTAL	06/29/2018	09/05/2020	SC	MET	NE
Method Code: 10156419	**Method Ref: SW-846 6020A**		**Revision:**		**Date: 2007**	
1000	ALUMINUM, TOTAL	02/21/2017	09/05/2020	SC	MET	NE
1005	ANTIMONY, TOTAL	02/21/2017	09/05/2020	SC	MET	NE
1010	ARSENIC, TOTAL	02/21/2017	09/05/2020	SC	MET	NE
1015	BARIUM, TOTAL	02/21/2017	09/05/2020	SC	MET	NE
1020	BERYLLIUM, TOTAL	02/21/2017	09/05/2020	SC	MET	NE
1030	CADMIUM, TOTAL	02/21/2017	09/05/2020	SC	MET	NE
1035	CALCIUM, TOTAL	02/21/2017	09/05/2020	SC	MET	NE

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1040	CHROMIUM, TOTAL	02/21/2017	09/05/2020	SC	MET	NE
1050	COBALT, TOTAL	02/21/2017	09/05/2020	SC	MET	NE
1055	COPPER, TOTAL	02/21/2017	09/05/2020	SC	MET	NE
1070	IRON, TOTAL	02/21/2017	09/05/2020	SC	MET	NE
1075	LEAD, TOTAL	02/21/2017	09/05/2020	SC	MET	NE
1085	MAGNESIUM, TOTAL	02/21/2017	09/05/2020	SC	MET	NE
1090	MANGANESE, TOTAL	02/21/2017	09/05/2020	SC	MET	NE
1100	MOLYBDENUM, TOTAL	02/21/2017	09/05/2020	SC	MET	NE
1105	NICKEL, TOTAL	02/21/2017	09/05/2020	SC	MET	NE
1125	POTASSIUM, TOTAL	02/21/2017	09/05/2020	SC	MET	NE
1140	SELENIUM, TOTAL	02/21/2017	09/05/2020	SC	MET	NE
1150	SILVER, TOTAL	02/21/2017	09/05/2020	SC	MET	NE
1155	SODIUM, TOTAL	02/21/2017	09/05/2020	SC	MET	NE
1160	STRONTIUM,TOTAL	02/21/2017	09/05/2020	SC	MET	NE
1165	THALLIUM, TOTAL	02/21/2017	09/05/2020	SC	MET	NE
1175	TIN, TOTAL	02/21/2017	09/05/2020	SC	MET	NE
1180	TITANIUM, TOTAL	02/21/2017	09/05/2020	SC	MET	NE
1185	VANADIUM, TOTAL	02/21/2017	09/05/2020	SC	MET	NE
1190	ZINC, TOTAL	02/21/2017	09/05/2020	SC	MET	NE
Method Code: 10156420	**Method Ref: SW-846 6020B**		**Revision:**		**Date: 2014**	
1000	ALUMINUM, TOTAL	06/29/2018	09/05/2020	SC	MET	NE
1005	ANTIMONY, TOTAL	06/29/2018	09/05/2020	SC	MET	NE
1010	ARSENIC, TOTAL	06/29/2018	09/05/2020	SC	MET	NE
1015	BARIUM, TOTAL	06/29/2018	09/05/2020	SC	MET	NE
1020	BERYLLIUM, TOTAL	06/29/2018	09/05/2020	SC	MET	NE
1030	CADMIUM, TOTAL	06/29/2018	09/05/2020	SC	MET	NE
1035	CALCIUM, TOTAL	06/29/2018	09/05/2020	SC	MET	NE
1040	CHROMIUM, TOTAL	06/29/2018	09/05/2020	SC	MET	NE
1050	COBALT, TOTAL	06/29/2018	09/05/2020	SC	MET	NE
1055	COPPER, TOTAL	06/29/2018	09/05/2020	SC	MET	NE
1070	IRON, TOTAL	06/29/2018	09/05/2020	SC	MET	NE
1075	LEAD, TOTAL	06/29/2018	09/05/2020	SC	MET	NE
1085	MAGNESIUM, TOTAL	06/29/2018	09/05/2020	SC	MET	NE
1090	MANGANESE, TOTAL	06/29/2018	09/05/2020	SC	MET	NE
1100	MOLYBDENUM, TOTAL	06/29/2018	09/05/2020	SC	MET	NE
1105	NICKEL, TOTAL	06/29/2018	09/05/2020	SC	MET	NE

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1125	POTASSIUM, TOTAL	06/29/2018	09/05/2020	SC	MET	NE
1140	SELENIUM, TOTAL	06/29/2018	09/05/2020	SC	MET	NE
1150	SILVER, TOTAL	06/29/2018	09/05/2020	SC	MET	NE
1155	SODIUM, TOTAL	06/29/2018	09/05/2020	SC	MET	NE
1160	STRONTIUM,TOTAL	06/29/2018	09/05/2020	SC	MET	NE
1165	THALLIUM, TOTAL	06/29/2018	09/05/2020	SC	MET	NE
1175	TIN, TOTAL	06/29/2018	09/05/2020	SC	MET	NE
1180	TITANIUM, TOTAL	06/29/2018	09/05/2020	SC	MET	NE
1185	VANADIUM, TOTAL	06/29/2018	09/05/2020	SC	MET	NE
1190	ZINC, TOTAL	06/29/2018	09/05/2020	SC	MET	NE
Method Code: 10166457	**Method Ref: SW-846 7471B**		**Revision:**		**Date: 2007**	
1095	MERCURY, TOTAL	12/29/2017	09/05/2020	SC	MET	NE
Method Code: 10167007	**Method Ref: SW-846 7474**		**Revision:**		**Date: 2007**	
1095	MERCURY, TOTAL	02/21/2017	09/05/2020	SC	MET	NE
Method Code: 10155905	**Method Ref: SW-846 6010C**		**Revision: 3**		**Date: 2007**	
2017	SULFUR	02/14/2017	09/05/2020	SC	NMI	NE
Method Code: 10155916	**Method Ref: SW-846 6010D**		**Revision:**		**Date: 2014**	
2017	SULFUR	06/29/2018	09/05/2020	SC	NMI	NE
Method Code: 10244823	**Method Ref: SW-846 9060A**		**Revision:**		**Date: 2004**	
2040	TOTAL ORGANIC CARBON (TOC)	02/21/2017	09/05/2020	SC	NMI	NE
Method Code: 20005203	**Method Ref: SM 2540 G**		**Revision: 18th ED**		**Date: 1992**	
1950	RESIDUE, TOTAL (TS)	02/21/2017	09/05/2020	SC	NMI	NE
Method Code: 60041001	**Method Ref: LLOYD KAHN**		**Revision:**		**Date: 1988**	
2040	TOTAL ORGANIC CARBON (TOC)	02/21/2017	09/05/2020	SC	NMI	NE
Method Code: 10135601	**Method Ref: SW-846 3050B**		**Revision: 2**		**Date: 1996**	
1400	ACID DIGESTION OF SOLIDS	02/21/2017	09/05/2020	SC	PRE	NE
Method Code: 10136002	**Method Ref: SW-846 3051A**		**Revision:**		**Date: 1998**	
1426	MICROWAVE DIGESTION OF SOLIDS	02/21/2017	09/05/2020	SC	PRE	NE
Method Code: 10140202	**Method Ref: SW-846 3540C**		**Revision: 3**		**Date: 1996**	
1452	SOXHLET EXTRACTION	02/21/2017	09/05/2020	SC	PRE	NE
Method Code: 10142708	**Method Ref: SW-846 3570**		**Revision:**		**Date: 2002**	
1429	MICROSCALE SOLVENT EXTRACTION	02/21/2017	09/05/2020	SC	PRE	NE
Method Code: 10143007	**Method Ref: SW-846 3580A**		**Revision: 1**		**Date: 1992**	
1470	WASTE DILUTION	02/21/2017	09/05/2020	SC	PRE	NE
Method Code: 10146802	**Method Ref: SW-846 3630C**		**Revision: 3**		**Date: 1996**	
1446	SILICA GEL CLEAN-UP	02/21/2017	09/05/2020	SC	PRE	NE
Method Code: 10147203	**Method Ref: SW-846 3640A**		**Revision: 1**		**Date: 1994**	
1418	GEL-PERMEATION CLEAN-UP	02/21/2017	09/05/2020	SC	PRE	NE

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Method Code: 10148400	**Method Ref: SW-846 3660B**		**Revision: 2**		**Date: 1996**	
1456	SULFUR CLEAN-UP	02/21/2017	09/05/2020	SC	PRE	NE
Method Code: 10148808	**Method Ref: SW-846 3665A**		**Revision: 1**		**Date: 1996**	
1458	SULFURIC ACID / PERMANGANATE CLEAN-UP	02/21/2017	09/05/2020	SC	PRE	NE
Method Code: 10186002	**Method Ref: SW-846 8270D**		**Revision:**		**Date: 2007**	
5155	1,2,4-TRICHLOROBENZENE	02/21/2017	09/05/2020	SC	VOC	NE
4610	1,2-DICHLOROBENZENE	02/21/2017	09/05/2020	SC	VOC	NE
4615	1,3-DICHLOROBENZENE	02/21/2017	09/05/2020	SC	VOC	NE
4620	1,4-DICHLOROBENZENE	02/21/2017	09/05/2020	SC	VOC	NE
4835	HEXACHLOROBUTADIENE	02/21/2017	09/05/2020	SC	VOC	NE
4840	HEXACHLOROETHANE	02/21/2017	09/05/2020	SC	VOC	NE
5005	NAPHTHALENE	02/21/2017	09/05/2020	SC	VOC	NE
5015	NITROBENZENE	02/21/2017	09/05/2020	SC	VOC	NE
5095	PYRIDINE	02/21/2017	09/05/2020	SC	VOC	NE
Method Code: 10242509	**Method Ref: SW-846 8270D SIM**		**Revision:**		**Date: 2007**	
4735	1,4-DIOXANE (1 4-DIETHYLENEOXIDE)	04/11/2019	09/05/2020	SC	VOC	NE
5005	NAPHTHALENE	02/21/2017	09/05/2020	SC	VOC	NE
Method Code: 10242543	**Method Ref: SW-846 8270E**		**Revision:**		**Date: 2017**	
5155	1,2,4-TRICHLOROBENZENE	07/13/2020	09/15/2020	SC	VOC	NE
4610	1,2-DICHLOROBENZENE	07/13/2020	09/15/2020	SC	VOC	NE
4615	1,3-DICHLOROBENZENE	07/13/2020	09/15/2020	SC	VOC	NE
4620	1,4-DICHLOROBENZENE	07/13/2020	09/15/2020	SC	VOC	NE
4835	HEXACHLOROBUTADIENE	07/13/2020	09/15/2020	SC	VOC	NE
4840	HEXACHLOROETHANE	07/13/2020	09/15/2020	SC	VOC	NE
5005	NAPHTHALENE	07/13/2020	09/15/2020	SC	VOC	NE
5015	NITROBENZENE	07/13/2020	09/15/2020	SC	VOC	NE
5095	PYRIDINE	07/13/2020	09/15/2020	SC	VOC	NE
Method Code: 10242565	**Method Ref: SW-846 8270E SIM**		**Revision:**		**Date: 2017**	
4735	1,4-DIOXANE (1 4-DIETHYLENEOXIDE)	07/13/2020	09/05/2020	SC	VOC	NE
5005	NAPHTHALENE	07/13/2020	09/05/2020	SC	VOC	NE
Method Code: 10305609	**Method Ref: SW-846 8015D**		**Revision:**		**Date: 2003**	
4750	ETHANOL	02/21/2017	09/05/2020	SC	VOC	NE
4785	ETHYLENE GLYCOL	02/21/2017	09/05/2020	SC	VOC	NE
4875	ISOBUTYL ALCOHOL (2-METHYL-1-PROPANOL)	02/21/2017	09/05/2020	SC	VOC	NE
4895	ISOPROPYL ALCOHOL (2-PROPANOL)	02/21/2017	09/05/2020	SC	VOC	NE
4930	METHANOL	02/21/2017	09/05/2020	SC	VOC	NE
5055	N-PROPANOL	02/21/2017	09/05/2020	SC	VOC	NE

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4368	T-AMYL ALCOHOL	02/21/2017	09/05/2020	SC	VOC	NE
4420	TERT-BUTYL ALCOHOL (TBA)	02/21/2017	09/05/2020	SC	VOC	NE
Method Code: 10178800	**Method Ref: SW-846 8081B**		**Revision:**		**Date: 2000**	
6275	HEXACHLOROBENZENE	02/21/2017	09/05/2020	SC	SBN	NE
Method Code: 10186002	**Method Ref: SW-846 8270D**		**Revision:**		**Date: 2007**	
6703	1,1'-BIPHENYL (BZ-0)	02/21/2017	09/05/2020	SC	SBN	NE
6715	1,2,4,5-TETRACHLOROBENZENE	02/21/2017	09/05/2020	SC	SBN	NE
6220	1,2-DIPHENYLHYDRAZINE	02/21/2017	09/05/2020	SC	SBN	NE
4659	2,2'-OXYBIS(1-CHLOROPROPANE)	02/21/2017	09/05/2020	SC	SBN	NE
6735	2,3,4,6-TETRACHLOROPHENOL	02/21/2017	09/05/2020	SC	SBN	NE
6835	2,4,5-TRICHLOROPHENOL	02/21/2017	09/05/2020	SC	SBN	NE
6840	2,4,6-TRICHLOROPHENOL	02/21/2017	09/05/2020	SC	SBN	NE
6000	2,4-DICHLOROPHENOL	02/21/2017	09/05/2020	SC	SBN	NE
6130	2,4-DIMETHYLPHENOL	02/21/2017	09/05/2020	SC	SBN	NE
6175	2,4-DINITROPHENOL	02/21/2017	09/05/2020	SC	SBN	NE
6185	2,4-DINITROTOLUENE (2 4-DNT)	02/21/2017	09/05/2020	SC	SBN	NE
6190	2,6-DINITROTOLUENE (2 6-DNT)	02/21/2017	09/05/2020	SC	SBN	NE
5795	2-CHLORONAPHTHALENE	02/21/2017	09/05/2020	SC	SBN	NE
5800	2-CHLOROPHENOL	02/21/2017	09/05/2020	SC	SBN	NE
6360	2-METHYL-4,6-DINITROPHENOL	02/21/2017	09/05/2020	SC	SBN	NE
6385	2-METHYLNAPHTHALENE	02/21/2017	09/05/2020	SC	SBN	NE
6400	2-METHYLPHENOL (O-CRESOL)	02/21/2017	09/05/2020	SC	SBN	NE
6460	2-NITROANILINE	02/21/2017	09/05/2020	SC	SBN	NE
6490	2-NITROPHENOL	02/21/2017	09/05/2020	SC	SBN	NE
5945	3,3-DICHLOROBENZIDINE	02/21/2017	09/05/2020	SC	SBN	NE
6405	3-METHYLPHENOL (M-CRESOL)	02/21/2017	09/05/2020	SC	SBN	NE
6465	3-NITROANILINE	02/21/2017	09/05/2020	SC	SBN	NE
5660	4-BROMOPHENYL PHENYL ETHER	02/21/2017	09/05/2020	SC	SBN	NE
5700	4-CHLORO-3-METHYLPHENOL	02/21/2017	09/05/2020	SC	SBN	NE
5745	4-CHLOROANILINE	02/21/2017	09/05/2020	SC	SBN	NE
5825	4-CHLOROPHENYL PHENYL ETHER	02/21/2017	09/05/2020	SC	SBN	NE
6410	4-METHYLPHENOL (P-CRESOL)	02/21/2017	09/05/2020	SC	SBN	NE
6470	4-NITROANILINE	02/21/2017	09/05/2020	SC	SBN	NE
6500	4-NITROPHENOL	02/21/2017	09/05/2020	SC	SBN	NE
5500	ACENAPHTHENE	02/21/2017	09/05/2020	SC	SBN	NE
5505	ACENAPHTHYLENE	02/21/2017	09/05/2020	SC	SBN	NE

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5510	ACETOPHENONE	02/21/2017	09/05/2020	SC	SBN	NE
5545	ANILINE	02/21/2017	09/05/2020	SC	SBN	NE
5555	ANTHRACENE	02/21/2017	09/05/2020	SC	SBN	NE
5570	BENZALDEHYDE	02/21/2017	09/05/2020	SC	SBN	NE
5595	BENZIDINE	02/21/2017	09/05/2020	SC	SBN	NE
5575	BENZO(A)ANTHRACENE	02/21/2017	09/05/2020	SC	SBN	NE
5580	BENZO(A)PYRENE	02/21/2017	09/05/2020	SC	SBN	NE
5585	BENZO(B)FLUORANTHENE	02/21/2017	09/05/2020	SC	SBN	NE
5590	BENZO(G,H,I)PERYLENE	02/21/2017	09/05/2020	SC	SBN	NE
5600	BENZO(K)FLUORANTHENE	02/21/2017	09/05/2020	SC	SBN	NE
5610	BENZOIC ACID	02/21/2017	09/05/2020	SC	SBN	NE
5630	BENZYL ALCOHOL	02/21/2017	09/05/2020	SC	SBN	NE
5670	BENZYL BUTYL PHTHALATE	02/21/2017	09/05/2020	SC	SBN	NE
5760	BIS(2-CHLOROETHOXY) METHANE	02/21/2017	09/05/2020	SC	SBN	NE
5765	BIS(2-CHLOROETHYL) ETHER	02/21/2017	09/05/2020	SC	SBN	NE
6065	BIS(2-ETHYLHEXYL) PHTHALATE	02/21/2017	09/05/2020	SC	SBN	NE
5680	CARBAZOLE	02/21/2017	09/05/2020	SC	SBN	NE
5855	CHRYSENE	02/21/2017	09/05/2020	SC	SBN	NE
5925	DI-N-BUTYL PHTHALATE	02/21/2017	09/05/2020	SC	SBN	NE
6200	DI-N-OCTYL PHTHALATE	02/21/2017	09/05/2020	SC	SBN	NE
5895	DIBENZO(A,H)ANTHRACENE	02/21/2017	09/05/2020	SC	SBN	NE
5905	DIBENZOFURAN	02/21/2017	09/05/2020	SC	SBN	NE
6070	DIETHYL PHTHALATE	02/21/2017	09/05/2020	SC	SBN	NE
6135	DIMETHYL PHTHALATE	02/21/2017	09/05/2020	SC	SBN	NE
6265	FLUORANTHENE	02/21/2017	09/05/2020	SC	SBN	NE
6270	FLUORENE	02/21/2017	09/05/2020	SC	SBN	NE
6275	HEXACHLOROBENZENE	02/21/2017	09/05/2020	SC	SBN	NE
6315	INDENO(1,2,3-CD)PYRENE	02/21/2017	09/05/2020	SC	SBN	NE
6320	ISOPHORONE	02/21/2017	09/05/2020	SC	SBN	NE
6545	N-NITROSODI-N-PROPYLAMINE	02/21/2017	09/05/2020	SC	SBN	NE
6530	N-NITROSODIMETHYLAMINE	02/21/2017	09/05/2020	SC	SBN	NE
6535	N-NITROSODIPHENYLAMINE	02/21/2017	09/05/2020	SC	SBN	NE
6600	PCNB (PENTACHLORONITROBENZENE)	02/21/2017	09/05/2020	SC	SBN	NE
6605	PENTACHLOROPHENOL	02/21/2017	09/05/2020	SC	SBN	NE
6615	PHENANTHRENE	02/21/2017	09/05/2020	SC	SBN	NE
6625	PHENOL	02/21/2017	09/05/2020	SC	SBN	NE

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6665	PYRENE	02/21/2017	09/05/2020	SC	SBN	NE
Method Code: 10242509	**Method Ref: SW-846 8270D SIM**		**Revision:**		**Date: 2007**	
6380	1-METHYLNAPHTHALENE	02/21/2017	09/05/2020	SC	SBN	NE
6385	2-METHYLNAPHTHALENE	02/21/2017	09/05/2020	SC	SBN	NE
5500	ACENAPHTHENE	02/21/2017	09/05/2020	SC	SBN	NE
5505	ACENAPHTHYLENE	02/21/2017	09/05/2020	SC	SBN	NE
9297	ALKALATED PAHS	02/21/2017	09/05/2020	SC	SBN	NE
5555	ANTHRACENE	02/21/2017	09/05/2020	SC	SBN	NE
5575	BENZO(A)ANTHRACENE	02/21/2017	09/05/2020	SC	SBN	NE
5580	BENZO(A)PYRENE	02/21/2017	09/05/2020	SC	SBN	NE
5585	BENZO(B)FLUORANTHENE	02/21/2017	09/05/2020	SC	SBN	NE
5590	BENZO(G,H,I)PERYLENE	02/21/2017	09/05/2020	SC	SBN	NE
5600	BENZO(K)FLUORANTHENE	02/21/2017	09/05/2020	SC	SBN	NE
5855	CHRYSENE	02/21/2017	09/05/2020	SC	SBN	NE
5895	DIBENZO(A,H)ANTHRACENE	02/21/2017	09/05/2020	SC	SBN	NE
6265	FLUORANTHENE	02/21/2017	09/05/2020	SC	SBN	NE
6270	FLUORENE	02/21/2017	09/05/2020	SC	SBN	NE
6315	INDENO(1,2,3-CD)PYRENE	02/21/2017	09/05/2020	SC	SBN	NE
6605	PENTACHLOROPHENOL	02/21/2017	09/05/2020	SC	SBN	NE
6615	PHENANTHRENE	02/21/2017	09/05/2020	SC	SBN	NE
6665	PYRENE	02/21/2017	09/05/2020	SC	SBN	NE
Method Code: 10242543	**Method Ref: SW-846 8270E**		**Revision:**		**Date: 2017**	
6703	1,1'-BIPHENYL (BZ-0)	07/13/2020	09/15/2020	SC	SBN	NE
6715	1,2,4,5-TETRACHLOROBENZENE	07/13/2020	09/15/2020	SC	SBN	NE
6220	1,2-DIPHENYLHYDRAZINE	07/13/2020	09/15/2020	SC	SBN	NE
4659	2,2'-OXYBIS(1-CHLOROPROPANE)	07/13/2020	09/15/2020	SC	SBN	NE
6735	2,3,4,6-TETRACHLOROPHENOL	07/13/2020	09/15/2020	SC	SBN	NE
6835	2,4,5-TRICHLOROPHENOL	07/13/2020	09/15/2020	SC	SBN	NE
6840	2,4,6-TRICHLOROPHENOL	07/13/2020	09/15/2020	SC	SBN	NE
6000	2,4-DICHLOROPHENOL	07/13/2020	09/15/2020	SC	SBN	NE
6130	2,4-DIMETHYLPHENOL	07/13/2020	09/15/2020	SC	SBN	NE
6175	2,4-DINITROPHENOL	07/13/2020	09/15/2020	SC	SBN	NE
6185	2,4-DINITROTOLUENE (2 4-DNT)	07/13/2020	09/15/2020	SC	SBN	NE
6190	2,6-DINITROTOLUENE (2 6-DNT)	07/13/2020	09/15/2020	SC	SBN	NE
5795	2-CHLORONAPHTHALENE	07/13/2020	09/15/2020	SC	SBN	NE
5800	2-CHLOROPHENOL	07/13/2020	09/15/2020	SC	SBN	NE

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6360	2-METHYL-4,6-DINITROPHENOL	07/13/2020	09/15/2020	SC	SBN	NE
6385	2-METHYLNAPHTHALENE	07/13/2020	09/15/2020	SC	SBN	NE
6400	2-METHYLPHENOL (O-CRESOL)	07/13/2020	09/15/2020	SC	SBN	NE
6460	2-NITROANILINE	07/13/2020	09/15/2020	SC	SBN	NE
6490	2-NITROPHENOL	07/13/2020	09/15/2020	SC	SBN	NE
5945	3,3-DICHLOROBENZIDINE	07/13/2020	09/15/2020	SC	SBN	NE
6405	3-METHYLPHENOL (M-CRESOL)	07/13/2020	09/15/2020	SC	SBN	NE
6465	3-NITROANILINE	07/13/2020	09/15/2020	SC	SBN	NE
5660	4-BROMOPHENYL PHENYL ETHER	07/13/2020	09/15/2020	SC	SBN	NE
5700	4-CHLORO-3-METHYLPHENOL	07/13/2020	09/15/2020	SC	SBN	NE
5745	4-CHLOROANILINE	07/13/2020	09/15/2020	SC	SBN	NE
5825	4-CHLOROPHENYL PHENYL ETHER	07/13/2020	09/15/2020	SC	SBN	NE
6410	4-METHYLPHENOL (P-CRESOL)	07/13/2020	09/15/2020	SC	SBN	NE
6470	4-NITROANILINE	07/13/2020	09/15/2020	SC	SBN	NE
6500	4-NITROPHENOL	07/13/2020	09/15/2020	SC	SBN	NE
5500	ACENAPHTHENE	07/13/2020	09/15/2020	SC	SBN	NE
5505	ACENAPHTHYLENE	07/13/2020	09/15/2020	SC	SBN	NE
5510	ACETOPHENONE	07/13/2020	09/15/2020	SC	SBN	NE
5545	ANILINE	07/13/2020	09/15/2020	SC	SBN	NE
5555	ANTHRACENE	07/13/2020	09/15/2020	SC	SBN	NE
5570	BENZALDEHYDE	07/13/2020	09/15/2020	SC	SBN	NE
5595	BENZIDINE	07/13/2020	09/15/2020	SC	SBN	NE
5575	BENZO(A)ANTHRACENE	07/13/2020	09/15/2020	SC	SBN	NE
5580	BENZO(A)PYRENE	07/13/2020	09/15/2020	SC	SBN	NE
5585	BENZO(B)FLUORANTHENE	07/13/2020	09/15/2020	SC	SBN	NE
5590	BENZO(G,H,I)PERYLENE	07/13/2020	09/15/2020	SC	SBN	NE
5600	BENZO(K)FLUORANTHENE	07/13/2020	09/15/2020	SC	SBN	NE
5610	BENZOIC ACID	07/13/2020	09/15/2020	SC	SBN	NE
5630	BENZYL ALCOHOL	07/13/2020	09/15/2020	SC	SBN	NE
5670	BENZYL BUTYL PHTHALATE	07/13/2020	09/15/2020	SC	SBN	NE
5760	BIS(2-CHLOROETHOXY) METHANE	07/13/2020	09/15/2020	SC	SBN	NE
5765	BIS(2-CHLOROETHYL) ETHER	07/13/2020	09/15/2020	SC	SBN	NE
6065	BIS(2-ETHYLHEXYL) PHTHALATE	07/13/2020	09/15/2020	SC	SBN	NE
5680	CARBAZOLE	07/13/2020	09/15/2020	SC	SBN	NE
5855	CHRYSENE	07/13/2020	09/15/2020	SC	SBN	NE
5925	DI-N-BUTYL PHTHALATE	07/13/2020	09/15/2020	SC	SBN	NE

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6200	DI-N-OCTYL PHTHALATE	07/13/2020	09/15/2020	SC	SBN	NE
5895	DIBENZO(A,H)ANTHRACENE	07/13/2020	09/15/2020	SC	SBN	NE
5905	DIBENZOFURAN	07/13/2020	09/15/2020	SC	SBN	NE
6070	DIETHYL PHTHALATE	07/13/2020	09/15/2020	SC	SBN	NE
6135	DIMETHYL PHTHALATE	07/13/2020	09/15/2020	SC	SBN	NE
6265	FLUORANTHENE	07/13/2020	09/15/2020	SC	SBN	NE
6270	FLUORENE	07/13/2020	09/15/2020	SC	SBN	NE
6275	HEXACHLOROBENZENE	07/13/2020	09/15/2020	SC	SBN	NE
6315	INDENO(1,2,3-CD)PYRENE	07/13/2020	09/15/2020	SC	SBN	NE
6320	ISOPHORONE	07/13/2020	09/15/2020	SC	SBN	NE
6545	N-NITROSODI-N-PROPYLAMINE	07/13/2020	09/15/2020	SC	SBN	NE
6530	N-NITROSODIMETHYLAMINE	07/13/2020	09/15/2020	SC	SBN	NE
6535	N-NITROSODIPHENYLAMINE	07/13/2020	09/15/2020	SC	SBN	NE
6600	PCNB (PENTACHLORONITROBENZENE)	07/13/2020	09/15/2020	SC	SBN	NE
6605	PENTACHLOROPHENOL	07/13/2020	09/15/2020	SC	SBN	NE
6615	PHENANTHRENE	07/13/2020	09/15/2020	SC	SBN	NE
6625	PHENOL	07/13/2020	09/15/2020	SC	SBN	NE
6665	PYRENE	07/13/2020	09/15/2020	SC	SBN	NE
Method Code: 10242565	**Method Ref: SW-846 8270E SIM**		**Revision:**		**Date: 2017**	
6380	1-METHYLNAPHTHALENE	07/13/2020	09/05/2020	SC	SBN	NE
6385	2-METHYLNAPHTHALENE	07/13/2020	09/05/2020	SC	SBN	NE
5500	ACENAPHTHENE	07/13/2020	09/05/2020	SC	SBN	NE
5505	ACENAPHTHYLENE	07/13/2020	09/05/2020	SC	SBN	NE
9297	ALKALATED PAHS	07/13/2020	09/05/2020	SC	SBN	NE
5555	ANTHRACENE	07/13/2020	09/05/2020	SC	SBN	NE
5575	BENZO(A)ANTHRACENE	07/13/2020	09/05/2020	SC	SBN	NE
5580	BENZO(A)PYRENE	07/13/2020	09/05/2020	SC	SBN	NE
5585	BENZO(B)FLUORANTHENE	07/13/2020	09/05/2020	SC	SBN	NE
5590	BENZO(G,H,I)PERYLENE	07/13/2020	09/05/2020	SC	SBN	NE
5600	BENZO(K)FLUORANTHENE	07/13/2020	09/05/2020	SC	SBN	NE
5855	CHRYSENE	07/13/2020	09/05/2020	SC	SBN	NE
5895	DIBENZO(A,H)ANTHRACENE	07/13/2020	09/05/2020	SC	SBN	NE
6265	FLUORANTHENE	07/13/2020	09/05/2020	SC	SBN	NE
6270	FLUORENE	07/13/2020	09/05/2020	SC	SBN	NE
6315	INDENO(1,2,3-CD)PYRENE	07/13/2020	09/05/2020	SC	SBN	NE
6605	PENTACHLOROPHENOL	07/13/2020	09/05/2020	SC	SBN	NE

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6615	PHENANTHRENE	07/13/2020	09/05/2020	SC	SBN	NE
6665	PYRENE	07/13/2020	09/05/2020	SC	SBN	NE
Method Code: 10305609	**Method Ref: SW-846 8015D**		**Revision:**		**Date: 2003**	
6657	PROPYLENE GLYCOL	02/21/2017	09/05/2020	SC	SBN	NE
Method Code: 10120602	**Method Ref: EPA 1613B**		**Revision:**		**Date: 1994**	
9519	1,2,3,4,6,7,8,9-OCTACHLORODIBENZO-P-DIOXIN (OCDD)	11/01/2018	09/05/2020	SC	SNO	NE
9516	1,2,3,4,6,7,8,9-OCTACHLORODIBENZOFURAN (OCDF)	11/01/2018	09/05/2020	SC	SNO	NE
9426	1,2,3,4,6,7,8-HEPTACHLORODIBENZO-P-DIOXIN (1,2,3,4,6,7,8-HPCDD)	11/01/2018	09/05/2020	SC	SNO	NE
9420	1,2,3,4,6,7,8-HEPTACHLORODIBENZOFURAN (1,2,3,4,6,7,8-HPCDF)	11/01/2018	09/05/2020	SC	SNO	NE
9423	1,2,3,4,7,8,9-HEPTACHLORODIBENZOFURAN (1,2,3,4,7,8,9-HPCDF)	11/01/2018	09/05/2020	SC	SNO	NE
9453	1,2,3,4,7,8-HEXACHLORODIBENZO-P-DIOXIN (1,2,3,4,7,8-HXCDD)	11/01/2018	09/05/2020	SC	SNO	NE
9471	1,2,3,4,7,8-HEXACHLORODIBENZOFURAN (1,2,3,4,7,8-HXCDF)	11/01/2018	09/05/2020	SC	SNO	NE
9456	1,2,3,6,7,8-HEXACHLORODIBENZO-P-DIOXIN(1,2,3,6,7,8-HXCDD)	11/01/2018	09/05/2020	SC	SNO	NE
9474	1,2,3,6,7,8-HEXACHLORODIBENZOFURAN (1,2,3,6,7,8-HXCDF)	11/01/2018	09/05/2020	SC	SNO	NE
9459	1,2,3,7,8,9-HEXACHLORODIBENZO-P-DIOXIN (1,2,3,7,8,9-HXCDD)	11/01/2018	09/05/2020	SC	SNO	NE
9477	1,2,3,7,8,9-HEXACHLORODIBENZOFURAN (1,2,3,7,8,9-HXCDF)	11/01/2018	09/05/2020	SC	SNO	NE
9540	1,2,3,7,8-PENTACHLORODIBENZO-P-DIOXIN (1,2,3,7,8-PECDD)	11/01/2018	09/05/2020	SC	SNO	NE
9543	1,2,3,7,8-PENTACHLORODIBENZOFURAN (1,2,3,7,8-PECDF)	11/01/2018	09/05/2020	SC	SNO	NE
9480	2,3,4,6,7,8-HEXACHLORODIBENZOFURAN	11/01/2018	09/05/2020	SC	SNO	NE
9549	2,3,4,7,8-PENTACHLORODIBENZOFURAN	11/01/2018	09/05/2020	SC	SNO	NE
9618	2,3,7,8-TETRACHLORODIBENZO- P-DIOXIN (2,3,7,8-TCDD)	11/01/2018	09/05/2020	SC	SNO	NE
9612	2,3,7,8-TETRACHLORODIBENZOFURAN (TCDF)	11/01/2018	09/05/2020	SC	SNO	NE
9438	TOTAL HEPTACHLORODIBENZO-P-DIOXIN (TOTAL HPCDD)	11/01/2018	09/05/2020	SC	SNO	NE
9444	TOTAL HEPTACHLORODIBENZOFURAN (TOTAL HPCDF)	11/01/2018	09/05/2020	SC	SNO	NE
9468	TOTAL HEXACHLORODIBENZO-P-DIOXIN (TOTAL HXCDD)	11/01/2018	09/05/2020	SC	SNO	NE
9483	TOTAL HEXACHLORODIBENZOFURAN (TOTAL HXCDF)	11/01/2018	09/05/2020	SC	SNO	NE
9555	TOTAL PENTACHLORODIBENZO-P-DIOXIN (TOTAL PECDD)	11/01/2018	09/05/2020	SC	SNO	NE
9552	TOTAL PENTACHLORODIBENZOFURAN (TOTAL PECDF)	11/01/2018	09/05/2020	SC	SNO	NE
9660	TOTAL POLYCHLORINATED DIBENZO-P-DIOXINS (TOTAL PCDDS)	11/01/2018	09/05/2020	SC	SNO	NE
9657	TOTAL POLYCHLORINATED DIBENZOFURANS (TOTAL PCDFS)	11/01/2018	09/05/2020	SC	SNO	NE
9609	TOTAL TETRACHLORODIBENZO-P-DIOXIN (TOTAL TCDD)	11/01/2018	09/05/2020	SC	SNO	NE

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9615	TOTAL TETRACHLORODIBENZOFURAN (TOTAL TCDF)	11/01/2018	09/05/2020	SC	SNO	NE
Method Code: 10187403	**Method Ref: EPA 8290A**		**Revision:**		**Date: 2007**	
9519	1,2,3,4,6,7,8,9-OCTACHLORODIBENZO-P-DIOXIN (OCDD)	11/01/2018	09/05/2020	SC	SNO	NE
9516	1,2,3,4,6,7,8,9-OCTACHLORODIBENZOFURAN (OCDF)	11/01/2018	09/05/2020	SC	SNO	NE
9426	1,2,3,4,6,7,8-HEPTACHLORODIBENZO-P-DIOXIN (1,2,3,4,6,7,8-HPCDD)	11/01/2018	09/05/2020	SC	SNO	NE
9420	1,2,3,4,6,7,8-HEPTACHLORODIBENZOFURAN (1,2,3,4,6,7,8-HPCDF)	11/01/2018	09/05/2020	SC	SNO	NE
9423	1,2,3,4,7,8,9-HEPTACHLORODIBENZOFURAN (1,2,3,4,7,8,9-HPCDF)	11/01/2018	09/05/2020	SC	SNO	NE
9453	1,2,3,4,7,8-HEXACHLORODIBENZO-P-DIOXIN (1,2,3,4,7,8-HXCDD)	11/01/2018	09/05/2020	SC	SNO	NE
9471	1,2,3,4,7,8-HEXACHLORODIBENZOFURAN (1,2,3,4,7,8-HXCDF)	11/01/2018	09/05/2020	SC	SNO	NE
9456	1,2,3,6,7,8-HEXACHLORODIBENZO-P-DIOXIN(1,2,3,6,7,8-HXCDD)	11/01/2018	09/05/2020	SC	SNO	NE
9474	1,2,3,6,7,8-HEXACHLORODIBENZOFURAN (1,2,3,6,7,8-HXCDF)	11/01/2018	09/05/2020	SC	SNO	NE
9459	1,2,3,7,8,9-HEXACHLORODIBENZO-P-DIOXIN (1,2,3,7,8,9-HXCDD)	11/01/2018	09/05/2020	SC	SNO	NE
9477	1,2,3,7,8,9-HEXACHLORODIBENZOFURAN (1,2,3,7,8,9-HXCDF)	11/01/2018	09/05/2020	SC	SNO	NE
9540	1,2,3,7,8-PENTACHLORODIBENZO-P-DIOXIN (1,2,3,7,8-PECDD)	11/01/2018	09/05/2020	SC	SNO	NE
9543	1,2,3,7,8-PENTACHLORODIBENZOFURAN (1,2,3,7,8-PECDF)	11/01/2018	09/05/2020	SC	SNO	NE
9480	2,3,4,6,7,8-HEXACHLORODIBENZOFURAN	11/01/2018	09/05/2020	SC	SNO	NE
9549	2,3,4,7,8-PENTACHLORODIBENZOFURAN	11/01/2018	09/05/2020	SC	SNO	NE
9618	2,3,7,8-TETRACHLORODIBENZO- P-DIOXIN (2,3,7,8-TCDD)	11/01/2018	09/05/2020	SC	SNO	NE
9612	2,3,7,8-TETRACHLORODIBENZOFURAN (TCDF)	11/01/2018	09/05/2020	SC	SNO	NE
9438	TOTAL HEPTACHLORODIBENZO-P-DIOXIN (TOTAL HPCDD)	11/01/2018	09/05/2020	SC	SNO	NE
9444	TOTAL HEPTACHLORODIBENZOFURAN (TOTAL HPCDF)	11/01/2018	09/05/2020	SC	SNO	NE
9468	TOTAL HEXACHLORODIBENZO-P-DIOXIN (TOTAL HXCDD)	11/01/2018	09/05/2020	SC	SNO	NE
9483	TOTAL HEXACHLORODIBENZOFURAN (TOTAL HXCDF)	11/01/2018	09/05/2020	SC	SNO	NE
9555	TOTAL PENTACHLORODIBENZO-P-DIOXIN (TOTAL PECDD)	11/01/2018	09/05/2020	SC	SNO	NE
9552	TOTAL PENTACHLORODIBENZOFURAN (TOTAL PECDF)	11/01/2018	09/05/2020	SC	SNO	NE
9660	TOTAL POLYCHLORINATED DIBENZO-P-DIOXINS (TOTAL PCDDS)	11/01/2018	09/05/2020	SC	SNO	NE
9657	TOTAL POLYCHLORINATED DIBENZOFURANS (TOTAL PCDFS)	11/01/2018	09/05/2020	SC	SNO	NE
9609	TOTAL TETRACHLORODIBENZO-P-DIOXIN (TOTAL TCDD)	11/01/2018	09/05/2020	SC	SNO	NE
9615	TOTAL TETRACHLORODIBENZOFURAN (TOTAL TCDF)	11/01/2018	09/05/2020	SC	SNO	NE
Method Code: 10305609	**Method Ref: SW-846 8015D**		**Revision:**		**Date: 2003**	
9369	DIESEL RANGE ORGANICS (DRO)	02/21/2017	09/05/2020	SC	SNO	NE

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NEW HAMPSHIRE ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM

29 Hazen Drive, PO Box 95, Concord, NH 03302 (603) 271-2998

PRIMARY ACCREDITATION ANALYTE LIST

ANALYTE LIST NUMBER: 206219-F

ALPHA ANALYTICAL (MANSFIELD) [#2062]
320 FORBES BLVD

MANSFIELD MA 02048
508-822-9300
Lab ID: 2062

Method Code: 10179201	**Method Ref: SW-846 8082A**		**Revision:**		**Date: 2000**	
9095	2,2',3,3',4,4',5,5',6-NONACHLOROBIPHENYL (BZ-206)	02/21/2017	09/05/2020	SC	SPC	NE
9065	2,2',3,3',4,4',5-HEPTACHLOROBIPHENYL (BZ-170)	02/21/2017	09/05/2020	SC	SPC	NE
9020	2,2',3,3',4,4'-HEXACHLOROBIPHENYL (BZ-128)	02/21/2017	09/05/2020	SC	SPC	NE
9080	2,2',3,4',5,5',6-HEPTACHLOROBIPHENYL (BZ-187)	02/21/2017	09/05/2020	SC	SPC	NE
9025	2,2',3,4,4', 5'-HEXACHLOROBIPHENYL (BZ-138)	02/21/2017	09/05/2020	SC	SPC	NE
9075	2,2',3,4,4',5',6-HEPTACHLOROBIPHENYL (BZ-183)	02/21/2017	09/05/2020	SC	SPC	NE
9134	2,2',3,4,4',5,5'-HEPTACHLORBIPHENYL (BZ-180)	02/21/2017	09/05/2020	SC	SPC	NE
8975	2,2',3,4,5'-PENTACHLOROBIPHENYL (BZ-87)	02/21/2017	09/05/2020	SC	SPC	NE
9030	2,2',3,4,5,5'-HEXACHLOROBIPHENYL (BZ-141)	02/21/2017	09/05/2020	SC	SPC	NE
8945	2,2',3,5'-TETRACHLOROBIPHENYL (BZ-44)	02/21/2017	09/05/2020	SC	SPC	NE
9035	2,2',3,5,5',6-HEXACHLOROBIPHENYL (BZ-151)	02/21/2017	09/05/2020	SC	SPC	NE
9040	2,2',4,4',5,5'-HEXACHLOROBIPHENYL (BZ-153)	02/21/2017	09/05/2020	SC	SPC	NE
8980	2,2',4,5,5'-PENTACHLOROBIPHENYL (BZ-101)	02/21/2017	09/05/2020	SC	SPC	NE
8955	2,2',5,5'-TETRACHLOROBIPHENYL (BZ-52)	02/21/2017	09/05/2020	SC	SPC	NE
8930	2,2',5-TRICHLOROBIPHENYL (BZ-18)	02/21/2017	09/05/2020	SC	SPC	NE
8995	2,3',4,4',5-PENTACHLOROBIPHENYL (BZ-118)	02/21/2017	09/05/2020	SC	SPC	NE
8960	2,3',4,4'-TETRACHLOROBIPHENYL (BZ-66)	02/21/2017	09/05/2020	SC	SPC	NE
8990	2,3,3',4',6-PENTACHLOROBIPHENYL (BZ-110)	02/21/2017	09/05/2020	SC	SPC	NE
8920	2,3-DICHLOROBIPHENYL (BZ-5)	02/21/2017	09/05/2020	SC	SPC	NE
8940	2,4',5-TRICHLOROBIPHENYL (BZ-31)	02/21/2017	09/05/2020	SC	SPC	NE
8915	2-CHLOROBIPHENYL (BZ-1)	02/21/2017	09/05/2020	SC	SPC	NE
8880	AROCLOR-1016 (PCB-1016)	02/21/2017	09/05/2020	SC	SPC	NE
8885	AROCLOR-1221 (PCB-1221)	02/21/2017	09/05/2020	SC	SPC	NE
8890	AROCLOR-1232 (PCB-1232)	02/21/2017	09/05/2020	SC	SPC	NE
8895	AROCLOR-1242 (PCB-1242)	02/21/2017	09/05/2020	SC	SPC	NE
8900	AROCLOR-1248 (PCB-1248)	02/21/2017	09/05/2020	SC	SPC	NE
8905	AROCLOR-1254 (PCB-1254)	02/21/2017	09/05/2020	SC	SPC	NE
8910	AROCLOR-1260 (PCB-1260)	02/21/2017	09/05/2020	SC	SPC	NE
8912	AROCLOR-1262 (PCB-1262)	02/21/2017	09/05/2020	SC	SPC	NE
8913	AROCLOR-1268 (PCB-1268)	02/21/2017	09/05/2020	SC	SPC	NE
Method Code: 10178800	**Method Ref: SW-846 8081B**		**Revision:**		**Date: 2000**	
7355	4,4-DDD	02/21/2017	09/05/2020	SC	SPE	NE
7360	4,4-DDE	02/21/2017	09/05/2020	SC	SPE	NE
7365	4,4-DDT	02/21/2017	09/05/2020	SC	SPE	NE
7025	ALDRIN	02/21/2017	09/05/2020	SC	SPE	NE

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ALPHA ANALYTICAL (MANSFIELD) [#2062]
320 FORBES BLVD

MANSFIELD MA 02048
508-822-9300
Lab ID: 2062

7110	ALPHA-BHC (ALPHA-HEXACHLOROCYCLOHEXANE)	02/21/2017	09/05/2020	SC	SPE	NE
7240	ALPHA-CHLORDANE	02/21/2017	09/05/2020	SC	SPE	NE
7115	BETA-BHC (BETA-HEXACHLOROCYCLOHEXANE)	02/21/2017	09/05/2020	SC	SPE	NE
7250	CHLORDANE (TECH.)	02/21/2017	09/05/2020	SC	SPE	NE
7105	DELTA-BHC	02/21/2017	09/05/2020	SC	SPE	NE
7470	DIELDRIN	02/21/2017	09/05/2020	SC	SPE	NE
7510	ENDOSULFAN I	02/21/2017	09/05/2020	SC	SPE	NE
7515	ENDOSULFAN II	02/21/2017	09/05/2020	SC	SPE	NE
7520	ENDOSULFAN SULFATE	02/21/2017	09/05/2020	SC	SPE	NE
7540	ENDRIN	02/21/2017	09/05/2020	SC	SPE	NE
7530	ENDRIN ALDEHYDE	02/21/2017	09/05/2020	SC	SPE	NE
7535	ENDRIN KETONE	02/21/2017	09/05/2020	SC	SPE	NE
7120	GAMMA-BHC (LINDANE)	02/21/2017	09/05/2020	SC	SPE	NE
7245	GAMMA-CHLORDANE	02/21/2017	09/05/2020	SC	SPE	NE
7685	HEPTACHLOR	02/21/2017	09/05/2020	SC	SPE	NE
7690	HEPTACHLOR EPOXIDE	02/21/2017	09/05/2020	SC	SPE	NE
7810	METHOXYCHLOR	02/21/2017	09/05/2020	SC	SPE	NE
7870	MIREX	02/21/2017	09/05/2020	SC	SPE	NE
8250	TOXAPHENE (CHLORINATED CAMPHENE)	02/21/2017	09/05/2020	SC	SPE	NE
Method Code: 10186002	**Method Ref: SW-846 8270D**		**Revision:**		**Date: 2007**	
7180	CAPROLACTAM	02/21/2017	09/05/2020	SC	SPE	NE
6285	HEXACHLOROCYCLOPENTADIENE	02/21/2017	09/05/2020	SC	SPE	NE
Method Code: 10242543	**Method Ref: SW-846 8270E**		**Revision:**		**Date: 2017**	
7180	CAPROLACTAM	07/13/2020	09/15/2020	SC	SPE	NE
6285	HEXACHLOROCYCLOPENTADIENE	07/13/2020	09/15/2020	SC	SPE	NE

ALPHA ANA 7/15/2020

Bill Hall
NH ELAP Program Manager
Issue Date: 07/15/2020

Matrix Legend: AE=Air; BT=Tissue; D=Drinking Water; N=Non-Potable Water; SC=Solid and Chemical Materials

Category Legend: MIC=Microbiology; MET=Metals; NMI=Non-Metal Inorganics; PRE=Preparation; VOC=Volatile Organic Compounds; SBN=SVOC-BNA; SHE=SVOC-Herbicides; SNO=SVOC-NOS; SPC=SVOC-PCB; SPE=SVOC-Pesticides; RAD=Radiochemistry; WET=Wet

Accreditation Legend: NE=NELAP; NH=NH State Certification; CE=State Certification; IN=Interim (NELAP); WI=Withdrawn; AP=Applied; RE=Revoked; SU=Suspended

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State of New Hampshire

Environmental Laboratory Accreditation Program

Awards

PRIMARY NH ELAP ACCREDITATION

to

ALPHA ANALYTICAL (WESTBOROUGH) [#2064]

of

WESTBOROUGH, MA

For the matrix, method and analytes listed on the latest Analyte List in accordance with the provisions on the 2009 TNI Standards and Env-C 300.

Certificate Number: 206420

Effective Date: 7/2/2020

Expiration Date: 7/1/2021

Laboratory ID: 2064

ALPHA AN7/1/2020

Bill Hall
NH ELAP Program Manager

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NEW HAMPSHIRE ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM

29 Hazen Drive, PO Box 95, Concord, NH 03302 (603) 271-2998

PRIMARY ACCREDITATION ANALYTE LIST

ANALYTE LIST NUMBER: 206420-A

ALPHA ANALYTICAL (WESTBOROUGH) [#2064]
8 WALKUP DRIVE

WESTBOROUGH MA 01581
508-898-9220
Lab ID: 2064

Analyte Code	Analyte Name	Effective Date	Expiration Date	Matrix	Category	Accr. Type
Method Code: 20181606	**Method Ref: SM 9215 B (PCA)-1994**		**Revision:**		**Date: 1994**	
2555	HETEROTROPHIC PLATE COUNT	05/10/2017	07/01/2021	D	MIC	NE
Method Code: 20212606	**Method Ref: SM 9223 B (COLILERT)-97**		**Revision: online**		**Date:**	
2525	ESCHERICHIA COLI	05/10/2017	07/01/2021	D	MIC	NE
2500	TOTAL COLIFORMS	05/10/2017	07/01/2021	D	MIC	NE
Method Code: 20213609	**Method Ref: SM 9223 B (COLILERT-18 QUANTI-TRAY)-1997**		**Revision:**		**Date: 1997**	
2525	ESCHERICHIA COLI	02/21/2017	07/01/2021	D	MIC	NE
2500	TOTAL COLIFORMS	02/21/2017	07/01/2021	D	MIC	NE
Method Code: 10011800	**Method Ref: EPA 180.1**		**Revision: 2**		**Date: 1993**	
2055	TURBIDITY	02/21/2017	07/01/2021	D	NMI	NE
Method Code: 10053200	**Method Ref: EPA 300.0**		**Revision: 2.1**		**Date: 1993**	
1575	CHLORIDE	02/21/2017	07/01/2021	D	NMI	NE
1730	FLUORIDE	02/21/2017	07/01/2021	D	NMI	NE
1810	NITRATE AS N	09/01/2017	07/01/2021	D	NMI	NE
2000	SULFATE	02/21/2017	07/01/2021	D	NMI	NE
Method Code: 10059742	**Method Ref: EPA 332**		**Revision: 1**		**Date: 2005**	
1895	PERCHLORATE	02/21/2017	07/01/2021	D	NMI	NE
Method Code: 10067604	**Method Ref: EPA 353.2**		**Revision: 2**		**Date: 1993**	
1810	NITRATE AS N	09/01/2017	07/01/2021	D	NMI	NE
1840	NITRITE AS N	09/01/2017	07/01/2021	D	NMI	NE
Method Code: 20039014	**Method Ref: SM 2120 B**		**Revision: 22ND ED**		**Date: 2011**	
1605	COLOR	10/31/2019	07/01/2021	D	NMI	NE
Method Code: 20039207	**Method Ref: SM 2120 B-1993**		**Revision:**		**Date: 1993**	
1605	COLOR	05/10/2017	07/01/2021	D	NMI	NE
Method Code: 20042619	**Method Ref: SM 2130 B**		**Revision: 22ND ED**		**Date: 2011**	
2055	TURBIDITY	10/31/2019	07/01/2021	D	NMI	NE
Method Code: 20042802	**Method Ref: SM 2130 B-1994**		**Revision:**		**Date: 1994**	
2055	TURBIDITY	05/10/2017	07/01/2021	D	NMI	NE
Method Code: 20043816	**Method Ref: SM 2150 B-2011**		**Revision:**		**Date: 2011**	
1855	ODOR	05/10/2017	07/01/2021	D	NMI	NE
Method Code: 20045618	**Method Ref: SM 2320 B-2011**		**Revision:**		**Date: 2011**	
1505	ALKALINITY	05/10/2017	07/01/2021	D	NMI	NE
Method Code: 20048617	**Method Ref: SM 2510 B-2011**		**Revision:**		**Date: 2011**	
1610	CONDUCTIVITY	05/10/2017	07/01/2021	D	NMI	NE
Method Code: 20050413	**Method Ref: SM 2540 C-2011**		**Revision:**		**Date: 2011**	
1955	RESIDUE, FILTERABLE (TDS)	05/10/2017	07/01/2021	D	NMI	NE

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NEW HAMPSHIRE ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM

29 Hazen Drive, PO Box 95, Concord, NH 03302 (603) 271-2998

PRIMARY ACCREDITATION ANALYTE LIST

ANALYTE LIST NUMBER: 206420-A

ALPHA ANALYTICAL (WESTBOROUGH) [#2064]
8 WALKUP DRIVE

WESTBOROUGH MA 01581
508-898-9220
Lab ID: 2064

Method Code: 20096213	**Method Ref: SM 4500-CN E**		**Revision: 22ND ED**		**Date: 2011**	
1645	CYANIDE, TOTAL	10/31/2019	07/01/2021	D	NMI	NE
Method Code: 20096406	**Method Ref: SM 4500-CN E-1997**		**Revision:**		**Date: 1997**	
1645	CYANIDE, TOTAL	05/10/2017	07/01/2021	D	NMI	NE
Method Code: 20102210	**Method Ref: SM 4500-F⁻ C**		**Revision: 22ND ED**		**Date: 2011**	
1730	FLUORIDE	10/31/2019	07/01/2021	D	NMI	NE
Method Code: 20102403	**Method Ref: SM 4500-F C-1997**		**Revision:**		**Date:**	
1730	FLUORIDE	05/10/2017	07/01/2021	D	NMI	NE
Method Code: 20105220	**Method Ref: SM 4500-H+ B-2011**		**Revision:**		**Date: 2011**	
1900	HYDROGEN ION (PH)	05/10/2017	07/01/2021	D	NMI	NE
Method Code: 20113115	**Method Ref: SM 4500-NO2 B-2011**		**Revision:**		**Date: 2011**	
1840	NITRITE AS N	07/17/2019	07/01/2021	D	NMI	NE
Method Code: 20116410	**Method Ref: SM 4500-NO3 F**		**Revision: 22ND ED**		**Date: 2011**	
1810	NITRATE AS N	10/31/2019	07/01/2021	D	NMI	NE
1820	NITRATE-NITRITE AS N	10/31/2019	07/01/2021	D	NMI	NE
1840	NITRITE AS N	10/31/2019	07/01/2021	D	NMI	NE
Method Code: 20117606	**Method Ref: SM 4500-NO3 F-1997**		**Revision:**		**Date:**	
1810	NITRATE AS N	05/10/2017	07/01/2021	D	NMI	NE
1820	NITRATE-NITRITE AS N	05/10/2017	07/01/2021	D	NMI	NE
1840	NITRITE AS N	05/10/2017	07/01/2021	D	NMI	NE
Method Code: 20124225	**Method Ref: SM 4500-P E-2011**		**Revision:**		**Date: 2011**	
1870	ORTHOPHOSPHATE AS P	09/01/2017	07/01/2021	D	NMI	NE
Method Code: 20138618	**Method Ref: SM 5310 C**		**Revision: 22ND ED**		**Date: 2011**	
1710	DISSOLVED ORGANIC CARBON (DOC)	10/31/2019	07/01/2021	D	NMI	NE
2040	TOTAL ORGANIC CARBON (TOC)	10/31/2019	07/01/2021	D	NMI	NE
Method Code: 20138801	**Method Ref: SM 5310 C-1996**		**Revision:**		**Date: 1996**	
1710	DISSOLVED ORGANIC CARBON (DOC)	09/01/2017	07/01/2021	D	NMI	NE
2040	TOTAL ORGANIC CARBON (TOC)	05/10/2017	07/01/2021	D	NMI	NE
Method Code: 20065663	**Method Ref: SM 4500-CN C-2011**		**Revision:**		**Date: 2011**	
1412	CYANIDE, MANUAL DISTILLATION	05/10/2017	07/01/2021	D	PRE	NE
Method Code: 10082801	**Method Ref: EPA 504.1**		**Revision: 1.1**		**Date: 1995**	
5180	1,2,3-TRICHLOROPROPANE	05/10/2019	07/01/2021	D	VOC	NE
4570	1,2-DIBROMO-3-CHLOROPROPANE (DBCP)	02/21/2017	07/01/2021	D	VOC	NE
4585	1,2-DIBROMOETHANE (EDB)	02/21/2017	07/01/2021	D	VOC	NE
Method Code: 10088809	**Method Ref: EPA 524.2**		**Revision: 4.1**		**Date: 1995**	
5105	1,1,1,2-TETRACHLOROETHANE	02/21/2017	07/01/2021	D	VOC	NE
5160	1,1,1-TRICHLOROETHANE	02/21/2017	07/01/2021	D	VOC	NE
5110	1,1,2,2-TETRACHLOROETHANE	02/21/2017	07/01/2021	D	VOC	NE
5165	1,1,2-TRICHLOROETHANE	02/21/2017	07/01/2021	D	VOC	NE

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ALPHA ANALYTICAL (WESTBOROUGH) [#2064]
8 WALKUP DRIVE

WESTBOROUGH MA 01581
508-898-9220
Lab ID: 2064

4630	1,1-DICHLOROETHANE	02/21/2017	07/01/2021	D	VOC	NE
4640	1,1-DICHLOROETHENE	02/21/2017	07/01/2021	D	VOC	NE
4670	1,1-DICHLOROPROPENE	02/21/2017	07/01/2021	D	VOC	NE
5150	1,2,3-TRICHLOROBENZENE	02/21/2017	07/01/2021	D	VOC	NE
5180	1,2,3-TRICHLOROPROPANE	02/21/2017	07/01/2021	D	VOC	NE
5155	1,2,4-TRICHLOROBENZENE	02/21/2017	07/01/2021	D	VOC	NE
5210	1,2,4-TRIMETHYLBENZENE	02/21/2017	07/01/2021	D	VOC	NE
4570	1,2-DIBROMO-3-CHLOROPROPANE (DBCP)	02/21/2017	07/01/2021	D	VOC	NE
4585	1,2-DIBROMOETHANE (EDB)	02/21/2017	07/01/2021	D	VOC	NE
4610	1,2-DICHLOROBENZENE	02/21/2017	07/01/2021	D	VOC	NE
4635	1,2-DICHLOROETHANE	02/21/2017	07/01/2021	D	VOC	NE
4655	1,2-DICHLOROPROPANE	02/21/2017	07/01/2021	D	VOC	NE
5215	1,3,5-TRIMETHYLBENZENE	02/21/2017	07/01/2021	D	VOC	NE
4615	1,3-DICHLOROBENZENE	02/21/2017	07/01/2021	D	VOC	NE
4660	1,3-DICHLOROPROPANE	02/21/2017	07/01/2021	D	VOC	NE
4620	1,4-DICHLOROBENZENE	02/21/2017	07/01/2021	D	VOC	NE
4665	2 2-DICHLOROPROPANE	02/21/2017	07/01/2021	D	VOC	NE
4410	2-BUTANONE (METHYL ETHYL KETONE MEK)	02/21/2017	07/01/2021	D	VOC	NE
4535	2-CHLOROTOLUENE	02/21/2017	07/01/2021	D	VOC	NE
4860	2-HEXANONE	02/21/2017	07/01/2021	D	VOC	NE
4540	4-CHLOROTOLUENE	02/21/2017	07/01/2021	D	VOC	NE
4910	4-ISOPROPYLTOLUENE (P-ISOPROPYLTOLUENE)	02/21/2017	07/01/2021	D	VOC	NE
4995	4-METHYL-2-PENTANONE (MIBK)	02/21/2017	07/01/2021	D	VOC	NE
4315	ACETONE	02/21/2017	07/01/2021	D	VOC	NE
4375	BENZENE	02/21/2017	07/01/2021	D	VOC	NE
4385	BROMOBENZENE	02/21/2017	07/01/2021	D	VOC	NE
4390	BROMOCHLOROMETHANE	02/21/2017	07/01/2021	D	VOC	NE
4395	BROMODICHLOROMETHANE	02/21/2017	07/01/2021	D	VOC	NE
4400	BROMOFORM	02/21/2017	07/01/2021	D	VOC	NE
4950	BROMOMETHANE (METHYL BROMIDE)	02/21/2017	07/01/2021	D	VOC	NE
4450	CARBON DISULFIDE	02/21/2017	07/01/2021	D	VOC	NE
4455	CARBON TETRACHLORIDE	02/21/2017	07/01/2021	D	VOC	NE
4475	CHLOROBENZENE	02/21/2017	07/01/2021	D	VOC	NE
4485	CHLOROETHANE	02/21/2017	07/01/2021	D	VOC	NE
4505	CHLOROFORM	02/21/2017	07/01/2021	D	VOC	NE
4960	CHLOROMETHANE (METHYL CHLORIDE)	02/21/2017	07/01/2021	D	VOC	NE

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4645	CIS-1 2-DICHLOROETHYLENE	02/21/2017	07/01/2021	D	VOC	NE
4680	CIS-1,3-DICHLOROPROPENE	02/21/2017	07/01/2021	D	VOC	NE
9375	DI-ISOPROPYLETHER (DIPE)	02/21/2017	07/01/2021	D	VOC	NE
4575	DIBROMOCHLOROMETHANE	02/21/2017	07/01/2021	D	VOC	NE
4595	DIBROMOMETHANE	02/21/2017	07/01/2021	D	VOC	NE
4625	DICHLORODIFLUOROMETHANE	02/21/2017	07/01/2021	D	VOC	NE
4975	DICHLOROMETHANE (METHYLENE CHLORIDE)	02/21/2017	07/01/2021	D	VOC	NE
4725	DIETHYL ETHER	02/21/2017	07/01/2021	D	VOC	NE
4770	ETHYL - T- BUTYL ETHER (ETBE)	02/21/2017	07/01/2021	D	VOC	NE
4765	ETHYLBENZENE	02/21/2017	07/01/2021	D	VOC	NE
4835	HEXACHLOROBUTADIENE	02/21/2017	07/01/2021	D	VOC	NE
4900	ISOPROPYLBENZENE	02/21/2017	07/01/2021	D	VOC	NE
5240	M/P-XYLENE	02/21/2017	07/01/2021	D	VOC	NE
5000	METHYL TERT-BUTYL ETHER (MTBE)	02/21/2017	07/01/2021	D	VOC	NE
4435	N-BUTYLBENZENE	02/21/2017	07/01/2021	D	VOC	NE
5090	N-PROPYLBENZENE	02/21/2017	07/01/2021	D	VOC	NE
5005	NAPHTHALENE	02/21/2017	07/01/2021	D	VOC	NE
5250	O-XYLENE	02/21/2017	07/01/2021	D	VOC	NE
4440	SEC-BUTYLBENZENE	02/21/2017	07/01/2021	D	VOC	NE
5100	STYRENE	02/21/2017	07/01/2021	D	VOC	NE
4370	TERT-AMYL METHYL ETHER (TAME)	02/21/2017	07/01/2021	D	VOC	NE
4420	TERT-BUTYL ALCOHOL (TBA)	02/21/2017	07/01/2021	D	VOC	NE
4445	TERT-BUTYLBENZENE	02/21/2017	07/01/2021	D	VOC	NE
5115	TETRACHLOROETHENE (PERCHLOROETHYLENE)	02/21/2017	07/01/2021	D	VOC	NE
5120	TETRAHYDROFURAN (THF)	02/21/2017	07/01/2021	D	VOC	NE
5140	TOLUENE	02/21/2017	07/01/2021	D	VOC	NE
4700	TRANS-1,2-DICLOROETHYLENE	02/21/2017	07/01/2021	D	VOC	NE
4685	TRANS-1,3-DICHLOROPROPENE	02/21/2017	07/01/2021	D	VOC	NE
5170	TRICHLOROETHENE (TRICHLOROETHYLENE)	02/21/2017	07/01/2021	D	VOC	NE
5175	TRICHLOROFLUOROMETHANE	02/21/2017	07/01/2021	D	VOC	NE
5235	VINYL CHLORIDE	02/21/2017	07/01/2021	D	VOC	NE
5260	XYLENE (TOTAL)	02/21/2017	07/01/2021	D	VOC	NE
Method Code: 10088809	**Method Ref: EPA 524.2**		**Revision: 4.1**		**Date: 1995**	
6800	1,3,5-TRICHLOROBENZENE	02/21/2017	07/01/2021	D	SBN	NE
Method Code: 20181606	**Method Ref: SM 9215 B (PCA)-1994**		**Revision:**		**Date: 1994**	
2555	HETEROTROPHIC PLATE COUNT	05/10/2017	07/01/2021	N	MIC	NE

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Method Code: 20196650	**Method Ref: SM 9221 E + C-2006**		**Revision:**		**Date: 2006**	
2530	FECAL COLIFORMS	02/21/2017	07/01/2021	N	MIC	NE
Method Code: 10162400	**Method Ref: SW-846 7196A**		**Revision: 1**		**Date: 1992**	
1045	CHROMIUM VI	02/21/2017	07/01/2021	N	MET	NE
Method Code: 20066266	**Method Ref: SM 3500-CR B-2011**		**Revision:**		**Date: 2011**	
1045	CHROMIUM VI	02/21/2017	07/01/2021	N	MET	NE
Method Code: 20069016	**Method Ref: SM 3500-FE B-2011**		**Revision:**		**Date: 2011**	
1073	IRON, FERROUS	09/06/2017	07/01/2021	N	MET	NE
Method Code: 10006403	**Method Ref: EPA 120.1**		**Revision:**		**Date: 1982**	
1610	CONDUCTIVITY	02/21/2017	07/01/2021	N	NMI	NE
Method Code: 10011800	**Method Ref: EPA 180.1**		**Revision: 2**		**Date: 1993**	
2055	TURBIDITY	02/21/2017	07/01/2021	N	NMI	NE
Method Code: 10053200	**Method Ref: EPA 300.0**		**Revision: 2.1**		**Date: 1993**	
1540	BROMIDE	02/21/2017	07/01/2021	N	NMI	NE
1575	CHLORIDE	02/21/2017	07/01/2021	N	NMI	NE
1730	FLUORIDE	02/21/2017	07/01/2021	N	NMI	NE
1810	NITRATE AS N	02/21/2017	07/01/2021	N	NMI	NE
2000	SULFATE	02/21/2017	07/01/2021	N	NMI	NE
Method Code: 10063602	**Method Ref: EPA 350.1**		**Revision: 2**		**Date: 1993**	
1515	AMMONIA AS N	02/21/2017	07/01/2021	N	NMI	NE
Method Code: 10064809	**Method Ref: EPA 351.1**		**Revision:**		**Date: 1978**	
1795	TOTAL KJELDAHL NITROGEN (TKN)	02/21/2017	07/01/2021	N	NMI	NE
Method Code: 10067604	**Method Ref: EPA 353.2**		**Revision: 2**		**Date: 1993**	
1810	NITRATE AS N	09/01/2017	07/01/2021	N	NMI	NE
1820	NITRATE-NITRITE AS N	09/01/2017	07/01/2021	N	NMI	NE
1840	NITRITE AS N	02/21/2017	07/01/2021	N	NMI	NE
Method Code: 10077404	**Method Ref: EPA 410.4**		**Revision: 2**		**Date: 1993**	
1565	COD	12/29/2017	07/01/2021	N	NMI	NE
Method Code: 10079400	**Method Ref: EPA 420.1**		**Revision:**		**Date: 1978**	
1905	PHENOLICS, TOTAL	02/21/2017	07/01/2021	N	NMI	NE
Method Code: 10127807	**Method Ref: EPA 1664A**		**Revision:**		**Date: 1999**	
1803	N-HEXANE EXTRACTABLE MATERIAL (O&G)	02/21/2017	07/01/2021	N	NMI	NE
1853	NON-POLAR EXTRACTABLE MATERIAL (TPH)	12/01/2017	07/01/2021	N	NMI	NE
Method Code: 10193803	**Method Ref: SW-846 9014**		**Revision: 0**		**Date: 1996**	
1645	CYANIDE, TOTAL	01/08/2016	07/01/2021	N	NMI	NE
Method Code: 10196608	**Method Ref: SW-846 9038**		**Revision: 0**		**Date: 1986**	
2000	SULFATE	01/08/2016	07/01/2021	N	NMI	NE
Method Code: 10198808	**Method Ref: SW-846 9050A**		**Revision: 1**		**Date: 1996**	
1610	CONDUCTIVITY	09/01/2017	07/01/2021	N	NMI	NE

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Method Code: 10199209	**Method Ref: SW-846 9056**		**Revision: 0**		**Date: 1994**	
1540	BROMIDE	12/29/2017	07/01/2021	N	NMI	NE
1575	CHLORIDE	12/29/2017	07/01/2021	N	NMI	NE
1730	FLUORIDE	12/29/2017	07/01/2021	N	NMI	NE
1810	NITRATE AS N	12/29/2017	07/01/2021	N	NMI	NE
2000	SULFATE	12/29/2017	07/01/2021	N	NMI	NE
Method Code: 10199607	**Method Ref: SW-846 9056A**		**Revision:**		**Date: 2000**	
1575	CHLORIDE	09/01/2017	07/01/2021	N	NMI	NE
1730	FLUORIDE	09/01/2017	07/01/2021	N	NMI	NE
1810	NITRATE AS N	09/01/2017	07/01/2021	N	NMI	NE
2000	SULFATE	09/01/2017	07/01/2021	N	NMI	NE
Method Code: 10200201	**Method Ref: SW-846 9060**		**Revision: 0**		**Date: 1986**	
2040	TOTAL ORGANIC CARBON (TOC)	02/21/2017	07/01/2021	N	NMI	NE
Method Code: 10200405	**Method Ref: SW-846 9065**		**Revision: 0**		**Date: 1986**	
1905	PHENOLICS, TOTAL	02/21/2017	07/01/2021	N	NMI	NE
Method Code: 10207406	**Method Ref: SW-846 9251**		**Revision: 0**		**Date: 1986**	
1575	CHLORIDE	01/08/2016	07/01/2021	N	NMI	NE
Method Code: 10234807	**Method Ref: SW-846 1010A**		**Revision:**		**Date:**	
6117	FLASHPOINT	02/21/2017	07/01/2021	N	NMI	NE
Method Code: 10243002	**Method Ref: SW-846 9010C**		**Revision:**		**Date:**	
1645	CYANIDE, TOTAL	02/21/2017	07/01/2021	N	NMI	NE
Method Code: 10243206	**Method Ref: SW-846 9012B**		**Revision:**		**Date:**	
1510	CYANIDE, AMENABLE	06/15/2018	07/01/2021	N	NMI	NE
1645	CYANIDE, TOTAL	01/08/2016	07/01/2021	N	NMI	NE
Method Code: 10244403	**Method Ref: SW-846 9040C**		**Revision:**		**Date:**	
1900	HYDROGEN ION (PH)	02/21/2017	07/01/2021	N	NMI	NE
Method Code: 10244823	**Method Ref: SW-846 9060A**		**Revision:**		**Date: 2004**	
2040	TOTAL ORGANIC CARBON (TOC)	09/01/2017	07/01/2021	N	NMI	NE
Method Code: 10261617	**Method Ref: EPA 1664B**		**Revision:**		**Date: 2010**	
1803	N-HEXANE EXTRACTABLE MATERIAL (O&G)	09/01/2017	07/01/2021	N	NMI	NE
1853	NON-POLAR EXTRACTABLE MATERIAL (TPH)	06/15/2018	07/01/2021	N	NMI	NE
Method Code: 10304800	**Method Ref: SW-846 6860**		**Revision:**		**Date:**	
1895	PERCHLORATE	09/01/2017	07/01/2021	N	NMI	NE
Method Code: 20039310	**Method Ref: SM 2120 B-2011**		**Revision:**		**Date: 2011**	
1605	COLOR	02/21/2017	07/01/2021	N	NMI	NE
Method Code: 20040088	**Method Ref: SM 2520 B-2011**		**Revision:**		**Date: 2011**	
1975	SALINITY	05/10/2017	07/01/2021	N	NMI	NE
Method Code: 20044615	**Method Ref: SM 2310 B-2011**		**Revision:**		**Date: 2011**	
1500	ACIDITY	02/21/2017	07/01/2021	N	NMI	NE

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Method Code: 20045618	**Method Ref: SM 2320 B-2011**		**Revision:**		**Date: 2011**	
1505	ALKALINITY	05/10/2017	07/01/2021	N	NMI	NE
Method Code: 20048220	**Method Ref: SM 2130 B-2011**		**Revision:**		**Date: 2011**	
2055	TURBIDITY	02/21/2017	07/01/2021	N	NMI	NE
Method Code: 20048617	**Method Ref: SM 2510 B-2011**		**Revision:**		**Date: 2011**	
1610	CONDUCTIVITY	02/21/2017	07/01/2021	N	NMI	NE
Method Code: 20049416	**Method Ref: SM 2540 B-2011**		**Revision:**		**Date: 2011**	
1950	RESIDUE, TOTAL (TS)	02/21/2017	07/01/2021	N	NMI	NE
Method Code: 20050413	**Method Ref: SM 2540 C-2011**		**Revision:**		**Date: 2011**	
1955	RESIDUE, FILTERABLE (TDS)	02/21/2017	07/01/2021	N	NMI	NE
Method Code: 20051212	**Method Ref: SM 2540 D-2011**		**Revision:**		**Date: 2011**	
1960	RESIDUE, NON-FILTERABLE (TSS)	02/21/2017	07/01/2021	N	NMI	NE
Method Code: 20051596	**Method Ref: SM 2540 E-2011**		**Revision:**		**Date: 2011**	
1970	RESIDUE, VOLATILE	02/21/2017	07/01/2021	N	NMI	NE
Method Code: 20052215	**Method Ref: SM 2540 F-2011**		**Revision:**		**Date: 2011**	
1965	RESIDUE, SETTLEABLE	02/21/2017	07/01/2021	N	NMI	NE
Method Code: 20086811	**Method Ref: SM 4500-CL⁻ E-2011**		**Revision:**		**Date: 2011**	
1575	CHLORIDE	02/21/2017	07/01/2021	N	NMI	NE
Method Code: 20096428	**Method Ref: SM 4500-CN E-2011**		**Revision:**		**Date: 2011**	
1645	CYANIDE, TOTAL	05/10/2017	07/01/2021	N	NMI	NE
Method Code: 20102414	**Method Ref: SM 4500-F C-2011**		**Revision:**		**Date: 2011**	
1730	FLUORIDE	02/21/2017	07/01/2021	N	NMI	NE
Method Code: 20105220	**Method Ref: SM 4500-H+ B-2011**		**Revision:**		**Date: 2011**	
1900	HYDROGEN ION (PH)	02/21/2017	07/01/2021	N	NMI	NE
Method Code: 20112214	**Method Ref: SM 4500-NH3 H-2011**		**Revision:**		**Date: 2011**	
1515	AMMONIA AS N	02/21/2017	07/01/2021	N	NMI	NE
1795	TOTAL KJELDAHL NITROGEN (TKN)	02/21/2017	07/01/2021	N	NMI	NE
Method Code: 20113115	**Method Ref: SM 4500-NO2 B-2011**		**Revision:**		**Date: 2011**	
1840	NITRITE AS N	02/21/2017	07/01/2021	N	NMI	NE
Method Code: 20117628	**Method Ref: SM 4500-NO3 F-2011**		**Revision:**		**Date: 2011**	
1810	NITRATE AS N	09/01/2017	07/01/2021	N	NMI	NE
1820	NITRATE-NITRITE AS N	02/21/2017	07/01/2021	N	NMI	NE
1840	NITRITE AS N	02/21/2017	07/01/2021	N	NMI	NE
Method Code: 20120836	**Method Ref: SM 4500-O C-2011**		**Revision:**		**Date: 2011**	
1880	OXYGEN, DISSOLVED	06/15/2018	07/01/2021	N	NMI	NE
Method Code: 20124225	**Method Ref: SM 4500-P E-2011**		**Revision:**		**Date: 2011**	
1870	ORTHOPHOSPHATE AS P	02/21/2017	07/01/2021	N	NMI	NE
1910	PHOSPHORUS, TOTAL	02/21/2017	07/01/2021	N	NMI	NE

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Method Code: 20125864	**Method Ref: SM 4500-S2 D-2011**		**Revision:**		**Date: 2011**	
2010	SULFIDE, TOTAL	02/21/2017	07/01/2021	N	NMI	NE
Method Code: 20130636	**Method Ref: SM 4500-SO_3^- B-2011**		**Revision:**		**Date: 2011**	
2015	SULFITE	02/21/2017	07/01/2021	N	NMI	NE
Method Code: 20132461	**Method Ref: SM 4500-SO4 E-2011**		**Revision:**		**Date: 2011**	
2000	SULFATE	05/10/2017	07/01/2021	N	NMI	NE
Method Code: 20135266	**Method Ref: SM 5210 B-2011**		**Revision:**		**Date: 2011**	
1530	BIOCHEMICAL OXYGEN DEMAND (BOD)	02/21/2017	07/01/2021	N	NMI	NE
1555	CARBONACEOUS BIOLOGICAL OXYGEN DEMAND	02/21/2017	07/01/2021	N	NMI	NE
Method Code: 20136816	**Method Ref: SM 5220 D-2011**		**Revision:**		**Date: 2011**	
1565	COD	02/21/2017	07/01/2021	N	NMI	NE
Method Code: 20138823	**Method Ref: SM 5310 C-2011**		**Revision:**		**Date: 2011**	
2040	TOTAL ORGANIC CARBON (TOC)	02/21/2017	07/01/2021	N	NMI	NE
Method Code: 20145066	**Method Ref: SM 5540 C-2011**		**Revision:**		**Date: 2011**	
2025	SURFACTANTS	02/21/2017	07/01/2021	N	NMI	NE
Method Code: 60006113	**Method Ref: LACHAT 10-107-06-2-D**		**Revision:**		**Date: 2003**	
1795	TOTAL KJELDAHL NITROGEN (TKN)	05/10/2017	07/01/2021	N	NMI	NE
Method Code: 60007138	**Method Ref: LACHAT 10-204-00-1-A**		**Revision:**		**Date: OCT-05**	
1645	CYANIDE, TOTAL	05/10/2017	07/01/2021	N	NMI	NE
Method Code: 10136604	**Method Ref: SW-846 3060A**		**Revision: 1**		**Date: 1996**	
1402	ALKALINE DIGESTION OF CR(VI)	02/21/2017	07/01/2021	N	PRE	NE
Method Code: 10138202	**Method Ref: SW-846 3510C**		**Revision: 3**		**Date: 1996**	
1444	SEPERATORY FUNNEL LIQUID-LIQUID EXTRACTION	02/21/2017	07/01/2021	N	PRE	NE
Method Code: 10146802	**Method Ref: SW-846 3630C**		**Revision: 3**		**Date: 1996**	
1446	SILICA GEL CLEAN-UP	02/21/2017	07/01/2021	N	PRE	NE
Method Code: 10148808	**Method Ref: SW-846 3665A**		**Revision: 1**		**Date: 1996**	
1458	SULFURIC ACID / PERMANGANATE CLEAN-UP	02/21/2017	07/01/2021	N	PRE	NE
Method Code: 10195605	**Method Ref: SW-846 9030B**		**Revision: 2**		**Date: 1996**	
1455	SULFIDE DISTILLATION	02/21/2017	07/01/2021	N	PRE	NE
Method Code: 10284603	**Method Ref: SW-846 5030C**		**Revision:**		**Date: 2003**	
1406	AQUEOUS-PHASE PURGE & TRAP	02/21/2017	07/01/2021	N	PRE	NE
Method Code: 20065663	**Method Ref: SM 4500-CN C-2011**		**Revision:**		**Date: 2011**	
1412	CYANIDE, MANUAL DISTILLATION	02/21/2017	07/01/2021	N	PRE	NE
Method Code: 20101411	**Method Ref: SM 4500-F B-2011**		**Revision:**		**Date: 2011**	
1416	FLUORIDE PRELIMINARY DISTILLATION	02/21/2017	07/01/2021	N	PRE	NE
Method Code: 20106018	**Method Ref: SM 4500-NH3 B-2011**		**Revision:**		**Date: 2011**	
1404	AMMONIA DISTILLATION	02/21/2017	07/01/2021	N	PRE	NE
Method Code: 20120018	**Method Ref: SM 4500-NORG C-2011**		**Revision:**		**Date: 2011**	
1462	TKN DIGESTION & DISTILLATION	11/22/2016	07/01/2021	N	PRE	NE

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Method Code: 20123368	**Method Ref: SM 4500-P B (5)-2011**		**Revision:**		**Date: 2011**	
1436	PHOSPHORUS, TOTAL (DIGESTION)	05/10/2017	07/01/2021	N	PRE	NE
Method Code: 10107207	**Method Ref: EPA 624**		**Revision:**		**Date: 1984**	
5160	1,1,1-TRICHLOROETHANE	12/29/2017	07/01/2021	N	VOC	NE
5110	1,1,2,2-TETRACHLOROETHANE	12/29/2017	07/01/2021	N	VOC	NE
5185	1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE (FREON 113)	12/29/2017	07/01/2021	N	VOC	NE
5165	1,1,2-TRICHLOROETHANE	12/29/2017	07/01/2021	N	VOC	NE
4630	1,1-DICHLOROETHANE	12/29/2017	07/01/2021	N	VOC	NE
4640	1,1-DICHLOROETHENE	12/29/2017	07/01/2021	N	VOC	NE
5150	1,2,3-TRICHLOROBENZENE	12/29/2017	07/01/2021	N	VOC	NE
5155	1,2,4-TRICHLOROBENZENE	12/29/2017	07/01/2021	N	VOC	NE
4570	1,2-DIBROMO-3-CHLOROPROPANE (DBCP)	12/29/2017	07/01/2021	N	VOC	NE
4585	1,2-DIBROMOETHANE (EDB)	12/29/2017	07/01/2021	N	VOC	NE
4610	1,2-DICHLOROBENZENE	12/29/2017	07/01/2021	N	VOC	NE
4635	1,2-DICHLOROETHANE	12/29/2017	07/01/2021	N	VOC	NE
4655	1,2-DICHLOROPROPANE	12/29/2017	07/01/2021	N	VOC	NE
4615	1,3-DICHLOROBENZENE	12/29/2017	07/01/2021	N	VOC	NE
4660	1,3-DICHLOROPROPANE	12/29/2017	07/01/2021	N	VOC	NE
4620	1,4-DICHLOROBENZENE	12/29/2017	07/01/2021	N	VOC	NE
4735	1,4-DIOXANE (1 4-DIETHYLENEOXIDE)	12/29/2017	07/01/2021	N	VOC	NE
4410	2-BUTANONE (METHYL ETHYL KETONE MEK)	12/29/2017	07/01/2021	N	VOC	NE
4500	2-CHLOROETHYLVINYL ETHER	12/29/2017	07/01/2021	N	VOC	NE
4860	2-HEXANONE	12/29/2017	07/01/2021	N	VOC	NE
4995	4-METHYL-2-PENTANONE (MIBK)	12/29/2017	07/01/2021	N	VOC	NE
4315	ACETONE	12/29/2017	07/01/2021	N	VOC	NE
4325	ACROLEIN (PROPENAL)	12/29/2017	07/01/2021	N	VOC	NE
4340	ACRYLONITRILE	12/29/2017	07/01/2021	N	VOC	NE
4375	BENZENE	12/29/2017	07/01/2021	N	VOC	NE
4395	BROMODICHLOROMETHANE	12/29/2017	07/01/2021	N	VOC	NE
4400	BROMOFORM	12/29/2017	07/01/2021	N	VOC	NE
4950	BROMOMETHANE (METHYL BROMIDE)	12/29/2017	07/01/2021	N	VOC	NE
4450	CARBON DISULFIDE	12/29/2017	07/01/2021	N	VOC	NE
4455	CARBON TETRACHLORIDE	12/29/2017	07/01/2021	N	VOC	NE
4475	CHLOROBENZENE	12/29/2017	07/01/2021	N	VOC	NE
4485	CHLOROETHANE	12/29/2017	07/01/2021	N	VOC	NE
4505	CHLOROFORM	12/29/2017	07/01/2021	N	VOC	NE

This analyte list supersedes all previously issued analyte lists. Method accreditation does not imply acceptance for NHDES compliance testing. Laboratory is required to use EPA-approved methods required by regulation. Continuing accreditation status is dependent on successful ongoing participation in the program. Customers may verify the laboratory's current accreditation status by calling (603) 271-2998 or by visiting the NH ELAP website (https://www.des.nh.gov/organization/divisions/water/dwgb/nhelap/index.htm).

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4960	CHLOROMETHANE (METHYL CHLORIDE)	12/29/2017	07/01/2021	N	VOC	NE
4645	CIS-1 2-DICHLOROETHYLENE	12/29/2017	07/01/2021	N	VOC	NE
4680	CIS-1,3-DICHLOROPROPENE	12/29/2017	07/01/2021	N	VOC	NE
4575	DIBROMOCHLOROMETHANE	12/29/2017	07/01/2021	N	VOC	NE
4595	DIBROMOMETHANE	12/29/2017	07/01/2021	N	VOC	NE
4625	DICHLORODIFLUOROMETHANE	12/29/2017	07/01/2021	N	VOC	NE
4975	DICHLOROMETHANE (METHYLENE CHLORIDE)	12/29/2017	07/01/2021	N	VOC	NE
4765	ETHYLBENZENE	12/29/2017	07/01/2021	N	VOC	NE
4900	ISOPROPYLBENZENE	12/29/2017	07/01/2021	N	VOC	NE
5000	METHYL TERT-BUTYL ETHER (MTBE)	12/29/2017	07/01/2021	N	VOC	NE
4965	METHYLCYCLOHEXANE	12/29/2017	07/01/2021	N	VOC	NE
4855	N-HEXANE (HEXANE)	12/29/2017	07/01/2021	N	VOC	NE
5100	STYRENE	12/29/2017	07/01/2021	N	VOC	NE
4370	TERT-AMYL METHYL ETHER (TAME)	12/29/2017	07/01/2021	N	VOC	NE
4420	TERT-BUTYL ALCOHOL (TBA)	12/29/2017	07/01/2021	N	VOC	NE
5115	TETRACHLOROETHENE (PERCHLOROETHYLENE)	12/29/2017	07/01/2021	N	VOC	NE
5140	TOLUENE	12/29/2017	07/01/2021	N	VOC	NE
4700	TRANS-1,2-DICLOROETHYLENE	12/29/2017	07/01/2021	N	VOC	NE
4685	TRANS-1,3-DICHLOROPROPENE	12/29/2017	07/01/2021	N	VOC	NE
5170	TRICHLOROETHENE (TRICHLOROETHYLENE)	12/29/2017	07/01/2021	N	VOC	NE
5175	TRICHLOROFLUOROMETHANE	12/29/2017	07/01/2021	N	VOC	NE
5225	VINYL ACETATE	12/29/2017	07/01/2021	N	VOC	NE
5235	VINYL CHLORIDE	12/29/2017	07/01/2021	N	VOC	NE
5260	XYLENE (TOTAL)	12/29/2017	07/01/2021	N	VOC	NE
Method Code: 10107401	**Method Ref: EPA 625**		**Revision:**		**Date: 1984**	
5155	1,2,4-TRICHLOROBENZENE	12/29/2017	07/01/2021	N	VOC	NE
4835	HEXACHLOROBUTADIENE	12/29/2017	07/01/2021	N	VOC	NE
4840	HEXACHLOROETHANE	12/29/2017	07/01/2021	N	VOC	NE
5005	NAPHTHALENE	12/29/2017	07/01/2021	N	VOC	NE
5015	NITROBENZENE	12/29/2017	07/01/2021	N	VOC	NE
5095	PYRIDINE	12/29/2017	07/01/2021	N	VOC	NE
Method Code: 10173009	**Method Ref: SW-846 8011**		**Revision:**		**Date: 1994**	
5180	1,2,3-TRICHLOROPROPANE	05/13/2019	07/01/2021	N	VOC	NE
4570	1,2-DIBROMO-3-CHLOROPROPANE (DBCP)	08/01/2017	07/01/2021	N	VOC	NE
4585	1,2-DIBROMOETHANE (EDB)	08/01/2017	07/01/2021	N	VOC	NE
Method Code: 10186002	**Method Ref: SW-846 8270D**		**Revision:**		**Date: 2007**	
5155	1,2,4-TRICHLOROBENZENE	02/21/2017	07/01/2021	N	VOC	NE

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4610	1,2-DICHLOROBENZENE	02/21/2017	07/01/2021	N	VOC	NE
4615	1,3-DICHLOROBENZENE	02/21/2017	07/01/2021	N	VOC	NE
4620	1,4-DICHLOROBENZENE	02/21/2017	07/01/2021	N	VOC	NE
4835	HEXACHLOROBUTADIENE	02/21/2017	07/01/2021	N	VOC	NE
4840	HEXACHLOROETHANE	02/21/2017	07/01/2021	N	VOC	NE
5005	NAPHTHALENE	02/21/2017	07/01/2021	N	VOC	NE
5015	NITROBENZENE	02/21/2017	07/01/2021	N	VOC	NE
5145	O-TOLUIDINE	09/05/2017	07/01/2021	N	VOC	NE
5095	PYRIDINE	02/21/2017	07/01/2021	N	VOC	NE
Method Code: 10188008	**Method Ref: SW-846 8315A**		**Revision: 1**		**Date: 1996**	
4815	FORMALDEHYDE	02/21/2017	07/01/2021	N	VOC	NE
Method Code: 10242509	**Method Ref: SW-846 8270D SIM**		**Revision:**		**Date: 2007**	
4835	HEXACHLOROBUTADIENE	02/21/2017	07/01/2021	N	VOC	NE
5005	NAPHTHALENE	02/21/2017	07/01/2021	N	VOC	NE
Method Code: 10242543	**Method Ref: SW-846 8270E**		**Revision:**		**Date: 2017**	
5155	1,2,4-TRICHLOROBENZENE	06/15/2020	07/01/2021	N	VOC	NE
4610	1,2-DICHLOROBENZENE	06/15/2020	07/01/2021	N	VOC	NE
4615	1,3-DICHLOROBENZENE	06/15/2020	07/01/2021	N	VOC	NE
4620	1,4-DICHLOROBENZENE	06/15/2020	07/01/2021	N	VOC	NE
4835	HEXACHLOROBUTADIENE	06/15/2020	07/01/2021	N	VOC	NE
4840	HEXACHLOROETHANE	06/15/2020	07/01/2021	N	VOC	NE
5005	NAPHTHALENE	06/15/2020	07/01/2021	N	VOC	NE
5015	NITROBENZENE	06/15/2020	07/01/2021	N	VOC	NE
5145	O-TOLUIDINE	06/15/2020	07/01/2021	N	VOC	NE
5095	PYRIDINE	06/15/2020	07/01/2021	N	VOC	NE
Method Code: 10242565	**Method Ref: SW-846 8270E SIM**		**Revision:**		**Date: 2017**	
4735	1,4-DIOXANE (1 4-DIETHYLENEOXIDE)	06/24/2020	07/01/2021	N	VOC	NE
4835	HEXACHLOROBUTADIENE	06/15/2020	07/01/2021	N	VOC	NE
5005	NAPHTHALENE	06/15/2020	07/01/2021	N	VOC	NE
Method Code: 10298121	**Method Ref: EPA 624.1**		**Revision:**		**Date: 2016**	
5160	1,1,1-TRICHLOROETHANE	07/02/2018	07/01/2021	N	VOC	NE
5110	1,1,2,2-TETRACHLOROETHANE	07/02/2018	07/01/2021	N	VOC	NE
5185	1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE (FREON 113)	07/02/2018	07/01/2021	N	VOC	NE
5165	1,1,2-TRICHLOROETHANE	07/02/2018	07/01/2021	N	VOC	NE
4630	1,1-DICHLOROETHANE	07/02/2018	07/01/2021	N	VOC	NE
4640	1,1-DICHLOROETHENE	07/02/2018	07/01/2021	N	VOC	NE
5150	1,2,3-TRICHLOROBENZENE	07/02/2018	07/01/2021	N	VOC	NE

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5155	1,2,4-TRICHLOROBENZENE	07/02/2018	07/01/2021	N	VOC	NE
4570	1,2-DIBROMO-3-CHLOROPROPANE (DBCP)	07/02/2018	07/01/2021	N	VOC	NE
4585	1,2-DIBROMOETHANE (EDB)	07/02/2018	07/01/2021	N	VOC	NE
4610	1,2-DICHLOROBENZENE	07/02/2018	07/01/2021	N	VOC	NE
4635	1,2-DICHLOROETHANE	07/02/2018	07/01/2021	N	VOC	NE
4655	1,2-DICHLOROPROPANE	07/02/2018	07/01/2021	N	VOC	NE
4615	1,3-DICHLOROBENZENE	07/02/2018	07/01/2021	N	VOC	NE
4660	1,3-DICHLOROPROPANE	07/02/2018	07/01/2021	N	VOC	NE
4620	1,4-DICHLOROBENZENE	07/02/2018	07/01/2021	N	VOC	NE
4735	1,4-DIOXANE (1 4-DIETHYLENEOXIDE)	07/02/2018	07/01/2021	N	VOC	NE
4410	2-BUTANONE (METHYL ETHYL KETONE MEK)	07/02/2018	07/01/2021	N	VOC	NE
4500	2-CHLOROETHYLVINYL ETHER	07/02/2018	07/01/2021	N	VOC	NE
4860	2-HEXANONE	07/02/2018	07/01/2021	N	VOC	NE
4995	4-METHYL-2-PENTANONE (MIBK)	07/02/2018	07/01/2021	N	VOC	NE
4315	ACETONE	07/02/2018	07/01/2021	N	VOC	NE
4325	ACROLEIN (PROPENAL)	07/02/2018	07/01/2021	N	VOC	NE
4340	ACRYLONITRILE	07/02/2018	07/01/2021	N	VOC	NE
4375	BENZENE	07/02/2018	07/01/2021	N	VOC	NE
4395	BROMODICHLOROMETHANE	07/02/2018	07/01/2021	N	VOC	NE
4400	BROMOFORM	07/02/2018	07/01/2021	N	VOC	NE
4950	BROMOMETHANE (METHYL BROMIDE)	07/02/2018	07/01/2021	N	VOC	NE
4450	CARBON DISULFIDE	07/02/2018	07/01/2021	N	VOC	NE
4455	CARBON TETRACHLORIDE	07/02/2018	07/01/2021	N	VOC	NE
4475	CHLOROBENZENE	07/02/2018	07/01/2021	N	VOC	NE
4485	CHLOROETHANE	07/02/2018	07/01/2021	N	VOC	NE
4505	CHLOROFORM	07/02/2018	07/01/2021	N	VOC	NE
4960	CHLOROMETHANE (METHYL CHLORIDE)	07/02/2018	07/01/2021	N	VOC	NE
4645	CIS-1 2-DICHLOROETHYLENE	07/02/2018	07/01/2021	N	VOC	NE
4680	CIS-1,3-DICHLOROPROPENE	07/02/2018	07/01/2021	N	VOC	NE
4575	DIBROMOCHLOROMETHANE	07/02/2018	07/01/2021	N	VOC	NE
4595	DIBROMOMETHANE	07/02/2018	07/01/2021	N	VOC	NE
4625	DICHLORODIFLUOROMETHANE	07/02/2018	07/01/2021	N	VOC	NE
4975	DICHLOROMETHANE (METHYLENE CHLORIDE)	07/02/2018	07/01/2021	N	VOC	NE
4765	ETHYLBENZENE	07/02/2018	07/01/2021	N	VOC	NE
4900	ISOPROPYLBENZENE	07/02/2018	07/01/2021	N	VOC	NE
5000	METHYL TERT-BUTYL ETHER (MTBE)	07/02/2018	07/01/2021	N	VOC	NE

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4965	METHYLCYCLOHEXANE	07/02/2018	07/01/2021	N	VOC	NE
4855	N-HEXANE (HEXANE)	07/02/2018	07/01/2021	N	VOC	NE
5100	STYRENE	07/02/2018	07/01/2021	N	VOC	NE
4370	TERT-AMYL METHYL ETHER (TAME)	07/02/2018	07/01/2021	N	VOC	NE
4420	TERT-BUTYL ALCOHOL (TBA)	07/02/2018	07/01/2021	N	VOC	NE
5115	TETRACHLOROETHENE (PERCHLOROETHYLENE)	07/02/2018	07/01/2021	N	VOC	NE
5140	TOLUENE	07/02/2018	07/01/2021	N	VOC	NE
4700	TRANS-1,2-DICLOROETHYLENE	07/02/2018	07/01/2021	N	VOC	NE
4685	TRANS-1,3-DICHLOROPROPENE	07/02/2018	07/01/2021	N	VOC	NE
5170	TRICHLOROETHENE (TRICHLOROETHYLENE)	07/02/2018	07/01/2021	N	VOC	NE
5175	TRICHLOROFLUOROMETHANE	07/02/2018	07/01/2021	N	VOC	NE
5225	VINYL ACETATE	07/02/2018	07/01/2021	N	VOC	NE
5235	VINYL CHLORIDE	07/02/2018	07/01/2021	N	VOC	NE
5260	XYLENE (TOTAL)	07/02/2018	07/01/2021	N	VOC	NE
Method Code: 10300024	**Method Ref: EPA 625.1**		**Revision:**		**Date: 2016**	
5155	1,2,4-TRICHLOROBENZENE	07/02/2018	07/01/2021	N	VOC	NE
4835	HEXACHLOROBUTADIENE	07/02/2018	07/01/2021	N	VOC	NE
4840	HEXACHLOROETHANE	07/02/2018	07/01/2021	N	VOC	NE
5005	NAPHTHALENE	07/02/2018	07/01/2021	N	VOC	NE
5015	NITROBENZENE	07/02/2018	07/01/2021	N	VOC	NE
5095	PYRIDINE	07/02/2018	07/01/2021	N	VOC	NE
Method Code: 10307003	**Method Ref: SW-846 8260C**		**Revision:**		**Date:**	
5105	1,1,1,2-TETRACHLOROETHANE	02/21/2017	07/01/2021	N	VOC	NE
5160	1,1,1-TRICHLOROETHANE	02/21/2017	07/01/2021	N	VOC	NE
5110	1,1,2,2-TETRACHLOROETHANE	02/21/2017	07/01/2021	N	VOC	NE
5185	1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE (FREON 113)	02/21/2017	07/01/2021	N	VOC	NE
5165	1,1,2-TRICHLOROETHANE	02/21/2017	07/01/2021	N	VOC	NE
4630	1,1-DICHLOROETHANE	02/21/2017	07/01/2021	N	VOC	NE
4640	1,1-DICHLOROETHENE	02/21/2017	07/01/2021	N	VOC	NE
4670	1,1-DICHLOROPROPENE	02/21/2017	07/01/2021	N	VOC	NE
5150	1,2,3-TRICHLOROBENZENE	02/21/2017	07/01/2021	N	VOC	NE
5180	1,2,3-TRICHLOROPROPANE	02/21/2017	07/01/2021	N	VOC	NE
5155	1,2,4-TRICHLOROBENZENE	02/21/2017	07/01/2021	N	VOC	NE
5210	1,2,4-TRIMETHYLBENZENE	02/21/2017	07/01/2021	N	VOC	NE
4570	1,2-DIBROMO-3-CHLOROPROPANE (DBCP)	02/21/2017	07/01/2021	N	VOC	NE
4585	1,2-DIBROMOETHANE (EDB)	02/21/2017	07/01/2021	N	VOC	NE

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4610	1,2-DICHLOROBENZENE	02/21/2017	07/01/2021	N	VOC	NE
4635	1,2-DICHLOROETHANE	02/21/2017	07/01/2021	N	VOC	NE
4655	1,2-DICHLOROPROPANE	02/21/2017	07/01/2021	N	VOC	NE
5215	1,3,5-TRIMETHYLBENZENE	02/21/2017	07/01/2021	N	VOC	NE
4615	1,3-DICHLOROBENZENE	02/21/2017	07/01/2021	N	VOC	NE
4660	1,3-DICHLOROPROPANE	02/21/2017	07/01/2021	N	VOC	NE
4620	1,4-DICHLOROBENZENE	02/21/2017	07/01/2021	N	VOC	NE
4735	1,4-DIOXANE (1 4-DIETHYLENEOXIDE)	02/21/2017	07/01/2021	N	VOC	NE
4665	2 2-DICHLOROPROPANE	02/21/2017	07/01/2021	N	VOC	NE
4410	2-BUTANONE (METHYL ETHYL KETONE MEK)	02/21/2017	07/01/2021	N	VOC	NE
4500	2-CHLOROETHYLVINYL ETHER	02/21/2017	07/01/2021	N	VOC	NE
4535	2-CHLOROTOLUENE	02/21/2017	07/01/2021	N	VOC	NE
4860	2-HEXANONE	02/21/2017	07/01/2021	N	VOC	NE
4540	4-CHLOROTOLUENE	02/21/2017	07/01/2021	N	VOC	NE
4910	4-ISOPROPYLTOLUENE (P-ISOPROPYLTOLUENE)	02/21/2017	07/01/2021	N	VOC	NE
4995	4-METHYL-2-PENTANONE (MIBK)	02/21/2017	07/01/2021	N	VOC	NE
4315	ACETONE	02/21/2017	07/01/2021	N	VOC	NE
4325	ACROLEIN (PROPENAL)	02/21/2017	07/01/2021	N	VOC	NE
4340	ACRYLONITRILE	02/21/2017	07/01/2021	N	VOC	NE
4375	BENZENE	02/21/2017	07/01/2021	N	VOC	NE
4385	BROMOBENZENE	02/21/2017	07/01/2021	N	VOC	NE
4390	BROMOCHLOROMETHANE	02/21/2017	07/01/2021	N	VOC	NE
4395	BROMODICHLOROMETHANE	02/21/2017	07/01/2021	N	VOC	NE
4400	BROMOFORM	02/21/2017	07/01/2021	N	VOC	NE
4950	BROMOMETHANE (METHYL BROMIDE)	02/21/2017	07/01/2021	N	VOC	NE
4450	CARBON DISULFIDE	02/21/2017	07/01/2021	N	VOC	NE
4455	CARBON TETRACHLORIDE	02/21/2017	07/01/2021	N	VOC	NE
4475	CHLOROBENZENE	02/21/2017	07/01/2021	N	VOC	NE
4485	CHLOROETHANE	02/21/2017	07/01/2021	N	VOC	NE
4505	CHLOROFORM	02/21/2017	07/01/2021	N	VOC	NE
4960	CHLOROMETHANE (METHYL CHLORIDE)	02/21/2017	07/01/2021	N	VOC	NE
4645	CIS-1 2-DICHLOROETHYLENE	02/21/2017	07/01/2021	N	VOC	NE
4680	CIS-1,3-DICHLOROPROPENE	02/21/2017	07/01/2021	N	VOC	NE
4555	CYCLOHEXANE	02/21/2017	07/01/2021	N	VOC	NE
4560	CYCLOHEXANONE	02/21/2017	07/01/2021	N	VOC	NE
9375	DI-ISOPROPYLETHER (DIPE)	02/21/2017	07/01/2021	N	VOC	NE

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4575	DIBROMOCHLOROMETHANE	02/21/2017	07/01/2021	N	VOC	NE
4595	DIBROMOMETHANE	02/21/2017	07/01/2021	N	VOC	NE
4625	DICHLORODIFLUOROMETHANE	02/21/2017	07/01/2021	N	VOC	NE
4975	DICHLOROMETHANE (METHYLENE CHLORIDE)	02/21/2017	07/01/2021	N	VOC	NE
4725	DIETHYL ETHER	02/21/2017	07/01/2021	N	VOC	NE
4750	ETHANOL	02/21/2017	07/01/2021	N	VOC	NE
4770	ETHYL - T- BUTYL ETHER (ETBE)	02/21/2017	07/01/2021	N	VOC	NE
4755	ETHYL ACETATE	02/21/2017	07/01/2021	N	VOC	NE
4810	ETHYL METHACRYLATE	02/21/2017	07/01/2021	N	VOC	NE
4765	ETHYLBENZENE	02/21/2017	07/01/2021	N	VOC	NE
4835	HEXACHLOROBUTADIENE	02/21/2017	07/01/2021	N	VOC	NE
4870	IODOMETHANE (METHYL IODIDE)	02/21/2017	07/01/2021	N	VOC	NE
4895	ISOPROPYL ALCOHOL (2-PROPANOL)	02/21/2017	07/01/2021	N	VOC	NE
4900	ISOPROPYLBENZENE	02/21/2017	07/01/2021	N	VOC	NE
5245	M-XYLENE	02/21/2017	07/01/2021	N	VOC	NE
4940	METHYL ACETATE	02/21/2017	07/01/2021	N	VOC	NE
4990	METHYL METHACRYLATE	02/21/2017	07/01/2021	N	VOC	NE
5000	METHYL TERT-BUTYL ETHER (MTBE)	02/21/2017	07/01/2021	N	VOC	NE
4965	METHYLCYCLOHEXANE	02/21/2017	07/01/2021	N	VOC	NE
4425	N-BUTYL ALCOHOL	02/21/2017	07/01/2021	N	VOC	NE
4435	N-BUTYLBENZENE	02/21/2017	07/01/2021	N	VOC	NE
4855	N-HEXANE (HEXANE)	12/29/2017	07/01/2021	N	VOC	NE
5090	N-PROPYLBENZENE	02/21/2017	07/01/2021	N	VOC	NE
5005	NAPHTHALENE	02/21/2017	07/01/2021	N	VOC	NE
5250	O-XYLENE	02/21/2017	07/01/2021	N	VOC	NE
5255	P-XYLENE	02/21/2017	07/01/2021	N	VOC	NE
5035	PENTACHLOROETHANE	02/21/2017	07/01/2021	N	VOC	NE
4440	SEC-BUTYLBENZENE	02/21/2017	07/01/2021	N	VOC	NE
5100	STYRENE	02/21/2017	07/01/2021	N	VOC	NE
4370	TERT-AMYL METHYL ETHER (TAME)	02/21/2017	07/01/2021	N	VOC	NE
4420	TERT-BUTYL ALCOHOL (TBA)	02/21/2017	07/01/2021	N	VOC	NE
4445	TERT-BUTYLBENZENE	02/21/2017	07/01/2021	N	VOC	NE
5115	TETRACHLOROETHENE (PERCHLOROETHYLENE)	02/21/2017	07/01/2021	N	VOC	NE
5120	TETRAHYDROFURAN (THF)	02/21/2017	07/01/2021	N	VOC	NE
5140	TOLUENE	02/21/2017	07/01/2021	N	VOC	NE
4700	TRANS-1,2-DICLOROETHYLENE	02/21/2017	07/01/2021	N	VOC	NE

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4685	TRANS-1,3-DICHLOROPROPENE	02/21/2017	07/01/2021	N	VOC	NE
4605	TRANS-1,4-DICHLORO-2-BUTENE	02/21/2017	07/01/2021	N	VOC	NE
5170	TRICHLOROETHENE (TRICHLOROETHYLENE)	02/21/2017	07/01/2021	N	VOC	NE
5175	TRICHLOROFLUOROMETHANE	02/21/2017	07/01/2021	N	VOC	NE
5225	VINYL ACETATE	02/21/2017	07/01/2021	N	VOC	NE
5235	VINYL CHLORIDE	02/21/2017	07/01/2021	N	VOC	NE
5260	XYLENE (TOTAL)	02/21/2017	07/01/2021	N	VOC	NE
Method Code: 10307105	**Method Ref: SW-846 8260C SIM**		**Revision:**		**Date:**	
4735	1,4-DIOXANE (1 4-DIETHYLENEOXIDE)	02/21/2017	07/01/2021	N	VOC	NE
Method Code: 10307127	**Method Ref: SW-846 8260D**		**Revision:**		**Date: 2018**	
5105	1,1,1,2-TETRACHLOROETHANE	06/15/2020	07/01/2021	N	VOC	NE
5160	1,1,1-TRICHLOROETHANE	06/15/2020	07/01/2021	N	VOC	NE
5110	1,1,2,2-TETRACHLOROETHANE	06/15/2020	07/01/2021	N	VOC	NE
5185	1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE (FREON 113)	06/15/2020	07/01/2021	N	VOC	NE
5165	1,1,2-TRICHLOROETHANE	06/15/2020	07/01/2021	N	VOC	NE
4630	1,1-DICHLOROETHANE	06/15/2020	07/01/2021	N	VOC	NE
4640	1,1-DICHLOROETHENE	06/15/2020	07/01/2021	N	VOC	NE
4670	1,1-DICHLOROPROPENE	06/15/2020	07/01/2021	N	VOC	NE
5150	1,2,3-TRICHLOROBENZENE	06/15/2020	07/01/2021	N	VOC	NE
5180	1,2,3-TRICHLOROPROPANE	06/15/2020	07/01/2021	N	VOC	NE
5155	1,2,4-TRICHLOROBENZENE	06/15/2020	07/01/2021	N	VOC	NE
5210	1,2,4-TRIMETHYLBENZENE	06/15/2020	07/01/2021	N	VOC	NE
4570	1,2-DIBROMO-3-CHLOROPROPANE (DBCP)	06/15/2020	07/01/2021	N	VOC	NE
4585	1,2-DIBROMOETHANE (EDB)	06/15/2020	07/01/2021	N	VOC	NE
4610	1,2-DICHLOROBENZENE	06/15/2020	07/01/2021	N	VOC	NE
4635	1,2-DICHLOROETHANE	06/15/2020	07/01/2021	N	VOC	NE
4655	1,2-DICHLOROPROPANE	06/15/2020	07/01/2021	N	VOC	NE
5215	1,3,5-TRIMETHYLBENZENE	06/15/2020	07/01/2021	N	VOC	NE
4615	1,3-DICHLOROBENZENE	06/15/2020	07/01/2021	N	VOC	NE
4660	1,3-DICHLOROPROPANE	06/15/2020	07/01/2021	N	VOC	NE
4620	1,4-DICHLOROBENZENE	06/15/2020	07/01/2021	N	VOC	NE
4735	1,4-DIOXANE (1 4-DIETHYLENEOXIDE)	06/15/2020	07/01/2021	N	VOC	NE
4665	2 2-DICHLOROPROPANE	06/15/2020	07/01/2021	N	VOC	NE
4410	2-BUTANONE (METHYL ETHYL KETONE MEK)	06/15/2020	07/01/2021	N	VOC	NE
4500	2-CHLOROETHYLVINYL ETHER	06/15/2020	07/01/2021	N	VOC	NE
4535	2-CHLOROTOLUENE	06/15/2020	07/01/2021	N	VOC	NE

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4860	2-HEXANONE	06/15/2020	07/01/2021	N	VOC	NE
4540	4-CHLOROTOLUENE	06/15/2020	07/01/2021	N	VOC	NE
4910	4-ISOPROPYLTOLUENE (P-ISOPROPYLTOLUENE)	06/15/2020	07/01/2021	N	VOC	NE
4995	4-METHYL-2-PENTANONE (MIBK)	06/15/2020	07/01/2021	N	VOC	NE
4315	ACETONE	06/15/2020	07/01/2021	N	VOC	NE
4325	ACROLEIN (PROPENAL)	06/15/2020	07/01/2021	N	VOC	NE
4340	ACRYLONITRILE	06/15/2020	07/01/2021	N	VOC	NE
4375	BENZENE	06/15/2020	07/01/2021	N	VOC	NE
4385	BROMOBENZENE	06/15/2020	07/01/2021	N	VOC	NE
4390	BROMOCHLOROMETHANE	06/15/2020	07/01/2021	N	VOC	NE
4395	BROMODICHLOROMETHANE	06/15/2020	07/01/2021	N	VOC	NE
4400	BROMOFORM	06/15/2020	07/01/2021	N	VOC	NE
4950	BROMOMETHANE (METHYL BROMIDE)	06/15/2020	07/01/2021	N	VOC	NE
4450	CARBON DISULFIDE	06/15/2020	07/01/2021	N	VOC	NE
4455	CARBON TETRACHLORIDE	06/15/2020	07/01/2021	N	VOC	NE
4475	CHLOROBENZENE	06/15/2020	07/01/2021	N	VOC	NE
4485	CHLOROETHANE	06/15/2020	07/01/2021	N	VOC	NE
4505	CHLOROFORM	06/15/2020	07/01/2021	N	VOC	NE
4960	CHLOROMETHANE (METHYL CHLORIDE)	06/15/2020	07/01/2021	N	VOC	NE
4645	CIS-1 2-DICHLOROETHYLENE	06/15/2020	07/01/2021	N	VOC	NE
4680	CIS-1,3-DICHLOROPROPENE	06/15/2020	07/01/2021	N	VOC	NE
4555	CYCLOHEXANE	06/15/2020	07/01/2021	N	VOC	NE
4560	CYCLOHEXANONE	06/15/2020	07/01/2021	N	VOC	NE
9375	DI-ISOPROPYLETHER (DIPE)	06/15/2020	07/01/2021	N	VOC	NE
4575	DIBROMOCHLOROMETHANE	06/15/2020	07/01/2021	N	VOC	NE
4595	DIBROMOMETHANE	06/15/2020	07/01/2021	N	VOC	NE
4625	DICHLORODIFLUOROMETHANE	06/15/2020	07/01/2021	N	VOC	NE
4975	DICHLOROMETHANE (METHYLENE CHLORIDE)	06/15/2020	07/01/2021	N	VOC	NE
4725	DIETHYL ETHER	06/15/2020	07/01/2021	N	VOC	NE
4750	ETHANOL	06/15/2020	07/01/2021	N	VOC	NE
4770	ETHYL - T- BUTYL ETHER (ETBE)	06/15/2020	07/01/2021	N	VOC	NE
4755	ETHYL ACETATE	06/15/2020	07/01/2021	N	VOC	NE
4810	ETHYL METHACRYLATE	06/15/2020	07/01/2021	N	VOC	NE
4765	ETHYLBENZENE	06/15/2020	07/01/2021	N	VOC	NE
4835	HEXACHLOROBUTADIENE	06/15/2020	07/01/2021	N	VOC	NE
4870	IODOMETHANE (METHYL IODIDE)	06/15/2020	07/01/2021	N	VOC	NE

This analyte list supersedes all previously issued analyte lists. Method accreditation does not imply acceptance for NHDES compliance testing. Laboratory is required to use EPA-approved methods required by regulation. Continuing accreditation status is dependent on successful ongoing participation in the program. Customers may verify the laboratory's current accreditation status by calling (603) 271-2998 or by visiting the NH ELAP website (https://www.des.nh.gov/organization/divisions/water/dwgb/nhelap/index.htm).

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4895	ISOPROPYL ALCOHOL (2-PROPANOL)	06/15/2020	07/01/2021	N	VOC	NE
4900	ISOPROPYLBENZENE	06/15/2020	07/01/2021	N	VOC	NE
5245	M-XYLENE	06/15/2020	07/01/2021	N	VOC	NE
4940	METHYL ACETATE	06/15/2020	07/01/2021	N	VOC	NE
4990	METHYL METHACRYLATE	06/15/2020	07/01/2021	N	VOC	NE
5000	METHYL TERT-BUTYL ETHER (MTBE)	06/15/2020	07/01/2021	N	VOC	NE
4965	METHYLCYCLOHEXANE	06/15/2020	07/01/2021	N	VOC	NE
4425	N-BUTYL ALCOHOL	06/15/2020	07/01/2021	N	VOC	NE
4435	N-BUTYLBENZENE	06/15/2020	07/01/2021	N	VOC	NE
4855	N-HEXANE (HEXANE)	06/15/2020	07/01/2021	N	VOC	NE
5090	N-PROPYLBENZENE	06/15/2020	07/01/2021	N	VOC	NE
5005	NAPHTHALENE	06/15/2020	07/01/2021	N	VOC	NE
5250	O-XYLENE	06/15/2020	07/01/2021	N	VOC	NE
5035	PENTACHLOROETHANE	06/15/2020	07/01/2021	N	VOC	NE
4440	SEC-BUTYLBENZENE	06/15/2020	07/01/2021	N	VOC	NE
5100	STYRENE	06/15/2020	07/01/2021	N	VOC	NE
4370	TERT-AMYL METHYL ETHER (TAME)	06/15/2020	07/01/2021	N	VOC	NE
4420	TERT-BUTYL ALCOHOL (TBA)	06/15/2020	07/01/2021	N	VOC	NE
4445	TERT-BUTYLBENZENE	06/15/2020	07/01/2021	N	VOC	NE
5115	TETRACHLOROETHENE (PERCHLOROETHYLENE)	06/15/2020	07/01/2021	N	VOC	NE
5120	TETRAHYDROFURAN (THF)	06/15/2020	07/01/2021	N	VOC	NE
5140	TOLUENE	06/15/2020	07/01/2021	N	VOC	NE
4700	TRANS-1,2-DICLOROETHYLENE	06/15/2020	07/01/2021	N	VOC	NE
4685	TRANS-1,3-DICHLOROPROPENE	06/15/2020	07/01/2021	N	VOC	NE
4605	TRANS-1,4-DICHLORO-2-BUTENE	06/15/2020	07/01/2021	N	VOC	NE
5170	TRICHLOROETHENE (TRICHLOROETHYLENE)	06/15/2020	07/01/2021	N	VOC	NE
5175	TRICHLOROFLUOROMETHANE	06/15/2020	07/01/2021	N	VOC	NE
5225	VINYL ACETATE	06/15/2020	07/01/2021	N	VOC	NE
5235	VINYL CHLORIDE	06/15/2020	07/01/2021	N	VOC	NE
5260	XYLENE (TOTAL)	06/15/2020	07/01/2021	N	VOC	NE
Method Code: 10307138	**Method Ref: SW-846 8260D SIM**		**Revision:**		**Date: 2018**	
4735	1,4-DIOXANE (1 4-DIETHYLENEOXIDE)	06/15/2020	07/01/2021	N	VOC	NE
Method Code: 90017202	**Method Ref: MADEP EPH**		**Revision: 1.1**		**Date: 2004**	
5005	NAPHTHALENE	08/20/2018	07/01/2021	N	VOC	NE
Method Code: 90017451	**Method Ref: MADEP VPH**		**Revision: 2.1**		**Date: 2018**	
4375	BENZENE	05/10/2019	07/01/2021	N	VOC	NE
4765	ETHYLBENZENE	05/10/2019	07/01/2021	N	VOC	NE

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5240	M/P-XYLENE	05/10/2019	07/01/2021	N	VOC	NE
5000	METHYL TERT-BUTYL ETHER (MTBE)	05/10/2019	07/01/2021	N	VOC	NE
5005	NAPHTHALENE	05/10/2019	07/01/2021	N	VOC	NE
5250	O-XYLENE	05/10/2019	07/01/2021	N	VOC	NE
5140	TOLUENE	05/10/2019	07/01/2021	N	VOC	NE
5304	VPH ALIPHATIC C5-C8	05/10/2019	07/01/2021	N	VOC	NE
5306	VPH ALIPHATIC C9-C12	05/10/2019	07/01/2021	N	VOC	NE
5311	VPH AROMATIC C9-C10	05/10/2019	07/01/2021	N	VOC	NE
5260	XYLENE (TOTAL)	05/10/2019	07/01/2021	N	VOC	NE
Method Code: NH0308	**Method Ref: MADEP EPH**		**Revision: 2.1**		**Date:**	
5005	NAPHTHALENE	06/15/2020	07/01/2021	N	VOC	NE
Method Code: 10107401	**Method Ref: EPA 625**		**Revision:**		**Date: 1984**	
6220	1,2-DIPHENYLHYDRAZINE	12/29/2017	07/01/2021	N	SBN	NE
4659	2,2'-OXYBIS(1-CHLOROPROPANE)	12/29/2017	07/01/2021	N	SBN	NE
6735	2,3,4,6-TETRACHLOROPHENOL	12/29/2017	07/01/2021	N	SBN	NE
6835	2,4,5-TRICHLOROPHENOL	12/29/2017	07/01/2021	N	SBN	NE
6840	2,4,6-TRICHLOROPHENOL	12/29/2017	07/01/2021	N	SBN	NE
6000	2,4-DICHLOROPHENOL	12/29/2017	07/01/2021	N	SBN	NE
6130	2,4-DIMETHYLPHENOL	12/29/2017	07/01/2021	N	SBN	NE
6175	2,4-DINITROPHENOL	12/29/2017	07/01/2021	N	SBN	NE
6185	2,4-DINITROTOLUENE (2 4-DNT)	12/29/2017	07/01/2021	N	SBN	NE
6190	2,6-DINITROTOLUENE (2 6-DNT)	12/29/2017	07/01/2021	N	SBN	NE
5795	2-CHLORONAPHTHALENE	12/29/2017	07/01/2021	N	SBN	NE
5800	2-CHLOROPHENOL	12/29/2017	07/01/2021	N	SBN	NE
6360	2-METHYL-4,6-DINITROPHENOL	12/29/2017	07/01/2021	N	SBN	NE
6385	2-METHYLNAPHTHALENE	12/29/2017	07/01/2021	N	SBN	NE
6400	2-METHYLPHENOL (O-CRESOL)	12/29/2017	07/01/2021	N	SBN	NE
6460	2-NITROANILINE	12/29/2017	07/01/2021	N	SBN	NE
6490	2-NITROPHENOL	12/29/2017	07/01/2021	N	SBN	NE
5945	3,3-DICHLOROBENZIDINE	12/29/2017	07/01/2021	N	SBN	NE
6405	3-METHYLPHENOL (M-CRESOL)	12/29/2017	07/01/2021	N	SBN	NE
6465	3-NITROANILINE	12/29/2017	07/01/2021	N	SBN	NE
5660	4-BROMOPHENYL PHENYL ETHER	12/29/2017	07/01/2021	N	SBN	NE
5700	4-CHLORO-3-METHYLPHENOL	12/29/2017	07/01/2021	N	SBN	NE
5745	4-CHLOROANILINE	12/29/2017	07/01/2021	N	SBN	NE
5825	4-CHLOROPHENYL PHENYL ETHER	12/29/2017	07/01/2021	N	SBN	NE

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6410	4-METHYLPHENOL (P-CRESOL)	12/29/2017	07/01/2021	N	SBN	NE
6470	4-NITROANILINE	12/29/2017	07/01/2021	N	SBN	NE
6500	4-NITROPHENOL	12/29/2017	07/01/2021	N	SBN	NE
5500	ACENAPHTHENE	12/29/2017	07/01/2021	N	SBN	NE
5505	ACENAPHTHYLENE	12/29/2017	07/01/2021	N	SBN	NE
5510	ACETOPHENONE	12/29/2017	07/01/2021	N	SBN	NE
5545	ANILINE	12/29/2017	07/01/2021	N	SBN	NE
5555	ANTHRACENE	12/29/2017	07/01/2021	N	SBN	NE
5595	BENZIDINE	12/29/2017	07/01/2021	N	SBN	NE
5575	BENZO(A)ANTHRACENE	12/29/2017	07/01/2021	N	SBN	NE
5580	BENZO(A)PYRENE	12/29/2017	07/01/2021	N	SBN	NE
5585	BENZO(B)FLUORANTHENE	12/29/2017	07/01/2021	N	SBN	NE
5590	BENZO(G,H,I)PERYLENE	12/29/2017	07/01/2021	N	SBN	NE
5600	BENZO(K)FLUORANTHENE	12/29/2017	07/01/2021	N	SBN	NE
5610	BENZOIC ACID	12/29/2017	07/01/2021	N	SBN	NE
5630	BENZYL ALCOHOL	12/29/2017	07/01/2021	N	SBN	NE
5670	BENZYL BUTYL PHTHALATE	12/29/2017	07/01/2021	N	SBN	NE
5760	BIS(2-CHLOROETHOXY) METHANE	12/29/2017	07/01/2021	N	SBN	NE
5765	BIS(2-CHLOROETHYL) ETHER	12/29/2017	07/01/2021	N	SBN	NE
6065	BIS(2-ETHYLHEXYL) PHTHALATE	12/29/2017	07/01/2021	N	SBN	NE
5680	CARBAZOLE	12/29/2017	07/01/2021	N	SBN	NE
5855	CHRYSENE	12/29/2017	07/01/2021	N	SBN	NE
5925	DI-N-BUTYL PHTHALATE	12/29/2017	07/01/2021	N	SBN	NE
6200	DI-N-OCTYL PHTHALATE	12/29/2017	07/01/2021	N	SBN	NE
5895	DIBENZO(A,H)ANTHRACENE	12/29/2017	07/01/2021	N	SBN	NE
5905	DIBENZOFURAN	12/29/2017	07/01/2021	N	SBN	NE
6070	DIETHYL PHTHALATE	12/29/2017	07/01/2021	N	SBN	NE
6135	DIMETHYL PHTHALATE	12/29/2017	07/01/2021	N	SBN	NE
6265	FLUORANTHENE	12/29/2017	07/01/2021	N	SBN	NE
6270	FLUORENE	12/29/2017	07/01/2021	N	SBN	NE
6275	HEXACHLOROBENZENE	12/29/2017	07/01/2021	N	SBN	NE
6315	INDENO(1,2,3-CD)PYRENE	12/29/2017	07/01/2021	N	SBN	NE
6320	ISOPHORONE	12/29/2017	07/01/2021	N	SBN	NE
5875	N-DECANE	12/29/2017	07/01/2021	N	SBN	NE
6545	N-NITROSODI-N-PROPYLAMINE	12/29/2017	07/01/2021	N	SBN	NE
6525	N-NITROSODIETHYLAMINE	12/29/2017	07/01/2021	N	SBN	NE

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6530	N-NITROSODIMETHYLAMINE	12/29/2017	07/01/2021	N	SBN	NE
6535	N-NITROSODIPHENYLAMINE	12/29/2017	07/01/2021	N	SBN	NE
6565	N-NITROSOPYRROLIDINE	12/29/2017	07/01/2021	N	SBN	NE
6580	N-OCTADECANE	12/29/2017	07/01/2021	N	SBN	NE
6605	PENTACHLOROPHENOL	12/29/2017	07/01/2021	N	SBN	NE
6615	PHENANTHRENE	12/29/2017	07/01/2021	N	SBN	NE
6625	PHENOL	12/29/2017	07/01/2021	N	SBN	NE
6665	PYRENE	12/29/2017	07/01/2021	N	SBN	NE
Method Code: 10186002	**Method Ref: SW-846 8270D**		**Revision:**		**Date: 2007**	
6703	1,1'-BIPHENYL (BZ-0)	02/21/2017	07/01/2021	N	SBN	NE
6715	1,2,4,5-TETRACHLOROBENZENE	02/21/2017	07/01/2021	N	SBN	NE
6220	1,2-DIPHENYLHYDRAZINE	02/21/2017	07/01/2021	N	SBN	NE
6160	1,3-DINITROBENZENE (1,3-DNB)	02/21/2017	07/01/2021	N	SBN	NE
6380	1-METHYLNAPHTHALENE	02/21/2017	07/01/2021	N	SBN	NE
4659	2,2'-OXYBIS(1-CHLOROPROPANE)	02/21/2017	07/01/2021	N	SBN	NE
6735	2,3,4,6-TETRACHLOROPHENOL	02/21/2017	07/01/2021	N	SBN	NE
6018	2,3-DIMETHYLANILINE	09/05/2017	07/01/2021	N	SBN	NE
6835	2,4,5-TRICHLOROPHENOL	02/21/2017	07/01/2021	N	SBN	NE
6880	2,4,5-TRIMETHYLANILINE	09/05/2017	07/01/2021	N	SBN	NE
6840	2,4,6-TRICHLOROPHENOL	02/21/2017	07/01/2021	N	SBN	NE
6000	2,4-DICHLOROPHENOL	02/21/2017	07/01/2021	N	SBN	NE
6130	2,4-DIMETHYLPHENOL	02/21/2017	07/01/2021	N	SBN	NE
6175	2,4-DINITROPHENOL	02/21/2017	07/01/2021	N	SBN	NE
6185	2,4-DINITROTOLUENE (2 4-DNT)	02/21/2017	07/01/2021	N	SBN	NE
6190	2,6-DINITROTOLUENE (2 6-DNT)	02/21/2017	07/01/2021	N	SBN	NE
5795	2-CHLORONAPHTHALENE	02/21/2017	07/01/2021	N	SBN	NE
5800	2-CHLOROPHENOL	02/21/2017	07/01/2021	N	SBN	NE
5869	2-ETHYLANILINE	09/05/2017	07/01/2021	N	SBN	NE
6360	2-METHYL-4,6-DINITROPHENOL	02/21/2017	07/01/2021	N	SBN	NE
6385	2-METHYLNAPHTHALENE	02/21/2017	07/01/2021	N	SBN	NE
6400	2-METHYLPHENOL (O-CRESOL)	02/21/2017	07/01/2021	N	SBN	NE
6430	2-NAPHTHYLAMINE	08/14/2019	07/01/2021	N	SBN	NE
6460	2-NITROANILINE	02/21/2017	07/01/2021	N	SBN	NE
6490	2-NITROPHENOL	02/21/2017	07/01/2021	N	SBN	NE
5945	3,3-DICHLOROBENZIDINE	02/21/2017	07/01/2021	N	SBN	NE
6036	3,4-DIMETHYLANILINE	09/05/2017	07/01/2021	N	SBN	NE

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6405	3-METHYLPHENOL (M-CRESOL)	02/21/2017	07/01/2021	N	SBN	NE
6465	3-NITROANILINE	02/21/2017	07/01/2021	N	SBN	NE
5660	4-BROMOPHENYL PHENYL ETHER	02/21/2017	07/01/2021	N	SBN	NE
5700	4-CHLORO-3-METHYLPHENOL	02/21/2017	07/01/2021	N	SBN	NE
0142NH	4-CHLORO-O-TOLUIDINE	09/05/2017	07/01/2021	N	SBN	NE
5745	4-CHLOROANILINE	02/21/2017	07/01/2021	N	SBN	NE
5825	4-CHLOROPHENYL PHENYL ETHER	02/21/2017	07/01/2021	N	SBN	NE
6410	4-METHYLPHENOL (P-CRESOL)	02/21/2017	07/01/2021	N	SBN	NE
6470	4-NITROANILINE	02/21/2017	07/01/2021	N	SBN	NE
6500	4-NITROPHENOL	02/21/2017	07/01/2021	N	SBN	NE
5500	ACENAPHTHENE	02/21/2017	07/01/2021	N	SBN	NE
5505	ACENAPHTHYLENE	02/21/2017	07/01/2021	N	SBN	NE
5510	ACETOPHENONE	02/21/2017	07/01/2021	N	SBN	NE
5545	ANILINE	02/21/2017	07/01/2021	N	SBN	NE
5555	ANTHRACENE	02/21/2017	07/01/2021	N	SBN	NE
5570	BENZALDEHYDE	02/21/2017	07/01/2021	N	SBN	NE
5595	BENZIDINE	02/21/2017	07/01/2021	N	SBN	NE
5575	BENZO(A)ANTHRACENE	02/21/2017	07/01/2021	N	SBN	NE
5580	BENZO(A)PYRENE	02/21/2017	07/01/2021	N	SBN	NE
5585	BENZO(B)FLUORANTHENE	02/21/2017	07/01/2021	N	SBN	NE
5590	BENZO(G,H,I)PERYLENE	02/21/2017	07/01/2021	N	SBN	NE
5600	BENZO(K)FLUORANTHENE	02/21/2017	07/01/2021	N	SBN	NE
5610	BENZOIC ACID	02/21/2017	07/01/2021	N	SBN	NE
5630	BENZYL ALCOHOL	02/21/2017	07/01/2021	N	SBN	NE
5670	BENZYL BUTYL PHTHALATE	02/21/2017	07/01/2021	N	SBN	NE
5760	BIS(2-CHLOROETHOXY) METHANE	02/21/2017	07/01/2021	N	SBN	NE
5765	BIS(2-CHLOROETHYL) ETHER	02/21/2017	07/01/2021	N	SBN	NE
6065	BIS(2-ETHYLHEXYL) PHTHALATE	02/21/2017	07/01/2021	N	SBN	NE
5680	CARBAZOLE	02/21/2017	07/01/2021	N	SBN	NE
5855	CHRYSENE	02/21/2017	07/01/2021	N	SBN	NE
5925	DI-N-BUTYL PHTHALATE	02/21/2017	07/01/2021	N	SBN	NE
6200	DI-N-OCTYL PHTHALATE	02/21/2017	07/01/2021	N	SBN	NE
5895	DIBENZO(A,H)ANTHRACENE	02/21/2017	07/01/2021	N	SBN	NE
5905	DIBENZOFURAN	02/21/2017	07/01/2021	N	SBN	NE
6070	DIETHYL PHTHALATE	02/21/2017	07/01/2021	N	SBN	NE
6135	DIMETHYL PHTHALATE	02/21/2017	07/01/2021	N	SBN	NE

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6205	DIPHENYLAMINE	02/21/2017	07/01/2021	N	SBN	NE
6265	FLUORANTHENE	02/21/2017	07/01/2021	N	SBN	NE
6270	FLUORENE	02/21/2017	07/01/2021	N	SBN	NE
6275	HEXACHLOROBENZENE	02/21/2017	07/01/2021	N	SBN	NE
6315	INDENO(1,2,3-CD)PYRENE	02/21/2017	07/01/2021	N	SBN	NE
6320	ISOPHORONE	02/21/2017	07/01/2021	N	SBN	NE
6545	N-NITROSODI-N-PROPYLAMINE	02/21/2017	07/01/2021	N	SBN	NE
6530	N-NITROSODIMETHYLAMINE	02/21/2017	07/01/2021	N	SBN	NE
6535	N-NITROSODIPHENYLAMINE	02/21/2017	07/01/2021	N	SBN	NE
6600	PCNB (PENTACHLORONITROBENZENE)	02/21/2017	07/01/2021	N	SBN	NE
6605	PENTACHLOROPHENOL	02/21/2017	07/01/2021	N	SBN	NE
6615	PHENANTHRENE	02/21/2017	07/01/2021	N	SBN	NE
6625	PHENOL	02/21/2017	07/01/2021	N	SBN	NE
6665	PYRENE	02/21/2017	07/01/2021	N	SBN	NE
Method Code: 10242509	**Method Ref: SW-846 8270D SIM**		**Revision:**		**Date: 2007**	
6360	2-METHYL-4,6-DINITROPHENOL	02/21/2017	07/01/2021	N	SBN	NE
6385	2-METHYLNAPHTHALENE	02/21/2017	07/01/2021	N	SBN	NE
5500	ACENAPHTHENE	02/21/2017	07/01/2021	N	SBN	NE
5505	ACENAPHTHYLENE	02/21/2017	07/01/2021	N	SBN	NE
5555	ANTHRACENE	02/21/2017	07/01/2021	N	SBN	NE
5575	BENZO(A)ANTHRACENE	02/21/2017	07/01/2021	N	SBN	NE
5580	BENZO(A)PYRENE	02/21/2017	07/01/2021	N	SBN	NE
5585	BENZO(B)FLUORANTHENE	02/21/2017	07/01/2021	N	SBN	NE
5590	BENZO(G,H,I)PERYLENE	02/21/2017	07/01/2021	N	SBN	NE
5600	BENZO(K)FLUORANTHENE	02/21/2017	07/01/2021	N	SBN	NE
5855	CHRYSENE	02/21/2017	07/01/2021	N	SBN	NE
5895	DIBENZO(A,H)ANTHRACENE	02/21/2017	07/01/2021	N	SBN	NE
6265	FLUORANTHENE	02/21/2017	07/01/2021	N	SBN	NE
6270	FLUORENE	02/21/2017	07/01/2021	N	SBN	NE
6275	HEXACHLOROBENZENE	02/21/2017	07/01/2021	N	SBN	NE
6315	INDENO(1,2,3-CD)PYRENE	02/21/2017	07/01/2021	N	SBN	NE
6605	PENTACHLOROPHENOL	02/21/2017	07/01/2021	N	SBN	NE
6615	PHENANTHRENE	02/21/2017	07/01/2021	N	SBN	NE
6665	PYRENE	02/21/2017	07/01/2021	N	SBN	NE
Method Code: 10242543	**Method Ref: SW-846 8270E**		**Revision:**		**Date: 2017**	
6703	1,1'-BIPHENYL (BZ-0)	06/15/2020	07/01/2021	N	SBN	NE



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6715	1,2,4,5-TETRACHLOROBENZENE	06/15/2020	07/01/2021	N	SBN	NE
6220	1,2-DIPHENYLHYDRAZINE	06/15/2020	07/01/2021	N	SBN	NE
6160	1,3-DINITROBENZENE (1,3-DNB)	06/15/2020	07/01/2021	N	SBN	NE
4659	2,2'-OXYBIS(1-CHLOROPROPANE)	06/15/2020	07/01/2021	N	SBN	NE
6735	2,3,4,6-TETRACHLOROPHENOL	06/15/2020	07/01/2021	N	SBN	NE
6018	2,3-DIMETHYLANILINE	06/15/2020	07/01/2021	N	SBN	NE
6835	2,4,5-TRICHLOROPHENOL	06/15/2020	07/01/2021	N	SBN	NE
6880	2,4,5-TRIMETHYLANILINE	06/15/2020	07/01/2021	N	SBN	NE
6840	2,4,6-TRICHLOROPHENOL	06/15/2020	07/01/2021	N	SBN	NE
6000	2,4-DICHLOROPHENOL	06/15/2020	07/01/2021	N	SBN	NE
6130	2,4-DIMETHYLPHENOL	06/15/2020	07/01/2021	N	SBN	NE
6175	2,4-DINITROPHENOL	06/15/2020	07/01/2021	N	SBN	NE
6185	2,4-DINITROTOLUENE (2 4-DNT)	06/15/2020	07/01/2021	N	SBN	NE
6190	2,6-DINITROTOLUENE (2 6-DNT)	06/15/2020	07/01/2021	N	SBN	NE
5795	2-CHLORONAPHTHALENE	06/15/2020	07/01/2021	N	SBN	NE
5800	2-CHLOROPHENOL	06/15/2020	07/01/2021	N	SBN	NE
5869	2-ETHYLANILINE	06/15/2020	07/01/2021	N	SBN	NE
6400	2-METHYLPHENOL (O-CRESOL)	06/15/2020	07/01/2021	N	SBN	NE
6430	2-NAPHTHYLAMINE	06/15/2020	07/01/2021	N	SBN	NE
6460	2-NITROANILINE	06/15/2020	07/01/2021	N	SBN	NE
6490	2-NITROPHENOL	06/15/2020	07/01/2021	N	SBN	NE
5945	3,3-DICHLOROBENZIDINE	06/15/2020	07/01/2021	N	SBN	NE
6036	3,4-DIMETHYLANILINE	06/15/2020	07/01/2021	N	SBN	NE
6405	3-METHYLPHENOL (M-CRESOL)	06/15/2020	07/01/2021	N	SBN	NE
6465	3-NITROANILINE	06/15/2020	07/01/2021	N	SBN	NE
5660	4-BROMOPHENYL PHENYL ETHER	06/15/2020	07/01/2021	N	SBN	NE
5700	4-CHLORO-3-METHYLPHENOL	06/15/2020	07/01/2021	N	SBN	NE
0142NH	4-CHLORO-O-TOLUIDINE	06/15/2020	07/01/2021	N	SBN	NE
5745	4-CHLOROANILINE	06/15/2020	07/01/2021	N	SBN	NE
5825	4-CHLOROPHENYL PHENYL ETHER	06/15/2020	07/01/2021	N	SBN	NE
6410	4-METHYLPHENOL (P-CRESOL)	06/15/2020	07/01/2021	N	SBN	NE
6470	4-NITROANILINE	06/15/2020	07/01/2021	N	SBN	NE
6500	4-NITROPHENOL	06/15/2020	07/01/2021	N	SBN	NE
5500	ACENAPHTHENE	06/15/2020	07/01/2021	N	SBN	NE
5505	ACENAPHTHYLENE	06/15/2020	07/01/2021	N	SBN	NE
5510	ACETOPHENONE	06/15/2020	07/01/2021	N	SBN	NE

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5545	ANILINE	06/15/2020	07/01/2021	N	SBN	NE
5555	ANTHRACENE	06/15/2020	07/01/2021	N	SBN	NE
5570	BENZALDEHYDE	06/15/2020	07/01/2021	N	SBN	NE
5595	BENZIDINE	06/15/2020	07/01/2021	N	SBN	NE
5585	BENZO(B)FLUORANTHENE	06/15/2020	07/01/2021	N	SBN	NE
5590	BENZO(G,H,I)PERYLENE	06/15/2020	07/01/2021	N	SBN	NE
5600	BENZO(K)FLUORANTHENE	06/15/2020	07/01/2021	N	SBN	NE
5610	BENZOIC ACID	06/15/2020	07/01/2021	N	SBN	NE
5630	BENZYL ALCOHOL	06/15/2020	07/01/2021	N	SBN	NE
5670	BENZYL BUTYL PHTHALATE	06/15/2020	07/01/2021	N	SBN	NE
5760	BIS(2-CHLOROETHOXY) METHANE	06/15/2020	07/01/2021	N	SBN	NE
5765	BIS(2-CHLOROETHYL) ETHER	06/15/2020	07/01/2021	N	SBN	NE
5680	CARBAZOLE	06/15/2020	07/01/2021	N	SBN	NE
5855	CHRYSENE	06/15/2020	07/01/2021	N	SBN	NE
5925	DI-N-BUTYL PHTHALATE	06/15/2020	07/01/2021	N	SBN	NE
6200	DI-N-OCTYL PHTHALATE	06/15/2020	07/01/2021	N	SBN	NE
5895	DIBENZO(A,H)ANTHRACENE	06/15/2020	07/01/2021	N	SBN	NE
5905	DIBENZOFURAN	06/15/2020	07/01/2021	N	SBN	NE
6070	DIETHYL PHTHALATE	06/15/2020	07/01/2021	N	SBN	NE
6135	DIMETHYL PHTHALATE	06/15/2020	07/01/2021	N	SBN	NE
6205	DIPHENYLAMINE	06/15/2020	07/01/2021	N	SBN	NE
6265	FLUORANTHENE	06/15/2020	07/01/2021	N	SBN	NE
6270	FLUORENE	06/15/2020	07/01/2021	N	SBN	NE
6275	HEXACHLOROBENZENE	06/15/2020	07/01/2021	N	SBN	NE
6545	N-NITROSODI-N-PROPYLAMINE	06/15/2020	07/01/2021	N	SBN	NE
6530	N-NITROSODIMETHYLAMINE	06/15/2020	07/01/2021	N	SBN	NE
6535	N-NITROSODIPHENYLAMINE	06/15/2020	07/01/2021	N	SBN	NE
6600	PCNB (PENTACHLORONITROBENZENE)	06/15/2020	07/01/2021	N	SBN	NE
6605	PENTACHLOROPHENOL	06/15/2020	07/01/2021	N	SBN	NE
6615	PHENANTHRENE	06/15/2020	07/01/2021	N	SBN	NE
6625	PHENOL	06/15/2020	07/01/2021	N	SBN	NE
6665	PYRENE	06/15/2020	07/01/2021	N	SBN	NE
Method Code: 10242565	**Method Ref: SW-846 8270E SIM**		**Revision:**		**Date: 2017**	
6360	2-METHYL-4,6-DINITROPHENOL	06/15/2020	07/01/2021	N	SBN	NE
6385	2-METHYLNAPHTHALENE	06/15/2020	07/01/2021	N	SBN	NE
5500	ACENAPHTHENE	06/15/2020	07/01/2021	N	SBN	NE

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5505	ACENAPHTHYLENE	06/15/2020	07/01/2021	N	SBN	NE
5555	ANTHRACENE	06/15/2020	07/01/2021	N	SBN	NE
5575	BENZO(A)ANTHRACENE	06/15/2020	07/01/2021	N	SBN	NE
5580	BENZO(A)PYRENE	06/15/2020	07/01/2021	N	SBN	NE
5585	BENZO(B)FLUORANTHENE	06/15/2020	07/01/2021	N	SBN	NE
5590	BENZO(G,H,I)PERYLENE	06/15/2020	07/01/2021	N	SBN	NE
5600	BENZO(K)FLUORANTHENE	06/15/2020	07/01/2021	N	SBN	NE
5855	CHRYSENE	06/15/2020	07/01/2021	N	SBN	NE
5895	DIBENZO(A,H)ANTHRACENE	06/15/2020	07/01/2021	N	SBN	NE
6265	FLUORANTHENE	06/15/2020	07/01/2021	N	SBN	NE
6270	FLUORENE	06/15/2020	07/01/2021	N	SBN	NE
6275	HEXACHLOROBENZENE	06/15/2020	07/01/2021	N	SBN	NE
6315	INDENO(1,2,3-CD)PYRENE	06/15/2020	07/01/2021	N	SBN	NE
6605	PENTACHLOROPHENOL	06/15/2020	07/01/2021	N	SBN	NE
6615	PHENANTHRENE	06/15/2020	07/01/2021	N	SBN	NE
6665	PYRENE	06/15/2020	07/01/2021	N	SBN	NE
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6220	1,2-DIPHENYLHYDRAZINE	07/02/2018	07/01/2021	N	SBN	NE
6380	1-METHYLNAPHTHALENE	07/02/2018	07/01/2021	N	SBN	NE
4659	2,2'-OXYBIS(1-CHLOROPROPANE)	07/02/2018	07/01/2021	N	SBN	NE
6735	2,3,4,6-TETRACHLOROPHENOL	07/02/2018	07/01/2021	N	SBN	NE
6835	2,4,5-TRICHLOROPHENOL	07/02/2018	07/01/2021	N	SBN	NE
6840	2,4,6-TRICHLOROPHENOL	07/02/2018	07/01/2021	N	SBN	NE
6000	2,4-DICHLOROPHENOL	07/02/2018	07/01/2021	N	SBN	NE
6130	2,4-DIMETHYLPHENOL	07/02/2018	07/01/2021	N	SBN	NE
6175	2,4-DINITROPHENOL	07/02/2018	07/01/2021	N	SBN	NE
6185	2,4-DINITROTOLUENE (2 4-DNT)	07/02/2018	07/01/2021	N	SBN	NE
6190	2,6-DINITROTOLUENE (2 6-DNT)	07/02/2018	07/01/2021	N	SBN	NE
5795	2-CHLORONAPHTHALENE	07/02/2018	07/01/2021	N	SBN	NE
5800	2-CHLOROPHENOL	07/02/2018	07/01/2021	N	SBN	NE
6360	2-METHYL-4,6-DINITROPHENOL	07/02/2018	07/01/2021	N	SBN	NE
6385	2-METHYLNAPHTHALENE	07/02/2018	07/01/2021	N	SBN	NE
6400	2-METHYLPHENOL (O-CRESOL)	07/02/2018	07/01/2021	N	SBN	NE
6460	2-NITROANILINE	07/02/2018	07/01/2021	N	SBN	NE
6490	2-NITROPHENOL	07/02/2018	07/01/2021	N	SBN	NE
5945	3,3-DICHLOROBENZIDINE	07/02/2018	07/01/2021	N	SBN	NE

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6412	3,4-METHYLPHENOLS (M,P-CRESOLS)	07/02/2018	07/01/2021	N	SBN	NE
6405	3-METHYLPHENOL (M-CRESOL)	07/02/2018	07/01/2021	N	SBN	NE
6465	3-NITROANILINE	07/02/2018	07/01/2021	N	SBN	NE
5660	4-BROMOPHENYL PHENYL ETHER	07/02/2018	07/01/2021	N	SBN	NE
5700	4-CHLORO-3-METHYLPHENOL	07/02/2018	07/01/2021	N	SBN	NE
5745	4-CHLOROANILINE	07/02/2018	07/01/2021	N	SBN	NE
5825	4-CHLOROPHENYL PHENYL ETHER	07/02/2018	07/01/2021	N	SBN	NE
6410	4-METHYLPHENOL (P-CRESOL)	07/02/2018	07/01/2021	N	SBN	NE
6470	4-NITROANILINE	07/02/2018	07/01/2021	N	SBN	NE
6500	4-NITROPHENOL	07/02/2018	07/01/2021	N	SBN	NE
5500	ACENAPHTHENE	07/02/2018	07/01/2021	N	SBN	NE
5505	ACENAPHTHYLENE	07/02/2018	07/01/2021	N	SBN	NE
5510	ACETOPHENONE	07/02/2018	07/01/2021	N	SBN	NE
5545	ANILINE	07/02/2018	07/01/2021	N	SBN	NE
5555	ANTHRACENE	07/02/2018	07/01/2021	N	SBN	NE
5562	AZOBENZENE	07/02/2018	07/01/2021	N	SBN	NE
5595	BENZIDINE	07/02/2018	07/01/2021	N	SBN	NE
5575	BENZO(A)ANTHRACENE	07/02/2018	07/01/2021	N	SBN	NE
5580	BENZO(A)PYRENE	07/02/2018	07/01/2021	N	SBN	NE
5585	BENZO(B)FLUORANTHENE	07/02/2018	07/01/2021	N	SBN	NE
5590	BENZO(G,H,I)PERYLENE	07/02/2018	07/01/2021	N	SBN	NE
5600	BENZO(K)FLUORANTHENE	07/02/2018	07/01/2021	N	SBN	NE
5610	BENZOIC ACID	07/02/2018	07/01/2021	N	SBN	NE
5630	BENZYL ALCOHOL	07/02/2018	07/01/2021	N	SBN	NE
5670	BENZYL BUTYL PHTHALATE	07/02/2018	07/01/2021	N	SBN	NE
5760	BIS(2-CHLOROETHOXY) METHANE	07/02/2018	07/01/2021	N	SBN	NE
5765	BIS(2-CHLOROETHYL) ETHER	07/02/2018	07/01/2021	N	SBN	NE
6065	BIS(2-ETHYLHEXYL) PHTHALATE	07/02/2018	07/01/2021	N	SBN	NE
5680	CARBAZOLE	07/02/2018	07/01/2021	N	SBN	NE
5855	CHRYSENE	07/02/2018	07/01/2021	N	SBN	NE
5925	DI-N-BUTYL PHTHALATE	07/02/2018	07/01/2021	N	SBN	NE
6200	DI-N-OCTYL PHTHALATE	07/02/2018	07/01/2021	N	SBN	NE
5895	DIBENZO(A,H)ANTHRACENE	07/02/2018	07/01/2021	N	SBN	NE
5905	DIBENZOFURAN	07/02/2018	07/01/2021	N	SBN	NE
6070	DIETHYL PHTHALATE	07/02/2018	07/01/2021	N	SBN	NE
6135	DIMETHYL PHTHALATE	07/02/2018	07/01/2021	N	SBN	NE

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6265	FLUORANTHENE	07/02/2018	07/01/2021	N	SBN	NE
6270	FLUORENE	07/02/2018	07/01/2021	N	SBN	NE
6275	HEXACHLOROBENZENE	07/02/2018	07/01/2021	N	SBN	NE
6315	INDENO(1,2,3-CD)PYRENE	07/02/2018	07/01/2021	N	SBN	NE
6320	ISOPHORONE	07/02/2018	07/01/2021	N	SBN	NE
5875	N-DECANE	07/02/2018	07/01/2021	N	SBN	NE
6545	N-NITROSODI-N-PROPYLAMINE	07/02/2018	07/01/2021	N	SBN	NE
6525	N-NITROSODIETHYLAMINE	07/02/2018	07/01/2021	N	SBN	NE
6530	N-NITROSODIMETHYLAMINE	07/02/2018	07/01/2021	N	SBN	NE
6535	N-NITROSODIPHENYLAMINE	07/02/2018	07/01/2021	N	SBN	NE
6565	N-NITROSOPYRROLIDINE	07/02/2018	07/01/2021	N	SBN	NE
6580	N-OCTADECANE	07/02/2018	07/01/2021	N	SBN	NE
6605	PENTACHLOROPHENOL	07/02/2018	07/01/2021	N	SBN	NE
6615	PHENANTHRENE	07/02/2018	07/01/2021	N	SBN	NE
6625	PHENOL	07/02/2018	07/01/2021	N	SBN	NE
6665	PYRENE	07/02/2018	07/01/2021	N	SBN	NE
Method Code: 10307003	**Method Ref: SW-846 8260C**		**Revision:**		**Date:**	
6800	1,3,5-TRICHLOROBENZENE	02/21/2017	07/01/2021	N	SBN	NE
Method Code: 10307127	**Method Ref: SW-846 8260D**		**Revision:**		**Date: 2018**	
6800	1,3,5-TRICHLOROBENZENE	06/15/2020	07/01/2021	N	SBN	NE
Method Code: 90014258	**Method Ref: NJ DEP EPH**		**Revision:**		**Date: 2010**	
3797	TOTAL EPH	02/21/2017	07/01/2021	N	SBN	NE
Method Code: 90017202	**Method Ref: MADEP EPH**		**Revision: 1.1**		**Date: 2004**	
6385	2-METHYLNAPHTHALENE	08/20/2018	07/01/2021	N	SBN	NE
5500	ACENAPHTHENE	08/20/2018	07/01/2021	N	SBN	NE
5505	ACENAPHTHYLENE	08/20/2018	07/01/2021	N	SBN	NE
5555	ANTHRACENE	08/20/2018	07/01/2021	N	SBN	NE
5575	BENZO(A)ANTHRACENE	08/20/2018	07/01/2021	N	SBN	NE
5580	BENZO(A)PYRENE	08/20/2018	07/01/2021	N	SBN	NE
5585	BENZO(B)FLUORANTHENE	08/20/2018	07/01/2021	N	SBN	NE
5590	BENZO(G,H,I)PERYLENE	08/20/2018	07/01/2021	N	SBN	NE
5600	BENZO(K)FLUORANTHENE	08/20/2018	07/01/2021	N	SBN	NE
5855	CHRYSENE	08/20/2018	07/01/2021	N	SBN	NE
5895	DIBENZO(A,H)ANTHRACENE	08/20/2018	07/01/2021	N	SBN	NE
6218	EPH ALIPHATIC C19-C36	08/20/2018	07/01/2021	N	SBN	NE
6222	EPH ALIPHATIC C9-C18	08/20/2018	07/01/2021	N	SBN	NE
6232	EPH AROMATIC C11-C22	08/20/2018	07/01/2021	N	SBN	NE

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6234	EPH AROMATIC C11-C22 UNADJUSTED	08/20/2018	07/01/2021	N	SBN	NE
6265	FLUORANTHENE	08/20/2018	07/01/2021	N	SBN	NE
6270	FLUORENE	08/20/2018	07/01/2021	N	SBN	NE
6315	INDENO(1,2,3-CD)PYRENE	08/20/2018	07/01/2021	N	SBN	NE
6615	PHENANTHRENE	08/20/2018	07/01/2021	N	SBN	NE
6665	PYRENE	08/20/2018	07/01/2021	N	SBN	NE
Method Code: NH0308	**Method Ref: MADEP EPH**		**Revision: 2.1**		**Date:**	
6385	2-METHYLNAPHTHALENE	06/15/2020	07/01/2021	N	SBN	NE
5500	ACENAPHTHENE	06/15/2020	07/01/2021	N	SBN	NE
5505	ACENAPHTHYLENE	06/15/2020	07/01/2021	N	SBN	NE
5555	ANTHRACENE	06/15/2020	07/01/2021	N	SBN	NE
5575	BENZO(A)ANTHRACENE	06/15/2020	07/01/2021	N	SBN	NE
5580	BENZO(A)PYRENE	06/15/2020	07/01/2021	N	SBN	NE
5585	BENZO(B)FLUORANTHENE	06/15/2020	07/01/2021	N	SBN	NE
5590	BENZO(G,H,I)PERYLENE	06/15/2020	07/01/2021	N	SBN	NE
5600	BENZO(K)FLUORANTHENE	06/15/2020	07/01/2021	N	SBN	NE
5855	CHRYSENE	06/15/2020	07/01/2021	N	SBN	NE
5895	DIBENZO(A,H)ANTHRACENE	06/15/2020	07/01/2021	N	SBN	NE
6218	EPH ALIPHATIC C19-C36	06/15/2020	07/01/2021	N	SBN	NE
6222	EPH ALIPHATIC C9-C18	06/15/2020	07/01/2021	N	SBN	NE
6232	EPH AROMATIC C11-C22	06/15/2020	07/01/2021	N	SBN	NE
6234	EPH AROMATIC C11-C22 UNADJUSTED	06/15/2020	07/01/2021	N	SBN	NE
6265	FLUORANTHENE	06/15/2020	07/01/2021	N	SBN	NE
6270	FLUORENE	06/15/2020	07/01/2021	N	SBN	NE
6315	INDENO(1,2,3-CD)PYRENE	06/15/2020	07/01/2021	N	SBN	NE
6615	PHENANTHRENE	06/15/2020	07/01/2021	N	SBN	NE
6665	PYRENE	06/15/2020	07/01/2021	N	SBN	NE
Method Code: 10183207	**Method Ref: SW-846 8151A**		**Revision:**		**Date: 1998**	
8655	2,4,5-T	02/21/2017	07/01/2021	N	SHE	NE
8650	2,4,5-TP (SILVEX)	02/21/2017	07/01/2021	N	SHE	NE
8545	2,4-D	02/21/2017	07/01/2021	N	SHE	NE
8560	2,4-DB	02/21/2017	07/01/2021	N	SHE	NE
8555	DALAPON	02/21/2017	07/01/2021	N	SHE	NE
8595	DICAMBA	02/21/2017	07/01/2021	N	SHE	NE
8605	DICHLOROPROP (DICHLORPROP)	02/21/2017	07/01/2021	N	SHE	NE
8620	DINOSEB (2-SEC-BUTYL-4 6-DINITROPHENOL DNBP)	07/02/2018	07/01/2021	N	SHE	CN

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Method Code: 10186002	**Method Ref: SW-846 8270D**		**Revision:**		**Date: 2007**	
5883	2,4-DIMETHYLANILINE	09/05/2017	07/01/2021	N	SNO	NE
Method Code: 10242543	**Method Ref: SW-846 8270E**		**Revision:**		**Date: 2017**	
5883	2,4-DIMETHYLANILINE	06/15/2020	07/01/2021	N	SNO	NE
Method Code: 10305609	**Method Ref: SW-846 8015D**		**Revision:**		**Date: 2003**	
9369	DIESEL RANGE ORGANICS (DRO)	07/02/2018	07/01/2021	N	SNO	NE
9408	GASOLINE RANGE ORGANICS (GRO)	07/02/2018	07/01/2021	N	SNO	NE
9506	RESIDUAL RANGE ORGANICS (RRO) - OIL RANGE ORGANICS	11/13/2018	07/01/2021	N	SNO	NE
Method Code: 20029316	**Method Ref: SM 5550 B**		**Revision: 20TH ED**		**Date: 1993**	
9597	TANNIN & LIGNIN	02/21/2017	07/01/2021	N	SNO	NE
Method Code: 10103603	**Method Ref: EPA 608**		**Revision:**		**Date: 1984**	
8880	AROCLOR-1016 (PCB-1016)	12/29/2017	07/01/2021	N	SPC	NE
8885	AROCLOR-1221 (PCB-1221)	12/29/2017	07/01/2021	N	SPC	NE
8890	AROCLOR-1232 (PCB-1232)	12/29/2017	07/01/2021	N	SPC	NE
8895	AROCLOR-1242 (PCB-1242)	12/29/2017	07/01/2021	N	SPC	NE
8900	AROCLOR-1248 (PCB-1248)	12/29/2017	07/01/2021	N	SPC	NE
8905	AROCLOR-1254 (PCB-1254)	12/29/2017	07/01/2021	N	SPC	NE
8910	AROCLOR-1260 (PCB-1260)	12/29/2017	07/01/2021	N	SPC	NE
Method Code: 10179201	**Method Ref: SW-846 8082A**		**Revision:**		**Date: 2000**	
8880	AROCLOR-1016 (PCB-1016)	02/21/2017	07/01/2021	N	SPC	NE
8885	AROCLOR-1221 (PCB-1221)	02/21/2017	07/01/2021	N	SPC	NE
8890	AROCLOR-1232 (PCB-1232)	02/21/2017	07/01/2021	N	SPC	NE
8895	AROCLOR-1242 (PCB-1242)	02/21/2017	07/01/2021	N	SPC	NE
8900	AROCLOR-1248 (PCB-1248)	02/21/2017	07/01/2021	N	SPC	NE
8905	AROCLOR-1254 (PCB-1254)	02/21/2017	07/01/2021	N	SPC	NE
8910	AROCLOR-1260 (PCB-1260)	02/21/2017	07/01/2021	N	SPC	NE
8912	AROCLOR-1262 (PCB-1262)	02/21/2017	07/01/2021	N	SPC	NE
8913	AROCLOR-1268 (PCB-1268)	02/21/2017	07/01/2021	N	SPC	NE
Method Code: 10296614	**Method Ref: EPA 608.3 GC-ECD**		**Revision:**		**Date: 2016**	
8880	AROCLOR-1016 (PCB-1016)	07/02/2018	07/01/2021	N	SPC	NE
8885	AROCLOR-1221 (PCB-1221)	07/02/2018	07/01/2021	N	SPC	NE
8890	AROCLOR-1232 (PCB-1232)	07/02/2018	07/01/2021	N	SPC	NE
8895	AROCLOR-1242 (PCB-1242)	07/02/2018	07/01/2021	N	SPC	NE
8900	AROCLOR-1248 (PCB-1248)	07/02/2018	07/01/2021	N	SPC	NE
8905	AROCLOR-1254 (PCB-1254)	07/02/2018	07/01/2021	N	SPC	NE
8910	AROCLOR-1260 (PCB-1260)	07/02/2018	07/01/2021	N	SPC	NE

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Method Code: 10103603	**Method Ref: EPA 608**		**Revision:**		**Date: 1984**	
7355	4,4-DDD	12/29/2017	07/01/2021	N	SPE	NE
7360	4,4-DDE	12/29/2017	07/01/2021	N	SPE	NE
7365	4,4-DDT	12/29/2017	07/01/2021	N	SPE	NE
7025	ALDRIN	12/29/2017	07/01/2021	N	SPE	NE
7110	ALPHA-BHC (ALPHA-HEXACHLOROCYCLOHEXANE)	12/29/2017	07/01/2021	N	SPE	NE
7240	ALPHA-CHLORDANE	12/29/2017	07/01/2021	N	SPE	NE
7115	BETA-BHC (BETA-HEXACHLOROCYCLOHEXANE)	12/29/2017	07/01/2021	N	SPE	NE
7250	CHLORDANE (TECH.)	12/29/2017	07/01/2021	N	SPE	NE
7105	DELTA-BHC	12/29/2017	07/01/2021	N	SPE	NE
7470	DIELDRIN	12/29/2017	07/01/2021	N	SPE	NE
7510	ENDOSULFAN I	12/29/2017	07/01/2021	N	SPE	NE
7515	ENDOSULFAN II	12/29/2017	07/01/2021	N	SPE	NE
7520	ENDOSULFAN SULFATE	12/29/2017	07/01/2021	N	SPE	NE
7540	ENDRIN	12/29/2017	07/01/2021	N	SPE	NE
7530	ENDRIN ALDEHYDE	12/29/2017	07/01/2021	N	SPE	NE
7535	ENDRIN KETONE	12/29/2017	07/01/2021	N	SPE	NE
7120	GAMMA-BHC (LINDANE)	12/29/2017	07/01/2021	N	SPE	NE
7685	HEPTACHLOR	12/29/2017	07/01/2021	N	SPE	NE
7690	HEPTACHLOR EPOXIDE	12/29/2017	07/01/2021	N	SPE	NE
7810	METHOXYCHLOR	12/29/2017	07/01/2021	N	SPE	NE
8250	TOXAPHENE (CHLORINATED CAMPHENE)	12/29/2017	07/01/2021	N	SPE	NE
7972	TRANS-CHLORDANE	12/29/2017	07/01/2021	N	SPE	NE
Method Code: 10107401	**Method Ref: EPA 625**		**Revision:**		**Date: 1984**	
6285	HEXACHLOROCYCLOPENTADIENE	12/29/2017	07/01/2021	N	SPE	NE
Method Code: 10178800	**Method Ref: SW-846 8081B**		**Revision:**		**Date: 2000**	
7355	4,4-DDD	02/21/2017	07/01/2021	N	SPE	NE
7360	4,4-DDE	02/21/2017	07/01/2021	N	SPE	NE
7365	4,4-DDT	02/21/2017	07/01/2021	N	SPE	NE
7005	ALACHLOR	02/21/2017	07/01/2021	N	SPE	NE
7025	ALDRIN	02/21/2017	07/01/2021	N	SPE	NE
7110	ALPHA-BHC (ALPHA-HEXACHLOROCYCLOHEXANE)	02/21/2017	07/01/2021	N	SPE	NE
7240	ALPHA-CHLORDANE	02/21/2017	07/01/2021	N	SPE	NE
7115	BETA-BHC (BETA-HEXACHLOROCYCLOHEXANE)	02/21/2017	07/01/2021	N	SPE	NE
7250	CHLORDANE (TECH.)	02/21/2017	07/01/2021	N	SPE	NE
7105	DELTA-BHC	02/21/2017	07/01/2021	N	SPE	NE
7470	DIELDRIN	02/21/2017	07/01/2021	N	SPE	NE

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7510	ENDOSULFAN I	02/21/2017	07/01/2021	N	SPE	NE
7515	ENDOSULFAN II	02/21/2017	07/01/2021	N	SPE	NE
7520	ENDOSULFAN SULFATE	02/21/2017	07/01/2021	N	SPE	NE
7540	ENDRIN	02/21/2017	07/01/2021	N	SPE	NE
7530	ENDRIN ALDEHYDE	02/21/2017	07/01/2021	N	SPE	NE
7535	ENDRIN KETONE	02/21/2017	07/01/2021	N	SPE	NE
7120	GAMMA-BHC (LINDANE)	02/21/2017	07/01/2021	N	SPE	NE
7245	GAMMA-CHLORDANE	02/21/2017	07/01/2021	N	SPE	NE
7685	HEPTACHLOR	02/21/2017	07/01/2021	N	SPE	NE
7690	HEPTACHLOR EPOXIDE	02/21/2017	07/01/2021	N	SPE	NE
7810	METHOXYCHLOR	02/21/2017	07/01/2021	N	SPE	NE
8250	TOXAPHENE (CHLORINATED CAMPHENE)	02/21/2017	07/01/2021	N	SPE	NE
Method Code: 10183207	**Method Ref: SW-846 8151A**		**Revision:**		**Date: 1998**	
7775	MCPA	02/21/2017	07/01/2021	N	SPE	NE
7780	MCPP	02/21/2017	07/01/2021	N	SPE	NE
Method Code: 10186002	**Method Ref: SW-846 8270D**		**Revision:**		**Date: 2007**	
7065	ATRAZINE	02/21/2017	07/01/2021	N	SPE	NE
7180	CAPROLACTAM	02/21/2017	07/01/2021	N	SPE	NE
6285	HEXACHLOROCYCLOPENTADIENE	02/21/2017	07/01/2021	N	SPE	NE
7955	PARATHION ETHYL	02/21/2017	07/01/2021	N	SPE	NE
8235	THIONAZIN (ZINOPHOS)	02/21/2017	07/01/2021	N	SPE	NE
Method Code: 10242543	**Method Ref: SW-846 8270E**		**Revision:**		**Date: 2017**	
7065	ATRAZINE	06/15/2020	07/01/2021	N	SPE	NE
7180	CAPROLACTAM	06/15/2020	07/01/2021	N	SPE	NE
6285	HEXACHLOROCYCLOPENTADIENE	06/15/2020	07/01/2021	N	SPE	NE
7955	PARATHION ETHYL	06/15/2020	07/01/2021	N	SPE	NE
8235	THIONAZIN (ZINOPHOS)	06/15/2020	07/01/2021	N	SPE	NE
Method Code: 10296614	**Method Ref: EPA 608.3 GC-ECD**		**Revision:**		**Date: 2016**	
7355	4,4-DDD	07/02/2018	07/01/2021	N	SPE	NE
7360	4,4-DDE	07/02/2018	07/01/2021	N	SPE	NE
7365	4,4-DDT	07/02/2018	07/01/2021	N	SPE	NE
7025	ALDRIN	07/02/2018	07/01/2021	N	SPE	NE
7110	ALPHA-BHC (ALPHA-HEXACHLOROCYCLOHEXANE)	07/02/2018	07/01/2021	N	SPE	NE
7240	ALPHA-CHLORDANE	07/02/2018	07/01/2021	N	SPE	NE
7115	BETA-BHC (BETA-HEXACHLOROCYCLOHEXANE)	07/02/2018	07/01/2021	N	SPE	NE
7250	CHLORDANE (TECH.)	07/02/2018	07/01/2021	N	SPE	NE
7105	DELTA-BHC	07/02/2018	07/01/2021	N	SPE	NE

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7470	DIELDRIN	07/02/2018	07/01/2021	N	SPE	NE
7510	ENDOSULFAN I	07/02/2018	07/01/2021	N	SPE	NE
7515	ENDOSULFAN II	07/02/2018	07/01/2021	N	SPE	NE
7520	ENDOSULFAN SULFATE	07/02/2018	07/01/2021	N	SPE	NE
7540	ENDRIN	07/02/2018	07/01/2021	N	SPE	NE
7530	ENDRIN ALDEHYDE	07/02/2018	07/01/2021	N	SPE	NE
7535	ENDRIN KETONE	07/02/2018	07/01/2021	N	SPE	NE
7120	GAMMA-BHC (LINDANE)	07/02/2018	07/01/2021	N	SPE	NE
7685	HEPTACHLOR	07/02/2018	07/01/2021	N	SPE	NE
7690	HEPTACHLOR EPOXIDE	07/02/2018	07/01/2021	N	SPE	NE
7810	METHOXYCHLOR	07/02/2018	07/01/2021	N	SPE	NE
7870	MIREX	05/13/2019	07/01/2021	N	SPE	NE
8250	TOXAPHENE (CHLORINATED CAMPHENE)	07/02/2018	07/01/2021	N	SPE	NE
7972	TRANS-CHLORDANE	07/02/2018	07/01/2021	N	SPE	NE
Method Code: 10300024	**Method Ref: EPA 625.1**		**Revision:**		**Date: 2016**	
7065	ATRAZINE	05/13/2019	07/01/2021	N	SPE	NE
6285	HEXACHLOROCYCLOPENTADIENE	07/02/2018	07/01/2021	N	SPE	NE
Method Code: 10305609	**Method Ref: SW-846 8015D**		**Revision:**		**Date: 2003**	
2050	TOTAL PETROLEUM HYDROCARBONS (TPH)	07/02/2018	07/01/2021	N	SHY	NE
Method Code: 10162400	**Method Ref: SW-846 7196A**		**Revision: 1**		**Date: 1992**	
1045	CHROMIUM VI	02/21/2017	07/01/2021	SC	MET	NE
Method Code: 10193803	**Method Ref: SW-846 9014**		**Revision: 0**		**Date: 1996**	
1645	CYANIDE, TOTAL	02/21/2017	07/01/2021	SC	NMI	NE
Method Code: 10195003	**Method Ref: SW-846 9023**		**Revision: 0**		**Date: 1996**	
1720	EXTRACTABLE ORGANIC HALIDES (EOX)	02/21/2017	07/01/2021	SC	NMI	NE
Method Code: 10196608	**Method Ref: SW-846 9038**		**Revision: 0**		**Date: 1986**	
2000	SULFATE	02/21/2017	07/01/2021	SC	NMI	NE
Method Code: 10198455	**Method Ref: SW-846 9045D**		**Revision:**		**Date: 2004**	
1900	HYDROGEN ION (PH)	12/29/2017	07/01/2021	SC	NMI	NE
Method Code: 10198808	**Method Ref: SW-846 9050A**		**Revision: 1**		**Date: 1996**	
1610	CONDUCTIVITY	02/21/2017	07/01/2021	SC	NMI	NE
Method Code: 10200405	**Method Ref: SW-846 9065**		**Revision: 0**		**Date: 1986**	
1905	PHENOLICS, TOTAL	02/21/2017	07/01/2021	SC	NMI	NE
Method Code: 10201806	**Method Ref: SW-846 9071B**		**Revision:**		**Date: 1998**	
1803	N-HEXANE EXTRACTABLE MATERIAL (O&G)	12/29/2017	07/01/2021	SC	NMI	NE
Method Code: 10207406	**Method Ref: SW-846 9251**		**Revision: 0**		**Date: 1986**	
1575	CHLORIDE	02/21/2017	07/01/2021	SC	NMI	NE

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Method Code: 10234807	**Method Ref: SW-846 1010A**		**Revision:**		**Date:**	
6117	FLASHPOINT	02/21/2017	07/01/2021	SC	NMI	NE
Method Code: 10243002	**Method Ref: SW-846 9010C**		**Revision:**		**Date:**	
1645	CYANIDE, TOTAL	02/21/2017	07/01/2021	SC	NMI	NE
Method Code: 10243206	**Method Ref: SW-846 9012B**		**Revision:**		**Date:**	
1510	CYANIDE, AMENABLE	06/15/2018	07/01/2021	SC	NMI	NE
1645	CYANIDE, TOTAL	02/21/2017	07/01/2021	SC	NMI	NE
Method Code: 10244403	**Method Ref: SW-846 9040C**		**Revision:**		**Date:**	
1900	HYDROGEN ION (PH)	02/21/2017	07/01/2021	SC	NMI	NE
Method Code: 10245600	**Method Ref: SW-846 9095B**		**Revision:**		**Date:**	
1434	PAINT FILTER LIQUIDS TEST	02/21/2017	07/01/2021	SC	NMI	NE
Method Code: 10304800	**Method Ref: SW-846 6860**		**Revision:**		**Date:**	
1895	PERCHLORATE	05/13/2019	07/01/2021	SC	NMI	NE
Method Code: 20005203	**Method Ref: SM 2540 G**		**Revision: 18th ED**		**Date: 1992**	
1950	RESIDUE, TOTAL (TS)	02/21/2017	07/01/2021	SC	NMI	NE
Method Code: NH0317	**Method Ref: SW-846 1030 (ALPHA SOP 2238)**		**Revision:**		**Date:**	
1780	IGNITABILITY	06/15/2020	07/01/2021	SC	NMI	NE
Method Code: 10118806	**Method Ref: SW-846 1311**		**Revision: 0**		**Date: 1992**	
1466	TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP)	02/21/2017	07/01/2021	SC	PRE	NE
Method Code: 10119003	**Method Ref: SW-846 1312**		**Revision: 0**		**Date: 1994**	
1460	SYNTHETIC PRECIPITATION LEACHING PROCEDURE (SCLP)	02/21/2017	07/01/2021	SC	PRE	NE
Method Code: 10136604	**Method Ref: SW-846 3060A**		**Revision: 1**		**Date: 1996**	
1402	ALKALINE DIGESTION OF CR(VI)	02/21/2017	07/01/2021	SC	PRE	NE
Method Code: 10140202	**Method Ref: SW-846 3540C**		**Revision: 3**		**Date: 1996**	
1452	SOXHLET EXTRACTION	02/21/2017	07/01/2021	SC	PRE	NE
Method Code: 10141205	**Method Ref: SW-846 3546**		**Revision:**		**Date: (11/00)**	
1428	MICROWAVE EXTRACTION	02/21/2017	07/01/2021	SC	PRE	NE
Method Code: 10142004	**Method Ref: SW-846 3550C**		**Revision:**		**Date: (11/00)**	
1468	ULTRASONIC EXTRACTION	02/21/2017	07/01/2021	SC	PRE	NE
Method Code: 10143007	**Method Ref: SW-846 3580A**		**Revision: 1**		**Date: 1992**	
1470	WASTE DILUTION	02/21/2017	07/01/2021	SC	PRE	NE
Method Code: 10146006	**Method Ref: SW-846 3620C**		**Revision:**		**Date: (11/00)**	
1414	FLORISIL CLEAN-UP	02/21/2017	07/01/2021	SC	PRE	NE
Method Code: 10146802	**Method Ref: SW-846 3630C**		**Revision: 3**		**Date: 1996**	
1446	SILICA GEL CLEAN-UP	02/21/2017	07/01/2021	SC	PRE	NE
Method Code: 10148400	**Method Ref: SW-846 3660B**		**Revision: 2**		**Date: 1996**	
1456	SULFUR CLEAN-UP	02/21/2017	07/01/2021	SC	PRE	NE
Method Code: 10148808	**Method Ref: SW-846 3665A**		**Revision: 1**		**Date: 1996**	
1458	SULFURIC ACID / PERMANGANATE CLEAN-UP	02/21/2017	07/01/2021	SC	PRE	NE

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Method Code: 10154004	**Method Ref: SW-846 5035**		**Revision: 0**		**Date: 1996**	
1450	SOLID-PHASE PURGE & TRAP	02/21/2017	07/01/2021	SC	PRE	NE
Method Code: 10284807	**Method Ref: SW-846 5035A**		**Revision:**		**Date:**	
1450	SOLID-PHASE PURGE & TRAP	07/02/2018	07/01/2021	SC	PRE	NE
Method Code: 30030718	**Method Ref: ASTM D3987-12**		**Revision:**		**Date: 2012**	
1386	SHAKE EXTRACTION OF SOLID WASTE WITH WATER	02/21/2017	07/01/2021	SC	PRE	NE
Method Code: 10186002	**Method Ref: SW-846 8270D**		**Revision:**		**Date: 2007**	
5155	1,2,4-TRICHLOROBENZENE	02/21/2017	07/01/2021	SC	VOC	NE
4610	1,2-DICHLOROBENZENE	02/21/2017	07/01/2021	SC	VOC	NE
4615	1,3-DICHLOROBENZENE	02/21/2017	07/01/2021	SC	VOC	NE
4620	1,4-DICHLOROBENZENE	02/21/2017	07/01/2021	SC	VOC	NE
4735	1,4-DIOXANE (1 4-DIETHYLENEOXIDE)	05/10/2019	07/01/2021	SC	VOC	NE
4835	HEXACHLOROBUTADIENE	02/21/2017	07/01/2021	SC	VOC	NE
4840	HEXACHLOROETHANE	02/21/2017	07/01/2021	SC	VOC	NE
5005	NAPHTHALENE	02/21/2017	07/01/2021	SC	VOC	NE
5015	NITROBENZENE	02/21/2017	07/01/2021	SC	VOC	NE
5095	PYRIDINE	02/21/2017	07/01/2021	SC	VOC	NE
Method Code: 10242509	**Method Ref: SW-846 8270D SIM**		**Revision:**		**Date: 2007**	
4835	HEXACHLOROBUTADIENE	02/21/2017	07/01/2021	SC	VOC	NE
5005	NAPHTHALENE	02/21/2017	07/01/2021	SC	VOC	NE
Method Code: 10242543	**Method Ref: SW-846 8270E**		**Revision:**		**Date: 2017**	
5155	1,2,4-TRICHLOROBENZENE	06/15/2020	07/01/2021	SC	VOC	NE
4610	1,2-DICHLOROBENZENE	06/15/2020	07/01/2021	SC	VOC	NE
4615	1,3-DICHLOROBENZENE	06/15/2020	07/01/2021	SC	VOC	NE
4620	1,4-DICHLOROBENZENE	06/15/2020	07/01/2021	SC	VOC	NE
4735	1,4-DIOXANE (1 4-DIETHYLENEOXIDE)	06/15/2020	07/01/2021	SC	VOC	NE
4835	HEXACHLOROBUTADIENE	06/15/2020	07/01/2021	SC	VOC	NE
4840	HEXACHLOROETHANE	06/15/2020	07/01/2021	SC	VOC	NE
5005	NAPHTHALENE	06/15/2020	07/01/2021	SC	VOC	NE
5015	NITROBENZENE	06/15/2020	07/01/2021	SC	VOC	NE
5095	PYRIDINE	06/15/2020	07/01/2021	SC	VOC	NE
Method Code: 10242565	**Method Ref: SW-846 8270E SIM**		**Revision:**		**Date: 2017**	
4835	HEXACHLOROBUTADIENE	06/15/2020	07/01/2021	SC	VOC	NE
5005	NAPHTHALENE	06/15/2020	07/01/2021	SC	VOC	NE
Method Code: 10307003	**Method Ref: SW-846 8260C**		**Revision:**		**Date:**	
5105	1,1,1,2-TETRACHLOROETHANE	02/21/2017	07/01/2021	SC	VOC	NE
5160	1,1,1-TRICHLOROETHANE	02/21/2017	07/01/2021	SC	VOC	NE

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5110	1,1,2,2-TETRACHLOROETHANE	02/21/2017	07/01/2021	SC	VOC	NE
5185	1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE (FREON 113)	02/21/2017	07/01/2021	SC	VOC	NE
5165	1,1,2-TRICHLOROETHANE	02/21/2017	07/01/2021	SC	VOC	NE
4630	1,1-DICHLOROETHANE	02/21/2017	07/01/2021	SC	VOC	NE
4640	1,1-DICHLOROETHENE	02/21/2017	07/01/2021	SC	VOC	NE
4670	1,1-DICHLOROPROPENE	02/21/2017	07/01/2021	SC	VOC	NE
5150	1,2,3-TRICHLOROBENZENE	02/21/2017	07/01/2021	SC	VOC	NE
5180	1,2,3-TRICHLOROPROPANE	02/21/2017	07/01/2021	SC	VOC	NE
5155	1,2,4-TRICHLOROBENZENE	02/21/2017	07/01/2021	SC	VOC	NE
5210	1,2,4-TRIMETHYLBENZENE	02/21/2017	07/01/2021	SC	VOC	NE
4570	1,2-DIBROMO-3-CHLOROPROPANE (DBCP)	02/21/2017	07/01/2021	SC	VOC	NE
4585	1,2-DIBROMOETHANE (EDB)	02/21/2017	07/01/2021	SC	VOC	NE
4610	1,2-DICHLOROBENZENE	02/21/2017	07/01/2021	SC	VOC	NE
4635	1,2-DICHLOROETHANE	02/21/2017	07/01/2021	SC	VOC	NE
4655	1,2-DICHLOROPROPANE	02/21/2017	07/01/2021	SC	VOC	NE
5215	1,3,5-TRIMETHYLBENZENE	02/21/2017	07/01/2021	SC	VOC	NE
4615	1,3-DICHLOROBENZENE	02/21/2017	07/01/2021	SC	VOC	NE
4660	1,3-DICHLOROPROPANE	02/21/2017	07/01/2021	SC	VOC	NE
4620	1,4-DICHLOROBENZENE	02/21/2017	07/01/2021	SC	VOC	NE
4735	1,4-DIOXANE (1 4-DIETHYLENEOXIDE)	02/21/2017	07/01/2021	SC	VOC	NE
4665	2 2-DICHLOROPROPANE	02/21/2017	07/01/2021	SC	VOC	NE
4410	2-BUTANONE (METHYL ETHYL KETONE MEK)	02/21/2017	07/01/2021	SC	VOC	NE
4500	2-CHLOROETHYLVINYL ETHER	02/21/2017	07/01/2021	SC	VOC	NE
4535	2-CHLOROTOLUENE	02/21/2017	07/01/2021	SC	VOC	NE
4860	2-HEXANONE	02/21/2017	07/01/2021	SC	VOC	NE
5020	2-NITROPROPANE	07/12/2019	07/01/2021	SC	VOC	NE
4540	4-CHLOROTOLUENE	02/21/2017	07/01/2021	SC	VOC	NE
4910	4-ISOPROPYLTOLUENE (P-ISOPROPYLTOLUENE)	02/21/2017	07/01/2021	SC	VOC	NE
4995	4-METHYL-2-PENTANONE (MIBK)	02/21/2017	07/01/2021	SC	VOC	NE
4315	ACETONE	02/21/2017	07/01/2021	SC	VOC	NE
4325	ACROLEIN (PROPENAL)	02/21/2017	07/01/2021	SC	VOC	NE
4340	ACRYLONITRILE	02/21/2017	07/01/2021	SC	VOC	NE
4355	ALLYL CHLORIDE (3-CHLOROPROPENE)	07/12/2019	07/01/2021	SC	VOC	NE
4375	BENZENE	02/21/2017	07/01/2021	SC	VOC	NE
4385	BROMOBENZENE	02/21/2017	07/01/2021	SC	VOC	NE
4390	BROMOCHLOROMETHANE	02/21/2017	07/01/2021	SC	VOC	NE

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4395	BROMODICHLOROMETHANE	02/21/2017	07/01/2021	SC	VOC	NE
4400	BROMOFORM	02/21/2017	07/01/2021	SC	VOC	NE
4950	BROMOMETHANE (METHYL BROMIDE)	02/21/2017	07/01/2021	SC	VOC	NE
4450	CARBON DISULFIDE	02/21/2017	07/01/2021	SC	VOC	NE
4455	CARBON TETRACHLORIDE	02/21/2017	07/01/2021	SC	VOC	NE
4475	CHLOROBENZENE	02/21/2017	07/01/2021	SC	VOC	NE
4577	CHLORODIFLUOROMETHANE (FREON-22)	07/12/2019	07/01/2021	SC	VOC	NE
4485	CHLOROETHANE	02/21/2017	07/01/2021	SC	VOC	NE
4505	CHLOROFORM	02/21/2017	07/01/2021	SC	VOC	NE
4960	CHLOROMETHANE (METHYL CHLORIDE)	02/21/2017	07/01/2021	SC	VOC	NE
4645	CIS-1 2-DICHLOROETHYLENE	02/21/2017	07/01/2021	SC	VOC	NE
4680	CIS-1,3-DICHLOROPROPENE	02/21/2017	07/01/2021	SC	VOC	NE
4555	CYCLOHEXANE	02/21/2017	07/01/2021	SC	VOC	NE
4560	CYCLOHEXANONE	02/21/2017	07/01/2021	SC	VOC	NE
9375	DI-ISOPROPYLETHER (DIPE)	02/21/2017	07/01/2021	SC	VOC	NE
4575	DIBROMOCHLOROMETHANE	02/21/2017	07/01/2021	SC	VOC	NE
4595	DIBROMOMETHANE	02/21/2017	07/01/2021	SC	VOC	NE
4625	DICHLORODIFLUOROMETHANE	02/21/2017	07/01/2021	SC	VOC	NE
4975	DICHLOROMETHANE (METHYLENE CHLORIDE)	02/21/2017	07/01/2021	SC	VOC	NE
4725	DIETHYL ETHER	02/21/2017	07/01/2021	SC	VOC	NE
4750	ETHANOL	02/21/2017	07/01/2021	SC	VOC	NE
4770	ETHYL - T- BUTYL ETHER (ETBE)	12/29/2017	07/01/2021	SC	VOC	NE
4755	ETHYL ACETATE	02/21/2017	07/01/2021	SC	VOC	NE
4810	ETHYL METHACRYLATE	02/21/2017	07/01/2021	SC	VOC	NE
4765	ETHYLBENZENE	02/21/2017	07/01/2021	SC	VOC	NE
4835	HEXACHLOROBUTADIENE	02/21/2017	07/01/2021	SC	VOC	NE
4840	HEXACHLOROETHANE	07/12/2019	07/01/2021	SC	VOC	NE
4870	IODOMETHANE (METHYL IODIDE)	08/14/2019	07/01/2021	SC	VOC	NE
4900	ISOPROPYLBENZENE	02/21/2017	07/01/2021	SC	VOC	NE
5245	M-XYLENE	02/21/2017	07/01/2021	SC	VOC	NE
4940	METHYL ACETATE	02/21/2017	07/01/2021	SC	VOC	NE
4990	METHYL METHACRYLATE	07/12/2019	07/01/2021	SC	VOC	NE
5000	METHYL TERT-BUTYL ETHER (MTBE)	02/21/2017	07/01/2021	SC	VOC	NE
4965	METHYLCYCLOHEXANE	02/21/2017	07/01/2021	SC	VOC	NE
4425	N-BUTYL ALCOHOL	02/21/2017	07/01/2021	SC	VOC	NE
4435	N-BUTYLBENZENE	02/21/2017	07/01/2021	SC	VOC	NE

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5090	N-PROPYLBENZENE	02/21/2017	07/01/2021	SC	VOC	NE
5005	NAPHTHALENE	02/21/2017	07/01/2021	SC	VOC	NE
5015	NITROBENZENE	07/12/2019	07/01/2021	SC	VOC	NE
5250	O-XYLENE	02/21/2017	07/01/2021	SC	VOC	NE
5255	P-XYLENE	02/21/2017	07/01/2021	SC	VOC	NE
4440	SEC-BUTYLBENZENE	02/21/2017	07/01/2021	SC	VOC	NE
5100	STYRENE	02/21/2017	07/01/2021	SC	VOC	NE
4370	TERT-AMYL METHYL ETHER (TAME)	02/21/2017	07/01/2021	SC	VOC	NE
4420	TERT-BUTYL ALCOHOL (TBA)	02/21/2017	07/01/2021	SC	VOC	NE
4445	TERT-BUTYLBENZENE	02/21/2017	07/01/2021	SC	VOC	NE
5115	TETRACHLOROETHENE (PERCHLOROETHYLENE)	02/21/2017	07/01/2021	SC	VOC	NE
5120	TETRAHYDROFURAN (THF)	02/21/2017	07/01/2021	SC	VOC	NE
5140	TOLUENE	02/21/2017	07/01/2021	SC	VOC	NE
4700	TRANS-1,2-DICLOROETHYLENE	02/21/2017	07/01/2021	SC	VOC	NE
4685	TRANS-1,3-DICHLOROPROPENE	02/21/2017	07/01/2021	SC	VOC	NE
4605	TRANS-1,4-DICHLORO-2-BUTENE	02/21/2017	07/01/2021	SC	VOC	NE
5170	TRICHLOROETHENE (TRICHLOROETHYLENE)	02/21/2017	07/01/2021	SC	VOC	NE
5175	TRICHLOROFLUOROMETHANE	02/21/2017	07/01/2021	SC	VOC	NE
5225	VINYL ACETATE	02/21/2017	07/01/2021	SC	VOC	NE
5235	VINYL CHLORIDE	02/21/2017	07/01/2021	SC	VOC	NE
5260	XYLENE (TOTAL)	02/21/2017	07/01/2021	SC	VOC	NE
Method Code: 10307127	**Method Ref: SW-846 8260D**		**Revision:**		**Date: 2018**	
5105	1,1,1,2-TETRACHLOROETHANE	06/15/2020	07/01/2021	SC	VOC	NE
5160	1,1,1-TRICHLOROETHANE	06/15/2020	07/01/2021	SC	VOC	NE
5110	1,1,2,2-TETRACHLOROETHANE	06/15/2020	07/01/2021	SC	VOC	NE
5185	1,1,2-TRICHLORO-1,2,2-TRIFLUOROETHANE (FREON 113)	06/15/2020	07/01/2021	SC	VOC	NE
5165	1,1,2-TRICHLOROETHANE	06/15/2020	07/01/2021	SC	VOC	NE
4630	1,1-DICHLOROETHANE	06/15/2020	07/01/2021	SC	VOC	NE
4640	1,1-DICHLOROETHENE	06/15/2020	07/01/2021	SC	VOC	NE
4670	1,1-DICHLOROPROPENE	06/15/2020	07/01/2021	SC	VOC	NE
5150	1,2,3-TRICHLOROBENZENE	06/15/2020	07/01/2021	SC	VOC	NE
5180	1,2,3-TRICHLOROPROPANE	06/15/2020	07/01/2021	SC	VOC	NE
5155	1,2,4-TRICHLOROBENZENE	06/15/2020	07/01/2021	SC	VOC	NE
5210	1,2,4-TRIMETHYLBENZENE	06/15/2020	07/01/2021	SC	VOC	NE
4570	1,2-DIBROMO-3-CHLOROPROPANE (DBCP)	06/15/2020	07/01/2021	SC	VOC	NE
4585	1,2-DIBROMOETHANE (EDB)	06/15/2020	07/01/2021	SC	VOC	NE

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4610	1,2-DICHLOROBENZENE	06/15/2020	07/01/2021	SC	VOC	NE
4635	1,2-DICHLOROETHANE	06/15/2020	07/01/2021	SC	VOC	NE
4655	1,2-DICHLOROPROPANE	06/15/2020	07/01/2021	SC	VOC	NE
5215	1,3,5-TRIMETHYLBENZENE	06/15/2020	07/01/2021	SC	VOC	NE
4615	1,3-DICHLOROBENZENE	06/15/2020	07/01/2021	SC	VOC	NE
4660	1,3-DICHLOROPROPANE	06/15/2020	07/01/2021	SC	VOC	NE
4620	1,4-DICHLOROBENZENE	06/15/2020	07/01/2021	SC	VOC	NE
4735	1,4-DIOXANE (1 4-DIETHYLENEOXIDE)	06/15/2020	07/01/2021	SC	VOC	NE
4480	1-CHLOROBUTANE	06/15/2020	07/01/2021	SC	VOC	NE
4665	2 2-DICHLOROPROPANE	06/15/2020	07/01/2021	SC	VOC	NE
4410	2-BUTANONE (METHYL ETHYL KETONE MEK)	06/15/2020	07/01/2021	SC	VOC	NE
4500	2-CHLOROETHYLVINYL ETHER	06/15/2020	07/01/2021	SC	VOC	NE
4535	2-CHLOROTOLUENE	06/15/2020	07/01/2021	SC	VOC	NE
4860	2-HEXANONE	06/15/2020	07/01/2021	SC	VOC	NE
5020	2-NITROPROPANE	06/15/2020	07/01/2021	SC	VOC	NE
4540	4-CHLOROTOLUENE	06/15/2020	07/01/2021	SC	VOC	NE
4910	4-ISOPROPYLTOLUENE (P-ISOPROPYLTOLUENE)	06/15/2020	07/01/2021	SC	VOC	NE
4995	4-METHYL-2-PENTANONE (MIBK)	06/15/2020	07/01/2021	SC	VOC	NE
4315	ACETONE	06/15/2020	07/01/2021	SC	VOC	NE
4325	ACROLEIN (PROPENAL)	06/15/2020	07/01/2021	SC	VOC	NE
4340	ACRYLONITRILE	06/15/2020	07/01/2021	SC	VOC	NE
4355	ALLYL CHLORIDE (3-CHLOROPROPENE)	06/15/2020	07/01/2021	SC	VOC	NE
4375	BENZENE	06/15/2020	07/01/2021	SC	VOC	NE
4385	BROMOBENZENE	06/15/2020	07/01/2021	SC	VOC	NE
4390	BROMOCHLOROMETHANE	06/15/2020	07/01/2021	SC	VOC	NE
4395	BROMODICHLOROMETHANE	06/15/2020	07/01/2021	SC	VOC	NE
4400	BROMOFORM	06/15/2020	07/01/2021	SC	VOC	NE
4950	BROMOMETHANE (METHYL BROMIDE)	06/15/2020	07/01/2021	SC	VOC	NE
4450	CARBON DISULFIDE	06/15/2020	07/01/2021	SC	VOC	NE
4455	CARBON TETRACHLORIDE	06/15/2020	07/01/2021	SC	VOC	NE
4475	CHLOROBENZENE	06/15/2020	07/01/2021	SC	VOC	NE
4577	CHLORODIFLUOROMETHANE (FREON-22)	06/15/2020	07/01/2021	SC	VOC	NE
4485	CHLOROETHANE	06/15/2020	07/01/2021	SC	VOC	NE
4505	CHLOROFORM	06/15/2020	07/01/2021	SC	VOC	NE
4960	CHLOROMETHANE (METHYL CHLORIDE)	06/15/2020	07/01/2021	SC	VOC	NE
4645	CIS-1 2-DICHLOROETHYLENE	06/15/2020	07/01/2021	SC	VOC	NE

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4680	CIS-1,3-DICHLOROPROPENE	06/15/2020	07/01/2021	SC	VOC	NE
4555	CYCLOHEXANE	06/15/2020	07/01/2021	SC	VOC	NE
4560	CYCLOHEXANONE	06/15/2020	07/01/2021	SC	VOC	NE
9375	DI-ISOPROPYLETHER (DIPE)	06/15/2020	07/01/2021	SC	VOC	NE
4575	DIBROMOCHLOROMETHANE	06/15/2020	07/01/2021	SC	VOC	NE
4595	DIBROMOMETHANE	06/15/2020	07/01/2021	SC	VOC	NE
4625	DICHLORODIFLUOROMETHANE	06/15/2020	07/01/2021	SC	VOC	NE
4975	DICHLOROMETHANE (METHYLENE CHLORIDE)	06/15/2020	07/01/2021	SC	VOC	NE
4725	DIETHYL ETHER	06/15/2020	07/01/2021	SC	VOC	NE
4750	ETHANOL	06/15/2020	07/01/2021	SC	VOC	NE
4770	ETHYL - T- BUTYL ETHER (ETBE)	06/15/2020	07/01/2021	SC	VOC	NE
4755	ETHYL ACETATE	06/15/2020	07/01/2021	SC	VOC	NE
4810	ETHYL METHACRYLATE	06/15/2020	07/01/2021	SC	VOC	NE
4765	ETHYLBENZENE	06/15/2020	07/01/2021	SC	VOC	NE
4835	HEXACHLOROBUTADIENE	06/15/2020	07/01/2021	SC	VOC	NE
4840	HEXACHLOROETHANE	06/15/2020	07/01/2021	SC	VOC	NE
4870	IODOMETHANE (METHYL IODIDE)	06/15/2020	07/01/2021	SC	VOC	NE
4900	ISOPROPYLBENZENE	06/15/2020	07/01/2021	SC	VOC	NE
5245	M-XYLENE	06/15/2020	07/01/2021	SC	VOC	NE
4940	METHYL ACETATE	06/15/2020	07/01/2021	SC	VOC	NE
4990	METHYL METHACRYLATE	06/15/2020	07/01/2021	SC	VOC	NE
5000	METHYL TERT-BUTYL ETHER (MTBE)	06/15/2020	07/01/2021	SC	VOC	NE
4965	METHYLCYCLOHEXANE	06/15/2020	07/01/2021	SC	VOC	NE
4425	N-BUTYL ALCOHOL	06/15/2020	07/01/2021	SC	VOC	NE
4435	N-BUTYLBENZENE	06/15/2020	07/01/2021	SC	VOC	NE
5090	N-PROPYLBENZENE	06/15/2020	07/01/2021	SC	VOC	NE
5005	NAPHTHALENE	06/15/2020	07/01/2021	SC	VOC	NE
5015	NITROBENZENE	06/15/2020	07/01/2021	SC	VOC	NE
5250	O-XYLENE	06/15/2020	07/01/2021	SC	VOC	NE
5255	P-XYLENE	06/15/2020	07/01/2021	SC	VOC	NE
4440	SEC-BUTYLBENZENE	06/15/2020	07/01/2021	SC	VOC	NE
5100	STYRENE	06/15/2020	07/01/2021	SC	VOC	NE
4370	TERT-AMYL METHYL ETHER (TAME)	06/15/2020	07/01/2021	SC	VOC	NE
4420	TERT-BUTYL ALCOHOL (TBA)	06/15/2020	07/01/2021	SC	VOC	NE
4445	TERT-BUTYLBENZENE	06/15/2020	07/01/2021	SC	VOC	NE
5115	TETRACHLOROETHENE (PERCHLOROETHYLENE)	06/15/2020	07/01/2021	SC	VOC	NE

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5120	TETRAHYDROFURAN (THF)	06/15/2020	07/01/2021	SC	VOC	NE
5140	TOLUENE	06/15/2020	07/01/2021	SC	VOC	NE
4700	TRANS-1,2-DICLOROETHYLENE	06/15/2020	07/01/2021	SC	VOC	NE
4685	TRANS-1,3-DICHLOROPROPENE	06/15/2020	07/01/2021	SC	VOC	NE
4605	TRANS-1,4-DICHLORO-2-BUTENE	06/15/2020	07/01/2021	SC	VOC	NE
5170	TRICHLOROETHENE (TRICHLOROETHYLENE)	06/15/2020	07/01/2021	SC	VOC	NE
5175	TRICHLOROFLUOROMETHANE	06/15/2020	07/01/2021	SC	VOC	NE
5225	VINYL ACETATE	06/15/2020	07/01/2021	SC	VOC	NE
5235	VINYL CHLORIDE	06/15/2020	07/01/2021	SC	VOC	NE
5260	XYLENE (TOTAL)	06/15/2020	07/01/2021	SC	VOC	NE
Method Code: 90017202	**Method Ref: MADEP EPH**		**Revision: 1.1**		**Date: 2004**	
5005	NAPHTHALENE	08/20/2018	07/01/2021	SC	VOC	NE
Method Code: 90017451	**Method Ref: MADEP VPH**		**Revision: 2.1**		**Date: 2018**	
4375	BENZENE	05/10/2019	07/01/2021	SC	VOC	NE
4765	ETHYLBENZENE	05/10/2019	07/01/2021	SC	VOC	NE
5240	M/P-XYLENE	05/10/2019	07/01/2021	SC	VOC	NE
5000	METHYL TERT-BUTYL ETHER (MTBE)	05/10/2019	07/01/2021	SC	VOC	NE
5005	NAPHTHALENE	05/10/2019	07/01/2021	SC	VOC	NE
5250	O-XYLENE	05/10/2019	07/01/2021	SC	VOC	NE
5140	TOLUENE	05/10/2019	07/01/2021	SC	VOC	NE
5304	VPH ALIPHATIC C5-C8	05/10/2019	07/01/2021	SC	VOC	NE
5306	VPH ALIPHATIC C9-C12	05/10/2019	07/01/2021	SC	VOC	NE
5311	VPH AROMATIC C9-C10	05/10/2019	07/01/2021	SC	VOC	NE
5260	XYLENE (TOTAL)	05/10/2019	07/01/2021	SC	VOC	NE
Method Code: NH0308	**Method Ref: MADEP EPH**		**Revision: 2.1**		**Date:**	
5005	NAPHTHALENE	06/15/2020	07/01/2021	SC	VOC	NE
Method Code: 10186002	**Method Ref: SW-846 8270D**		**Revision:**		**Date: 2007**	
6703	1,1'-BIPHENYL (BZ-0)	02/21/2017	07/01/2021	SC	SBN	NE
6715	1,2,4,5-TETRACHLOROBENZENE	02/21/2017	07/01/2021	SC	SBN	NE
6220	1,2-DIPHENYLHYDRAZINE	02/21/2017	07/01/2021	SC	SBN	NE
6380	1-METHYLNAPHTHALENE	02/21/2017	07/01/2021	SC	SBN	NE
4659	2,2'-OXYBIS(1-CHLOROPROPANE)	02/21/2017	07/01/2021	SC	SBN	NE
6735	2,3,4,6-TETRACHLOROPHENOL	02/21/2017	07/01/2021	SC	SBN	NE
6835	2,4,5-TRICHLOROPHENOL	02/21/2017	07/01/2021	SC	SBN	NE
6840	2,4,6-TRICHLOROPHENOL	02/21/2017	07/01/2021	SC	SBN	NE
6000	2,4-DICHLOROPHENOL	02/21/2017	07/01/2021	SC	SBN	NE
6130	2,4-DIMETHYLPHENOL	02/21/2017	07/01/2021	SC	SBN	NE

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6175	2,4-DINITROPHENOL	02/21/2017	07/01/2021	SC	SBN	NE
6185	2,4-DINITROTOLUENE (2 4-DNT)	02/21/2017	07/01/2021	SC	SBN	NE
6190	2,6-DINITROTOLUENE (2 6-DNT)	02/21/2017	07/01/2021	SC	SBN	NE
5795	2-CHLORONAPHTHALENE	02/21/2017	07/01/2021	SC	SBN	NE
5800	2-CHLOROPHENOL	02/21/2017	07/01/2021	SC	SBN	NE
6360	2-METHYL-4,6-DINITROPHENOL	02/21/2017	07/01/2021	SC	SBN	NE
6385	2-METHYLNAPHTHALENE	02/21/2017	07/01/2021	SC	SBN	NE
6400	2-METHYLPHENOL (O-CRESOL)	02/21/2017	07/01/2021	SC	SBN	NE
6460	2-NITROANILINE	02/21/2017	07/01/2021	SC	SBN	NE
6490	2-NITROPHENOL	02/21/2017	07/01/2021	SC	SBN	NE
5945	3,3-DICHLOROBENZIDINE	02/21/2017	07/01/2021	SC	SBN	NE
6405	3-METHYLPHENOL (M-CRESOL)	02/21/2017	07/01/2021	SC	SBN	NE
6465	3-NITROANILINE	02/21/2017	07/01/2021	SC	SBN	NE
5660	4-BROMOPHENYL PHENYL ETHER	02/21/2017	07/01/2021	SC	SBN	NE
5700	4-CHLORO-3-METHYLPHENOL	02/21/2017	07/01/2021	SC	SBN	NE
5745	4-CHLOROANILINE	02/21/2017	07/01/2021	SC	SBN	NE
5825	4-CHLOROPHENYL PHENYL ETHER	02/21/2017	07/01/2021	SC	SBN	NE
6410	4-METHYLPHENOL (P-CRESOL)	02/21/2017	07/01/2021	SC	SBN	NE
6470	4-NITROANILINE	02/21/2017	07/01/2021	SC	SBN	NE
6500	4-NITROPHENOL	02/21/2017	07/01/2021	SC	SBN	NE
5500	ACENAPHTHENE	02/21/2017	07/01/2021	SC	SBN	NE
5505	ACENAPHTHYLENE	02/21/2017	07/01/2021	SC	SBN	NE
5510	ACETOPHENONE	02/21/2017	07/01/2021	SC	SBN	NE
5545	ANILINE	02/21/2017	07/01/2021	SC	SBN	NE
5555	ANTHRACENE	02/21/2017	07/01/2021	SC	SBN	NE
5570	BENZALDEHYDE	02/21/2017	07/01/2021	SC	SBN	NE
5595	BENZIDINE	02/21/2017	07/01/2021	SC	SBN	NE
5575	BENZO(A)ANTHRACENE	02/21/2017	07/01/2021	SC	SBN	NE
5580	BENZO(A)PYRENE	02/21/2017	07/01/2021	SC	SBN	NE
5585	BENZO(B)FLUORANTHENE	02/21/2017	07/01/2021	SC	SBN	NE
5590	BENZO(G,H,I)PERYLENE	02/21/2017	07/01/2021	SC	SBN	NE
5600	BENZO(K)FLUORANTHENE	02/21/2017	07/01/2021	SC	SBN	NE
5610	BENZOIC ACID	02/21/2017	07/01/2021	SC	SBN	NE
5630	BENZYL ALCOHOL	02/21/2017	07/01/2021	SC	SBN	NE
5670	BENZYL BUTYL PHTHALATE	02/21/2017	07/01/2021	SC	SBN	NE
5760	BIS(2-CHLOROETHOXY) METHANE	02/21/2017	07/01/2021	SC	SBN	NE

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5765	BIS(2-CHLOROETHYL) ETHER	02/21/2017	07/01/2021	SC	SBN	NE
6065	BIS(2-ETHYLHEXYL) PHTHALATE	02/21/2017	07/01/2021	SC	SBN	NE
5680	CARBAZOLE	02/21/2017	07/01/2021	SC	SBN	NE
5855	CHRYSENE	02/21/2017	07/01/2021	SC	SBN	NE
5925	DI-N-BUTYL PHTHALATE	02/21/2017	07/01/2021	SC	SBN	NE
6200	DI-N-OCTYL PHTHALATE	02/21/2017	07/01/2021	SC	SBN	NE
5895	DIBENZO(A,H)ANTHRACENE	02/21/2017	07/01/2021	SC	SBN	NE
5905	DIBENZOFURAN	02/21/2017	07/01/2021	SC	SBN	NE
6070	DIETHYL PHTHALATE	02/21/2017	07/01/2021	SC	SBN	NE
6135	DIMETHYL PHTHALATE	02/21/2017	07/01/2021	SC	SBN	NE
6205	DIPHENYLAMINE	02/21/2017	07/01/2021	SC	SBN	NE
6265	FLUORANTHENE	02/21/2017	07/01/2021	SC	SBN	NE
6270	FLUORENE	02/21/2017	07/01/2021	SC	SBN	NE
6275	HEXACHLOROBENZENE	02/21/2017	07/01/2021	SC	SBN	NE
6315	INDENO(1,2,3-CD)PYRENE	02/21/2017	07/01/2021	SC	SBN	NE
6320	ISOPHORONE	02/21/2017	07/01/2021	SC	SBN	NE
6545	N-NITROSODI-N-PROPYLAMINE	02/21/2017	07/01/2021	SC	SBN	NE
6530	N-NITROSODIMETHYLAMINE	02/21/2017	07/01/2021	SC	SBN	NE
6535	N-NITROSODIPHENYLAMINE	02/21/2017	07/01/2021	SC	SBN	NE
6600	PCNB (PENTACHLORONITROBENZENE)	02/21/2017	07/01/2021	SC	SBN	NE
6605	PENTACHLOROPHENOL	02/21/2017	07/01/2021	SC	SBN	NE
6615	PHENANTHRENE	02/21/2017	07/01/2021	SC	SBN	NE
6625	PHENOL	02/21/2017	07/01/2021	SC	SBN	NE
6665	PYRENE	02/21/2017	07/01/2021	SC	SBN	NE
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6380	1-METHYLNAPHTHALENE	02/21/2017	07/01/2021	SC	SBN	NE
6360	2-METHYL-4,6-DINITROPHENOL	02/21/2017	07/01/2021	SC	SBN	NE
6385	2-METHYLNAPHTHALENE	02/21/2017	07/01/2021	SC	SBN	NE
5500	ACENAPHTHENE	02/21/2017	07/01/2021	SC	SBN	NE
5505	ACENAPHTHYLENE	02/21/2017	07/01/2021	SC	SBN	NE
5555	ANTHRACENE	02/21/2017	07/01/2021	SC	SBN	NE
5575	BENZO(A)ANTHRACENE	02/21/2017	07/01/2021	SC	SBN	NE
5580	BENZO(A)PYRENE	02/21/2017	07/01/2021	SC	SBN	NE
5585	BENZO(B)FLUORANTHENE	02/21/2017	07/01/2021	SC	SBN	NE
5590	BENZO(G,H,I)PERYLENE	02/21/2017	07/01/2021	SC	SBN	NE
5600	BENZO(K)FLUORANTHENE	02/21/2017	07/01/2021	SC	SBN	NE

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5855	CHRYSENE	02/21/2017	07/01/2021	SC	SBN	NE
5895	DIBENZO(A,H)ANTHRACENE	02/21/2017	07/01/2021	SC	SBN	NE
6265	FLUORANTHENE	02/21/2017	07/01/2021	SC	SBN	NE
6270	FLUORENE	02/21/2017	07/01/2021	SC	SBN	NE
6275	HEXACHLOROBENZENE	02/21/2017	07/01/2021	SC	SBN	NE
6315	INDENO(1,2,3-CD)PYRENE	02/21/2017	07/01/2021	SC	SBN	NE
6605	PENTACHLOROPHENOL	02/21/2017	07/01/2021	SC	SBN	NE
6615	PHENANTHRENE	02/21/2017	07/01/2021	SC	SBN	NE
6665	PYRENE	02/21/2017	07/01/2021	SC	SBN	NE
Method Code: 10242543	**Method Ref: SW-846 8270E**		**Revision:**		**Date: 2017**	
6703	1,1'-BIPHENYL (BZ-0)	06/15/2020	07/01/2021	SC	SBN	NE
6715	1,2,4,5-TETRACHLOROBENZENE	06/15/2020	07/01/2021	SC	SBN	NE
6220	1,2-DIPHENYLHYDRAZINE	06/15/2020	07/01/2021	SC	SBN	NE
6380	1-METHYLNAPHTHALENE	06/15/2020	07/01/2021	SC	SBN	NE
4659	2,2'-OXYBIS(1-CHLOROPROPANE)	06/15/2020	07/01/2021	SC	SBN	NE
6735	2,3,4,6-TETRACHLOROPHENOL	06/15/2020	07/01/2021	SC	SBN	NE
6835	2,4,5-TRICHLOROPHENOL	06/15/2020	07/01/2021	SC	SBN	NE
6840	2,4,6-TRICHLOROPHENOL	06/15/2020	07/01/2021	SC	SBN	NE
6000	2,4-DICHLOROPHENOL	06/15/2020	07/01/2021	SC	SBN	NE
6130	2,4-DIMETHYLPHENOL	06/15/2020	07/01/2021	SC	SBN	NE
6175	2,4-DINITROPHENOL	06/15/2020	07/01/2021	SC	SBN	NE
6185	2,4-DINITROTOLUENE (2 4-DNT)	06/15/2020	07/01/2021	SC	SBN	NE
6190	2,6-DINITROTOLUENE (2 6-DNT)	06/15/2020	07/01/2021	SC	SBN	NE
5795	2-CHLORONAPHTHALENE	06/15/2020	07/01/2021	SC	SBN	NE
5800	2-CHLOROPHENOL	06/15/2020	07/01/2021	SC	SBN	NE
6360	2-METHYL-4,6-DINITROPHENOL	06/15/2020	07/01/2021	SC	SBN	NE
6385	2-METHYLNAPHTHALENE	06/15/2020	07/01/2021	SC	SBN	NE
6400	2-METHYLPHENOL (O-CRESOL)	06/15/2020	07/01/2021	SC	SBN	NE
6460	2-NITROANILINE	06/15/2020	07/01/2021	SC	SBN	NE
6490	2-NITROPHENOL	06/15/2020	07/01/2021	SC	SBN	NE
5945	3,3-DICHLOROBENZIDINE	06/15/2020	07/01/2021	SC	SBN	NE
6405	3-METHYLPHENOL (M-CRESOL)	06/15/2020	07/01/2021	SC	SBN	NE
6465	3-NITROANILINE	06/15/2020	07/01/2021	SC	SBN	NE
5660	4-BROMOPHENYL PHENYL ETHER	06/15/2020	07/01/2021	SC	SBN	NE
5700	4-CHLORO-3-METHYLPHENOL	06/15/2020	07/01/2021	SC	SBN	NE
0142NH	4-CHLORO-O-TOLUIDINE	06/15/2020	07/01/2021	SC	SBN	NE

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5745	4-CHLOROANILINE	06/15/2020	07/01/2021	SC	SBN	NE
5825	4-CHLOROPHENYL PHENYL ETHER	06/15/2020	07/01/2021	SC	SBN	NE
6410	4-METHYLPHENOL (P-CRESOL)	06/15/2020	07/01/2021	SC	SBN	NE
6470	4-NITROANILINE	06/15/2020	07/01/2021	SC	SBN	NE
6500	4-NITROPHENOL	06/15/2020	07/01/2021	SC	SBN	NE
5500	ACENAPHTHENE	06/15/2020	07/01/2021	SC	SBN	NE
5505	ACENAPHTHYLENE	06/15/2020	07/01/2021	SC	SBN	NE
5510	ACETOPHENONE	06/15/2020	07/01/2021	SC	SBN	NE
5545	ANILINE	06/15/2020	07/01/2021	SC	SBN	NE
5555	ANTHRACENE	06/15/2020	07/01/2021	SC	SBN	NE
5570	BENZALDEHYDE	06/15/2020	07/01/2021	SC	SBN	NE
5595	BENZIDINE	06/15/2020	07/01/2021	SC	SBN	NE
5575	BENZO(A)ANTHRACENE	06/15/2020	07/01/2021	SC	SBN	NE
5580	BENZO(A)PYRENE	06/15/2020	07/01/2021	SC	SBN	NE
5585	BENZO(B)FLUORANTHENE	06/15/2020	07/01/2021	SC	SBN	NE
5590	BENZO(G,H,I)PERYLENE	06/15/2020	07/01/2021	SC	SBN	NE
5600	BENZO(K)FLUORANTHENE	06/15/2020	07/01/2021	SC	SBN	NE
5610	BENZOIC ACID	06/15/2020	07/01/2021	SC	SBN	NE
5630	BENZYL ALCOHOL	06/15/2020	07/01/2021	SC	SBN	NE
5670	BENZYL BUTYL PHTHALATE	06/15/2020	07/01/2021	SC	SBN	NE
5760	BIS(2-CHLOROETHOXY) METHANE	06/15/2020	07/01/2021	SC	SBN	NE
5765	BIS(2-CHLOROETHYL) ETHER	06/15/2020	07/01/2021	SC	SBN	NE
6065	BIS(2-ETHYLHEXYL) PHTHALATE	06/15/2020	07/01/2021	SC	SBN	NE
5680	CARBAZOLE	06/15/2020	07/01/2021	SC	SBN	NE
5855	CHRYSENE	06/15/2020	07/01/2021	SC	SBN	NE
5925	DI-N-BUTYL PHTHALATE	06/15/2020	07/01/2021	SC	SBN	NE
6200	DI-N-OCTYL PHTHALATE	06/15/2020	07/01/2021	SC	SBN	NE
5895	DIBENZO(A,H)ANTHRACENE	06/15/2020	07/01/2021	SC	SBN	NE
5905	DIBENZOFURAN	06/15/2020	07/01/2021	SC	SBN	NE
6070	DIETHYL PHTHALATE	06/15/2020	07/01/2021	SC	SBN	NE
6135	DIMETHYL PHTHALATE	06/15/2020	07/01/2021	SC	SBN	NE
6205	DIPHENYLAMINE	06/15/2020	07/01/2021	SC	SBN	NE
6265	FLUORANTHENE	06/15/2020	07/01/2021	SC	SBN	NE
6270	FLUORENE	06/15/2020	07/01/2021	SC	SBN	NE
6275	HEXACHLOROBENZENE	06/15/2020	07/01/2021	SC	SBN	NE
6315	INDENO(1,2,3-CD)PYRENE	06/15/2020	07/01/2021	SC	SBN	NE

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6320	ISOPHORONE	06/15/2020	07/01/2021	SC	SBN	NE
6545	N-NITROSODI-N-PROPYLAMINE	06/15/2020	07/01/2021	SC	SBN	NE
6530	N-NITROSODIMETHYLAMINE	06/15/2020	07/01/2021	SC	SBN	NE
6535	N-NITROSODIPHENYLAMINE	06/15/2020	07/01/2021	SC	SBN	NE
6600	PCNB (PENTACHLORONITROBENZENE)	06/15/2020	07/01/2021	SC	SBN	NE
6605	PENTACHLOROPHENOL	06/15/2020	07/01/2021	SC	SBN	NE
6615	PHENANTHRENE	06/15/2020	07/01/2021	SC	SBN	NE
6625	PHENOL	06/15/2020	07/01/2021	SC	SBN	NE
6665	PYRENE	06/15/2020	07/01/2021	SC	SBN	NE
Method Code: 10242565	**Method Ref: SW-846 8270E SIM**		**Revision:**		**Date: 2017**	
6380	1-METHYLNAPHTHALENE	06/15/2020	07/01/2021	SC	SBN	NE
6360	2-METHYL-4,6-DINITROPHENOL	06/15/2020	07/01/2021	SC	SBN	NE
6385	2-METHYLNAPHTHALENE	06/15/2020	07/01/2021	SC	SBN	NE
5500	ACENAPHTHENE	06/15/2020	07/01/2021	SC	SBN	NE
5505	ACENAPHTHYLENE	06/15/2020	07/01/2021	SC	SBN	NE
5555	ANTHRACENE	06/15/2020	07/01/2021	SC	SBN	NE
5575	BENZO(A)ANTHRACENE	06/15/2020	07/01/2021	SC	SBN	NE
5580	BENZO(A)PYRENE	06/15/2020	07/01/2021	SC	SBN	NE
5585	BENZO(B)FLUORANTHENE	06/15/2020	07/01/2021	SC	SBN	NE
5590	BENZO(G,H,I)PERYLENE	06/15/2020	07/01/2021	SC	SBN	NE
5600	BENZO(K)FLUORANTHENE	06/15/2020	07/01/2021	SC	SBN	NE
5855	CHRYSENE	06/15/2020	07/01/2021	SC	SBN	NE
5895	DIBENZO(A,H)ANTHRACENE	06/15/2020	07/01/2021	SC	SBN	NE
6265	FLUORANTHENE	06/15/2020	07/01/2021	SC	SBN	NE
6270	FLUORENE	06/15/2020	07/01/2021	SC	SBN	NE
6315	INDENO(1,2,3-CD)PYRENE	06/15/2020	07/01/2021	SC	SBN	NE
6605	PENTACHLOROPHENOL	06/15/2020	07/01/2021	SC	SBN	NE
6615	PHENANTHRENE	06/15/2020	07/01/2021	SC	SBN	NE
6665	PYRENE	06/15/2020	07/01/2021	SC	SBN	NE
Method Code: 10307003	**Method Ref: SW-846 8260C**		**Revision:**		**Date:**	
6800	1,3,5-TRICHLOROBENZENE	02/21/2017	07/01/2021	SC	SBN	NE
Method Code: 10307127	**Method Ref: SW-846 8260D**		**Revision:**		**Date: 2018**	
6800	1,3,5-TRICHLOROBENZENE	06/15/2020	07/01/2021	SC	SBN	NE
Method Code: 90014258	**Method Ref: NJ DEP EPH**		**Revision:**		**Date: 2010**	
3797	TOTAL EPH	02/21/2017	07/01/2021	SC	SBN	NE
Method Code: 90017202	**Method Ref: MADEP EPH**		**Revision: 1.1**		**Date: 2004**	
6385	2-METHYLNAPHTHALENE	08/20/2018	07/01/2021	SC	SBN	NE

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5500	ACENAPHTHENE	08/20/2018	07/01/2021	SC	SBN	NE
5505	ACENAPHTHYLENE	08/20/2018	07/01/2021	SC	SBN	NE
5555	ANTHRACENE	08/20/2018	07/01/2021	SC	SBN	NE
5575	BENZO(A)ANTHRACENE	08/20/2018	07/01/2021	SC	SBN	NE
5580	BENZO(A)PYRENE	08/20/2018	07/01/2021	SC	SBN	NE
5585	BENZO(B)FLUORANTHENE	08/20/2018	07/01/2021	SC	SBN	NE
5590	BENZO(G,H,I)PERYLENE	08/20/2018	07/01/2021	SC	SBN	NE
5600	BENZO(K)FLUORANTHENE	08/20/2018	07/01/2021	SC	SBN	NE
5855	CHRYSENE	08/20/2018	07/01/2021	SC	SBN	NE
5895	DIBENZO(A,H)ANTHRACENE	08/20/2018	07/01/2021	SC	SBN	NE
6218	EPH ALIPHATIC C19-C36	08/20/2018	07/01/2021	SC	SBN	NE
6222	EPH ALIPHATIC C9-C18	08/20/2018	07/01/2021	SC	SBN	NE
6232	EPH AROMATIC C11-C22	08/20/2018	07/01/2021	SC	SBN	NE
6234	EPH AROMATIC C11-C22 UNADJUSTED	08/20/2018	07/01/2021	SC	SBN	NE
6265	FLUORANTHENE	08/20/2018	07/01/2021	SC	SBN	NE
6270	FLUORENE	08/20/2018	07/01/2021	SC	SBN	NE
6315	INDENO(1,2,3-CD)PYRENE	08/20/2018	07/01/2021	SC	SBN	NE
6615	PHENANTHRENE	08/20/2018	07/01/2021	SC	SBN	NE
6665	PYRENE	08/20/2018	07/01/2021	SC	SBN	NE
Method Code: NH0308	**Method Ref: MADEP EPH**		**Revision: 2.1**		**Date:**	
6385	2-METHYLNAPHTHALENE	06/15/2020	07/01/2021	SC	SBN	NE
5500	ACENAPHTHENE	06/15/2020	07/01/2021	SC	SBN	NE
5505	ACENAPHTHYLENE	06/15/2020	07/01/2021	SC	SBN	NE
5555	ANTHRACENE	06/15/2020	07/01/2021	SC	SBN	NE
5575	BENZO(A)ANTHRACENE	06/15/2020	07/01/2021	SC	SBN	NE
5580	BENZO(A)PYRENE	06/15/2020	07/01/2021	SC	SBN	NE
5585	BENZO(B)FLUORANTHENE	06/15/2020	07/01/2021	SC	SBN	NE
5590	BENZO(G,H,I)PERYLENE	06/15/2020	07/01/2021	SC	SBN	NE
5600	BENZO(K)FLUORANTHENE	06/15/2020	07/01/2021	SC	SBN	NE
5855	CHRYSENE	06/15/2020	07/01/2021	SC	SBN	NE
5895	DIBENZO(A,H)ANTHRACENE	06/15/2020	07/01/2021	SC	SBN	NE
6218	EPH ALIPHATIC C19-C36	06/15/2020	07/01/2021	SC	SBN	NE
6222	EPH ALIPHATIC C9-C18	06/15/2020	07/01/2021	SC	SBN	NE
6232	EPH AROMATIC C11-C22	06/15/2020	07/01/2021	SC	SBN	NE
6234	EPH AROMATIC C11-C22 UNADJUSTED	06/15/2020	07/01/2021	SC	SBN	NE
6265	FLUORANTHENE	06/15/2020	07/01/2021	SC	SBN	NE

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6270	FLUORENE	06/15/2020	07/01/2021	SC	SBN	NE
6315	INDENO(1,2,3-CD)PYRENE	06/15/2020	07/01/2021	SC	SBN	NE
6615	PHENANTHRENE	06/15/2020	07/01/2021	SC	SBN	NE
6665	PYRENE	06/15/2020	07/01/2021	SC	SBN	NE
Method Code: 10183207	**Method Ref: SW-846 8151A**		**Revision:**		**Date: 1998**	
8655	2,4,5-T	02/21/2017	07/01/2021	SC	SHE	NE
8650	2,4,5-TP (SILVEX)	02/21/2017	07/01/2021	SC	SHE	NE
8545	2,4-D	02/21/2017	07/01/2021	SC	SHE	NE
8560	2,4-DB	02/21/2017	07/01/2021	SC	SHE	NE
8555	DALAPON	02/21/2017	07/01/2021	SC	SHE	NE
8595	DICAMBA	02/21/2017	07/01/2021	SC	SHE	NE
8605	DICHLOROPROP (DICHLORPROP)	02/21/2017	07/01/2021	SC	SHE	NE
Method Code: 10305609	**Method Ref: SW-846 8015D**		**Revision:**		**Date: 2003**	
9369	DIESEL RANGE ORGANICS (DRO)	07/02/2018	07/01/2021	SC	SNO	NE
9408	GASOLINE RANGE ORGANICS (GRO)	07/02/2018	07/01/2021	SC	SNO	NE
9506	RESIDUAL RANGE ORGANICS (RRO) - OIL RANGE ORGANICS	11/13/2018	07/01/2021	SC	SNO	NE
Method Code: 10179201	**Method Ref: SW-846 8082A**		**Revision:**		**Date: 2000**	
8880	AROCLOR-1016 (PCB-1016)	02/21/2017	07/01/2021	SC	SPC	NE
8885	AROCLOR-1221 (PCB-1221)	02/21/2017	07/01/2021	SC	SPC	NE
8890	AROCLOR-1232 (PCB-1232)	02/21/2017	07/01/2021	SC	SPC	NE
8895	AROCLOR-1242 (PCB-1242)	02/21/2017	07/01/2021	SC	SPC	NE
8900	AROCLOR-1248 (PCB-1248)	02/21/2017	07/01/2021	SC	SPC	NE
8905	AROCLOR-1254 (PCB-1254)	02/21/2017	07/01/2021	SC	SPC	NE
8910	AROCLOR-1260 (PCB-1260)	02/21/2017	07/01/2021	SC	SPC	NE
8912	AROCLOR-1262 (PCB-1262)	02/21/2017	07/01/2021	SC	SPC	NE
8913	AROCLOR-1268 (PCB-1268)	02/21/2017	07/01/2021	SC	SPC	NE
Method Code: 10178800	**Method Ref: SW-846 8081B**		**Revision:**		**Date: 2000**	
7355	4,4-DDD	02/21/2017	07/01/2021	SC	SPE	NE
7360	4,4-DDE	02/21/2017	07/01/2021	SC	SPE	NE
7365	4,4-DDT	02/21/2017	07/01/2021	SC	SPE	NE
7005	ALACHLOR	02/21/2017	07/01/2021	SC	SPE	NE
7025	ALDRIN	02/21/2017	07/01/2021	SC	SPE	NE
7110	ALPHA-BHC (ALPHA-HEXACHLOROCYCLOHEXANE)	02/21/2017	07/01/2021	SC	SPE	NE
7240	ALPHA-CHLORDANE	02/21/2017	07/01/2021	SC	SPE	NE
7115	BETA-BHC (BETA-HEXACHLOROCYCLOHEXANE)	02/21/2017	07/01/2021	SC	SPE	NE
7250	CHLORDANE (TECH.)	02/21/2017	07/01/2021	SC	SPE	NE
7105	DELTA-BHC	02/21/2017	07/01/2021	SC	SPE	NE

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7470	DIELDRIN	02/21/2017	07/01/2021	SC	SPE	NE
7510	ENDOSULFAN I	02/21/2017	07/01/2021	SC	SPE	NE
7515	ENDOSULFAN II	02/21/2017	07/01/2021	SC	SPE	NE
7520	ENDOSULFAN SULFATE	02/21/2017	07/01/2021	SC	SPE	NE
7540	ENDRIN	02/21/2017	07/01/2021	SC	SPE	NE
7530	ENDRIN ALDEHYDE	02/21/2017	07/01/2021	SC	SPE	NE
7535	ENDRIN KETONE	02/21/2017	07/01/2021	SC	SPE	NE
7120	GAMMA-BHC (LINDANE)	02/21/2017	07/01/2021	SC	SPE	NE
7245	GAMMA-CHLORDANE	02/21/2017	07/01/2021	SC	SPE	NE
7685	HEPTACHLOR	02/21/2017	07/01/2021	SC	SPE	NE
7690	HEPTACHLOR EPOXIDE	02/21/2017	07/01/2021	SC	SPE	NE
7810	METHOXYCHLOR	02/21/2017	07/01/2021	SC	SPE	NE
8250	TOXAPHENE (CHLORINATED CAMPHENE)	02/21/2017	07/01/2021	SC	SPE	NE
Method Code: 10183207	**Method Ref: SW-846 8151A**		**Revision:**		**Date: 1998**	
7775	MCPA	02/21/2017	07/01/2021	SC	SPE	NE
7780	MCPP	02/21/2017	07/01/2021	SC	SPE	NE
Method Code: 10186002	**Method Ref: SW-846 8270D**		**Revision:**		**Date: 2007**	
7065	ATRAZINE	02/21/2017	07/01/2021	SC	SPE	NE
7180	CAPROLACTAM	02/21/2017	07/01/2021	SC	SPE	NE
6285	HEXACHLOROCYCLOPENTADIENE	02/21/2017	07/01/2021	SC	SPE	NE
7955	PARATHION ETHYL	02/21/2017	07/01/2021	SC	SPE	NE
Method Code: 10242543	**Method Ref: SW-846 8270E**		**Revision:**		**Date: 2017**	
7065	ATRAZINE	06/15/2020	07/01/2021	SC	SPE	NE
7180	CAPROLACTAM	06/15/2020	07/01/2021	SC	SPE	NE
6285	HEXACHLOROCYCLOPENTADIENE	06/15/2020	07/01/2021	SC	SPE	NE
7955	PARATHION ETHYL	06/15/2020	07/01/2021	SC	SPE	NE

ALPHA ANA 7/1/2020

Bill Hall
NH ELAP Program Manager
Issue Date: 07/01/2020

This analyte list supersedes all previously issued analyte lists. Method accreditation does not imply acceptance for NHDES compliance testing. Laboratory is required to use EPA-approved methods required by regulation. Continuing accreditation status is dependent on successful ongoing participation in the program. Customers may verify the laboratory's current accreditation status by calling (603) 271-2998 or by visiting the NH ELAP website (https://www.des.nh.gov/organization/divisions/water/dwgb/nhelap/index.htm).

NEW HAMPSHIRE ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM

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PRIMARY ACCREDITATION ANALYTE LIST

ANALYTE LIST NUMBER: 206420-A

ALPHA ANALYTICAL (WESTBOROUGH) [#2064]
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Matrix Legend: AE=Air; BT=Tissue; D=Drinking Water; N=Non-Potable Water; SC=Solid and Chemical Materials

Category Legend: MIC=Microbiology; MET=Metals; NMI=Non-Metal Inorganics; PRE=Preparation; VOC=Volatile Organic Compounds; SBN=SVOC-BNA; SHE=SVOC-Herbicides; SNO=SVOC-NOS; SPC=SVOC-PCB; SPE=SVOC-Pesticides; RAD=Radiochemistry; WET=Wet

Accreditation Legend: NE=NELAP; NH=NH State Certification; CE=State Certification; IN=Interim (NELAP); WI=Withdrawn; AP=Applied; RE=Revoked; SU=Suspended

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Nitrogen, Ammonia

Reference Methods: **SM4500 NH_3-BH,** Standard Methods for the Examination of Water and Wastewater. APHA-AWWA-WEF. Standard Methods Online.

Method 350.1, Methods for the Chemical Analysis of Water and Wastes, EPA 600/ 4-82-055, Revision 2.0, August 1993.

Method 10-107-06-1-A, Methods for Automated Ion Analyzers, May 20, 1998.

1. Scope and Application

Matrices: Ammonia nitrogen can be determined in potable, surface, and saline waters as well as domestic and industrial wastewaters and in soils.

Definitions: See Alpha Laboratories Quality Manual Appendix A.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Laboratory Services Manager, Laboratory Director, or Quality Assurance Officer

This method is restricted to use by or under the supervision of analysts experienced in the operation of the Tecator and/or Lachat Instrument and in the interpretation of Lachat data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

In waters and wastewaters the forms of nitrogen of greatest interest are, in order of decreasing oxidation state: nitrate, nitrite, ammonia, and organic nitrogen. All these forms of nitrogen, as well as nitrogen gas (N_2), are biochemically interconvertible and are components of the nitrogen cycle. They are of interest for many reasons. Organic nitrogen is defined functionally as organically bound nitrogen in the trinegative oxidation state. It does not include all organic nitrogen compounds. Analytically, organic nitrogen and ammonia can be determined together and have been referred to as “kjeldahl nitrogen,” a term that reflects the technique used in their determination. Organic nitrogen includes such natural materials as proteins and peptides, nucleic acids and urea, and numerous synthetic organic materials. Typical organic nitrogen concentrations vary from a few hundred micrograms per liter in some lakes to more than 20mg/L in raw sewage.

Ammonia is present naturally in surface and wastewaters. Its concentration generally is low in groundwaters because it adsorbs to soil particles and clays and is not leached readily from soils. It is produced largely by deamination of organic nitrogen containing compounds and by hydrolysis of urea. At some water treatment plants ammonia is added to react with chlorine to form a combined chlorine residual.

In the chlorination of wastewater effluents containing ammonia, virtually no free residual chlorine is obtained until the ammonia has been oxidized. Rather, the chlorine reacts with ammonia to form mono- and dichloramines. Ammonia concentrations encountered in water vary from less than 10µg ammonia nitrogen/L in some natural surface and groundwaters to more than 30mg/L in some wastewaters.

In this discussion, organic nitrogen is referred to as organic N, nitrate nitrogen as NO_3-N, nitrite nitrogen as NO_2-N, and ammonia nitrogen as NH_3-N.

2. Summary of Method

The sample is buffered at pH 9.5 with a borate buffer to decrease hydrolysis of cyanates and organic nitrogen compounds. It is distilled into a solution of boric acid. The ammonia in the distillate is determined colorimetrically by the phenate method.

The phenate method is base on the Berthelot reaction. Ammonia reacts with alkaline phenol, then with sodium hypochlorite to form indophenol blue. Sodium nitroprusside (nitroferricyanide) is added to enhance sensitivity. The absorbance of the reaction product is measured at 630nm, and is directly proportional to the original ammonia concentration.

2.1 Method Modifications from Reference

Method was modified to include soil sample distillation. Samples are buffered at pH 9.5. pH is checked using pH paper 7.5-14 (not pH meter)

3. Detection Limits

The laboratory follows the procedure found in 40CFR Part 136 to determine the MDL on an annual basis. The method detection limits determined by the laboratory are on file for review.

The RDL is determined to be 0.075mg/L based on a 100mL sample size for waters and 7.5 mg/kg based on 1g of soil material used for distillation.

4. Interferences

Most of the following interferences refer to the direct analysis of a water sample. The distillation steps remove most of these interferences.

4.1 Instrument

Samples with a high concentration of NH_3 may carry-over into the next sample and therefore yield false high results in that next sample. If a sample with a low concentration follows a sample with a high concentration, re-analyze the low sample to ensure results are accurate.

4.2 Parameters

4.2.1 Glycine, urea, glutamic acid, cyanates, and acetamide hydrolyze very slowly in solution on standing but, of these, only urea and cyanates will hydrolyze on distillation at pH of 9.5. Hydrolysis amounts to about 7% at this pH for urea and about 5% for cyanates. Some of the organic compounds that may interfere, such as formaldehyde, may be eliminated by boiling off at a low pH before nesslerization. Remove residual chlorine by sample pretreatment.

4.2.2 Color and turbidity of the sample distillate may interfere with analysis. These interferences are removed by manual filtration of the distillate prior to Lachat analysis, or by dilution of the sample prior to distillation.

5. Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

Note: Phenol used in this method is hazardous and general laboratory safety practices must be observed.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, and Handling

6.1 Sample Collection

The most reliable results are obtained on fresh samples. Use plastic or glass containers.

6.2 Sample Preservation

If prompt analysis is impossible, preserve samples with approximately 0.8mL concentrated H_2SO_4/L sample. The pH of the acid-preserved samples should be <2. (Some wastewaters may require more concentrated H_2SO_4 to achieve this pH.) Store samples at 4ºC.

Soils samples are received without preservation and stored at 4ºC

6.3 Sample Handling

Samples (both waters and soils) are analyzed within 28 days after collection.

7. Equipment and Supplies

7.1 Distillation apparatus: Tecator Instruments Automatic Distillation Unit. Follow the instrument manufacturer's instructions for proper operation.

7.2 pH Strips: Range 7.5 to 14 units.

7.3 250mL Disposable Polypropylene cups with covers.

7.4 Automated Ion Analyzer: Lachat Instruments.

7.5 Helium gas, He: 140kPa (20 lb/in^2).

7.6 Volumetric flasks, various volumes.

7.7 Pipets, various volumes.

7.8 Analytical Balance

7.9 Centrifuge Tubes: 50mL volume.

8. Standards and Reagents

8.1 Borate buffer solution: Add 88mL 0.1N NaOH solution to 500mL of approximately 0.025M sodium tetraborate ($Na_2B_4O_7$) solution (9.5g $Na_2B_4O_7 \cdot 10\ H_2O$/L) and dilute to 1L with DI water. Store at room temperature. Expires one month from date of preparation.

8.2 Sodium thiosulfate (dechlorinating reagent): Dissolve 0.35g $Na_2S_2O_3 \cdot 5H_2O$ in DI water and dilute to 100mL. Prepare fresh daily as needed.

8.3 Neutralization agents: Prepare with DI water:

8.3.1 Sodium hydroxide, NaOH, 6N: In a 1L volumetric flask, carefully mix 240g of NaOH (Section 8.11) pellets with 500mL DI water. Dissolve and bring to volume with DI water. Store at room temperature. Expires one month from date of preparation.

8.3.2 Sulfuric acid, H_2SO_4, 10%: To a 1L volumetric flask, add 500mL DI water. Carefully add 100mL of concentrated Sulfuric Acid (Section 8.12). Bring to volume with DI water. Store at room temperature. Expires one month from date of preparation.

8.4 Sodium Phenolate: **CAUTION!** Wear gloves. Phenol causes severe burns and is rapidly absorbed into the body through the skin. In a 1L volumetric flask, dissolve 88mL of 88% liquified phenol or 83g crystalline phenol (C_6H_5OH) in approximately 600mL DI water. While stirring, slowly add 32g sodium hydroxide (NaOH). Cool, dilute to the mark, and invert three times. Do **not** degas this reagent. Store at room temperature. Expires one month from date of preparation.

8.5 Sodium Hypochlorite (approximate 2.6%): In a 500mL volumetric flask, dilute 250mL Regular Chlorine bleach [5.25% sodium hypochlorite (NaOCl)] to the mark with DI water. Invert three times to mix. Store at room temperature. Expires one month from date of preparation.

8.6 Sodium Nitroprusside (coloring agent): In a 1L volumetric flask, dissolve 3.50g sodium nitroprusside (Sodium Nitroferricyanide $[Na_2Fe(CN)_5NO_2 \cdot H_2O]$) dilute to the mark with DI water. Degas with helium to prevent bubble formation. Use He at 140kPa (20 lb/in^2) through a helium degassing tube. Bubble He vigorously through the solution for one minute. Store refrigerated at 4 ± 2 °C. Expires one month from date of preparation.

8.7 Boric 1.5% Boric Acid Solution: To a 1000mL volumetric flask add 15g Boric Acid. Dilute to the mark with DI water. Store at room temperature. Expires one month from date of preparation.

8.8 0.2% Boric Acid Solution (Carrier Solution): To a 2L volumetric flask, dissolve 4g Boric Acid (H_3BO_3) in DI water. Degas by bubbling vigorously with Helium for one minute. Store at room temperature. Expires one month from date of preparation.

8.9 Stock Standard, 1000ppm as NH_3 (for calibration and spike solutions): Commercially prepared. Certificate of analysis is required. Store refrigerated at 4 ± 2 °C. Expires upon manufacturer's specified date.

8.9.1 Intermediate Calibration Stock Standard, 100ppm as NH_3: To a 100mL volumetric flask, add 10.0mL of Stock Standard (Section 8.9) and dilute to the mark with 0.2% Boric Acid solution (Section 8.8). Invert three times. Store refrigerated at 4 ± 2 °C. Expires one month from date of preparation.

8.9.1.1 Nine Working Calibration Standards: The following standards are prepared in volumetric flasks fresh each day of use:

8.9.1.1.1 **20.0ppm:** 40mL of 100ppm standard (Section 8.9.1) to 200mL with 0.2% Boric Acid solution (Section 8.8).

8.9.1.1.2 **10.0ppm:** 20mL of 100ppm standard (Section 8.9.1) to 200mL with 0.2% Boric Acid solution (Section 8.8). Prepare fresh each day of use.

8.9.1.1.3 **8.00ppm:** 8mL of 100ppm standard (Section 8.9.1) to 100mL with 0.2% Boric Acid solution (Section 8.8).

8.9.1.1.4 **4.00ppm:** 8mL of 100ppm standard (Section 8.9.1) to 200mL with 0.2% Boric Acid solution (Section 8.8).

8.9.1.1.5 **2.00ppm:** 2mL of 100ppm standard (Section 8.9.1) to 100mL with 0.2% Boric Acid solution (Section 8.8).

8.9.1.1.6 **1.00ppm:** 1mL of 100ppm standard (Section 8.9.1) to 100mL with 0.2% Boric Acid solution (Section 8.8).

8.9.1.1.7 **0.400ppm:** 4mL of 20ppm standard (Section 8.9.1.1.1) to 200mL with 0.2% Boric Acid solution (Section 8.8).

8.9.1.1.8 **0.200ppm:** 1mL of 20ppm standard (Section 8.9.1.1.1) to 100mL with 0.2% Boric Acid solution (Section 8.8). Alternately, this standard may be prepared utilizing autodilution of the 20ppm standard on the autosampler.

8.9.1.1.9 **0.100ppm:** 1mL of 20ppm standard (Section 8.9.1.1.1) to 200mL with 0.2% Boric Acid solution (Section 8.8). Alternately, this standard may be prepared utilizing autodilution of the 20ppm standard on the autosampler.

8.9.1.1.10 **0.050ppm:** 20mL of 0.100ppm standard (Section 8.9.1.1.8) to 40mL with 0.2% Boric Acid solution (Section 8.8). Alternately, this standard may be prepared utilizing autodilution of the 20ppm standard on the autosampler.

8.9.1.2 Continuing Calibration Standards:

8.9.1.2.1 **0.400ppm Low CCV:** 4mL of 20ppm standard (Section 8.9.1.1.1) to 200mL with 0.2% Boric Acid solution (Section 8.8).

8.9.1.2.2 **4.00ppm Hi CCV:** 8mL of 100ppm standard (Section 8.9.1) to 200mL with 0.2% Boric Acid solution (Section 8.8).

8.9.2 Spiking solution, 200ppm as NH_3: To a 100mL volumetric flask add 20mL of 1000ppm Stock Standard (Section 8.9) and dilute to the mark with DI water.

8.10 Stock Standard Solution, 1000ppm as NH_3 (for ICV and LCS): Commercially prepared. Certificate of analysis is required. This must be from a different source than that used for Stock Standard (Section 8.9). Store refrigerated at 4 ± 2 °C. Expires upon manufacturer's specified date.

8.10.1 Initial Calibration Verification Standards (ICV):

8.10.1.1 Hi ICV, 10ppm: To a 100mL volumetric flask add 1mL of 1000ppm standard (Section 8.10). Dilute to the mark with 0.2% Boric Acid Solution (Section 8.8). Expires one month from date of preparation.

8.10.1.2 Hi ICV, 8.0ppm: To a 100mL volumetric flask add 0.8mL of 1000ppm standard (Section 8.10). Dilute to the mark with 0.2% Boric Acid Solution (Section 8.8). Expires one month from date of preparation

8.10.1.3 Low ICV, 1.0ppm: To a 100mL volumetric flask add 10mL of 10ppm ICV (Section 8.10.1.1). Dilute to the mark with 0.2% Boric Acid Solution (Section 8.8). Expires one month from date of preparation.

8.10.2 LCS solution, 200ppm as NH_3: To a 100mL volumetric flask add 20mL of 1000ppm Stock Standard (Section 8.10) and dilute to the mark with DI water. Store refrigerated at 4 ± 2 °C. Expires one month from date of preparation.

8.11 NaOH Pellets: ACS Grade. Store well sealed at room temperature. This salt does not have an expiration date.

8.12 Sulfuric Acid (Concentrated): Store at room temperature. Expires upon manufacturer's specified date. Use extreme caution when handling.

8.13 Ottawa sand: store at room temperature. Expires upon manufacturer's specified date.

8.14 Thymol Blue Sodium Salt 0.04% water solution: used as indicator during pH adjustment prior distillation. Store at room temperature. Expires upon manufacturer's specified date.

9. Procedure

9.1 SET-UP

9.1.1 Prior to analysis, organize samples in order from low to high odor.

9.1.2 Clean 250mL Tecator tubes by rinsing twice with approximately 0.5mL of 6N NaOH solution and 100mL RO water. Rinse twice again with DI water.

9.2 Initial Calibration

Calibrate the Lachat ion analyzer according to manufacturer's instructions.

9.2.1 Calibration

Two boards are used to calibrate the Lachat instrument. Each curve has 7 calibration points. The correlation coefficient of each curve must be $\geq$ 0.995, otherwise re-calibration is necessary. Prepare standard curves by plotting the peak areas of standards processed through the manifold against NH_3-N concentrations in standards.

9.2.1.1 Channel 1 is used to generate a calibration curve on the low range from 0.00 – 2ppm.

9.2.1.2 Channel 2 is used to generate a calibration curve on the high range from 0 – 20ppm.

Alternative method: One board can be used to calibrate the Lachat instrument. !0 point calibration curve will be used with calibration standards 10.0, 8.0, 4.0, 2.0, 1.0,04, 0.2, 0.1, 0.05 mg/l each and blank. The correlation coefficient must be $\geq$ 0.995, otherwise re-calibration is necessary. Prepare standard curves by plotting

the peak areas of standards processed through the manifold against NH_3-N concentrations in standards.
Calibration coefficient will be calculated using Lachat software. All calibration points are back calculated by Lachat software and should be within 10% from true concentration, except 2 lowest points of calibration curve. %recoveries for low range will be wider, but shouldn't exceed 100% and correlation coefficient will not be worse then 0.995.

9.2.2 Initial Calibration Verification (ICV)
Prior to sample analysis, an ICV is analyzed at 1.0ppm to verify the low calibration curve on Channel 1. Another ICV is analyzed at 10ppm to verify the high calibration curve on Channel 2. Both ICVs must yield results ± 10% of their true value, otherwise re-calibration is necessary.

Note: if instrument is calibrated using one board calibration, then both ICV's (Low and High), will be evaluated and high ICV will be 8.0 mg/l ICV standard (Section 8.10.1.2)

9.2.3 Initial Calibration Blank (ICB)
Following the ICV is the analysis of an ICB. The ICB consists of an aliquot of 0.2% Boric Acid (Section 8.8). Results must be less than the Reporting Limit.

9.3 Standardization (Continuing Calibration Verification)

Analyze the following after every 10 samples and at the completion of analysis:

0.400ppm Low CCV, (Section 8.9.1.2.1)

4.0ppm Hi CCV, (Section 8.9.1.2.2)

Blank, 0.2% Boric Acid Solution. (Section 8.8)

9.4 Equipment Operation and Sample Analysis

9.4.1 Sample preparation

9.4.1.1 Sample Aliquots

9.4.1.1.1 Aqueous Samples
Add 100mL dechlorinated sample or a portion diluted to 100mL with DI water, to pre-washed Tecator tubes that are numbered to correspond with the samples.

9.4.1.1.1 Soil Samples
One gram of a well-homogenized sample is weighed out into a centrifuge tube (record weight in notebook). The sample is then transferred into a Tecator tube with 100 mL of DI.

9.4.1.1.2 QC Samples
In a similar manner, prepare the QC samples to be distilled with the batch (refer to Sections 10.2.1, 10.3, 10.5 and 10.8).

9.4.1.2 Add approximately 1.0mL of $Na_2S_2O_3$ to each tube, followed by 5.0mL of Borate Buffer solution.

9.4.1.2.1 Aqueous Samples

Add small amounts of 6N NaOH until the pH is adjusted to 9.5. 2-3 drops of indicator (sec 8.14) can be used for pH adjustment. Color of solution will change from pink to blue at pH 9.5. Use pH paper to confirm pH reading.

9.4.1.2.2 Soil Samples

No pH adjustment is needed.

9.4.2 Distillation

9.4.2.1 Equipment preparation: To clean any prior contamination, first rinse the Tecator distillation unit by running a series of 3 rinse tubes. The first rinse containing 90mL DI and 10mL 6N NaOH. The second and third rinses are with100mL DI and 0.5mL 6N NaOH.

9.4.2.2. Distillation: Begin by distilling rinse tubes, Blank, Low and Hi LCS, and then Samples and QC Samples using the Tecator Instruments Automatic Distillation Unit according to the manufacturer's instructions.

Distillate is collected from the Tecator unit in 250mL disposable polypropylene cups that contain 20mL of 1.5% Boric acid (Section 8.7). Distillation is complete after collection of approximately 140mL. Fill each cup to 150mL with DI water. Cover and store at 1-4º C if Lachat analysis is to be delayed.

9.4.3 Ammonia analysis of distillate: Follow the manufacturer's instructions for the proper operation of the Lachat ion analyzer. The following are specific notes for this analysis.

Sample throughput:	90 samples/hr; 40sec/sample
Pump speed:	35
Cycle period:	40 s
Inject to start of peak period:	25 s
Inject to end of peak period:	63 ± s

9.4.4 System Notes:

9.4.4.1 Allow 15 minutes for heating unit to warm up to 60ºC.

9.4.4.2 System IV GAIN: 175 X 1

9.4.4.3 If standards are not distilled, samples should be multiplied by 1.5 to correct for the increase in volume after the distillation.

9.4.4.4 If baseline drifts, peaks are too wide, or other problems with precision arise, clean the manifold by the following procedure:

9.4.4.4.1 Place all reagent lines in deionized water and pump to clear reagents (2-5 minutes).

9.4.4.4.2 Place reagent lines and carrier in 1M hydrochloric acid (1 volume concentrated HCl added to 11 volumes of deionized water) and pump for several minutes.

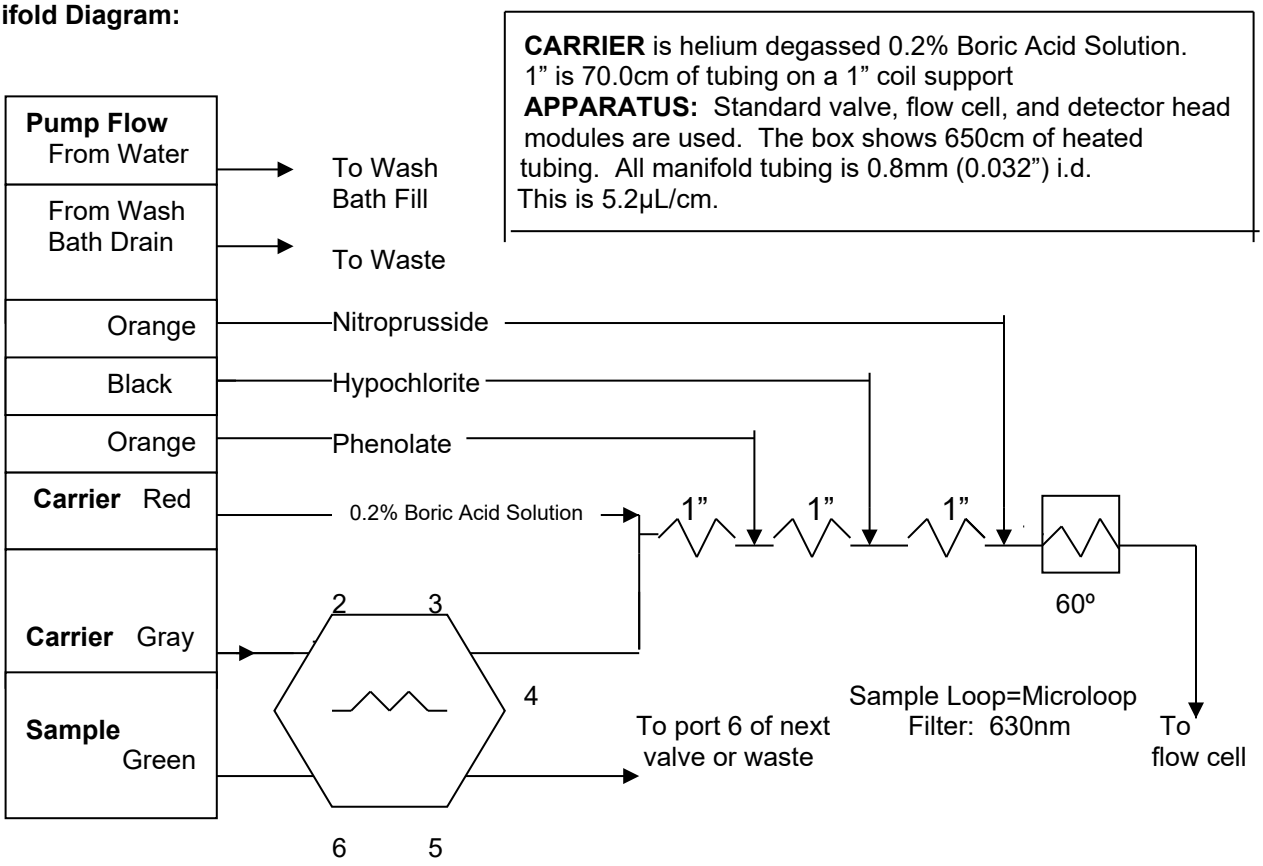
9.4.4.4.3 Place all lines in deionized water and pump until the HCl is thoroughly washed out.

9.4.4.4.4 Resume pumping reagents.

9.4.4.4.5 If sample distillate is colored or turbid, discard the sample distillate and re-distill the original sample at a higher dilution (Sections 9.4.1 and 9.4.2).

If there is not sufficient original sample volume for re-distillation, the colored/turbid distillate may be filtered through a 0.45µm filter and diluted on the Lachat instrument.

Manifold Diagram:



9.5 Preventative Maintenance

9.5.1 All lines are flushed at the end of each run.

9.5.2 All equipment is kept clean.

9.6 Calculations

Compute sample NH_3-N concentration by comparing sample peak areas with standard curve, as determined by the Lachat instrument software.

9.6.1 If the sample has a concentration of less than 2ppm, calculate results by using the low curve generated on Channel 1.

9.6.2 If the sample concentration is greater than 2ppm but less than 20ppm, calculate results by using the high curve generated on Channel 2.

9.6.3 If the concentration is greater than 20ppm, dilute the sample and reanalyze.

NOTE: For soil samples Lachat reading is multiplied by extraction volume (150ml) and divided by sample weight (the exact weight is used as recorded in the laboratory notebook). Calculations are done using Lachat software.

10. Quality Control and Data Assessment

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a calibration verification standard is used to confirm the measurements were performed in an in-control mode of operation.

10.1 Demonstration of Capability

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method. Each time a method modification is made, the analyst is required to repeat the procedure.

When one or more of the parameters tested fail at least one of the acceptance criteria, the analyst must locate and correct the source of the problem and repeat the test for failed parameters of the method.

Repeated failure confirms a general problem with the measurement system or analytical technique of the analyst. If the failure repeats, locate and correct the source of the problem and repeat the test for all parameters listed in the method.

10.2 Blank

10.2.1 The Distillation Blank is 100 mL DI for waters or 1g of Ottawa sand for soils. Distill one per batch of 20 samples or less. Results for the Distillation Blank must be less than the Reporting Limit. If results for the Distillation Blank are greater than the reporting limit, and all sample results for the batch are greater than 10x the Blank result, sample results may be reported with a narrative to be included on the final report. Otherwise, all samples must be redistilled and reanalyzed.

10.2.2 The Analytical Blank (ICB) for the Lachat analysis is not distilled and is 0.2% Boric Acid Solution (Section 8.8).

The ICB is run after the initial calibration verification standards (ICV) and another is run after the continuing calibration standards (CCV).

If results for the Analytical Blank are greater than the reporting limit, the ICB is reinjected. If failure continues the instrument must be recalibrated. Samples are not to be analyzed until an acceptable ICB is obtained.

10.3 Laboratory Control Samples (LCS)

10.3.1 Distillation: Distill a Low and a Hi LCS with each batch of 20 samples or less. Recovery of the LCSs must be within +/- 10% of true value for method 350.1 for waters, and within control charts for method 4500 and soils. If recovery is outside of this range, the standard is reanalyzed once. If recovery still fails criteria, the entire sample batch must be redistilled and reanalyzed.

10.3.1.1 Low 2ppm LCS: Add 1mL of 200ppm LCS solution (Section 8.10.2) to 100mL DI for waters or for 1.0g of Ottawa sand for soils. This is used for the Low 0 – 2ppm curve.

10.3.1.2 Hi 20ppm LCS: Add 10mL of 200ppm LCS solution (Section 8.10.2) to 100mL DI for waters or for 1.0g of Ottawa sand for soils. This is used for the Hi 2 – 20ppm curve.

10.4 Initial Calibration Verification Standards

10.4.1 Lachat Analysis: The ICVs are not distilled. Analyze the following Hi and Low ICVs after calibration of the Lachat instrument. Recoveries must be within 10% of the true value. If results are outside of this range, the ICV may be reinjected once. If failure continues, recalibration of the instrument is necessary. Samples are not to be analyzed until acceptable ICV results are obtained.

10.4.1.1 Low ICV, 1.0ppm (Section 8.10.1.2)

10.4.1.2 Hi ICV, 10ppm (Section 8.10.1.1). **HI ICV** will be **8.0** ppm (Section 8.10.1.2) in case of one board calibration.

10.5 Continuing Calibration Verification Standards

10.5.1 Lachat Analysis: The CCVs are not distilled. Analyze the following Low and Hi CCVs after every ten samples and at the completion of analysis. Recoveries must be within 10% of the true value.

If recoveries fall outside of this range, the CCV is reanalyzed. If recovery is acceptable, the analysis may continue. If recovery is again unacceptable, the cause for the failure is determined and corrected, and the instrument is recalibrated. All samples that were analyzed since the last CCV that was within range are reanalyzed.

10.5.1.1 0.4ppm Low CCV (Section 8.9.1.2.1)

10.5.1.2 4.0ppm Hi CCV (Section 8.9.1.2.2)

10.6 Interference Check Standards

None.

10.7 Matrix Spike

One matrix spike is prepped and analyzed per batch of **20 samples** or less for **method 4500**. Matrix spike has to be analyzed **for every 10 samples** or **less for method 350.1**. Prior to distillation, use 2mL of 200ppm Intermediate Stock Standard (Section 8.9.2) per 100mL of sample for waters or 1.0g of sample for soils. Recovery of the Matrix Spike must be within +/- 10% of true value for waters and within control charts for soils. If the recovery is outside of acceptance criteria, and the LCS is within acceptance criteria, matrix interference may be assumed. The associated data is reported with a narrative to be included on the final report.

10.8 Duplicates

Distill one duplicate sample per batch of 20 samples or less. The %RPD for the duplicate sample must be within Control Chart limits. If results are outside of the acceptance criteria, a narrative to be included on the final report.

10.9 Control Limits

The laboratory maintains performance records to document the quality of data that is generated. Method accuracy for samples is assessed and records maintained.

Control limits for the method parameters are generated. The control limits are calculated based on in-house performance data. The limits are compared to the control limits found in the reference method.

10.10 Analytical Sequences

10.10.1 Distillation Sequence:

3 Rinse tubes
Blank
Low LCS
Hi LCS
Rinse
Samples
Rinse after suspected high samples
Duplicate
Spike
Rinse
Shut-Down

10.10.2 Lachat Analytical Sequence:

Instrument Calibration
CCV: Low, Hi,CCB
Low ICV

Hi ICV
IC Blank
Samples
CCV: Low, Hi, CC Blank every 10 samples and at completion of analysis
Shut-Down

11. Method Performance

11.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

Note: Method **350.1** required MDL studies to be performed and evaluated every 6 month.

11.2 Demonstration of Capability Studies

Refer to Alpha SOP/1734, 1739 for further information regarding IDC/DOC Generation.

11.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

11.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

12. Corrective Actions

Holding time exceedence, improper preservation and observed sample headspace are noted on the nonconformance report form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

Review of standards, blanks and standard response for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form.

If the CV or LCS recovery of any parameter falls outside the designated acceptance range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked samples is suspect and is only reported for regulatory compliance purposes with the appropriate nonconformance action form. Immediate corrective action includes reanalyzing all affected samples by using any retained sample before the expiration of the holding time and/or recalibration of the instrument.

13. Pollution Prevention

NOTE: Lachat Phenol waste is hazardous and must be disposed of in "Hazmat" Lachat waste drums.

See Chemical Hygiene Plan for pollution prevention operations.

14. Waste Management

See Chemical Hygiene Plan for waste handling and disposal SOP/1728

Nitrate, Nitrite and Nitrate/Nitrite Nitrogen

Reference: **Methods 353.2:** Methods for the Determination of Inorganic Substances in Environmental Samples, EPA 600/ R-93/ 100. August, 1993.

Methods 4500NO_3-F, 4500NO_2-B: Standard Methods for the Examination of Water and Wastewater. APHA-AWWA-WEF. Standard Methods Online.

Method 10-107-04-1, Lachat Instruments, 6645 West Mill Road, Milwaukee, WI 53218, 1992

1. Scope and Application

Matrices: This method is limited to optically clear water samples with a total concentration of nitrite and nitrate below 8mg N/L.

Definitions: Refer to Alpha Analytical Quality Manual.

In waters and wastewaters, the forms of nitrogen of greatest interest are, in order of decreasing oxidation state, nitrate, nitrite, ammonia, and organic nitrogen. All these forms of nitrogen, as well as nitrogen gas (N_2), are biochemically interconvertible and are components of the nitrogen cycle. They are of interest for many reasons.

Organic nitrogen is defined functionally as organically bound nitrogen in the trinegative oxidation state. It does not include all organic nitrogen compounds. Analytically, organic nitrogen and ammonia can be determined together and have been referred to as “kjeldahl nitrogen,” a term that reflects the technique used in their determination. Organic nitrogen includes such natural materials as proteins and peptides, nucleic acids and urea. Numerous concentrations vary from a few hundred micrograms per liter in some lakes to more than 20mg/L in raw sewage.

Total oxidized nitrogen is the sum of nitrate and nitrite nitrogen. Nitrate generally occurs in trace quantities in surface water but many attain high levels in some groundwater. In excessive amounts, it contributes to the illness known as methemoglobinemia in infants. A limit of 10mg nitrate as nitrogen/L has been imposed on drinking water to prevent this disorder. Nitrate is found only in small amounts in fresh domestic wastewater but in the effluent of nitrifying biological treatment plants, nitrate may be found in concentrations of up to 30mg nitrate as nitrogen/L. It is an essential nutrient for many photosynthetic autotrophs and has been identified as a growth-limiting nutrient.

Nitrite is an intermediate oxidation state of nitrogen, both in the oxidation of ammonia to nitrate and in the reduction of nitrate. Such oxidation and reduction may occur in wastewater treatment plants, water distribution systems, and natural waters. Nitrite can enter a water supply system through its use as a corrosion inhibitor in industrial process water. Nitrite is the actual etiologic agent of methemoglobinemia. Nitrous acid, which is formed from nitrite in acidic solution, can react with secondary amines (RR’NH) to form nitrosamines (RR’N-NO), many of which are known to be carcinogens. The toxicologic significance of nitrosation reactions in vivo and in the natural environment is the subject of much current concern and research.

Within this SOP, organic nitrogen is referred to as organic N, nitrate nitrogen as NO_3^--N, and nitrite nitrogen as NO_2^--N.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the Lachat Analyzer and in the interpretation of Lachat data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

Nitrate is quantitatively reduced to nitrite by passage of the sample through a copperized cadmium column. The nitrite (reduced nitrate plus original nitrite) is then determined by diazotizing with sulfanilamide followed by coupling with N-(1-naphthyl)ethylenediamine dihydrochloride. The resulting water-soluble dye has a magenta color, which is read at 520nm. Nitrite alone can be determined by removing the cadmium column. The nitrate is calculated as the difference between the reduced and non-reduced sample

2.1 Method Modifications from Reference

Soils can be analyzed using 1:10 ratio soil to water extraction, following filtration.

3. Reporting Limits

This method has an analytical range of 0.1 to 8.0mg N/L in the form of nitrate, and 0.05 to 8.0mg N/L in the form of nitrite.

The Reporting Limit is 0.1mg/L for Nitrate and 0.05 mg/L for Nitrite. Reporting limit is 1.0 mg/kg for soils

4. Interferences

4.1 Suspended matter in the column will restrict sample flow.

4.2 For turbid samples, filter through 0.45µm membrane filter prior to analysis.

4.3 Low results would be obtained for samples that contain high concentrations of iron, copper or other metals. In this method, EDTA is added to the buffer to reduce this interference.

4.4 Samples that contain large concentrations of oil and grease will coat the surface of the cadmium. In this case, only the water phase of the sample is used for analysis and a narrative is submitted with the data. Dilutions are performed as necessary.

4.5 Residual chlorine can interfere by oxidizing the Cd column, reducing its efficiency. Prior to analysis, check wastewater and drinking water samples for residual chlorine and record results in the Laboratory Notebook. If residual chlorine is present, and the samples are preserved with H_2SO_4, the sample may be analyzed for NO_3/NO_2 determination. However, NO_2 must be performed by a manual method. If it is not possible to analyze NO_2 by a manual method, the result is reported as NA and a narrative is submitted.

4.6 Sample color interferes if it is absorbed at about 540nm.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples are collected in glass or plastic bottles; 250mL minimum volume. Soils can be collected in plastic or glass containers.

6.2 Sample Preservation

Refrigerate samples at 4 ± 2 °C.

For Nitrate/Nitrite analysis, the samples are preserved with 1:1 H_2SO_4.

6.3 Sample Shipping

No specific requirement.

6.4 Sample Handling

Begin NO_3^- and/or NO_2^- determinations promptly after sampling. If storage is necessary, store for up to 48 hours at 4 ± 2 °C.

NOTE: If the 48-hour hold time cannot be met, the sample is to be handled as follows, only in an emergency situation. These instructions are not to be used on a regular basis.

Prior to the expiration of the 48-hour hold time, the following three steps are executed:

1. A manually colored Nitrite test is performed by Method 354.2. Results are recorded in the Laboratory Notebook.
2. A 50mL aliquot of the sample is preserved to a pH of <2 with concentrated H_2SO_4. Preservation is recorded in the Laboratory Notebook.

Prior to analysis, within 14 days of preservation, the preserved sample is neutralized using 6N NaOH. The sample is analyzed using only the Lachat Instrument.

CAUTION! Samples must NOT be preserved with mercuric chloride or thiosulfate because this will degrade the cadmium column

7. Equipment and Supplies

7.1 Lachat 8000 Automated Ion Analyzer or Lachat QuickChem 8500 Automated Ion Analyzer

7.2 Nitrate+Nitrite Lachat Board

7.3 Nitrite Lachat Board

7.4 Pre-packed Cadmium Columns: Available from Lachat.

7.5 Ottawa sand.

7.6 Disposable Culture Tubes 13x100 ml

7.7 Disposable pipettes.

8. Reagents and Standards

8.1 Stock Nitrate Standard, 1000mg N/L as NO_3^-: Purchased commercially prepared with certificate of analysis. Expires upon manufacturer's expiration date. There must be different manufacturers for calibration stock and ICV/LCS stock.

8.1.1 Stock Nitrate Standard, 200.0mg N/L as NO_3^-: Pipet 50mL of 1000ppm standard (Section 8.1) into 250mL volumetric flask and bring to volume with DI.

Alternately, in a 1L volumetric flask, dissolve 1.444g potassium nitrate (KNO_3) in about 600mL DI. Add 2mL chloroform. Dilute to the mark with DI and invert to mix. Refrigerate at 4±2°C. This solution is stable for six months.

8.2 Stock Nitrite Standard, 1000mg N/L as NO_2^-: Purchased commercially prepared with certificate of analysis. Expires upon manufacturer's expiration date. There must be different manufacturers for calibration stock and ICV/LCS stock.

8.2.1 Stock Nitrite Standard, 200.0mg N/L as NO_2^-: Pipet 50mL of 1000ppm standard (Section 8.2) into 250mL volumetric flask and bring to volume with DI.

Alternately, in a 1L volumetric flask, dissolve 0.986g sodium nitrite ($NaNO_2$) or 1.214g potassium nitrite (KNO_2) in approximately 800mL DI. Add 2mL chloroform. Dilute to the mark with DI and invert to mix. Refrigerate at 4±2°C. This solution is stable for six months.

8.3 Intermediate Nitrate Working Standard, 20 mg N/L as Nitrate: To a 250mL volumetric flask, add 25.0mL of the 200mg N/L NO_3^- stock standard. Dilute to the mark with DI and invert to mix. These solutions are stable for two weeks. Refrigerate at 4±2°C.

8.4 Intermediate Nitrite Working Standard, 20 mg N/L as Nitrite: To a 250mL volumetric flask, add 25.0mL of the 200mg N/L NO_2^- stock standard. Dilute to the mark with DI and invert to mix. These solutions are stable for two weeks. Refrigerate at 4±2°C.

8.5 Set of Six Calibration NO_3^- Standards, 8.0, 4.0, 1.00, 0.40, 0.20 and 0.1mg N/L as Nitrate: These standards are stable for 2 weeks. Refrigerate at 4±2°C.

To four 200mL volumetric flasks, add respectively: 8.0, 4.0, 1.0 and 0.4mL of the 200mg N/L NO_3^- stock standard. Bring to volume with DI water.

To two 200mL volumetric flasks, add respectively: 2.0 and 1.0mL of the 20mg N/L NO_3^- intermediate standard. Bring to volume with DI water.

Alternatively, an autodiluter can be used to make the standards during calibration, in which case only 8.0ppm and 1.0 ppm need to be manually prepared. If an autodiluter is used then it must be checked in an analytical tray by autodiluting 8.0mg N/L as Nitrite. The recovery for NO2 must be within 10% of the true value.

8.6 Set of Six Calibration NO_2^- Standards, 8.0, 4.0, 1.00, 0.40, 0.10 and 0.05mg N/L as Nitrite: These standards are stable for 2 weeks. Refrigerate at 4±2°C.

To three 200mL volumetric flasks, add respectively: 8.0, 4.0 and 1.0 of the 200mg N/L NO_2^- stock standard. Bring to volume with DI water.

To three 200mL volumetric flasks, add respectively:4.0, 1.0mL and 0.5mL of the 20mg N/L NO_2^- intermediate standard. Bring to volume with DI water.

Alternatively, an autodiluter can be used to make the standards during calibration, in which case only 8.0ppm and 1.0 ppm need to be manually prepared.

8.7 Ammonium Chloride Buffer, pH 8.5: In a 2L volumetric flask, dissolve 170g ammonium chloride (NH_4Cl) and 2.0g disodium ethylenediamine tetraacetic acid dihydrate ($Na_2EDTA•2H_2O$) in about 800mL water. Dilute to the mark with DI water and invert to mix. Adjust the pH to 8.5 with concentrated ammonium hydroxide. This solution is prepared monthly and stored at room temperature.

8.8 Sulfanilamide Color Reagent: To a 2L volumetric flask add about 1200mL water. Then add 200mL of 85% phosphoric acid (H_3PO_4), 80.0g sulfanilamide, and 2.0g N^-(1-naphthyl)ethylenediamine dihydrochloride (NED). Shake to wet, and stir to dissolve for 30 minutes. Dilute to the mark with DI water and invert to mix. Store in a dark bottle. This solution is stable for one month. Store at room temperature.

8.9 200ppm Nitrate Stock Standard, (for ICV/LCS): Pipet 50mL of 1000ppm standard (Section 8.1) into 250mL volumetric flask and bring to volume with DI. Store refrigerated at 4±2°C. Expires six months from preparation or upon manufacturer's expiration date.

8.10 200ppm Nitrite Stock Standard: Pipet 50mL of 1000ppm standard (Section 8.2) into 250mL volumetric flask and bring to volume with DI. Store refrigerated at 4±2°C. Expires six months from preparation or upon manufacturer's expiration date.

8.11 Initial Calibration Verification Standard (ICV)/Laboratory Control Sample (LCS): Store refrigerated at 4±2°C. Expiration is 2 weeks from date of preparation.

8.11.1 Nitrate LCS, 5.0ppm: Pipet 5.0mL of 200ppm stock (Section 8.9) into a 200mL volumetric flask and bring to volume with DI.

8.11.2 Nitrate ICV, 0.5ppm: Pipet 10.0mL of 5.0ppm standard (Section 8.11.1) into a 100mL volumetric flask and bring to volume with DI.

8.11.3 Nitrite LCS, 5.0ppm: Pipet 5.0mL of 200ppm stock (Section 8.9) into a 200mL volumetric flask and bring to volume with DI.

8.11.4 Nitrite ICV, 0.5ppm: Pipet 10.0mL of 5.0ppm standard (Section 8.11.3) into a 100mL volumetric flask and bring to volume with DI.

8.12 DPD Free Chlorine Reagent Powder Pillows: HACH brand, for 25mL sample. Store at room temperature. Expires upon manufacturer's expiration date.

8.13 1N Hydrochloric acid (HCL): To a 1L volumetric flask add about 600mL DI. Then add 83mL of concentrated hydrochloric acid (HCL) Stir to dissolve. Dilute to the mark with DI water and invert to mix. This solution is stable for six month. Store at room temperature.

8.14 1N Sodium Hydroxide (NaOH): To a 1L volumetric flask add about 600mL DI. Then add 40 g of sodium Hydroxide. Stir to dissolve. Dilute to the mark with DI water and invert to mix. This solution is stable for six month. Store at room temperature

8.15 6N Sulfuric Acid (H2SO4): To a 1L volumetric flask add about 600mL DI. Then add 140 ml of concentrated Sulfuric Acid (H2SO4). Stir to dissolve. Dilute to the mark with DI water and invert to mix. This solution is stable for six month. Store at room temperature

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank

One Method Blank is analyzed per batch of 20 samples or less The Method Blank consists of DI.

For soils: 5g of Ottawa sand extracted with 50 ml of DI. Results must be < 0.1mg/L. If this criterion is not met, the blank is re-analyzed. If there is still failure, the problem must be found and corrected prior to any sample analysis

9.2 Laboratory Control Sample (LCS)

The 5ppm ICV is reported as the LCS for the batch. See Section 9.3.

For soil LCS: 5g of Ottawa sand extracted with 0.25 ml of 1000 mg/l nitrate (8.1) (or 1000 mg/l Nitrite standard (8.2)) and 50 ml DI. The nitrate standard is used for spikes for Nitrate-N as well as Nitrate/Nitrite-N. LCS recoveries must be recovered within ± 10% of the true value. If these criteria are not met, LCS's must be re-analyzed. If failure continues, the batch has to be re-extracted and re-analyzed.

9.3 Initial Calibration Verification (ICV)

Two ICVs are analyzed at the beginning of the analytical sequence. One is at a concentration of 0.5ppm, and the other is at a concentration of 5.0ppm.

Both must be recovered within ± 10% of the true value. If these criteria are not met, the ICVs must be re-analyzed. If failure continues, the ICVs are to be re-made and/or a new calibration curve is to be generated.

The 5ppm ICV is reported as the LCS for the batch.

9.4 Continuing Calibration Verification (CCV)

At the beginning of the first tray, after every ten samples and at the end of every analytical sequence, a CCV and a CCB pair must be analyzed to verify both calibration curves.

1.0ppm Nitrate CCV (Section 8.5)

1.0ppm Nitrite ICV (Section 8.6)

Calibration Blank (DI)

The results of the CCVs must be within ±10% of the true value, otherwise re-calibration is required.

The results of the CCBs must be less than our standard limit of detection, otherwise the analysis is stopped and the problem corrected.

9.5 Matrix Spike

One Matrix Spike is analyzed per batch of 20 samples or less. Separate spikes are performed for Nitrate and Nitrite. In a 25mL volumetric flask, 0.5mL of 200ppm stock calibration standard (Section 8.1 or 8.2) is added to the sample. The final concentration of the matrix spike is

4.0ppm. The nitrate standard is used for spikes for Nitrate-N as well as Nitrate/Nitrite-N. The nitrite standard is used for spikes for Nitrite-N.

For soils: weigh 5.0 g of sample, add 2.0 ml of 200 mg/l Nitrate or Nitrite standard and 48 ml of DI. The final concentration of the matrix spike is 80.0 mg/kg. The nitrate standard is used for spikes for Nitrate-N as well as Nitrate/Nitrite-N. The nitrite standard is used for spikes for Nitrite-N.

% Recovery for the Matrix Spike must be within in-house control limits. If acceptance criteria are not met, the Matrix Spike is reanalyzed. If failure continues, a narrative is included with the data for inclusion on the Client report.

Note: For samples, analyzed by method **353.2** (NO2-353 and NO3-353) maximum batch size is 10 samples; every 10 samples required separate matrix spike (MS) to be analyzed. % Recovery for the Matrix Spike must be within +/- 10% of true value. If acceptance criteria are not met, the Matrix Spike is reanalyzed. If failure continues, a narrative is included with the data for inclusion on the Client report.

9.6 Laboratory Duplicate

One Duplicate sample is analyzed per batch of 20 samples or less. A separate aliquot of the sample is analyzed for this purpose.

% RPD for the Duplicate must be within in-house control limits. If acceptance criteria are not met, the Duplicate is reanalyzed. If failure continues, a narrative is included with the data for inclusion on the Client report

9.7 Method-specific Quality Control Samples

None.

9.8 Method Sequence

- Calibration
- ICV/LCS – both levels
- Sample analysis
- CCV – every ten samples and at the end of the analytical sequence

10. Procedure

10.1 Equipment Set-up

10.1.1 Preparation

10.1.1.1 Place the Nitrate+Nitrite board (containing the cadmium column) in Channel 1. Place the Nitrite board in Channel 2. Make sure the valve to the cadmium column is closed prior to starting to pump the reagents.

10.1.1.2 Commence pumping of reagents.

10.1.1.3 Once the lines are full of reagent and free of gas bubbles, open the valve to allow reagent to flow through the cadmium column.

NOTE: Be sure to switch the valve back before rinsing the manifold with DI water at the completion of the run.

NOTE: DO NOT LET AIR ENTER THE CADMIUM COLUMN.

10.1.2 Column Efficiency Procedure

10.1.2.1 Visually inspect the column. Check for air bubbles in the column or lines, gaps in the column or any change in the cadmium surface characteristics, (cadmium granules should be dark gray). If air bubbles are present in column, connect the column into the manifold, turn the pump on maximum and tap firmly with a screwdriver handle, being careful not to break the column, working up the column until all air is removed. If air cannot be removed, the column should be repacked. Cadmium columns should be stored filled with buffer. If air enters the column, efficiency will decrease. Check the flow efficiency by disconnecting the cadmium column from the manifold and reconnecting to a green pump tube. Pump buffer through the packed column and collect in a graduated cylinder. The flow rate with the column connected should be greater than 4.0 mL/minute.

10.1.2.2 **Column Efficiency – Slope Ratio Method:** Calibrate with the mid-range NO_3 – N standards. Calibrate with a matching concentration range of NO_2–N standards. The column efficiency is determined by the equation:

$$E = \frac{S_{NO3\text{-}N}}{S_{NO2\text{-}N}} \times 100$$

where:

$S_{NO3\text{-}N}$ = slope of NO_3 calibration
$S_{NO2\text{-}N}$ = slope of NO_2 calibration
E = % efficiency

10.1.2.3 **Column Efficiency – Concentration Ratio Method:** Calibrate with the mid-range NO_2-N and NO_3-N standards. Run a known concentration NO_2-N standard. Run a matching concentration NO_3-N standard. The column efficiency is determined by the following equation:

$$E = \frac{C_{NO3\text{-}N}}{C_{NO2\text{-}N}} \times 100$$

where:

$C_{NO3\text{-}N}$ = concentration of NO_3 standard
$C_{NO2\text{-}N}$ = concentration of NO_2 standard
E = % efficiency

10.1.2.4 **Column Efficiency Result:** If the efficiency is <75%, the column is repacked. All results are recorded and maintained on file in the QC department.

10.1.3 Residual Chlorine Screening

Check all wastewater and drinking water samples for residual chlorine prior to analysis.

10.1.3.1 Add 1 DPD Free Chlorine powder pillow (Section 8.12) to 25mL of sample in a centrifuge tube. An immediate color change to pink indicates residual chlorine is present. If residual chlorine is present, add a small amount of ascorbic acid to a sample aliquot (record this in logbook) and check for residual chlorine presence again. If residual chlorine remains, notify the Department Manager and/or the Laboratory Director. Results will be reported as Not Applicable (N/A).

If residual chlorine is not present, continue with sample analysis.

10.2 Initial Calibration

Calibrate the Lachat ion analyzer according to manufacturer's instructions.

10.2.1 Calibration

Two boards are used to calibrate the Lachat instrument. Each curve has seven calibration points. The correlation coefficient of each curve must be $\geq$0.995, otherwise recalibration is necessary. Prepare standard curves by plotting the peak areas of standards processed through the manifold against NO_3+NO_2 as N and NO_2 as N concentrations in standards.

10.2.1.1 Channel 1 is used to generate a calibration curve for Nitrate/Nitrite ranging from 0 to 8.0ppm.

10.2.1.2 Channel 2 is used to generate a calibration curve for Nitrite ranging from 0 to 8.0ppm.

Note: Instrument is calibrated daily, fixed calibration range is used; linearity is verified daily; three standards are used for linear calibration verification (low ICV (0.5 mg/l), High ICV (5.0 mg/l) and CCV (1.0 mg/l)). All standards must be within 10% of true value

10.2.2 Initial Calibration Verification (ICV)

10.2.2.1 Prior to sample analysis, the following ICVs must be analyzed to verify both calibration curves.

10.2.2.1.1 Nitrate ICV, 0.5ppm (Section 8.12.2)

10.2.2.1.2 Nitrate ICV, 5.0ppm (Section 8.12.1)

10.2.2.1.3 Nitrite ICV, 0.5ppm (Section 8.12.4)

10.2.2.1.4 Nitrite ICV, 5.0ppm (Section 8.12.3)

10.2.2.2 The results must be within $\pm$10% of the true value, otherwise re-calibration is required.

10.3 Equipment Operation and Sample Processing

Follow the manufacturer's directions for the operation of the Lachat 8000.

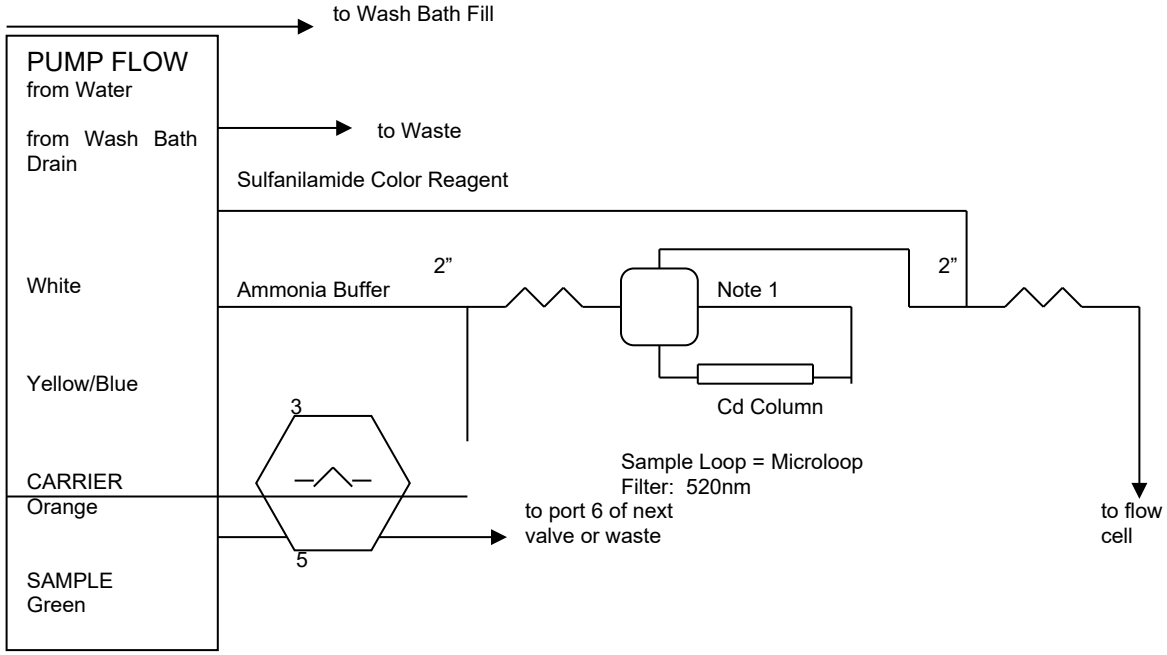
All samples have to be inspected prior to analysis. Samples that are turbid or have sediment have to be filtered prior to analysis.

Check pH of the samples. If pH is less then 5 or greater then 9, then adjust pH using 1N Hydrochloric Acid (HCl) (8.13), 6N Sulfuric Acid (H2SO4) (8.15) or 1N Sodium Hydroxide (NaOH) (8.14). Record pH adjustment in the log book.

For soils: extract soils samples prior to analysis: take 5g of sample, add 50 ml of DI, extract for 30 min, then filter thorough 0.45 nm filter. Record all weights for calculations.

Note: if samples are filtered, then Method blank also have to be filtered

The Manifold Diagram follows:



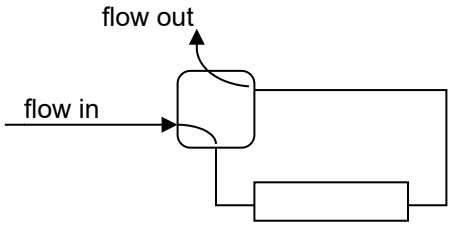
CARRIER is water.

2" is 135cm of tubing on a 2-inch coil support.
APPARATUS: Standard valve, flow cell, and detector head modules are used.

All manifold tubing is 0.8mm (0.032") i.d. This is 5.2µL/cm.

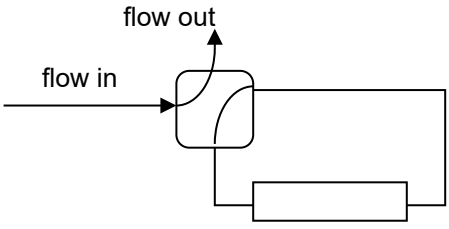
NOTE 1: This is a two-state switching valve used to place the cadmium column in line with the manifold.

State 1: Nitrate + Nitrite



Cd Column

State 2: Nitrite Only



Cd Column

10.4 Continuing Calibration

See Section 9.4

10.5 Preventive Maintenance

Tubing is changed monthly or as needed.

At the end of each analytical sequence, the valve to the column is closed. DI is rinsed through the Lachat for five minutes followed by five minutes of air.

All maintenance is documented in the Instrument Maintenance Logbook.

11. Data Evaluation, Calculations and Reporting

11.1 Nitrate/Nitrite: When the software is set up according to the manufacturer's recommendations, the concentration of nitrate plus nitrite in mg NO_3/NO_2-N/L is reported directly when the Cd column is included in the sample train in Channel 1.

11.2 Nitrite: When the software is set up according to the manufacturer's recommendations, the concentration nitrite in mg NO_2-N/L is reported directly when the Cd is not included in the sample train in Channel 2.

11.3 Nitrate: The concentration of nitrate is determined by the subtraction of the nitrite concentration, (Section 11.2 above), from the nitrate-nitrite concentration, (Section 11.1 above).

11.3.1 If the sample was preserved initially as described in Section 6.3, subtract the Nitrite value generated manually from the Nitrate/Nitrite value generated by the Lachat Instrument. This value is reported as the Nitrate result.

When the sample is preserved initially as described in Section 6.3, the value generated by the Lachat instrument for Nitrite is invalid and therefore disregarded.

11.4 If any sample exceeds a concentration of 8.0 mg/L, the sample must be diluted and re-analyzed. All sample concentrations must fall within the calibration curve.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedance and improper preservation are noted on the nonconformance report form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

Review of standards, blanks and standard response for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form.

If the CV or LCS recovery of any parameter falls outside the designated acceptance range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked samples is suspect and is only reported for regulatory compliance purposes with the appropriate nonconformance action form. Immediate corrective action includes reanalyzing all affected samples by using any retained sample before the expiration of the holding time.

13. Method Performance

13.1 Detection Limit Study (DL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the DL, LOD, and/or LOQ as outlined in Alpha SOP ID 1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP ID 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Hazardous Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

2121 Chemical Hygiene Plan

1732 Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ) SOP

1739 Demonstration of Capability (DOC) Generation SOP

1728 Hazardous Waste Management and Disposal SOP

16. Attachments

None.

Orthophosphate - Colorimetric, Combined Reagent

Reference: SM 4500P-E, Standard Methods for the Examination of Water and Wastewater. APHA-AWWA-WEF. Standard Methods Online.

1. Scope and Application

Matrices: Water and wastewater samples.

Definitions: Refer to Alpha Analytical Quality Manual.

Phosphorus occurs in natural waters and in wastewaters almost solely as phosphates. These are classified as orthophosphates, condensed phosphates (pyro-, meta-, and other polyphosphates), and organically bound phosphates. They occur in solution, in particles or detritus, or in the bodies of aquatic organisms.

These forms of phosphate arise from a variety of sources. Small amounts of certain condensed phosphates are added to some water supplies during treatment. Larger quantities of the same compounds may be added when the water is used for laundering or other cleaning, because these materials are major constituents of many commercial cleaning preparations. Phosphates are used extensively in the treatment of boiler waters. Orthophosphates applied to agricultural or residential cultivated land as fertilizers are carried into surface waters with storm run-off and to a lesser extent with melting snow. Organic phosphates are formed primarily by biological processes. They are contributed to sewage by body wastes and food residues, and also may be formed from orthophosphates in biological treatment processes or by receiving water biota.

Phosphorus is essential to the growth of organisms and can be the nutrient that limits the primary productivity of a body of water. In instances where phosphate is a growth-limiting nutrient, the discharge of raw or treated wastewater, agricultural drainage, or certain industrial wastes to that water may stimulate the growth of photosynthetic aquatic micro- and macro-organisms in nuisance quantities.

Phosphates also occur in bottom sediments and in biological sludges, both as precipitated inorganic forms and incorporated into organic compounds.

Phosphorus analyses embody two general procedural steps: (a) conversion of the phosphorus form of interest to dissolved orthophosphate, and (b) colorimetric determination of dissolved orthophosphate. The separation of phosphorus into its various forms is defined analytically but the analytical differentiations have been selected so that they may be used for interpretive purposes.

Filtration through a 0.45-µm-pore-diameter membrane filter separates dissolved from suspended forms of phosphorus. No claim is made that filtration through 0.45-µm filters is a true separation of suspended and dissolved forms of phosphorus; it is merely a convenient and replicable analytical technique designed to make a gross separation.

Phosphates that respond to colorimetric tests without preliminary hydrolysis or oxidative digestion of the sample are termed “reactive phosphorus.” While reactive phosphorus is largely a measure of orthophosphate, a small fraction of any condensed phosphate present usually is hydrolyzed unavoidably in the procedure. Reactive phosphorus occurs in both dissolved and suspended forms.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel

before performing the modification: Area Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

The ascorbic acid method is used for the determination of orthophosphate in environmental samples. Ammonium molybdate and potassium antimonyl tartrates react in acid medium with orthophosphate to form a heteropoly acid-phosphomolybdic acid that is reduced to intensely colored molybdenum blue by ascorbic acid. Samples are analyzed at 880nm using a spectrophotometer.

2.1 Method Modifications from Reference

None.

3. Reporting Limits

The Reporting Limit is 0.005mg/L.

4. Interferences

Correction for Turbidity or Interfering Color: The natural color of water generally does not interfere at the high wavelength used. For highly colored or turbid waters, prepare a blank by adding all reagents except ascorbic acid and potassium antimonyl tartrate to the sample. Subtract the blank absorbance from the absorbance of each sample.

In some cases, the background color or turbidity can be eliminated by a dilution, but this will raise the reporting limit.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples are collected in 250mL plastic containers.

6.2 Sample Preservation

Samples are not preserved. Do not add either acid or $CHCl_3$ as a preservative.

6.3 Sample Shipping

No specific requirement.

6.4 Sample Handling

Samples are stored under refrigeration at 4 ±2 °C. Analysis must be performed within 48 hours of collection.

7. Equipment and Supplies

7.1 Spectrophotometer, with infrared phototube for use at 880nm, providing a light path of 2.5cm.

7.2 Acid-washed Glassware: Use acid-washed glassware for determining low concentration of phosphorus. Phosphate contamination is common because of its absorption on glass surfaces. Avoid using commercial detergents containing phosphate. Clean all glassware with 1:1 HCl and rinse well with distilled water. The glassware should only be used for phosphate determination.

7.3 Centrifuge Tubes: 50mL volume. (Must be new and disposable.)

7.4 0.45µm Acrodisc filters with disposable syringes

7.5 Pipets or Pipettor: Various sizes, new and disposable or acid-rinsed glass

8. Reagents and Standards

8.1 Stock Phosphate Standard: 1000 mgP/L This stock solution is certified and purchased commercially prepared. Store at room temperature. Expires upon manufacturer's specified date.

8.2 Intermediate Phosphate Standard: 50 mgP/L Dilute 50.0mL stock phosphate solution to 1000mL with DI water, 1.00mL = 2.50µg P. Store refrigerated at 4 ±2 °C. Expires 6 months from date of preparation.

8.3 Working Standard: 1.0 mgP/L Add 2mL of 50 mgP/L intermediate standard (Section 8.2) to 100mL volumetric flask and dilute to volume with DI water. Prepare fresh on each day of use.

8.4 Calibration Standards: Follow table below for preparation instructions. Prepare fresh on each day of use. Use a calibrated pipettor and bring up to 25mL with DI.

Volume of 1.0 mg/L Working Standard (Section 8.3)	Final Volume (mL)	Calibration Standard Final Concentration (mgP/L)
0	25	0.0
0.125 mL	25	0.005
0.250 mL	25	0.01
1.250 mL	25	0.05
2.50 mL	25	0.10
12.5 mL	25	0.50
25 mL	25	1.00

8.5 ICV-LCS-CCV Stock Standard: 1000 mgP/L Second source standard than that used in Section 8.1.

8.6 ICV-LCS-CCV Intermediate Standard: 25 mgP/L Add 5mL of 1000 mgP/L stock standard (Section 8.5) to 200mL volumetric flask and dilute to volume with DI water. Store refrigerated at 4 ±2 °C. Expires 6 months from date of preparation.

8.7 ICV-LCS-CCV Working Standards: Prepare fresh on each day of use.

8.7.1 0.5 mgP/L: Add 0.25mL of 50 mg/L intermediate standard (Section 8.6) to 25mL volumetric flask and dilute to volume with DI water.

8.8 Matrix Spike Solution: Intermediate Phosphate Standard (Section 8.2) is utilized for matrix spike solution. 0.25mL of the 50 mgP/L standard added to 25mL of sample will afford a 0.5mgP/l matrix spike concentration.

8.9 Sulfuric Acid, H2SO4, 5N: Dilute 70mL concentrated sulfuric acid to 500mL with DI. Store at room temperature. Expires 6 months from date of preparation. Alternatively, 1L of 5N H2SO4 can be made: dilute140 ml of concentrated sulfuric acid to 1000mL with DI. Expires 6 months from date of preparation.

8.10 Potassium Antimonyl Tartrate Solution: Dissolve 1.3715g K(SbO) $C_4H_4O_6 \cdot ½H_2O$ in 400mL DI water in a 500mL volumetric flask and dilute to volume. Store in a glass-stoppered bottle at room temperature. Expires one month from date of preparation.

8.11 Ammonium Molybdate Solution: Dissolve 20g $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$ in 500mL DI water. Store in a glass-stoppered bottle at room temperature. Expires one month from date of preparation.

8.12 Ascorbic Acid, 0.1M: Dissolve 3.52g ascorbic acid in 200mL DI water. The solution is stable for about 1 week at 4 ±2ºC. Alternatively, 100 ml of Ascorbic Acid can be made: Dissolve 1.76g ascorbic acid in 100mL DI water. The solution is stable for 1 week at 4 ±2ºC.

8.13 Combined Reagent: Mix the above reagents in the following proportions for 100mL of the combined reagent: 50mL 5N H_2SO_4 (Section 8.9), 5mL potassium antimonyl tartrate solution (Section 8.10), 15mL ammonium molybdate solution (Section 8.11), and 30mL ascorbic acid solution (section 8.12). Mix after addition of each reagent. Let all reagents reach room temperature before they are mixed and mix in the order given. If turbidity forms in the combined reagent, shake and let stand for a few minutes until turbidity disappears before proceeding. The reagent is stable for 4 hours. Discard reagent if it turns blue or black in color.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blanks

9.1.1 **ICB or Method Blank** - One method blank, which consists of 25mL DI water filtered through a 0.45 micron Acrodisc filter, must be analyzed per batch of 20 samples or less. The method blank result must be less than the Reporting Limit.

9.1.2 **Continuing Calibration Blank** – The ICB should be re-read after every 10 samples (thus becoming the CCB) and at the end of the batch The CCB result must be less than the Reporting Limit. If the CCB is greater than the RL, all samples analyzed since the last passing CCB must be recolored and reanalyzed.

9.2 Laboratory Control Sample (LCS)

Analyze one LCS per batch of 20 samples or less. The calibration curve must be verified by a second source standard prior to performing any sample analysis. The LCS must be recovered at 90 – 110% of the true value.

If the LCS fails, re-analyze. If failure remains, stop analysis, correct problem and perform re-calibration.

9.3 Initial Calibration Verification (ICV)

The calibration curve must be verified by a second source standard prior to performing any sample analysis. The ICV must be recovered at 90 – 110% of the true value.

If the ICV fails, re-analyze. If failure remains, stop analysis, correct problem and perform re-calibration.

9.4 Continuing Calibration Verification (CCV)

The calibration curve must be verified by a second source standard prior to performing any sample analysis. The CCV must be recovered at 90 – 110% of the true value.

If the CCV fails, re-analyze. If failure remains, stop analysis, correct problem and perform re-calibration.

The CCV must be re-read every 10 samples and at the end of the batch

9.5 Matrix Spike

Analyze one per batch of 20 samples or less. Concentration is 0.5 mg P /L. The matrix spike must be recovered at 80 – 120% of the true value. If the MS recovery is outside of acceptance limits, the sample and its spike are reanalyzed. If the MS failure continues, a narrative is submitted with the data for inclusion on the Client report.

9.6 Laboratory Duplicate

Analyze one sample in duplicate, per batch of 20 samples or less. The RPD must be $\leq$ 20%. If the RPD is outside of acceptance limits, the sample and its duplicate are reanalyzed. If the RPD failure continues, a narrative is submitted with the data for inclusion on the Client report.

9.7 Method-specific Quality Control Samples

None.

9.8 Method Sequence

- Calibration curve generation or verification of existing curve.
- Acid-rinsing of glassware
- Sample filtration including blank
- Add sample aliquot to a new centrifuge tube
- Add combined reagent to samples
- Read sample absorbance after 10-30 minutes
- Analyze CCV and CCB after every 10 samples to verify curve
- End sequence with CCV and CCB
- Calculate results

10. Procedure

10.1 Equipment Set-up

10.1.1 Preparation of calibration curve: Prepare individual calibration curve from a series of six standards and DI (0.005 mgP/L to 1.0 mgP/L) on a daily basis when samples are to be analyzed. Use a DI water blank to zero the instrument before taking photometric readings for the calibration curve. Plot a curve of absorbance vs. phosphate concentration. The curve is acceptable if the calibration coefficient is $\geq$ 0.995. The calibration curve is prepared fresh each day of analysis.

All calibration points are back calculated and should be within 10% from true concentration, except 2 lowest points of calibration curve. %recoveries for low range will be wider, but shouldn't exceed 100% and correlation coefficient will not be worse than 0.995.

10.2 Initial Calibration

See Section 9.3

10.3 Equipment Operation and Sample Processing

10.3.1 Filter samples and QC samples through 0.45µm Acrodisc filters. 1 µm Acrodisc filter may be used to prefilter hard-to-filter samples.

10.3.2 Pour 25mL of each clear filtered sample, a duplicate and matrix spike sample into a corresponding new (never used) centrifuge tube.

10.3.3 Add 4.0mL combined reagent to all 25mL samples and QC sample aliquots and mix thoroughly.

10.3.4 After at least 10 minutes but no more than 30 minutes, measure absorbance of each sample at 880nm with a 2.5cm cell. Follow the procedure described in the Wet Chemistry Electronic Notebook Work Instructions (WI/2516).

10.3.5 Compare with a standard curve prepared from a range of standards and carried through this procedure. Sample concentration must fall within the range of the calibration curve.

10.3.6 If the sample concentration is greater than the highest calibration standard concentration, dilute the original sample with DI water and recolor and reanalyze as outlined above (Section 10.3.1 – 10.3.6).

10.4 Continuing Calibration

See Section 9.4.

10.5 Preventive Maintenance

The Spectrophotometers are calibrated on a semi-annual basis by an instrument service company. Certificates are kept on file.

11. Data Evaluation, Calculations and Reporting

mg P/L = mg P (from calibration curve) x dilution

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedances or improper preservation are noted on the nonconformance report form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

Review of standards, blanks and standard response for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form.

If the CV or LCS recovery of any parameter falls outside the designated acceptance range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked samples is suspect and is only reported for regulatory compliance purposes with the appropriate nonconformance action form. Immediate corrective action includes reanalyzing all affected samples by using any retained sample before the expiration of the holding time.

13. Method Performance

13.1 Detection Limit Study (DL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the DL, LOD, and/or LOQ as outlined in Alpha SOP ID 1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP ID 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Hazardous Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

2121 Chemical Hygiene Plan

1732 Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ) SOP

1739 Demonstration of Capability (DOC) Generation SOP

1728 Hazardous Waste Management and Disposal SOP

2516 Electronic Laboratory Notebook Work Instructions

16. Attachments

None.

Organochlorine Pesticides
By Capillary Column Gas Chromatography

Reference Method No.: **Method 8081B**

References: SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update IV, 2007.

SW-846 Pesticides by 8081, State of Connecticut Department of Environmental Protection Reasonable Confidence Protocol (RCP), Version 1.0, July 2005

1. Scope and Application

Method 8081B is used to determine the concentrations of various organochlorine pesticides in extracts from solid and liquid matrices. This SOP details the analysis for these compounds using fused-silica, open-tubular, capillary columns with electron capture detectors (ECD).

Matrices: Extracts from solid and liquid matrices.

Definitions: See Alpha Analytical Quality Manual

Regulatory Parameter List: The compounds listed below are determined by this method:

Parameter	CAS
Aldrin	309-00-2
Alpha-BHC	319-84-6
Beta-BHC	319-85-7
Gamma-BHC (Lindane)	58-89-9
Delta-BHC	319-86-8
Alpha-chlordane	5103-71-9
Gamma-chlordane	5103-74-2
4,4'-DDD	72-54-8
4,4'-DDE	72-55-9
4,4'-DDT	50-29-3
Dieldrin	60-57-1
Endosulfan I	959-98-8
Endosulfan II	33213-65-9
Endosulfan Sulfate	1031-07-8
Endrin	72-20-8
Endrin Aldehyde	7421-93-4
Endrin Ketone	53494-70-5
Heptachlor	76-44-8
Heptachlor Epoxide	1024-57-3
Methoxychlor	72-43-5
Toxaphene	8001-35-2
Chlordane	12789-03-6
Alachlor	15972-60-8

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Laboratory Services Manager, Laboratory Director, or Quality Assurance Officer

This method is restricted to use by or under the supervision of analysts experienced in the operation of the gas chromatograph (GC) and in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability (see section 13), analyzing a proficiency test sample and completing the record of training.

After initial demonstration, ongoing demonstration is based on acceptable laboratory performance of at least a quarterly laboratory control sample or acceptable performance from an annual proficiency test sample. A major modification to this procedure requires demonstration of performance. The identification of major method modification requiring performance demonstration is directed by the QA Officer and/or Laboratory Director on a case-by-case basis.

2. Summary of Method

A measured volume or weight of sample (for liquids, 500 mls or 1L, for solids, 15g to 30g) is extracted using the appropriate matrix-specific sample extraction technique.

Liquid samples are extracted at neutral pH with methylene chloride using Method 3510C (separatory funnel), or other appropriate technique.

Solid samples are extracted with methylene chloride: acetone (1:1) using Method 3540C (Soxhlet), Method 3546 (microwave extraction), or other appropriate technique.

Wipe samples are extracted with methylene chloride: acetone (1:1) using Method 3540C (Soxhlet) or other appropriate technique.

Oil samples are diluted with hexane following the procedure outlined in the extraction SOP.

After cleanup, the extract is analyzed by injecting a 1µL sample into a gas chromatograph equipped with narrow- or wide-bore fused silica capillary columns and electron capture (GC/ECD) detectors.

2.1 Method Modifications from Reference

None.

3. Reporting Limits

Table 1 lists our routine reporting limits.

4. Interferences

4.1 Only high purity gases are used in the GC system to eliminate this source of possible contamination. Both the hydrogen (carrier gas – 99.999%) and argon-methane (detector make-up gas) are certified by the gas supplier.

4.2 Preventive instrument maintenance is performed routinely, and whenever highly contaminated extracts are analyzed that could result in chromatographic interferences or result in degradation of system performance. Section 9.5 details the maintenance steps.

4.3 Glassware must be scrupulously cleaned. This procedure is detailed in the extraction SOPs. Store dry glassware in a clean environment.

4.4 All solvents used are pesticide grade or equivalent, and reagents are purchased as certified contaminant free. All of these materials are routinely determined to be free of interferences by analysis of extraction blanks with every extraction batch performed.

4.5 Certain compounds (i.e. phthalates) can be extracted from the sample matrix and be detected by the ECD that could possibly result in false positive results or complicate the data interpretation. The use of the cleanup procedures detailed in the extraction SOPs minimize these possible interferences. Analyst experience is also crucial in making compound determinations.

4.6 Interferences co-extracted from the samples will vary considerably from waste to waste. While general cleanup techniques are referenced or provided as part of the method, unique samples may require additional cleanup approaches to achieve desired degrees of discrimination and quantitation.

4.7 Interferences by phthalate esters introduced during sample preparation can pose a major problem in pesticide determinations.

4.7.1 Common flexible plastics contain varying amounts of phthalate esters which are easily extracted or leached from such materials during laboratory operations.

4.7.2 Cross-contamination of clean glassware routinely occurs when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled.

4.7.3 Interferences from phthalate esters are minimized by avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination.

4.8 The presence of elemental sulfur will result in broad peaks that interfere with the detection of early-eluting organochlorine pesticides. Sulfur contamination is often seen in sediment and some soil samples.

4.9 Other halogenated pesticides or industrial chemicals may interfere with the analysis of pesticides. Coeluting chlorophenols are eliminated by using Method 3620B (florisil).

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. The following analytes covered by this method have been tentatively classified as known or suspected human or mammalian carcinogens: 4,4'-DDT, 4,4'-DDD, and BHCs. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

5.1 Lab coats, safety glasses, and gloves must be worn when handling samples, extracts, standards or solvents.

5.2 All solvent and extract transfers must be handled in the vented bench area in the GC laboratory.

5.3 All stock standards, working standards, and vialed sample extracts must be placed into the waste bucket in the lab, for future disposal by the Hazardous Waste Manager. The container must be labeled properly with hazard warning labels indicating the container contents.

5.4 Bottles containing flammable solvents must be stored in the flammables cabinet.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Aqueous samples are collected in either two 500 ml amber glass jars or two 1L amber glass jars with teflon-lined lids. Solid samples are collected in one 250 mL wide-mouth glass jar with a teflon-lined lid. All containers are purchased pre-cleaned and certified from commercial vendors.

6.2 Sample Preservation

Both aqueous and solid samples are then preserved by packing in coolers with ice or ice packs, to maintain a temperature of 4 ± 2° C. Upon receipt at the laboratory, the samples are transferred into sample storage refrigerators to maintain at a temperature of 4 ± 2° C.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

Aqueous samples must be extracted within 7 days of sample collection, solid samples within 14 days of collection. Once extracted, the samples must be analyzed within 40 days of the extraction date.

7. Equipment and Supplies

7.1 Gas Chromatograph, Hewlett Packard 6890 (or equivalent): An analytical system complete with gas chromatograph configured for split-splitless injection and all required accessories including syringes, analytical columns, gases, electron capture detectors (ECD), and data system.

7.1.1 Data System: A computer system is interfaced to the GC for collection of data. Data acquisition is performed using Agilent Technologies GC ChemStation (Rev. A.10.02 [1757]) and/or Perkin Elmer Corp. Turbochrom software (6.1.0.1:F04).

7.2 GC Columns: Alpha utilizes dual-column analyses. The dual-column approach involves either a single injection that is split between two columns that are mounted in a single gas chromatograph, or dual injections of the split extract on a single GC equipped with two columns. Typical column pairs used are listed below. Other columns may be used as long as method performance criteria can be met.

7.2.1 Column pair 1

30m x 0.32mm ID fused silica capillary column (RTX-clpesticide), 0.32µm film thickness.

30m x 0.32mm ID fused silica capillary column (RTX-clpesticideII) 0.25µm film thickness.

7.2.2 Column pair 2

30m x 0.32mm ID fused silica capillary column (STX-clpesticide), 0.32µm film thickness.

30m x 0.32mm ID fused silica capillary column (STX-clpesticideII) 0.25µm film thickness.

7.3 Volumetric Flasks: 10mL and 25mL, for the preparation of standards.

7.4 Microsyringes/Wiretrol syringes: 10 µL – 1000 µL

7.5 Disposable Borosilicate Pipets

7.6 Vials: 2 mL clear glass, crimp-top and screw-cap.

8. Reagents and Standards

Reagent grade or pesticide grade chemicals are used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficient high purity to permit its use without lessening the accuracy of the determination.

NOTE: Store the standard solutions (stock, composite, calibration, internal, and surrogate) at 4 $\pm$ 2°C in Teflon(R)-sealed containers in the dark. When a lot of standards is prepared, aliquots of that lot are stored in individual small vials. All stock standard solutions must be replaced after one year or sooner if routine QC tests indicate a problem. All other standard solutions must be replaced after six months or sooner if routine QC indicates a problem.

8.1 n-Hexane: Pesticide quality or equivalent.

8.2 Acetone: Pesticide quality or equivalent.

8.3 Methylene chloride: Pesticide quality or equivalent.

8.4 Organic-free Reagent Water: All references to water in this method refer to organic-free reagent water from Alpha's RO water treatment system.

8.5 Stock Standard Solutions: All stock standard solutions are purchased from commercial vendors as ampulated certified solutions. When an ampulated stock solution is

opened, it is transferred to a labeled amber screw-cap vial. The expiration date of the stock solution is either the vendor specified expiration date, or 1 year from the date the ampule was opened, whichever is sooner. Typical stock standard concentrations are listed in Table 2.

8.6 Calibration Standards: Calibration standards are prepared volumetrically by diluting the appropriate stock standard(s) with hexane. Calibration standards expire 6 months from the date of preparation, or on the earliest expiration date of any of the stock solutions used to prepare the calibration standard. Calibrations are performed at 10 concentration levels for individual pesticides and 9 concentration levels for Chlordane/Toxaphene mix; concentrations for all levels are listed in Table 2. Below are suggested procedures for preparation of individual pesticides (Sec. 8.6.1) and Chlordane/Toxaphene (Sec. 8.6.2) initial calibration standards.

8.6.1 Preparation of individual pesticides initial calibration standards;

(syringes needed: *100µL [A], 500µL [B]*)
(solvent: *hexane)*
(glassware: *1x25mL, 10x10mL, volumetric flasks*)

STOCK #1: *2000µg/L; 25mL*
50µL[A] of Accustandard M-8081-SC (Pesticides mix; 1000µg/mL)
250µL[B] of Accustandard CLP-032-R (TCMX, Deca; 200µg/mL)
500µL[B] of Accustandard P-1025 (Alachlor; 100µg/mL)
500µL[B] of Accustandard APP-9-112 (Hexachlorobenzene; 100µg/mL)

STOCK #2 (=LEVEL 9)**:** *100µg/L; 10mL.*
500µL[B] of STOCK #1
…..

LEVEL 1, 0.5 µg/L: 50µL[A] of STOCK #2, 10mL
LEVEL 2, 1.0 µg/L: 100µL[A] of STOCK #2, 10mL
LEVEL 3, 2.0 µg/L: 200µL[B] of STOCK #2, 10mL
LEVEL 4, 3.0 µg/L: 300µL[B] of STOCK #2, 10mL
LEVEL 5, 4.0 µg/L: 400µL[B] of STOCK #2, 10mL
LEVEL 6, 5.0 µg/L: 500µL[B] of STOCK #2, 10mL
LEVEL 7, 10 µg/L: 50µL[A] of STOCK #1, 10mL
LEVEL 8, 50 µg/L: 250µL[B] of STOCK #1, 10mL
LEVEL 9, 100 µg/L: 500µL[B] of STOCK #1, 10mL
LEVEL 10, 200 µg/L: 1mL[B] of STOCK #1, 10mL

8.6.2 Preparation of Chlordane/Toxaphene initial calibration standards;

(syringes needed: *100µL [A], 500µL [B]*)
(solvent: *hexane)*
(glassware: *10x10mL, volumetric flasks*)

STOCK #1: *50/100µg/mL (Chlordane/Toxaphene);10mL*
100µL[A] of Ultra Scientific EPA-1086 (Chlordane Solution; 5000µg/mL)
1mL[B] of Restek 32005 (Toxaphene Standard; 1000µg/mL)

STOCK #2 (=LEVEL 7)**:** *1000/2000µg/L; 10mL.*

200µL[A] of STOCK #1

LEVEL 1, 5/10 µg/L: 50µL[A] of STOCK #2, 10mL
LEVEL 2, 10/20 µg/L: 100µL[A] of STOCK #2, 10mL
LEVEL 3, 50/100 µg/L: 500µL[B] of STOCK #2, 10mL
LEVEL 4, 100/200 µg/L: 1mL[B] of STOCK #2, 10mL
LEVEL 5, 250/500 µg/L: 50µL[A] of STOCK #1, 10mL
LEVEL 6, 500/1000 µg/L: 100µL[A] of STOCK #1, 10mL
LEVEL 7, 1000/2000 µg/L: 200µL[A] of STOCK #1, 10mL
LEVEL 8, 2500/5000 µg/L: 500µL[B] of STOCK #1, 10mL
LEVEL 9, 5000/10000 µg/L: 1mL[B] of STOCK #1, 10mL

8.7 Internal Standard Solution: 1-Bromo-2-nitrobenzene is used as the internal standard, and is added to all single-component calibration standards and sample extracts to achieve a concentration of 0.25 µg/mL. Standard solution expires 6 months after the date of preparation.

8.8 Surrogate Standards: Tetrachloro-m-xylene and decachlorobiphenyl are used as surrogates. They are added to the calibration standards at the concentrations listed in Table 2, and are spiked into all samples and QC samples prior to extraction. The spiking solution is prepared in acetone at the concentrations listed in Table 2. Standard expires 6 months after the date of preparation.

8.9 LCS/MS Spiking Solutions: The LCS/MS spiking solutions are prepared volumetrically by diluting the appropriate stock standards in acetone. The spiking solution concentrations are listed in Table 2. Spiking solution expires 6 months after the date of preparation.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

Extraction blanks are performed with each extraction batch of 20 or less samples, according to the extraction SOPs. The extraction blank must not contain any of the reportable analytes above the reporting limit. If any reportable analytes are detected in the blank, the entire extraction batch is suspect and re-extraction of all associated samples is required.

9.2 Laboratory Control Sample (LCS)

A Laboratory Control Sample (LCS) is extracted with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the single component pesticide analytes. The concentration of the spiking solution is listed in Table 2. The recovery acceptance criteria are listed in Table 3. If any recovery criteria are not met, the extract should be reanalyzed. If the criteria are still not met, the entire batch should be re-extracted. If this is not possible, due to insufficient sample or holding time exceedence, the analyst must write up the failure on a narrative sheet for inclusion in the client report.

9.2.1 LCS Duplicate (LCSD)

A Laboratory Control Sample Duplicate is extracted with each analytical batch. The LCSD consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCSD is spiked with the single component pesticide analytes. The concentration of the spiking solution is listed in Table 2. The recovery acceptance criteria is listed in Table 3. If Recovery limits are outside acceptance criteria, the non-conformance is narrated.

9.2.2 Connecticut RCP LCS Requirements

For CT- Reasonable Confidence Protocol (RCP) 8081B samples only, the LCS recovery criteria is 40 – 140%.

9.3 Initial Calibration Verification (ICV)

Refer to Section 10.2.

9.4 Continuing Calibration Verification (CCV)

Refer to Section 10.4

9.5 Matrix Spike

A matrix spike (MS)/matrix spike duplicate (MSD) is extracted and analyzed upon Client request for each batch of 20 or less samples. The spike compounds and levels are listed in Table 2. The recovery acceptance criteria are 30-150%. If the recovery criteria are not met, but are met in the LCS, the failure may be attributed to sample matrix effects and must be noted on a narrative sheet for inclusion in the client report.

9.6 Laboratory Duplicate

A duplicate sample is extracted and analyzed upon Client request for each batch of 20 or less samples. The % RPD criteria are listed in Table 3. If the %RPD is not met, the failure may be attributed to sample matrix effects and must be noted on a narrative sheet for inclusion in the client report.

9.7 Method-specific Quality Control Samples

9.7.1 Surrogates

All extracted samples and associated QC are spiked with surrogates at the levels listed in Table 2. The laboratory must evaluate surrogate recovery data from individual samples and QC samples versus the surrogate control limits listed in Table 3. If the surrogate limits are not met, the extract should be reanalyzed to determine if the failure was due to an instrument problem. If the criteria are still not met, the affected samples should be re-extracted to confirm that the failure was due to sample matrix. If matrix effect is confirmed, this must be noted on a narrative sheet for inclusion in the client report.

9.8 Method Sequence

Initial calibration:

1. Instrument Blank (may be omitted to conserve tray space)
2. Degradation check standard
3. Std Level 1
4. Std Level 2

5. Std Level 3
6. Std Level 4
7. Std Level 5
8. Std Level 6
9. Std Level 7
10. Std Level 8
11. Std Level 9
12. Std Level 10 (not applicable to chlordane and toxaphene)
13. Initial Calibration Verification Standard (ICV)
14. Repeat std Levels 1 –9 for other mixes until curve is complete.

    [**NOTE:** If multiple calibration mixtures are analyzed, it is acceptable to analyze appropriate ICVs after all calibration standards have been injected.]

Typical Daily sequence:

1. Degradation Check Standard
2. Pesticide Continuing Calibration Standard
3. Chlordane/Toxaphene Continuing Calibration Standard
4. Extraction Blank
5. Laboratory Control Sample
6. Laboratory Control Sample Duplicate
7. Matrix Spike (Upon Client Request)
8. Duplicate/Matrix Spike Duplicate (Upon Client Request)
9. Samples (up to 20 may be analyzed)
10. Cal check
11. Repeat 1 – 9 (as needed)

10. Procedure

10.1 Equipment Set-up

10.1.1 Sample Extraction

Water samples are extracted at a neutral pH with methylene chloride using a separatory funnel (Method 3510C). See extraction SOP for details.

Solid samples are extracted with methylene chloride: acetone (1:1) using Soxhlet extraction (Method 3540C) or microwave extraction (Method 3546). See extraction SOP for details.

10.1.2 Extract Cleanup

Cleanup procedures may not be necessary for a relatively clean sample matrix, but most extracts from environmental and waste samples will require additional preparation before analysis. The specific cleanup procedure used will depend on the nature of the sample to be analyzed and the data quality objectives for the measurements. See extraction SOPs for details.

10.1.3 GC Conditions:

The dual-column / dual-detector approach involves the use of the columns listed in section 7.2. The columns are connected to an injection tee or dual injection GC, and separate electron capture detectors. Typical GC conditions for the HP 6890 instruments are listed below, but may be altered as long as method performance criteria are met.

Temperature 1:	120 °C
Time 1:	0 minute
Ramp 1:	45 °C/minute
Temperature 2	200 °C
Time 2:	0 minutes
Ramp 2:	15 ° C/minute
Temperature 3:	230 °C
Time 3:	0 minutes
Injector temperature:	250 °C
Ramp 3:	30 °C/minute
Final Temperature:	330 °C
Final Time:	2.0 minutes

Injector mode:	Pulsed Split
	2:1 split, 0.75 min pulse
Injector Flow:	6.0 mL/min split flow
Detector temperature:	375 °C
Carrier gas:	Hydrogen
Carrier flow:	17 mL/min
Carrier mode:	Constant flow
Makeup gas:	Argon/methane (P5)
Total detector flow:	55 mL/min
Injection volume:	1 µL

10.1.4 DDT and Endrin Breakdown

The breakdown of DDT and Endrin must be measured before samples are analyzed and at the beginning of each 12-hour shift. Injector maintenance must be completed if the breakdown in greater than 15% for either compound (See Section 10.5.1). Both analytical columns must pass DDT/Endrin breakdown criteria prior to sample analysis.

10.2 Initial Calibration

10.2.1 Prepare calibration standards using the procedures in Section 8.6 and the concentrations listed in Table 2. The calibration standards are aliquoted into autosampler vials and capped prior to loading onto the autosampler tray.

10.2.2 Establish the GC operating conditions by loading the appropriate GC method. Typical instrument conditions are listed in section 10.1.3. The same operating conditions are used for calibrations and sample analyses. Create the analytical sequence using the Turbochrom or Agilent Chemstation data acquisition software.

10.2.2.1 Record the calibration standard, unique lab identifier code (lot), the analytical sequence list.

10.2.3 A 1µL injection volume of each calibration standard is typically used. Other injection volumes may be employed, provided that the analyst can demonstrate adequate sensitivity for the compounds of interest. The same injection volume must be used for all standards and samples.

10.2.4 Because of the low concentration of pesticide standards injected on a GC/ECD, column adsorption may be a problem when the GC has not been used for a day or more or after system maintenance. The GC column may be primed (or deactivated) by injecting a pesticide standard mixture approximately 20 times more concentrated than the mid-concentration standard. Inject this standard mixture prior to beginning the initial calibration or calibration verification.

Alternately, the system may be primed by baking at the final analytical temperature for approximately 30 minutes.

Several analytes may be observed in the injection just following system priming. Always run an instrument blank after system priming.

10.2.5 Calibration Factors

Internal standard calibration techniques are employed in this method.

10.2.5.1 Internal Standard Procedure. In each standard, calculate the response factor (RF) for each analyte, the average RF, and the relative standard deviation (RSD) of the RFs, using the Enviroquant data processing software. The calculations are performed automatically, using the formulae listed in Alpha's Quality Manual.

10.2.6 Initial Calibration Criteria

If the RSD for an analyte is $\leq$ 20%, then the response of the instrument for this compound is considered linear over the range and the mean calibration factor can be used to quantitate sample results.

If the RSD for any analyte is > 20%, then linearity through the origin cannot be assumed. The mean response factor cannot be used for quantitation. An alternative calculation may be done by the use of linearity as long as the correlation coefficient is $\geq$0.995. If both of these quantitation methods fail criteria for any compound in the initial calibration, then the system must be reevaluated and a new calibration curve must be analyzed.

10.2.7 Retention Time Windows

10.2.7.1 The retention time windows used for the identification of target analytes are calculated using the procedure recommended in Method 8000 and were found to be ± 0.015 minutes.

10.2.7.2 The windows listed above are used as guidance; however the experience of the analyst weighs heavily in the interpretation of the chromatograms. For example, it has been observed that certain oil matrices can cause the retention times to shift more dramatically. Additionally, if any positive results are questionable and at sufficiently high concentration, GC/MS analysis is used for confirmation.

10.2.8 Initial Calibration Verification

An initial calibration verification standard must be run immediately after each initial calibration, near the midpoint of the curve. This standard must be prepared using a second source that is different than the source used for the initial calibration. The %D for each analyte to be quantitated must not exceed a ± 20% difference when compared to the initial calibration curve.

10.3 Equipment Operation and Sample Processing

10.3.1 The same GC operating conditions used for the initial calibration must be employed for sample analyses, including sample injection volume (Section 10.1.3).

10.3.2 Tentative identification of an analyte occurs when a peak from a sample extract falls within the retention time window for the compound. Each tentative identification is confirmed using a second GC column of dissimilar stationary phase. Confirmation is positive when the %RPD is $\leq$ 40% from the results of the two columns. In particularly difficult matrices, confirmation by GC/MS may be advisable (see Section 10.3.11).

10.3.3 The concentration reported for an identified target analyte in an extract is calculated using the Enviroquant data processing software. The Enviroquant methods have been configured to utilize the quantitation formulas found in Alpha's Quality Manual. Proper quantitation requires the appropriate selection of a baseline from which the peak area or height can be determined. See the Manual Integration SOP for integration guidelines.

10.3.3.1 If the responses exceed the calibration range of the system, dilute the extract and reanalyze.

10.3.4 Each sample analysis must begin with an acceptable initial calibration, calibration verification standard(s) (each 12-hour analytical shift), or calibration standards interspersed within the samples. The Internal Standard area of the samples must be within 50%-200% of the starting CCV. When a calibration verification standard fails to meet the QC criteria, all samples that were injected after the last standard that last met the QC criteria must be re-injected.

10.3.5 Sample injections may continue for as long as the calibration verification standards and standards interspersed with the samples meet instrument QC requirements. The sequence ends when the set of samples has been injected or when qualitative and/or quantitative QC criteria are exceeded.

10.3.6 Use the calibration standards analyzed during the sequence to evaluate retention time stability. The retention time windows are established using the absolute retention time of each analyte in the mid-concentration standard during the initial calibration as the mid-point of the window. The widths of the windows are defined in Section 10.2.7.

10.3.7 Each subsequent injection of a standard during the 12-hour analytical shift (i.e., those standards injected every 20 samples, or more frequently) must be checked against the retention time windows. If any of these subsequent standards fall outside their absolute retention time windows, the GC system is out of control. Determine the cause of the problem and correct it. If the problem cannot be corrected, a new initial calibration must be performed.

10.3.8 Identification of mixtures (i.e. Chlordane and Toxaphene) is based on the characteristic 'fingerprint' retention time and shape of the indicator peak(s); and quantitation is based on the area under the characteristic peaks as compared to the area under the corresponding calibration peaks(s) of the same retention time and shape generated using internal calibration procedures.

10.3.9 If compound identification or quantitation is precluded due to interference (e.g., broad, rounded peaks or ill-defined baselines are present) cleanup of the extract may be needed. If instrument problems are suspected, rerun the extract on another instrument to determine if the problem results from analytical hardware or the sample matrix. Refer to the extraction SOPs for the procedures to be followed in sample cleanup.

10.3.10 For secondary column analysis, a second dissimilar column is utilized to confirm positive pesticide results. The laboratory must report the higher of the two results unless obvious interference is present on one of the columns. All required QA/QC parameters (e.g. calibrations, LCSs, etc.) must be met on the second column as well.

10.3.11 GC/MS Confirmation

GC/MS confirmation may be used in conjunction with either single-column or dual-column analysis if the concentration is sufficient for detection by GC/MS.

10.3.11.1 Full-scan GC/MS will normally require a concentration of approximately 10ng/µL in the final extract for each single-component compound.

10.3.11.2 The GC/MS must be calibrated for the specific target pesticides when it is used for quantitative analysis.

10.3.11.3 GC/MS may not be used for confirmation when concentrations are below the sensitivity of the instrument.

10.3.11.4 GC/MS confirmation should be accomplished by analyzing the same extract that is used for GC/ECD analysis.

10.3.11.5 The base/neutral/acid extract and the associated blank may be used for GC/MS confirmation if the surrogates and internal standards do not interfere and if it is demonstrated that the analyte is stable during acid/base partitioning. However, if the compounds are not detected in the base/neutral/acid extract, then GC/MS analysis of the pesticide extract should be performed.

10.3.11.6 A QC reference sample containing the compound should also be analyzed by GC/MS. The concentration of the QC reference sample must demonstrate that those pesticides identified by GC/ECD can be confirmed by GC/MS.

10.4 Continuing Calibration Verification

10.4.1 Verify calibration each 12-hour shift by injecting calibration verification standards (CCV) prior to conducting any sample analyses. A calibration standard must be injected at intervals of not less than once every twenty samples (after every 4 - 8 samples is routine practice to minimize the number of samples requiring re-injection when QC limits are exceeded) and at the end of the analysis sequence.

10.4.1.1 The calibration factor (for external standard compounds) and response factor (for internal standard compounds) for each analyte to be quantitated must not exceed a ± 20% difference when compared to the initial calibration curve. The Enviroquant data processing software automatically calculates the %D for all analytes according to the formulae in Alpha's Quality Manual. The Internal Standard area of the CCV must be within 50%-150% of the Initial Calibration Internal Standard area.

10.4.1.2 If this criterion is exceeded, inspect the gas chromatographic system to determine the cause and perform whatever maintenance is necessary before verifying calibration and proceeding with sample analysis.

10.4.1.3 If routine maintenance does not return the instrument performance to meet the QC requirements (Section 10) based on the last initial calibration, then a new initial calibration must be performed. Due to the large number of analytes present, allowances may be made for a CF or RF that drifts out high, as long as there are no positive hits for that particular analyte in any of the associated samples. Any QC failures must be written up by the analyst on narrative sheets for inclusion with the sample data.

10.4.2 Compare the retention time of each analyte in the calibration standard with the absolute retention time windows described in section 10.2.7. The center of the absolute retention time window for each analyte is its retention time in the mid-concentration standard analyzed during the initial calibration. Each analyte in each standard must fall within its respective retention time window. If not, the gas chromatographic system must either be adjusted so that a second analysis of the standard does result in all analytes falling within their retention time windows, or a new initial calibration must be performed and new retention time windows established.

10.5 Preventive Maintenance

Routine preventive maintenance should be performed to maintain GC system performance. This includes periodic replacement of injector septa, replacement of injector liner(s), and replacement of injector seals.

10.5.1 Other Maintenance

Additional maintenance may be required if system performance degrades.

10.5.1.1 GC injector ports are of critical concern, especially in the analysis of DDT and Endrin.

10.5.1.1.1 Injectors that are contaminated or chemically active can cause the degradation ("breakdown") of the analytes. Endrin and DDT breakdown to Endrin aldehyde, Endrin ketone, DDD, or DDE.

Check for degradation problems by injecting a standard containing only 4,4'-DDT and Endrin. Presence of 4,4'-DDE, 4,4'-DDD, Endrin ketone or Endrin indicates breakdown. If degradation of either DDT or Endrin exceeds 15%, take corrective action before proceeding with calibration.

When such breakdown is observed, replacement of the injector liner and seal may solve the problem. If not, clip approximately 3 – 6 inches from the injector end of the GC column. If the degradation does not improve, it may be necessary to replace the column(s).

Calculate percent breakdown as follows:

$$\% \text{ breakdown of DDT} = \frac{\text{sum of degradation peak areas (DDD+DDE)}}{\text{sum of all peak areas (DDT+DDE+DDD)}} \times 100$$

$$\% \text{ breakdown of Endrin} = \frac{\text{sum of degradation peak areas (aldehyde+ketone)}}{\text{sum of all peak areas (Endrin+aldehyde+ketone)}} \times 100$$

10.5.1.2 ECD detectors may also become contaminated, requiring bake out at elevated temperatures, or repair by the manufacturer.

11. Data Evaluation, Calculations and Reporting

11.1 Quantitation of Single Component Pesticides

The single component pesticide compounds are calculated as described in Section 10.3.3, and reported in µg/L or µg/Kg units. After performing technical data review, validating that all QC criteria have been met and confirming all positive hits, the data report is sent electronically to the LIMS computer for generation of the client report. There are two levels of review of the data in the LIMS system prior to release of data. These reviews should be done by two separate individuals.

11.1.1 Quantitation of Multiple-Component Analytes

Quantitation is based on use of 3-5 of the major peaks present for chlordane and 4-6 of the major peaks present for toxaphene. Each of these peaks is individually calibrated with a 9 point calibration based on average response factors. The %RSD must meet the criteria of ≤20%. The 3 to 5 or 4 – 6 major peaks are calculated as described in Section 10.3.3. After individual calculation meets criteria, the average of the major 3-5 or 4 – 6 peaks is used to determine the final concentration.

11.1.1.1 Toxaphene

Toxaphene is quantitated by the internal standard method, using the 4 – 6 largest peaks found in the standard and averaging the resulting concentrations.

11.1.1.2 Chlordane

Chlordane is a mixture of at least 11 major components and 30 or more minor components. Trans- and cis-Chlordane (alpha and gamma, respectively), are the two major components of Chlordane. However, the exact percentage of each in the chlordane material is not completely defined, and is not consistent from batch to batch.

11.1.1.2.1 The GC pattern of a Chlordane residue may differ considerably from that of the chlordane standard. Depending on the sample substrate and its history, residues of Chlordane can consist of almost any combination of constituents

from the Chlordane, plant and/or animal metabolites, and products of degradation caused by exposure to environmental factors such as water and sunlight.

11.1.1.2.2 Whenever possible, when Chlordane residue does not resemble Chlordane, the analyst should quantitate the peaks of alpha-Chlordane, gamma-Chlordane, Heptachlor, and trans-Nonachlor separately against the appropriate reference materials, and report the individual residues.

11.1.1.2.3 When the GC pattern of the residue resembles that of Chlordane, the analyst may quantitate Chlordane residues by comparing the total area of the Chlordane chromatogram using the 3-5 major peaks

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedance and/or improper preservation are noted on the nonconformance report form.

Perform instrument maintenance as described throughout this SOP as needed when instrument calibration criteria are not met. Record all maintenance in the instrument logbook.

All batch and sample specific QC criteria outlined in Section 10 are evaluated by the analyst prior to approval of the data. When any QC criteria fail, the cause for the failure must be identified and corrected. This may include instrument recalibration followed by sample reanalysis, sample cleanup, or sample re-extraction. If it is determined that the failure is due to sample matrix effects, a project narrative report is written by the analyst for inclusion in the data report. If there is insufficient sample volume to perform the re-analysis for confirmation, this is also noted in the narrative and included in the client report.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Qualtrax ID 1732. These studies performed by the laboratory are maintained on file for review

13.2 Demonstration of Capability Studies

Refer to Qualtrax ID 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

Qualtrax ID 1732 MDL/LOD/LOQ Generation

Qualtrax ID1739 IDC/DOC Generation

Qualtrax ID 1728 Waste Management and Disposal SOP

16. Attachments

Table 1: REPORTING LIMITS

Table 2: STANDARD SOLUTIONS

Table 3: QC ACCEPTANCE CRITERIA

TABLE 1

REPORTING LIMITS*

	RL (Aqueous)	RL (Soil)
Pesticides		
Alpha-BHC	0.02 µg/L	3.33 µg/Kg
Gamma-BHC (Lindane)	0.02 µg/L	2.67 µg/Kg
Heptachlor	0.02 µg/L	4 µg/Kg
Endosulfan I	0.02 µg/L	10 µg/Kg
Dieldrin	0.04 µg/L	5 µg/Kg
Endrin	0.04 µg/L	3.33 µg/Kg
4, 4'-DDD	0.04 µg/L	10 µg/Kg
4, 4'-DDT	0.04 µg/L	15 µg/Kg
Methoxychlor	0.2 µg/L	15 µg/Kg
Aldrin	0.02 µg/L	10 µg/Kg
Beta-BHC	0.02 µg/L	10 µg/Kg
Delta-BHC	0.02 µg/L	10 µg/Kg
Heptachlor Epoxide	0.02 µg/L	15 µg/Kg
trans-Chlordane	0.02 µg/L	10 µg/Kg
cis-Chlordane	0.02 µg/L	10 µg/Kg
4, 4'- DDE	0.04 µg/L	15 µg/Kg
Endosulfan II	0.04 µg/L	15 µg/Kg
Endrin Aldehyde	0.04 µg/L	10 µg/Kg
Endosulfan Sulfate	0.04 µg/L	3.33 µg/Kg
Endrin Ketone	0.04 µg/L	10 µg/Kg
Chlordane	0.2 µg/L	65 µg/Kg
Toxaphene	0.2 µg/L	150 µg/Kg
Alachlor [+]	0.1 µg/L	10 µg/Kg

*** RL:** Typical laboratory reporting limit. Actual RLs may be higher depending on sample matrix. RLs are not adjusted for % Moisture.

[+] CT-RCP- only compound.

TABLE 2

STANDARD SOLUTIONS

	Stock 1 solution (µg/mL)	Stock 2 solution (µg/mL)	Level 10 (µg/L)	Level 9 (µg/L)	Level 8 (µg/L)	Level 7 (µg/L)	Level 6 (µg/L)	Level 5 (µg/L)	Level 4 (µg/L)	Level 3 (µg/L)	Level 2 (µg/L)	Level 1 (µg/L)	Std. Spike Solution (µg/mL)	Std. LCS Solution (µg/mL)
Hexachlorobenzene	2	0.1	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Alpha-BHC	2	0.1	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Lindane	2	0.1	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Heptachlor	2	0.1	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Endosulfan I	2	0.1	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Dieldrin	2	0.1	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Endrin	2	0.1	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
4, 4'-DDD	2	0.1	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
4, 4'-DDT	2	0.1	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Methoxychlor	2	0.1	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Tetrachloro-m-Xylene *	2	0.1	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Decachloro-Biphenyl *	2	0.1	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Aldrin	2	0.1	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Beta-BHC	2	0.1	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Delta-BHC	2	0.1	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Heptachlor Epoxide	2	0.1	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Trans-Chlordane	2	0.1	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Cis-Chlordane	2	0.1	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
4, 4'- DDE	2	0.1	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Endosulfan II	2	0.1	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Endrin Aldehyde	2	0.1	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Endosulfan Sulfate	2	0.1	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Endrin Ketone	2	0.1	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Alachlor	2	0.1	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Chlordane	50000	1000	N/A	5000	2500	1000	500	250	100	50	10	5		
Toxaphene	100000	2000	N/A	10000	5000	2000	1000	500	200	100	20	10		
Internal Standard														
1-Bromo-2-Nitrobenzene	5000	5000	25	25	25	25	25	25	25	25	25	25		

* - surrogates

TABLE 3

QC ACCEPTANCE CRITERIA**

	Aqueous		Soil	
Surrogate % Recovery*	**Lower Control Limit**	**Upper Control Limit**	**Lower Control Limit**	**Upper Control Limit**
2,4,5,6-Tetrachloro-m-xylene	30%	150%	30%	150%
Decachlorobiphenyl	30%	150%	30%	150%

*One surrogate on one column is allowed to be outside of the control limits and still be within criteria.

Compound	**LCS % Recovery (Aqueous)**		**LCS % Recovery (Soil)**		**Duplicate and/or MSD %RPD (Aqueous and Soil)**	
	Lower Control Limit	**Upper Control Limit**	**Target Analyte Limits**	**Marginal Exceedance Limits**	**Target Analyte Limits**	**Marginal Exceedance Limits**
Target Analytes LCS/LCSD	30%	150%	30%	150%	30%	50%
Target Analytes MS/MSD	30%	150%	30%	150%	30%	50%

** **NOTE:** CT-RCP, NJ-8081, and MCP-8081 % Recovery LCS/ LCSD Acceptance Criteria is 40-140%

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PROCEDURES AND CRITERIA
ANALYSIS OF FLUORESCENT DYES
IN WATER AND CHARCOAL SAMPLERS:

FLUORESCEIN, EOSINE, RHODAMINE WT, AND SULFORHODAMINE B DYES

Revision Date:
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INTRODUCTION

This document describes standard procedures and criteria currently in use at the Ozark Underground Laboratory (OUL) as of the date shown on the title page. Some samples may be subjected to different procedures and criteria because of unique conditions; such non-standard procedures and criteria are identified in reports for those samples. Standard procedures and criteria change as knowledge and experience increases and as equipment is improved or up-graded. The OUL maintains a summary of changes in standard procedures and criteria.

TRACER DYES AND SAMPLE TYPES

Dye Nomenclature

Dye manufacturers and retailers use a myriad of names for the dyes. This causes confusion among dye users and report readers. The primary dyes used at the OUL and described in this document are included in Table 1 below.

Table 1. Primary OUL Dye Nomenclature.

OUL Common Name	Color Index Number	Color Index Name	Other Names
Fluorescein	45350	Acid Yellow 73	uranine, uranine C, sodium fluorescein, fluorescein LT and fluorescent yellow/green
Eosine	45380	Acid Red 87	eosin, eosine OJ, and D&C Red 22
Rhodamine WT	None assigned	Acid Red 388	fluorescent red (but not the same as rhodamine B)
Sulforhodamine B	45100	Acid Red 52	pontacyl brilliant pink B, lissamine red 4B, and fluoro brilliant pink

The OUL routinely provides dye for tracing projects. Dyes purchased for groundwater tracing are always mixtures that contain both dye and an associated diluent. Diluents enable the manufacturer to standardize the dye mixture so that there are minimal differences among batches. Additionally, diluents are often designed to make it easier to dissolve the dye mixture in water, or to produce a product which meets a particular market need (groundwater tracing is only a tiny fraction of the dye market). The percent of dye in "as-sold" dye mixtures often varies dramatically among manufacturers and retailers, and retailers are sometimes incorrect about the percent of dye in their products. The OUL subjects all of its dyes to strict quality control (QC) testing. Table 2 summarizes the as-sold dye mixtures used by the OUL.

Table 2. As-Sold Dye Mixtures at the OUL.

OUL Common Name	Form	Dye Equivalent
Fluorescein	Powder	75% dye equivalent, 25% diluent
Eosine	Powder	75% dye equivalent, 25% diluent
Rhodamine WT	Liquid	20% dye equivalent, 80% diluent
Sulforhodamine B	Powder	75% dye equivalent, 25% diluent

Analytical results are based on the as-sold weights of the dyes provided by the OUL. The use of dyes from other sources is discouraged due to the wide variability of dye equivalents within the market. However, if alternate source dyes are used, a sample should be provided to the OUL for quality control and to determine if a correction factor is necessary for the analytical results.

Types of Samples

Typical samples that are collected for fluorescent tracer dye analysis include charcoal samplers (also called activated carbon or charcoal packets) and water samples.

The charcoal samplers are packets of fiberglass screening partially filled with 4.25 grams of activated coconut charcoal. The charcoal used by the OUL is Calgon 207C coconut shell carbon, 6 to 12 mesh, or equivalent. The most commonly used charcoal samplers are about 4 inches long by 2 inches wide. A cigar-shaped sampler is made for use in very small diameter wells (such as 1-inch diameter piezometers); this is a special order item and should be specifically requested in advance when needed. All of the samplers are closed by heat sealing.

In specialized projects, soil samples have been collected from soil cores and analyzed for fluorescent tracer dyes. Project-specific procedures have been developed for projects such as these. For additional information, please contact the OUL.

FIELD PROCEDURES

Field procedures included in this section are intended as guidance, and not firm requirements. Placement of samplers and other field procedures require adjustment to field conditions. Personnel at the OUL are available to provide additional assistance for implementation of field procedures specific to specialized field conditions.

Placement of Samplers

Charcoal samplers are placed so as to be exposed to as much water as possible. Water should flow through the packet. In springs and streams they are typically attached to a rock or other anchor in a riffle area. Attachment of the packets often uses plastic tie wires. In swifter water galvanized wire (such as electric fence wire) is often used. Other types of anchoring wire can be used. Electrical wire with plastic insulation is also good. Packets are attached so that they extend outward from the anchor rather than laying flat against it. Two or more separately anchored packets are typically used for sampling springs and streams. The placement of multiple packets is recommended in order to minimize the chance of loss during the sampling period. The use of fewer packets is discouraged except when the spring or stream is so small that there is not appropriate space for placing multiple packets.

When pumping wells are being sampled, the samplers are typically placed in sample holders made of plastic pipe fittings. Brass hose fittings can be at the end of the sample holders so that the sample holders can be installed on outside hose bibs and water which has run through the samplers can be directed to waste through a connected garden hose. The samplers can be unscrewed in the middle so that charcoal packets can be changed. The middle portions of the samplers consist of 1.5 inch diameter pipe and pipe fitting.

Charcoal packets can be lowered into monitoring wells for sampling purposes. In general, if the well is screened, samplers should be placed approximately in the middle of the screened interval. Due to the typically lower volume of water that flows through a well, only one charcoal sampler should be used per well. However, multiple packets can be placed in a single well at depths to test different depth horizons when desirable. A weight should be added near the charcoal packet to ensure that it will not float. The weight should be of such a nature that it will not affect water quality. One common approach is to anchor the packets with a white or uncolored plastic cable tie to the top of a dedicated weighted disposable bailer. We typically run nylon cord from the top of the well to the charcoal packet and its weight. ***Do not use colored cord*** since some of them are colored with fluorescent dyes. Nylon fishing line should not be used since it can be readily cut by a sharp projection in the well.

In some cases, especially with small diameter wells and appreciable well depths, the weighted disposable bailers sink very slowly or may even fail to sink because of friction and floating of the anchoring cord. In such cases a weight may be added to the top of the disposable bailer. Stainless steel weights are ideal, but are not needed in all cases. All weights should be cleaned prior to use; the cleaning approach should comply with decontamination procedures in use at the project site.

Optional Preparation of Charcoal Samplers

Charcoal packets routinely contain some fine powder that washes off rapidly when they are placed in water. While not usually necessary, the following optional preparation step is suggested if the fine charcoal powder is problematic.

Charcoal packets can be triple rinsed with distilled, demineralized, or reagent water known to be free of tracer dyes. This rinsing is typically done by soaking. With this approach,

approximately 25 packets are placed in one gallon of water and soaked for at least 10 minutes. The packets are then removed from the water and excess water is shaken off the packets. The packets are then placed in a second gallon of water and again soaked for at least 10 minutes. After this soaking they are removed from the water and excess water is shaken off the packets. The packets are then placed in a third gallon of water and the procedure is again repeated. Rinsed packets are placed in plastic bags and are placed at sampling stations within three days. Packets can also be rinsed in jets of water for about one minute; this requires more water and is typically difficult to do in the field with water known to be free of tracer dyes.

Collection and Replacement of Samplers

Samplers are routinely collected and replaced at each of the sampling stations. The frequency of sampler collection and replacement is determined by the nature of the study. Collections at one week intervals are common, but shorter or longer collection frequencies are acceptable and sometimes more appropriate. Shorter sampling frequencies are often used in the early phases of a study to better characterize time of travel. As an illustration, we often collect and change charcoal packets 1, 2, 4, and 7 days after dye injection. Subsequent sampling is then weekly.

The sampling interval in wells at hazardous wastes sites should generally be no longer than about a week. Contaminants in the water can sometimes use up sorption sites on the charcoal that would otherwise adsorb the dye. This is especially important if the dye might pass in a relatively short duration pulse.

Where convenient, the collected samplers should be briefly rinsed in the water being sampled to remove dirt and accumulated organic material. This is not necessary with well samples. The packets are shaken to remove excess water. Next, the packet (or packets) are placed in a plastic bag (Whirl-Pak® bags are ideal). The bag is labeled on the outside with a black permanent type felt marker pen, such as a Sharpie®. ***Use only pens that have black ink***; colored inks may contain fluorescent dyes. The notations include station name or number and the date and time of collection. Labels must not be inserted inside the sample bags.

Collected samplers are kept in the dark to minimize algal growth on the charcoal prior to analysis work. New charcoal samplers are routinely placed when used charcoal packets are collected. The last set of samplers placed at a stream or spring is commonly not collected.

Water Samples

Water samples are often collected. They should be collected in either glass or plastic; the OUL routinely uses 50 milliliter (mL) research-grade polypropylene copolymer Perfector Scientific vials (Catalog Number 2650) for such water samples. No more than 30 mL of water is required for analysis. The sides of the vials should be labeled with the project name, sample ID, sample date and time with a black permanent felt tip pen. ***Do not label the lid only***. The vials should be placed in the dark and refrigerated immediately after collection, and maintained under refrigeration until shipment. The OUL supplies vials for the collection of water samples.

Sample Shipment

When water or charcoal samplers are collected for shipment to the OUL they should be shipped promptly. We prefer (and in some studies require) that samples be refrigerated with frozen re-usable ice packs upon collection and that they be shipped refrigerated with frozen ice packs by overnight express. ***Do not ship samplers packed in wet ice*** since this can create a potential for cross contamination when the ice melts. Our experience indicates that it is not essential for samplers to be maintained under refrigeration; yet maintaining them under refrigeration clearly minimizes some potential problems. A product known as "green ice" should not be used for maintaining the samples in a refrigerated condition since this product contains a dye which could contaminate samples if the "green ice" container were to break or leak.

We receive good overnight and second day air service from both UPS and FedEx. The U.S. Postal Service does not typically provide next day service to us. DHL does not provide overnight service to us. FedEx is recommended for international shipments. The OUL does not receive Saturday delivery.

Each shipment of charcoal samplers or water samples ***must be accompanied by a sample custody document***. The OUL provides a sheet (which bears the title "Samples for Fluorescence Analysis") that can be used if desired. These sheets can be augmented by a client's chain-of-custody forms or any other relevant documentation. OUL's custody document works well for charcoal samplers because it allows for both the placement date and time as well as the collection date and time. Many other standard chain-of-custody documents do not allow for these types of samples. Attachment 1 includes a copy of OUL's Sample Collection Data Sheet.

Please write legibly on the custody documents and ***use black ink***. Check the accuracy of the sample sheet against the samples prior to shipment to identify and correct errors that may delay the analysis of your samples following receipt at the laboratory.

Supplies Provided by the OUL

The OUL provides supplies for the collection of fluorescent tracer dyes. Supplies provided upon request are charcoal packets, Whirl-Pak® bags (to contain the charcoal packets after collection for shipment to the laboratory), and water vials. These supplies are subjected to strict QA/QC procedures to ensure the materials are free of any potential tracer dye contaminants. The charge for these materials is included in the cost of sample analysis. Upon request, coolers and re-freezable ice packs are also provided for return shipment of samples.

The OUL also has tracer dyes available for purchase. These dyes are subject to strict QA/QC testing. All analytical work is based upon the OUL as-sold weight of the dyes.

LABORATORY PROCEDURES

The following procedures are followed upon receipt of samples at the laboratory.

Receipt of Samples

Samplers shipped to the OUL are logged in and refrigerated upon receipt. Prior to cleaning and analysis, samplers are assigned a laboratory identification number.

It sometimes occurs that there are discrepancies between the sample collection data sheet and the actual samples received. When this occurs, a "Discrepancy Sheet" form is completed and sent to the shipper of the sample for resolution. The purpose of the form is to help resolve discrepancies, even when they may be minor. Many discrepancies arise from illegible custody documents. ***Please write legibly*** on the custody documents and ***use black ink***. Check the accuracy of the sample sheet against the samples prior to shipment to identify and correct errors that may delay the analysis of your samples following receipt at the laboratory.

Cleaning of Charcoal Samplers

Samplers are cleaned by spraying them with jets of clean water from a laboratory well in a carbonate aquifer. OUL uses non-chlorinated water for the cleansing to minimize dye deterioration. We do not wash samplers in public water supplies. Effective cleansing cannot generally be accomplished simply by washing in a conventional laboratory sink even if the sink is equipped with a spray unit.

The duration of packet washing depends upon the condition of the sampler. Very clean samplers may require less than a minute of washing; dirtier samplers may require several minutes of washing.

Elution of the Charcoal

There are various eluting solutions that can be used for the recovery of tracer dyes. The solutions typically include an alcohol, water, and a strong basic solution such as aqueous ammonia and /or potassium hydroxide.

The standard elution solution used at the OUL is a mixture of 5% aqua ammonia and 95% isopropyl alcohol solution and sufficient potassium hydroxide pellets to saturate the solution. The isopropyl alcohol solution is 70% alcohol and 30% water. The aqua ammonia solution is 29% ammonia. The potassium hydroxide is added until a super-saturated layer is visible in the bottom of the container. This super-saturated layer is not used for elution. Preparation of eluting solutions uses dedicated glassware which is never used in contact with dyes or dye solutions.

The eluting solution will elute fluorescein, eosine, rhodamine WT, and sulforhodamine B dyes. It is also suitable for separating fluorescein peaks from peaks of some naturally present materials found in may be found in samplers.

Fifteen mL of the eluting solution is poured over the washed charcoal in a disposable sample beaker. The sample beaker is capped. The sample is allowed to stand for 60 minutes. After this time, the liquid is carefully poured off the charcoal into a new disposable beaker which has been appropriately labeled with the laboratory identification number. A few grains of charcoal may inadvertently pass into the second beaker; no attempt is made to remove these from the second sample beaker. After the pouring, a small amount of the elutant will remain in the initial sample beaker. After the transfer of the elutant to the second sample beaker, the contents of the first sample beaker (the eluted charcoal) are discarded. Samples are kept refrigerated until analyzed.

pH Adjustment of Water Samples

The fluorescence intensity of several of the commonly used fluorescent tracer dyes is pH dependent. The pH of samples analyzed for fluorescein, eosine, and pyranine dyes are adjust to a target pH of greater than 9.5 in order to obtain maximum fluorescence intensities.

Adjustment of pH is achieved by placing samples in a high ammonia atmosphere for at least two hours in order to increase the pH of the sample. Reagent water standards are placed in the same atmosphere as the samples. If dye concentrations in a sample are off-scale and require dilution for quantification of the dye concentration, the diluting water used is OUL reagent water that has been pH adjusted in a high ammonia atmosphere. Samples that are only analyzed for rhodamine WT or sulforhodamine B are not required to be pH adjusted.

Analysis on the Shimadzu RF-5301

The OUL uses a Shimadzu spectrofluorophotometer model RF-5301. This instrument is capable of synchronous scanning. The OUL also owns a Shimadzu RF-540 spectrofluorometers that is occasionally used for special purposes.

A sample of the elutant or water is withdrawn from the sample container using a disposable polyethylene pipette. Approximately 3 mL of the sample is then placed in disposable rectangular polystyrene cuvette. The cuvette has a maximum capacity of 3.5 mL. The cuvette is designed for fluorometric analysis; all four sides and the bottom are clear. The acceptable spectral range of these cuvettes is 340 to 800 nm. The pipettes and cuvettes are discarded after one use.

The cuvette is then placed in the RF-5301. This instrument is controlled by a programmable computer and operated by proprietary software developed for dye tracing applications.

Our instruments are operated and maintained in accordance with the manufacturer's recommendations. On-site installation of our first instrument and a training session on its use was provided by the instrument supplier. Repairs are made by a Shimadzu-authorized repairman.

Our typical analysis of an elutant sample where fluorescein, eosine, rhodamine WT, or sulforhodamine B dyes may be present includes synchronous scanning of excitation and emission spectra with a 17 nm separation between excitation and emission wavelengths. For these dyes,

the excitation scan is from 443 to 613 nm; the emission scan is from 460 to 630 nm. The emission fluorescence from the scan is plotted on a graph. The typical scan speed setting is “fast” on the RF-5301. The typical sensitivity setting used is "high."

Table 3. Excitation and emission slit width settings routinely used for dye analysis.

Parameter	**Excitation Slit (nm)**	**Emission Slit (nm)**
ES, FL, RWT, and SRB in elutant	3	1.5
ES, FL, RWT, and SRB in water	5	3

Note: ES = Eosine. FL = Fluorescein. RWT = Rhodamine WT. SRB = Sulforhodamine B.

The instrument produces a plot of the synchronous scan for each sample; the plot shows emission fluorescence only. The synchronous scans are subjected to computer peak picks using proprietary software; peaks are picked to the nearest 0.1 nm. Instrument operators have the ability to manually adjust peaks as necessary based upon computer-picked peaks and experience. All samples run on the RF-5301 are stored electronically with sample information. All samples analyzed are recorded in a bound journal.

Quantification

We calculate the magnitude of fluorescence peaks for fluorescein, eosine, rhodamine WT, and sulforhodamine B dyes in both elutant and water samples. Dye quantities are expressed in microgram per liter (parts per billion; ppb). The dye concentrations are calculated by separating fluorescence peaks due to dyes from background fluorescence on the charts, and then calculating the area within the fluorescence peak. This area is proportional to areas obtained from standard solutions.

We run dye concentration standards each day the RF-5301 is used. Six standards are used; the standard or standards appropriate for the analysis work being conducted are selected. All standards are based upon the as-sold weights of the dyes. The standards are as follows:

1) 10 ppb fluorescein and 100 ppb rhodamine WT in well water from the Jefferson City-Cotter Formation
2) 10 ppb eosine in well water from the Jefferson City-Cotter Formation
3) 100 ppb sulforhodamine B in well water from the Jefferson City-Cotter Formation.
4) 10 ppb fluorescein and 100 ppb rhodamine WT in elutant.
5) 10 ppb eosine in elutant.
6) 100 ppb sulforhodamine B in elutant.

Preparation of Standards

Dye standards are prepared as follows:

Step 1. A small sample of the as-sold dye is placed in a pre-weighed sample vial and the vial is again weighed to determine the weight of the dye. We attempt to use a sample weighing between 1 and 5 grams. This sample is then diluted with well water to make a 1% dye solution by weight (based upon the as-sold weight of the dye). The resulting dye solution is allowed to sit for at least four hours to ensure that all dye is fully dissolved.

Step 2. One part of each dye solution from Step 1 is placed in a mixing container with 99 parts of well water. Separate mixtures are made for fluorescein, rhodamine WT, eosine, and sulforhodamine B. The resulting solutions contain 100 mg/L dye (100 parts per million dye mixture). The typical prepared volume of this mixture is appropriate for the sample bottles being used; we commonly prepare about 50 mL of the Step 2 solutions. The dye solution from Step 1 that is used in making the Step 2 solution is withdrawn with a digital Finnpipette which is capable of measuring volumes between 0.200 and 1.000 mL at intervals of 0.005 mL. The calibration certificate with this instrument indicates that the accuracy (in percent) is as follows:

At 0.200 mL, 0.90%

At 0.300 mL, 0.28%

At 1.000 mL, 0.30%

The Step 2 solution is called the long term standard. OUL experience indicates that Step 2 solutions, if kept refrigerated, will not deteriorate appreciably over periods of less than a year. Furthermore, these Step 2 solutions may last substantially longer than one year.

Step 3. A series of intermediate-term dye solutions are made. Approximately 45 mL. of each intermediate-term dye solution is made. All volume measurements of less than 5 mL are made with a digital Finnpipette. (see description in Step 2). All other volume measurements are made with Rheinland Kohn Geprufte Sicherheit 50 mL capacity pump dispenser which will pump within plus or minus 1% of the set value. The following solutions are made; all concentrations are based on the as-sold weight of the dyes:

1) 1 ppm fluorescein dye and 10 ppm rhodamine WT dye.

2) 1 ppm eosine.

3) 10 ppm sulforhodamine B dye.

Step 4. A series of six short-term dye standards are made from solutions in Step 3. These standards were identified earlier in this section. In the experience of the OUL these standards have a useful shelf life in excess of one week. However, in practice, Step 4 elutant standards are made weekly, and Step 4 water standards are made daily.

Dilution of Samples

Samples with peaks that have arbitrary fluorescence unit values of 500 or more are diluted a hundred fold to ensure accurate quantification.

Some water samples have high turbidity or color which interferes with accurate detection and measurement of dye concentrations. It is often possible to dilute these samples and then measure the dye concentration in the diluted sample.

The typical dilutions are either 10 fold (1:10) or 100 fold (1:100). A 1:10 dilution involves combining one part of the test sample with 9 parts of water (if the sample is water) or elutant (if the sample is elutant). A 1:100 dilution involves combining one part of the test sample is combined with 99 parts of water or elutant, based upon the sample media. Typically, 0.300 mL of the test solution is combined with 29.700 mL of water (or elutant as appropriate) to yield a new test solution.

All volume measurements of less than 5 mL are made with a digital Finnpipette. All other volume measurements are made with Rheinland Kohn Geprufte Sicherheit 50 mL capacity pump dispenser which will pump within plus or minus 1% of the set value.

The water used for dilution is from a carbonate aquifer. All dilution water is pH adjusted to greater than pH 9.5 by holding it in open containers in a high ammonia concentration chamber. This adjustment takes a minimum of two hours.

Quality Control

Laboratory blanks are run for every sample where the last two digits of the laboratory numbers are 00, 20, 40, 60, or 80. A charcoal packet is placed in a pumping well sampler and at least 25 gallons of unchlorinated water is passed through the sampler at a rate of about 2.5 gallons per minute. The sampler is then subjected to the same analytical protocol as all other samplers.

System functioning tests of the analytical instruments are conducted in accordance with the manufacturer's recommendations. Spiked samples are also analyzed when appropriate for quality control purposes.

All materials used in sampling and analysis work are routinely analyzed for the presence of any compounds that might create fluorescence peaks in or near the acceptable wavelength ranges for any of the tracer dyes. This testing includes approximately 1% of materials used.

Project specific QA/QC samples may include sample replicates and sample duplicates. A replicate sample is when a single sample is analyzed twice. A sample duplicate is where two samples are collected in a single location and both are analyzed. Sample replicates and duplicates are run for QA/QC purposes upon request of the client. These results are reported in the Certificate of Analysis.

Reports

Sample analysis results are typically reported in a Certificate of Analysis. However, specialized reports are provided in accordance with the needs of the client. Certificates of Analysis typically provide a listing of station number, sample ID, and dye concentrations if detected. Standard data format includes deliverables in MS Excel and Adobe Acrobat (.pdf)

format. Hard copy of the data package, and copies of the analytical charts are available upon request.

Work at the OUL is directed by Mr. Thomas Aley. Mr. Aley has 45 years of professional experience in hydrology and hydrogeology. He is certified as a Professional Hydrogeologist (Certificate #179) by the American Institute of Hydrology and licenced as a Professional Geologist in Missouri, Arkansas, Kentucky, and Alabama. Additional details regarding laboratory qualifications are available upon request.

Waste Disposal

All laboratory wastes are disposed of according to applicable state and federal regulations. Waste elutant and water samples are collected in 15 gallon poly drums and disposed with a certified waste disposal facilityas non-hazardous waste.

In special cases, wastes for a particular project may be segregated and returned to the client upon completion of the project. These projects may have samples that contain contaminants that the client must account for all materials generated and disposed. These situations are managed on a case-by-case basis.

CRITERIA FOR DETERMINATION OF POSITIVE DYE RECOVERIES

Normal Emission Ranges and Detection Limits

The OUL has established normal emission fluorescence wavelength ranges for each of the four dyes described in this document. The normal acceptable range equals mean values plus and minus two standard deviations. These values are derived from actual groundwater tracing studies conducted by the OUL.

The detection limits are based upon concentrations of dye necessary to produce emission fluorescence peaks where the signal to noise ratio is 3. The detection limits are realistic for most field studies since they are based upon results from actual field samples rather than being based upon values from spiked samples in a matrix of reagent water or the elutants from unused activated carbon samplers. In some cases detection limits may be smaller than reported if the water being sampled has very little fluorescent material in it. In some cases detection limits may be greater than reported; this most commonly occurs if the sample is turbid due to suspended material or a coloring agent such as tannic compounds. Turbid samples are typically allowed to settle, centrifuged, or, if these steps are not effective, diluted prior to analysis.

Table 4 provides normal emission wavelength ranges and detection limits for the four dyes when analyzed on the OUL's RF-5301 for samples analyzed as of March 3, 2015.

Table 4. RF-5301 Spectrofluorophotometer. Normal emission wavelength ranges and detection limits for fluorescein, eosine, rhodamine WT, and sulforhodamine B dyes in water and elutant samples.

Fluorescent Dye	**Normal Acceptable Emission Wavelength Range (nm)**		**Detection Limit (ppb)**	
	Elutant	**Water**	**Elutant**	**Water**
Eosine	539.3 to 545.1	532.5 to 537.0	0.050	0.015
Fluorescein	514.1 to 519.2	505.9 to 509.7	0.025	0.002
Rhodamine WT	564.6 to 571.2	571.9 to 577.2	0.170	0.015
Sulforhodamine B	575.2 to 582.0	580.1 to 583.7	0.080	0.008

Note: Detection limits are based upon the as-sold weight of the dye mixtures normally used by the OUL.
Fluorescein and eosine detection limits in water are based on samples pH adjusted to greater than 9.5.

It is important to note that the normal acceptable emission wavelength ranges are subject to change based on instrument maintenance, a change in instrumentation, or slight changes in dye formulation. Significant changes in normal acceptable emission wavelength ranges will be updated in this document as they occur.

Fluorescence Background

Due to the nature of fluorescence analysis, it is important to identify and characterize any potential background fluorescence at dye introduction and monitoring locations prior to the introduction of any tracer dyes.

There is generally little or no detectable fluorescence background in or near the general range of eosine, rhodamine WT, and sulforhodamine B dyes encountered in most groundwater tracing studies. There is often some fluorescence background in or near the range of fluorescein dye present at some of the stations used in groundwater tracing studies.

Criteria for Determining Dye Recoveries

The following sections identify normal criteria used by the OUL for determining dye recoveries. The primary instrument in use is a Shimadzu RF-5301.

EOSINE

Normal Criteria Used by the OUL for Determining Eosine Dye Recoveries in Elutants from Charcoal Samplers

Criterion 1. There must be at least one fluorescence peak in the range of 540.0 to 545.8 nm in the sample.

Criterion 2. The dye concentration associated with the fluorescence peak must be at least 3 times the detection limit. The eosine detection limit in elutant samples is 0.050 ppb, thus this dye concentration limit equals 0.150 ppb.

Criterion 3. The dye concentration must be at least 10 times greater than any other concentration reflective of background at the sampling station in question.

Criterion 4. The shape of the fluorescence peak must be typical of eosine. Much background fluorescence yields low, broad, and asymmetrical fluorescence peaks rather than the more narrow and symmetrical fluorescence peaks typical of eosine. In addition, there must be no other factors which suggest that the fluorescence peak may not be eosine dye from our groundwater tracing work.

Normal Criteria Used by the OUL for Determining Eosine Dye Recoveries in Water Samples

Criterion 1. In most cases, the associated charcoal samplers for the station should also contain eosine dye in accordance with the criteria listed above. This criterion may be waived if no charcoal sampler exists.

Criterion 2. There must be no factors which suggest that the fluorescence peak may not be eosine dye from our groundwater tracing work. The fluorescence peak should generally be in the range of 532.8 to 537.3 nm.

Criterion 3. The dye concentration associated with the fluorescence peak must be at least three times the detection limit. Our eosine detection limit in water samples is 0.015 ppb, thus this dye concentration limit equals 0.045 ppb.

Criterion 4. The dye concentration must be at least 10 times greater than any other concentration reflective of background at the sampling station in question.

FLUORESCEIN

Normal Criteria Used by the OUL for Determining Fluorescein Dye Recoveries in Elutants from Charcoal Samplers

Criterion 1. There must be at least one fluorescence peak in the range of 514.5 to 519.6 nm in the sample.

Criterion 2. The dye concentration associated with the fluorescence peak must be at least 3 times the detection limit. The fluorescein detection limit in elutant samples is 0.025 ppb, thus this dye concentration limit equals 0.075 ppb.

Criterion 3. The dye concentration must be at least 10 times greater than any other concentration reflective of background at the sampling station in question.

Criterion 4. The shape of the fluorescence peak must be typical of fluorescein. Much background fluorescence yields low, broad, and asymmetrical fluorescence peaks rather than the more narrow and symmetrical fluorescence peaks typical of fluorescein. In addition, there must be no other factors which suggest that the fluorescence peak may not be fluorescein dye from our groundwater tracing work.

Normal Criteria Used by the OUL for Determining Fluorescein Dye Recoveries in Water Samples

Criterion 1. In most cases, the associated charcoal samplers for the station should also contain fluorescein dye in accordance with the criteria listed above. This criterion may be waived if no charcoal sampler exists.

Criterion 2. There must be no factors which suggest that the fluorescence peak may not be fluorescein dye from our groundwater tracing work. The fluorescence peak should generally be in the range of 506.8 to 510.6 nm.

Criterion 3. The dye concentration associated with the fluorescence peak must be at least three times the detection limit. Our fluorescein detection limit in water samples is 0.002 ppb, thus this dye concentration limit equals 0.006 ppb.

Criterion 4. The dye concentration must be at least 10 times greater than any other concentration reflective of background at the sampling station in question.

RHODAMINE WT

Normal Criteria Used by the OUL for Determining Rhodamine WT Dye Recoveries in Elutants from Charcoal Samplers

Criterion 1. There must be at least one fluorescence peak in the sample in the range of 565.2 to 571.8 nm.

Criterion 2. The dye concentration associated with the rhodamine WT peak must be at least 3 times the detection limit. The detection limit in elutant samples is 0.170 ppb, thus this dye concentration limit equals 0.510 ppb.

Criterion 3. The dye concentration must be at least 10 times greater than any other concentration reflective of background at the sampling station in question.

Criterion 4. The shape of the fluorescence peak must be typical of rhodamine WT. In addition, there must be no other factors which suggest that the fluorescence peak may not be dye from the groundwater tracing work under investigation.

Normal Criteria Used by the OUL for Determining Rhodamine WT Dye Recoveries in Water Samples

Criterion 1. In most cases, the associated charcoal samplers for the station should also contain rhodamine WT dye in accordance with the criteria listed above. These criteria may be waived if no charcoal sampler exists.

Criterion 2. There must be no factors which suggest that the fluorescence peak may not be rhodamine WT dye from the tracing work under investigation. The fluorescence peak should generally be in the range of 572.4 to 577.7 nm.

Criterion 3. The dye concentration associated with the fluorescence peak must be at least three times the detection limit. Our rhodamine WT detection limit in water samples is 0.015 ppb, thus this dye concentration limit is 0.045 ppb.

Criterion 4. The dye concentration must be at least 10 times greater than any other concentration reflective of background at the sampling station in question.

SULFORHODAMINE B

Normal Criteria Used by the OUL for Determining Sulforhodamine B Dye Recoveries in Elutants from Charcoal Samplers

Criterion 1. There must be at least one fluorescence peak in the sample in the range of 576.4 to 583.2 nm.

Criterion 2. The dye concentration associated with the sulforhodamine B peak must be at least 3 times the detection limit. The detection limit in elutant samples is 0.080 ppb, thus this dye concentration limit equals 0.240 ppb.

Criterion 3. The dye concentration must be at least 10 times greater than any other concentration reflective of background at the sampling station in question.

Criterion 4. The shape of the fluorescence peak must be typical of sulforhodamine B. In addition, there must be no other factors which suggest that the fluorescence peak may not be dye from the groundwater tracing work under investigation.

Normal Criteria Used by the OUL for Determining Sulforhodamine B dye Recoveries in Water Samples

Criterion 1. In most cases, the associated charcoal samplers for the station should also contain sulforhodamine B dye in accordance with the criteria listed earlier. This criterion may be waived if no charcoal sampler exists.

Criterion 2. There must be no factors which suggest that the fluorescence peak may not be sulforhodamine B dye from the tracing work under investigation. The fluorescence peak should generally be in the range of 580.8 to 584.4 nm.

Criterion 3. The dye concentration associated with the fluorescence peak must be at least three times the detection limit. The detection limit in water is 0.008 ppb, thus this dye concentration limit equals 0.024 ppb.

Criterion 4. The dye concentration must be at least 10 times greater than any other concentration reflective of background at the sampling station in question.

Standard Footnotes

Sometimes not all the criteria are met for a straight forward determination of tracer dye in a sample. For these reasons, the emission graph is scrutinized carefully by the analytical technician and again during the QA/QC process. Sometimes the emission graphs require interpretation as to whether or not a fluorescence peak represents the tracer dye or not. Background samples from each of the sampling stations aid in the interpretation of the emission fluorescence graphs. When the results do not meet all the criteria for a positive dye detection, often the fluorescence peak is quantified and flagged with a footnote to the result as not meeting all the criteria for a positive dye detection. Standard footnotes are as follows:

Single asterisk (*): A fluorescence peak is present that does not meet all the criteria for a positive dye recovery. However, it has been calculated as though it were the tracer dye.

Double asterisk (**): A fluorescence peak is present that does not meet all the criteria for this dye. However, it has been calculated as a positive dye recovery.

Other footnotes specific to the fluorescence signature are sometimes also used. These footnotes are often developed for a specific project.

The quantification of fluorescence peaks that do not meet all the criteria for a positive dye detection can be important for interpretation of the dataset as a whole.

ATTACHMENT 1

Sample Collection Data Sheet

OZARK UNDERGROUND LABORATORY, INC.

1572 Aley Lane Protem, MO 65733 (417) 785-4289 fax (417) 785-4290 email: contact@ozarkundergroundlab.com

SAMPLE COLLECTION DATA SHEET for FLUORESCENCE ANALYSIS

Project _____ **Week No:** ___ **Samples Collected By:** _____

Samples Shipped By: _____ **Samples Received By:** _____

Date Samples Shipped: _____ **Date Samples Received:** _____ **Time Samples Received:** _____ **Return Cooler? Yes** ☐ **No** ☐

Bill to: _____ **Send Results to:** _____

Analyze for: ☐ **Fluorescein** ☐ **Eosine** ☐ **Rhodamine WT** ☐ **Other** _____ **Ship cooler to:** _____

OUL use only		***Please indicate stations where dye was visible in the field*** *for field technician use - use black ink only*						*OUL use only*
# CHAR REC'D	**LAB NUMBER**	**STATION NUMBER 1-4 Numbers**	**STATION NAME**	**PLACED**		**COLLECTED**		**# WATER REC'D**
				DATE	**TIME**	**DATE**	**TIME**	

COMMENTS _____

_____

This sheet filled out by OUL staff? Yes ☐ **No** ☐ **Charts for samples on this page proofed by OUL:** _____

OUL Project No._____ **Date Analyzed:**_____ **Analyzed By:**_____

PCBs

By Capillary Column Gas Chromatography

Reference Methods: Method 8082A SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update IV, 2007.

Quality Control Requirements and Performance Standards for Analysis of Polychlorinated Biphenyls (PCBs) by Gas Chromatography (GC) in Support of Response Action under the Massachusetts Contingency Plan (MCP), Revision No.1, July 1, 2010.

State of Connecticut, Department of Environmental Protection, RRCP, Version 2.0, July 2006.

1. Scope and Application

Method 8082A is used to determine the concentrations of Polychlorinated Biphenyls (PCBs) as Aroclors in extracts from solid and liquid matrices. This SOP details the analysis for PCBs using fused-silica, open-tubular, capillary columns with electron capture detectors (ECD).
Matrices: Extracts from solid and liquid matrices.

Definitions: See Alpha Laboratories Quality Manual Appendix A

Regulatory Parameter List: The standard compounds listed below are determined by this method.

Parameter	CAS#
Aroclor 1016	12674-11-2
Aroclor 1221	11104-28-2
Aroclor 1232	11141-16-5
Aroclor 1242	53469-21-9
Aroclor 1248	12672-29-6
Aroclor 1254	11097-69-1
Aroclor 1260	11096-82-5
Aroclor 1262	37324-23-5
Aroclor 1268	11100-14-4

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Laboratory Director, or Quality Assurance Officer.
This method is restricted to use by or under the supervision of analysts experienced in the operation of the gas chromatograph (GC) and in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability (see section 13.2).

2. Summary of Method

A measured volume or weight of sample (volumes and weights can vary but approximately 1L or 125 mls (LVI – Low Volume Initiative) for liquids, 15g to 30g for solids) is extracted using the appropriate matrix-specific sample extraction technique.

Liquid samples are extracted at neutral pH with methylene chloride using Method 3510C (separatory funnel), or other appropriate technique. See extraction SOP for details.

Solid samples are extracted with methylene chloride: acetone (1:1) using Method 3540C (Soxhlet), or other appropriate technique. Solid samples may also be extracted with hexane:acetone (1:1) using Method 3546 (microwave). See extraction SOP for details.

Wipe samples are extracted with methylene chloride: acetone (1:1) using Method 3540C (Soxhlet) or other appropriate technique. See extraction SOP for details.

Oil samples are diluted with hexane following the procedure outlined in the extraction SOP.

Sulfuric acid cleanup (Method 3665A), Copper cleanup (Method 3660B) and Silica Gel cleanup (Method 3630) are utilized for PCB extracts. See extraction SOP for details.

After cleanup, the extract is analyzed by injecting 1µL into a gas chromatograph equipped with narrow- or wide-bore fused silica capillary columns and electron capture (GC/ECD) detectors.

2.1 Method Modifications from Reference

Not applicable.

3. Reporting Limits

The reporting limits for this method as outlined is as follows:

- Aqueous samples: 0.25 ug/L / Aroclor (based on a 1L extraction or 125 ml LVI extraction)
- Soil Samples: 33.3 ug/kg / Aroclor (based on a 15g extraction)
- Solid of Difficult Matrices (i.e Caulking, Concrete, etc. are logged using the Alpha Low Level 8082 products): based on a 15g extraction
    - Aroclors 1016, 1221, 1232, 1242, 1254: 20 ug/kg
    - Aroclors 1248, 1260: 13.3 ug/kg
    - Aroclors 1262, 1268: 6.67 ug/kg

4. Interferences

4.1 Instrumental

4.1.1 Only high purity gases are used in the GC system to eliminate this source of possible contamination. Both the helium (carrier gas – 99.999%) and argon-methane (detector make-up gas) are certified by the gas supplier.

4.1.2 Preventive instrument maintenance is performed routinely, and whenever highly contaminated extracts are analyzed that could result in chromatographic interferences or result in degradation of system performance. Section 10.5 details the maintenance steps.

4.1.3 Glassware must be scrupulously cleaned. This procedure is detailed in the Organic Extraction Cleaning and Handling SOP/1953. Store dry glassware in a clean environment.

4.2 Parameters

4.2.1 All solvents used are pesticide grade or equivalent, and reagents are purchased as certified contaminant free. All of these materials are routinely determined to be free of interferences by analysis of extraction blanks with every extraction batch performed.

4.2.2 Certain compounds (i.e. phthalates) can be extracted from the sample matrix and be detected by the ECD that could possibly result in false positive results or complicate the data interpretation. The use of the cleanup procedures detailed in the extraction SOP minimizes these possible interferences. Analyst experience is also crucial in making compound determinations.

4.2.3 Interferences co-extracted from the samples will vary considerably from waste to waste. While a general cleanup technique is referenced or provided as part of the method, unique samples may require additional cleanup approaches to achieve desired degrees of discrimination and quantitation.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound must be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. PCBs have been tentatively classified as known or suspected human or mammalian carcinogens. Additional references to laboratory safety are available in the Chemical Hygiene Plan.
All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

5.1 Lab coats, safety glasses, and gloves must be worn when handling samples, extracts, standards or solvents.

5.2 All solvent and extract transfers must be handled in the vented bench area in the GC laboratory.

5.3 All stock standards, working standards, and vialed sample extracts must be placed into the waste bucket in the lab, for future disposal by the Hazardous Waste Manager. The container must be labeled properly with hazard warning labels indicating the container contents.

5.4 Bottles containing flammable solvents must be stored in the flammables cabinet.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Aqueous samples are collected in two 1L or two 125 ml (LVI) amber glass jars with teflon-lined lids. Solid samples are collected in one 250 mL wide-mouth glass jar with a teflon-lined lid. All containers are purchased pre-cleaned and certified from commercial vendors.

6.2 Sample Preservation

Both aqueous and solid samples are then preserved by packing in coolers with ice or ice packs, to maintain a temperature of 4 ± 2° C. Upon receipt at the laboratory, the samples are transferred into sample storage refrigerators to maintain at a temperature of 4 ± 2° C.

6.3 Sample Handling

Aqueous samples must be extracted within 7 days of sample collection, solid samples within 14 days of collection. Once extracted, the samples must be analyzed within 40 days of the extraction date.

7. Equipment and Supplies

7.1 Gas Chromatograph, Agilent 6890, 7890: An analytical system complete with gas chromatograph configured for split-splitless injection and all required accessories including syringes, analytical columns, gases, electron capture detectors (ECD), and data system.

7.2 GC Columns: Alpha utilizes dual-column analyses. The dual-column approach involves either a single injection that is split between two columns that are mounted in a single gas chromatograph. Typical column pair used is listed below. Other columns may be used as long as method performance criteria can be met.

Column pair:

RTX-CLP: Cat. #11141 from Restek or equivalent; 30m, 0.32mm, 0.32µm

RTX-CLPII Cat. #11324 from Restek or equivalent; 30m, 0.32mm, 0.25µm

7.3 Guard Column: Cat. #10027 from Restek or equivalent; 5m, 0.32mm

7.4 Class "A" Volumetric Flasks: 10mL and 25mL (and other volumes), for standards preparation

7.5 Microsyringes/Wiretrol syringes: 10 µL – 1000 µL

7.6 Gooseneck splitless injecton liner, Cat #20799-214.5 from Restek or equivalent

7.7 Universal "Y" Press-tight tee split: Cat. #20406 from Restek or equivalent / **Siltek MXT Connector:** Cat. #21388 from Restek or equivalent

8. Reagents and Standards

Reagent grade or pesticide grade chemicals are used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficient high purity to permit its use without lessening the accuracy of the determination.
NOTE: Store the standard solutions (stock, composite, calibration, internal, and surrogate) at 4 ± 2° C in Teflon(R)-sealed containers in the dark. When a Lot of standards is prepared, aliquots of that Lot are stored in individual small vials. All stock standard solutions must be replaced after one year or sooner if routine QC tests indicate a problem. All other standard solutions must be replaced after six months or sooner if routine QC indicates a problem.

8.1 n-Hexane: Pesticide quality or equivalent.

8.2 Acetone: Pesticide quality or equivalent.

8.3 Organic-free Reagent Water: All references to water in this method refer to organic-free reagent water from Alpha's RO water treatment system.

8.4 Stock Standard Solutions: All stock standard solutions are purchased from commercial vendors as ampulated certified solutions. When an ampulated stock solution is opened, it is transferred to a labeled amber screw-cap vial. The expiration date of the stock solution is either the vendor specified expiration date, or 1 year from the date the ampule was opened, whichever is sooner.

8.5 Calibration Standards: Calibration standards are prepared volumetrically by diluting the appropriate stock standard(s) with hexane. Calibration standards expire 6 months from the date of preparation, or on the earliest expiration date of any of the stock solutions used to prepare the calibration standard. Calibrations are performed at the 6 concentration levels listed in Table 1. The list of ampulated calibration standards are obtain from **Ultra**:

- Aroclor 1016, Cat. #PP-282, at 100ug/ml
- Aroclor 1260, Cat. #PP-361, at 100ug/ml
- Aroclor 1262, Cat. #PP-371, at 100ug/ml
- Aroclor 1268, Cat. #PP-382, at 100ug/ml
- Aroclor 1221, Cat. #PP-292, at 100ug/ml
- Aroclor 1232, Cat. #PP-302, at 100ug/ml
- Aroclor 1242, Cat. #PP-312, at 100ug/ml
- Aroclor 1248, Cat. #PP-342, at 100ug/ml
- Aroclor 1254, Cat. #PP-351, at 100ug/ml

8.6 Second Source Standards: (ICV/CCAL) Continuing Calibration standards are prepared volumetrically by diluting the appropriate stock standard(s) with hexane. Continuing Calibration standards expire 6 months from the date of preparation, or on the earliest expiration date of any of the stock solutions used to prepare the standard. The list of ampulated standards are obtain from **Accustandard**:

- Aroclor 1016, Cat. #C-216S-H-10X, at 1000ug/ml
- Aroclor 1260, Cat. #C-260S-H-10X, at 1000ug/ml
- Aroclor 1262, Cat. #C-262S-H-10X, at 1000ug/ml
- Aroclor 1268, Cat. #C-268S-H-10X, at 1000ug/ml
- Aroclor 1221, Cat. #C-221S-H-10X, at 1000ug/ml
- Aroclor 1232, Cat. #C-232S-H-10X, at 1000ug/ml
- Aroclor 1242, Cat. #C-242S-H-10X, at 1000ug/ml
- Aroclor 1248, Cat. #C-248S-H-10X, at 1000ug/ml
- Aroclor 1254, Cat. #C-254S-H-10X, at 1000ug/ml

8.7 Internal Standard Solution: 1-Bromo-2-nitrobenzene (Ultra, Cat. #PPS-351) is used as the internal standard, and is added to all single-component calibration standards and sample extracts to achieve a concentration of 0.25µg/mL. For LVI, this solution is diluted 10X more, achieving a concentration of 0.025µg/mL.

8.8 Surrogate Standards: Tetrachloro-m-xylene (TCMX) and Decachlorobiphenyl (DCB) are used as surrogates for Aroclor analysis. They are added to the calibration standards at the concentrations listed in Table 1, Continuing Calibration Standards and are spiked into all samples and QC samples prior to extraction.

- **ICAL Surrogates Stock:** is prepared by diluting of 500ul of Pesticides Surrogates Standard Spiking Solution (Ultra, Cat. #ISM-320-1) and 500ul of Decachlorobiphenyl (Accustandard, Cat. #CLP-032-R-01) to 20ml of Hexane to achieve concentration of TCMX at 5ug/ml and DCB at 10ug/ml. For LVI, this solution is diluted 10X more, achieving a concentration of 0.5 ug/ml for TCMX and 0.1 ug/ml for DCB.
- **CCAL Surrogates Stock:** is prepared by diluting of 1ml of TCMX&DCB (Accustandard, Cat. #CLP-032-R) and 1ml of Decachlorobiphenyl (Accustandard, Cat. #CLP-032-R-01) to 20ml of Hexane to achieve concentration of TCMX at 10ug/ml and DCB at 20ug/ml. For LVI, this solution is diluted 10X more, achieving a concentration of 1 ug/ml for TCMX and 2 ug/ml for DCB.
- **Extraction Surrogates Stock:** is prepared by diluting of 10ml of TCMX&DCB (Accustandard, Cat. #CLP-032-R) to 1000ml of Acetone to achieve concentration of TCMX and DCB at 2ug/ml. For LVI, this solution is diluted 10X more, achieving a concentration of 0.2 ug/ml for both TCMX and DCB.

8.9 LCS/MS Spiking Solutions: The LCS/MS spiking solution is prepared by diluting of 6.25ml of Arochlor 1016/1260 (Restek, Cat. #32039) to 500ml of Acetone to achieve concentration of Arochlor 1016/1260 at 12.5ug/ml. For LVI, 1.25 ml of the stock solution is diluted to 500 mls of Acetone to achieve a concentration of Aroclor 1016/1260 at 2.5 ug/ml.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

A Method Blank is an aliquot of a clean reference matrix (reagent water for water samples or Ottawa sand for soil/sediment samples) that is carried through the entire analytical procedure. Extraction blanks are performed with each extraction batch of 20 or less samples, according to the extraction SOPs. The extraction blank must not contain any of the reportable analytes above the reporting limit. If any reportable analytes are detected in the blank, the entire extraction batch is suspect and re-extraction of all associated samples is required, unless the associated samples are non-detect or concentration of the analyte in the samples is 10 times greater than the concentration of this analyte in the blank. The surrogate recoveries must also be within the acceptance criteria listed in Table 2. If surrogate acceptance criteria are exceeded, the extraction batch must be evaluated to determine if re-extraction or re-analysis is necessary.

9.2 Laboratory Control Sample (LCS)

A Laboratory Control Sample (LCS)/ Laboratory Control Sample Duplicate (LCSD) pair is extracted with each analytical batch. The LCS/LCSD consist of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. For Aroclor analysis, the LCS/LCSD are spiked with a mixture of Aroclor 1016 and 1260. The recovery acceptance criteria are listed in Table 2. If any recovery criteria are not met, the extract may be re-analyzed. If the criteria are still not met, the **entire batch is re-extracted**, unless the recoveries are high and the associated samples are non-detect. If this is not possible, due to insufficient sample or holding time exceedances, the analyst must narrate the failure in the LIMS for inclusion in the client report.

9.3 Initial Calibration Verification (ICV)

Refer to Section 10.2.7.

9.4 Continuing Calibration Verification (CCV)

Refer to Section 10.4.

9.5 Matrix Spike

Upon client request, a matrix spike and matrix spike duplicate pair are extracted and analyzed with each batch of 20 or less samples. The MS/MSD pair is extracted and analyzed for standard PCB analysis. The recovery acceptance criteria are listed in Table 2. If the recovery criteria are not met, but are met in the LCS, the failure may be attributed to sample matrix effects and must be narrated for inclusion in the client report.

9.6 Laboratory Duplicate

Upon client request, a Laboratory Duplicate is extracted and analyzed with each batch of 20 or less samples. The relative percent difference (RPD) acceptance criteria are listed in Table 2. If the RPD criteria are not met, the failure may be attributed to matrix effect and must be narrated for inclusion in the client report.

9.7 Surrogates

All extracted samples and associated QC are spiked with Extraction Surrogates Stock to achieve concentration of TCMX and DCB at 0.5ug/ml (0.2 ug/ml for LVI). The laboratory must evaluate surrogate recovery data from individual samples and QC samples versus the surrogate control limits listed in Table 2. If the surrogate limits are not met, the extract may be reanalyzed to determine if the failure was due to an instrument problem. If the criteria are still not met, the affected samples must be re-extracted to confirm that the failure was due to sample matrix, unless the surrogate recovery is high and the associated sample is non-detect. If matrix effect is confirmed, this must be noted on a narrative sheet for inclusion in the client report.

9.8 Method Sequence

Typical Initial calibration (each level to identified with the standard lot number)

1. Prime
2. Blank
3. Standard Level 1
4. Standard Level 2
5. Standard Level 3

6. Standard Level 4
7. Standard Level 5
8. Standard Level 6
9. Initial Calibration Verification Standard (ICV)

Repeat steps 3 – 9 as needed for each Aroclor necessary for calibration.

NOTE: If multiple calibration mixtures are analyzed, it is acceptable to analyze appropriate ICVs after all calibration standards have been injected.

Typical Daily Sequence

1.1016/1260 Continuing Calibration Standard **(identified with the standard lot number)**
2. Extraction Blank
3. Laboratory Control Sample
4. Matrix Spike / Matrix Spike Duplicate (if requested by Client)
5. Duplicate (if included with batch QC)
6. Samples up to 16
7. Repeat 1 – 6 as needed.

10. Procedure

10.1 Equipment Set-up

10.1.1 GC Conditions:

The dual-column / dual-detector approach involves the use of the columns listed in section 7.2. The columns are connected to an injection tee or dual injection GC, and separate electron capture detectors. Alpha typical GC conditions are listed below, but may be altered as long as method performance criteria are met.

Temperature1: 120 ºC	Injector temerature: 250ºC
Time1: 0 minutes	Injector mode: Pulsed Split
Ramp1: 45ºC/minute	1.4:1 split, 0.20 min pulse
Temperature2: 200ºC	Injector Flow: 5.7 ml/min split flow
Time2: 0 minutes	Detector temperature: 350ºC
Ramp2: 15ºC/minute	Carrier gas: Helium
Temperature3: 230ºC	Carrier flow: 20ml/min
Time3: 0 minutes	Carrier mode: Constant flow
Ramp3: 30ºC/minute	Makeup gas: Argon/methane (P5)
Final temperature 330ºC	Total detector flow: 55ml/min
Final time: 2 minutes	Injection Volume: 1 µL

10.2 Initial Calibration

10.2.1 Prepare calibration standards using the standards listed in Section 8.5 to achieve the concentrations from Table 1. Alternatively, a standard containing a mixture of Aroclor 1016 and Aroclor 1260 will include many of the peaks represented in the other five Aroclor mixtures. As a result, a multi-point initial calibration employing a mixture of Aroclors 1016 and 1260 at five concentrations should be sufficient to demonstrate the linearity of the detector response without the necessity of performing multi-point initial calibrations for each of the seven Aroclors. In addition, such a mixture can be used as a standard to demonstrate that a sample does not contain peaks that represent any one of the Aroclors. Single standards of each of the other seven Aroclors are required to aid the analyst in pattern recognition. Assuming that the Aroclor 1016/1260 standards have been used to demonstrate the linearity of the detector, these single standards of the remaining seven Aroclors also may be used to determine the calibration factor for each Aroclor when a linear calibration model through the origin is chosen. Prepare a standard for each of the other Aroclors. The concentrations should generally correspond to the mid-point of the linear range of the detector, but lower concentrations may be employed.

10.2.2 Establish the GC operating conditions by loading the appropriate GC method. Typical instrument conditions are listed in section 10.1.1. The same operating conditions are used for calibrations and sample analyses. Create the analytical sequence using the Agilent Chemstation data acquisition software. Record the calibration standard, unique lot number (PP#) and analyst's initials in the analytical sequence list.

10.2.3 A 1µL injection volume of each calibration standard is typically used. Other injection volumes may be employed, provided that the analyst can demonstrate adequate sensitivity for the compounds of interest. The same injection volume must be used for all standards and samples.

10.2.4 Column adsorption may be a problem when the GC has not been used for a day or more or after system maintenance. The GC column may be primed (or deactivated) by injecting a PCB standard mixture approximately 20 times more concentrated than the mid-concentration standard. Inject this standard mixture prior to beginning the initial calibration or calibration verification.

Alternately, the system may be primed by baking at the final analytical temperature for approximately 30 minutes.

Several analytes may be observed in the injection just following system priming. Always run an instrument blank after system priming.

10.2.5 **Calibration Factor:** Internal standard calibration techniques are employed in this method.

10.2.5.1 Internal Standard Procedure. In each standard, calculate the response factor (RF) for each analyte, the average RF, and the relative standard deviation (RSD) of the RFs, using the Enviroquant data processing software. The calculations are performed automatically, using the formula listed in Alpha's Quality Manual.

Alternatively, standards of the other seven Aroclors are necessary for pattern recognition. When employing the traditional model of a linear calibration through the origin, these standards are also used to determine a single-point calibration factor for each Aroclor, assuming that the Aroclor 1016/1260 mixture has been

used to describe the detector response. The standards for these seven Aroclors should be analyzed before the analysis of any samples with hits above the RL. For example, an Aroclor 1254 standard should be analyzed before a sample with a hit of Aroclor 1254.

10.2.6 Initial Calibration Criteria

- If the **RSD for an analyte is < 20%**, then the response of the instrument for this compound is considered linear over the range and the mean calibration factor can be used to quantitate sample results.
- If the **RSD for any analyte is > 20%**, then linearity through the origin cannot be assumed. The mean response factor cannot be used for quantitation. An alternative calculation may be done by the use of **linear regression** or **quadratic regression** (minimum of six ICAL points are needed and regression must be weighted inversely proportional to concentration) as long as the correlation coefficient is **>0.990**. If both of these quantitation methods fail criteria for any compound in the initial calibration, then the system must be reevaluated and a new calibration curve must be analyzed. If quadratic regression is used for calibration, this must be noted in the laboratory narrative.
- **MCP requirement**: minimum of five unique peaks must be evaluated for Aroclors 1016 and 1260.
- **MCP requirement:** If linear or non-linear regression is used, RL must to be verified by recalculating concentrations in the lowest calibration standard using the final calibration curve. Recoveries must be **70-130%.**
- **MCP requirement:** Minimum of five standards (or six if non-linear regression used) must be used.

Initial Calibration Verification

An initial calibration verification standard must be run immediately after each initial calibration, near the midpoint of the curve. The standard must be prepared using a second source that is different than the source used for the initial calibration. (Standards listed in Section 8.6). The **%D** has to be within **20%** (**15% for CT RCP**) when compared to the mean response factor from the initial calibration.

10.2.7 Retention Time Window

10.2.7.1 The retention time window used for the identification of target analytes is ± 0.07 minutes. These criteria have been adopted from the EPA CLP Statement of Work (OLM04.2). It has been found that these limits work well, being wide enough to eliminate false-negatives while being tight enough to eliminate false-positives. Windows that are calculated using the procedure recommended in Method 8000 tend to be very narrow, creating the risk of false negative results.

10.2.7.2 The windows listed above are used as guidance; however the experience of the analyst weighs heavily in the interpretation of the chromatograms. For example, it has been observed that certain oil matrices can cause the retention times to shift more dramatically.

10.3 Sample Processing

The determination of PCB Aroclors is accomplished by comparing the sample chromatogram to that of the most similar Aroclor standard. The use of PCB overlays is extremely helpful, either by using hardcopies of chromatograms or by utilizing the Enviroquant software. A choice must be made as to which Aroclor is most similar and whether that standard is truly representative of the PCB in the sample. Both retention time and pattern are important when determining PCBs in a sample.

Samples that contained weathered PCB present special analytical challenges. Weathering could alter the Aroclor pattern to the extent that different peaks have to be selected for quantitation. Samples that contained more than one Aroclor present similar problems. For these samples, the Analyst may have to consider selecting the earlier eluting peaks for the lower boiling Aroclor and selecting the later eluting peaks for the higher boiling Aroclors to minimize overlapping peaks. Minimum of 3 peaks must be chosen for each Aroclor. In these instances, the Analyst may need request the assistance of someone with more expertise in determining the presence of PCB Aroclor.

If compound identification or quantitation is precluded due to interference (e.g., broad, rounded peaks or ill-defined baselines are present) cleanup of the extract may be needed. If instrument problems are suspected, rerun the extract on another instrument to determine if the problem results from analytical hardware or the sample matrix. Refer to the extraction SOPs for the procedures to be followed in sample cleanup.

The laboratory must report the ***HIGHER*** of the two results unless obvious interference is present on of the columns.

10.4 Continuing Calibration

10.4.1 Verify calibration each **12-hours** shift by injecting calibration verification standards prior to conducting any sample analyses. A calibration standard must also be injected at intervals of not less than **once every twenty injections**. A bracketing CCV is not required with the use of internal standard calibration (Method 8082A 11.6.8) with the exception of samples ran under CT RCP method. For Aroclor analysis, the calibration verification standard should be a mixture of Aroclor 1016 and 1260. The calibration verification process does not require analysis of the other Aroclor standards used for pattern recognition (Method 8082A 11.6.2). However, if the one-point calibration is used for the seven other Aroclor, a calibration standard must be analyzed before the sample for any hits.

10.4.2 The response factor (for internal standard compounds) for each analyte to be quantitated must not exceed a **± 20% difference** when compared to the initial calibration curve (**± 15% for CT RCP**). The Target data processing software automatically calculates the %D for all analytes according to the formulae in Alpha's Quality Manual. A retention time shift >30 seconds for the internal standard necessitates reanalysis of all affected samples.

10.5 Internal Standard

The use of internal standard calibration does not require that all sample results be bracketed with CCV standard. However, when internal standard calibration is used, the

retention times of internal standards and the area response of internal standards should be checked for each analysis.

10.5.1 **IS in CCAL** – The measured area of the internal standard must be no more than ± 50% different from the average area calculated during initial calibration **(-50 to 150%).**

10.5.2 **IS in samples** - The measured area of the internal standard must be no more than -50% to +100% different from the area calculated from opening CCV **(-50 to 200%)**

Retention time shifts of more than 30 sec from the retention time of the most recent calibration standard are cause for concern and must be investigated.

10.6 Preventive Maintenance

10.6.1 **Preventive Maintenance:** Routine preventive maintenance is performed to maintain GC system performance. This includes periodic replacement of injector septa, replacement of injector liner(s), and replacement of injector seals.

10.6.2 **Other Maintenance:** ECD detectors may become contaminated, requiring bake out at elevated temperatures, (no greater than 375°C) or repair by the manufacturer.

11. Data Evaluation, Calculations and Reporting

11.1 Quantitation of Aroclors

Per Method 8082A, quantitation is based on the use of a minimum of 3 of the major peaks present in the analyte, although the use of 5 of the major peaks is recommended. Each of these peaks is individually calibrated with a **minimum of five calibration points** based on average response factors. The %RSD must meet the criteria of ≤20% for the ICAL. The five major peaks are calculated as described below. After individual calculation meets criteria, the average of the peaks selected for quantitation is used to determine the final concentration.

11.1.1 Aqueous samples

$$\text{Concentration } (\mu g/L) = \frac{C \times DF \times Vf \times 1000}{Vo}$$

where:

C = Extract concentration (µg/mL), **NOTE**: ng on column = ng/injection volume = ng/uL = ug/mL

DF = Dilution factor

Vf = Final extract volume (mL)
Vo = Sample volume (mL)

11.1.2 Soil/sediment samples

$$\text{Concentration (µg/Kg, dry weight)} = \frac{C \times DF \times Vf \times 1000}{W\ (gm)} \div \%S$$

where:
C = Extract concentration (µg/mL), **NOTE**: ng on column = ng/injection volume = ng/uL = ug/mL
DF = Dilution factor
Vf = Final extract volume (mL)
W = Weight of the sample extracted (10g for high, 30g for low)
%S = Percent solids, as a decimal value

11.1.3 Reporting Results

11.1.3.1 After performing technical data review, validating that all QC criteria have been met and confirming all positive hits, the data report is sent electronically to the LIMS computer for generation of the client report. There are two levels of review of the data in the LIMS system prior to release of data. These reviews must be done by two separate individuals.

11.1.3.2 Reporting Results for PCBs in Caulk Samples

If in the screen sample Aroclor concentration as calculated above is **> 20000ppm**, the Client is contacted by a Customer Service Representative and these results are sent to the LIMS and reported to the Client.

If the sample concentration as calculated above for any Aroclor is **< 20000ppm**, the sample is sent for re-extraction by Method 3540C (Alpha SOP/1954).

11.1.3.3 Summation Rules

"TOTAL" concentrations are calculated for **ALL samples and Quality Control Samples** (i.e. LCS, MS, DUP, BLK).

TOTAL = sum of "reportable" Aroclors

Reportable- all Aroclors reported for associated project.

For dual-column analysis, Total is reported as part of column "A" data, unless all individuals are reported from "B" column. "Total" is calculated based on the associated "Report List". See Work Instruction #14335 for details.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedance and/or improper preservation are noted on the nonconformance report form.

Perform instrument maintenance as described throughout this SOP as needed when instrument calibration criteria are not met. Record all maintenance in the instrument logbook.

All batch and sample specific QC criteria outlined in Section 10 are evaluated by the analyst prior to approval of the data. When any QC criteria fail, the cause for the failure must be identified and corrected. This may include instrument recalibration followed by sample reanalysis, sample cleanup, or sample re-extraction. If it is determined that the failure is due to sample matrix effects, a project narrative report is written into the LIMS by the analyst for inclusion in the data report. If there is insufficient sample volume to perform the re-analysis for confirmation, this is also noted in the narrative and included in the client report.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/1732 MDL/LOD/LOQ Generation

SOP/1739 IDC/DOC Generation

SOP/1728 Waste Management and Disposal SOP

16. Attachments

Table 1: STANDARD SOLUTIONS

Table 2: QC ACCEPTANCE CRITERIA

TABLE 1

STANDARD SOLUTIONS – Suggested Concentrations

STANDARD SOLUTIONS	Stock solution (ug/mL)	Level 1 (ug/mL)	Level 2 (ug/mL)	Level 3 (ug/mL)	Level 4 (ug/mL)	Level 5 (ug/mL)	Level 6 (ug/mL)	Spike Solution (ug/mL)	LCS Solution (ug/mL)
PCB									
Aroclor 1016/1260	100	0.1	0.5	1	2.5	5	10	12.5	12.5
Aroclors 1221, 1232, 1242, 1254, 1262, 1268	100	0.1	0.5	1	2.5	5	10		
LVI		0.01	0.05	0.1	0.25	0.5	1	2.5	2.5
Internal Standard									
1-Bromo-2-Nitrobenzene	5000	0.25	0.25	0.25	0.25	0.25	0.25		
LVI		0.025	0.025	0.025	0.025	0.025	0.025		
Surrogates:									
Tetrachloro-m-xylene	2.0	0.0064	0.032	0.064	0.16	0.32	0.64	2	2
Decachlorobiphenyl	2.0	0.0126	0.064	0.128	0.32	0.64	1.28	2	2
LVI – 10X less								0.2	0.2

LVI is spiked 10X lower

TABLE 2

QC ACCEPTANCE CRITERIA

	Aqueous, Soils	
Surrogate % Recovery	**Lower Control Limit**	**Upper Control Limit**
2,4,5,6-Tetrachloro-m-xylene	30%	150%
Decachlorobiphenyl	30%	150%

	Aqueous, Soils % Recovery		Duplicate and/or MSD	
MS/MSD and LCS	**Lower Control Limit**	**Upper Control Limit**	**Aqueous RPD**	**Soil RPD**
Aroclor 1016, 1260	40%	140%	30%	50%

Determination of Selected Perfluorinated Alkyl Substances by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry Isotope Dilution (LC/MS/MS)

Reference: EPA Method 537, Version 1.1, September 2009, EPA Document #: EPA/600/R-08/09

EPA Method 537.1, Version 1, November 2018, EPA Document #: EPA/600/R-18/352

Department of Defense, Quality Systems Manual for Environmental Laboratories, Version 5.2, .2019

1. Scope and Application

Matrices: Drinking water, Non-potable Water, and Soil Matrices

Definitions: Refer to Alpha Analytical Quality Manual.

1.1 This is a liquid chromatography/tandem mass spectrometry (LC/MS/MS) method for the determination of selected perfluorinated alkyl substances (PFAS) in Non-Drinking Water and soil Matrices. Accuracy and precision data have been generated in reagent water, and finished ground and surface waters for the compounds listed in Table 1.

1.2 The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

1.3 This method is restricted to use by or under the supervision of analysts experienced in the operation of the LC/MS/MS and in the interpretation of LC/MS/MS data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

2.1 A 250-mL water sample is fortified with extracted internal standards (EIS) and passed through a solid phase extraction (WAX) cartridge containing a mixed mode, Weak Anion Exchange, reversed phase, water-wettable polymer to extract the method analytes and isotopically-labeled compounds. The compounds are eluted from the solid phase in two fractions with methanol followed by a small amount of 2% ammonium hydroxide in methanol solution. The extract is concentrated with nitrogen in a heated water bath, and then adjusted to a 1-mL volume with 80:20% (vol/vol) methanol:water. A 3 µl injection is made into an LC equipped with a C18 column that is interfaced to an MS/MS. The analytes are separated and identified by comparing the acquired mass spectra and retention times to reference spectra and retention times for calibration standards acquired under identical LC/MS/MS conditions. The concentration of each analyte is determined by using the isotope dilution technique. Extracted Internal Standards (EIS) analytes are used to monitor the extraction efficiency of the method analytes.

2.2 Method Modifications from Reference

None.

Table 1

Parameter	Acronym	CAS
PERFLUOROALKYL ETHER CARBOXYLIC ACIDS (PFECAs)		
Tetrafluoro-2-(heptafluoropropoxy)propanoic acid	HFPO-DA	62037-80-3
4,8-dioxa-3H-perfluorononanoic acid	ADONA	919005-14-4
PERFLUOROALKYLCARBOXILIC ACIDS (PFCAs)		
Perfluorobutanoic acid	PFBA	375-22-4
Perfluoropentanoic acid	PFPeA	2706-90-3
Perfluorohexanoic acid	PFHxA *	307-24-4
Perfluoroheptanoic acid	PFHpA *	375-85-9
Perfluorooctanoic acid	PFOA *	335-67-1
Perfluorononanoic acid	PFNA *	375-95-1
Perfluorodecanoic acid	PFDA *	335-76-2
Perfluoroundecanoic acid	PFUnA *	2058-94-8
Perfluorododecanoic acid	PFDoA *	307-55-1
Perfluorotridecanoic acid	PFTrDA *	72629-94-8
Perfluorotetradecanoic acid	PFTA *	376-06-7
Perfluorohexadecanoic acid	PFHxDA	67905-19-5
Perfluorooctadecanoic acid	PFODA	16517-11-6
PERFLUOROALKYLSULFONATES (PFASs)		
Perfluorobutanesulfonic acid	PFBS *	375-73-5
Perfluoropentanesulfonic acid	PFPeS	2706-91-4
Perfluorohexanesulfonic acid	PFHxS *	355-46-4
Perfluoroheptanesulfonic acid	PFHpS	375-92-8
Perfluorooctanesulfonic acid	PFOS *	1763-23-1
Perfluorononanesulfonic acid	PFNS	68259-12-1
Perfluorodecanesulfonic acid	PFDS	335-77-3
Perfluorododecanesulfonic acid	PFDoS	79780-39-5

*** also reportable via the standard 537 method**

Table 1 Cont.

Parameter	Acronym	CAS
CHLORO-PERFLUOROALKYLSULFONATE		
11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	11Cl-PF3OUdS	763051-92-9
9-chlorohexadecafluoro-3-oxanone-1-sulfonic acid	9Cl-PF3ONS	756426-58-1
PERFLUOROOCTANESULFONAMIDES (FOSAs)		
Perfluorooctanesulfonamide	PFOSA	754-91-6
N-methylperfluoro-1-octanesulfonamide	NMeFOSA	31506-32-8
N-ethylperfluoro-1-octanesulfonamide	NEtFOSA	4151-50-2
TELOMER SULFONATES		
1H,1H,2H,2H-perfluorohexane sulfonate (4:2)	4:2FTS	27619-93-8
1H,1H,2H,2H-perfluorooctane sulfonate (6:2)	6:2FTS	27619-97-2
1H,1H,2H,2H-perfluorodecane sulfonate (8:2)	8:2FTS	39108-34-4
1H,1H,2H,2H-perfluorododecane sulfonate (10:2)	10:2FTS	120226-60-0
PERFLUOROOCTANESULFONAMIDOACETIC ACIDS		
N-methyl perfluorooctanesulfonamidoacetic acid	NMeFOSAA *	2355-31-9
N-ethyl perfluorooctanesulfonamidoacetic acid	NEtFOSAA *	2991-50-6
NATIVE PERFLUOROOCTANESULFONAMIDOETHANOLS (FOSEs)		
2-(N-methylperfluoro-1-octanesulfonamido)-ethanol	NMeFOSE	24448-09-7
2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol	NEtFOSE	1691-99-2

*** also reportable via the standard 537 method**

3. Reporting Limits

The reporting limit for PFAS's is 2 ng/L for aqueous samples (20 ng/L for HFPO-DA) and 1 ng/g (10 ng/g for HFPO-DA) for soil samples.

4. Interferences

4.1 PFAS standards, extracts and samples should not come in contact with any glass containers or pipettes as these analytes can potentially adsorb to glass surfaces. PFAS analyte and EIS standards commercially purchased in glass ampoules are acceptable; however, all subsequent transfers or dilutions performed by the analyst must be prepared and stored in polypropylene containers.

4.2 Method interferences may be caused by contaminants in solvents, reagents (including reagent water), sample bottles and caps, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the chromatograms. The method analytes in this method can also be found in many common laboratory supplies and equipment, such

as PTFE (polytetrafluoroethylene) products, LC solvent lines, methanol, aluminum foil, SPE sample transfer lines, etc. All items such as these must be routinely demonstrated to be free from interferences (less than 1/3 the RL for each method analyte) under the conditions of the analysis by analyzing laboratory reagent blanks as described in Section 9.2. **Subtracting blank values from sample results is not permitted.**

4.3 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the water. Humic and/or fulvic material can be co-extracted during SPE and high levels can cause enhancement and/or suppression in the electrospray ionization source or low recoveries on the SPE sorbent. Total organic carbon (TOC) is a good indicator of humic content of the sample.

4.4 SPE cartridges can be a source of interferences. The analysis of field and laboratory reagent blanks can provide important information regarding the presence or absence of such interferences. Brands and lots of SPE devices should be tested to ensure that contamination does not preclude analyte identification and quantitation.

5. Health and Safety

5.1 The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

5.2 All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

5.3 PFOA has been described as “likely to be carcinogenic to humans.” Pure standard materials and stock standard solutions of these method analytes should be handled with suitable protection to skin and eyes, and care should be taken not to breathe the vapors or ingest the materials.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection for Aqueous Samples

6.1.1 Samples must be collected in two (2) 250-mL high density polyethylene (HDPE) container with an unlined plastic screw cap.

6.1.2 The sample handler must wash their hands before sampling and wear nitrile gloves while filling and sealing the sample bottles. PFAS contamination during sampling can occur from a number of common sources, such as food packaging and certain foods and beverages. Proper hand washing and wearing nitrile gloves will aid in minimizing this type of accidental contamination of the samples.

6.1.3 Open the tap and allow the system to flush until the water temperature has stabilized (approximately 3 to 5 min). Collect samples from the flowing system.

6.1.4 Fill sample bottles. Samples do not need to be collected headspace free.

6.1.5 After collecting the sample and cap the bottle. Keep the sample sealed from time of collection until extraction.

6.1.6 Field Reagent Blank (FRB)

6.1.6.1 A FRB must be handled along with each sample set. The sample set is composed of samples collected from the same sample site and at the same time. At the laboratory, fill the field blank sample bottle with reagent water and preservatives, seal, and ship to the sampling site along with the sample bottles. For each FRB shipped, an empty sample bottle (no preservatives) must also be shipped. At the sampling site, the sampler must open the shipped FRB and pour the reagent water into the empty shipped sample bottle, seal and label this bottle as the FRB. The FRB is shipped back to the laboratory along with the samples and analyzed to ensure that PFAS's were not introduced into the sample during sample collection/handling.

The reagent water used for the FRBs must be initially analyzed for method analytes as a MB and must meet the MB criteria in Section 9.2.1 prior to use. This requirement will ensure samples are not being discarded due to contaminated reagent water rather than contamination during sampling.

6.2 Sample Collection for Soil and Sediment samples.

Grab samples are collected in polypropylene containers. Sample containers and contact surfaces containing PTFE shall be avoided.

6.3 Sample Preservation

Not applicable.

6.4 Sample Shipping

Samples must be chilled during shipment and must not exceed 10 °C during the first 48 hours after collection. Sample temperature must be confirmed to be at or below 10 °C when the samples are received at the laboratory. Samples stored in the lab must be held at or below 6 °C until extraction, but should not be frozen.

NOTE: Samples that are significantly above 10° C, at the time of collection, may need to be iced or refrigerated for a period of time, in order to chill them prior to shipping. This will allow them to be shipped with sufficient ice to meet the above requirements.

6.5 Sample Handling

6.5.1 Holding Times

6.5.1.1 Water samples should be extracted as soon as possible but must be extracted within 14 days. Soil samples should be extracted within 28 days. Extracts are stored at < 10 ° C and analyzed within 28 days after extraction.

7. Equipment and Supplies

7.1 SAMPLE CONTAINERS – 250-mL high density polyethylene (HDPE) bottles fitted with unlined screw caps. Sample bottles must be discarded after use.

7.2 POLYPROPYLENE BOTTLES – 4-mL narrow-mouth polypropylene bottles.

7.3 CENTRIFUGE TUBES – 50-mL conical polypropylene tubes with polypropylene screw caps for storing standard solutions and for collection of the extracts.

7.4 AUTOSAMPLER VIALS – Polypropylene 0.7-mL autosampler vials with polypropylene caps.

7.4.1 NOTE: Polypropylene vials and caps are necessary to prevent contamination of the sample from PTFE coated septa. However, polypropylene caps do not reseal, so evaporation occurs after injection. Thus, multiple injections from the same vial are not possible.

7.5 POLYPROPYLENE GRADUATED CYLINDERS – Suggested sizes include 25, 50, 100 and 1000-mL cylinders.

7.6 Auto Pipets – Suggested sizes include 5, 10, 25, 50, 100, 250, 500, 1000, 5000 and 10,000-µls.

7.7 PLASTIC PIPETS – Polypropylene or polyethylene disposable pipets.

7.8 ANALYTICAL BALANCE – Capable of weighing to the nearest 0.0001 g.

7.9 SOLID PHASE EXTRACTION (SPE) APPARATUS FOR USING CARTRIDGES

7.9.1 SPE CARTRIDGES – 0.5 g SPE cartridges containing a reverse phase copolymer characterized by a weak anion exchanger (WAX) sorbent phase.

7.9.2 VACUUM EXTRACTION MANIFOLD – A manual vacuum manifold with large volume sampler for cartridge extractions, or an automatic/robotic sample preparation system designed for use with SPE cartridges, may be used if all QC requirements discussed in Section 9 are met. Extraction and/or elution steps may not be changed or omitted to accommodate the use of an automated system. Care must be taken with automated SPE systems to ensure the PTFE commonly used in these systems does not contribute to unacceptable analyte concentrations in the MB (Sect. 9.2.1).

7.9.3 SAMPLE DELIVERY SYSTEM – Use of a polypropylene transfer tube system, which transfers the sample directly from the sample container to the SPE cartridge, is recommended, but not mandatory. Standard extraction manifolds come equipped with PTFE transfer tube systems. These can be replaced with 1/8” O.D. x 1/16” I.D. polypropylene or polyethylene tubing cut to an appropriate length to ensure no sample contamination from the sample transfer lines. Other types of non-PTFE tubing may be used provided it meets the MB (Sect. 9.2.1) and LCS (Sect. 9.3) QC requirements. The PTFE transfer tubes may be used, but an MB must be run on each PFTE transfer tube and the QC requirements in Section 13.2.2 must be met. In the case of automated SPE, the removal of PTFE lines may not be feasible; therefore, MBs will need to be rotated among the ports and must meet the QC requirements of Sections 13.2.2 and 9.2.1.

7.10 Extract Clean-up Cartridge – 250 mg 6ml SPE Cartridge containing graphitized polymer carbon

7.11 EXTRACT CONCENTRATION SYSTEM – Extracts are concentrated by evaporation with nitrogen using a water bath set no higher than 65 °C.

7.12 LABORATORY OR ASPIRATOR VACUUM SYSTEM – Sufficient capacity to maintain a vacuum of approximately 10 to 15 inches of mercury for extraction cartridges.

7.13 LIQUID CHROMATOGRAPHY (LC)/TANDEM MASS SPECTROMETER (MS/MS) WITH DATA SYSTEM

7.13.1 LC SYSTEM – Instrument capable of reproducibly injecting up to 10-µL aliquots, and performing binary linear gradients at a constant flow rate near the flow rate used for development of this method (0.4 mL/min). The LC must be capable of pumping the water/methanol mobile phase without the use of a degasser which pulls vacuum on the mobile phase bottle (other types of degassers are acceptable). Degassers which pull vacuum on the mobile phase bottle will volatilize the ammonium acetate mobile phase causing the analyte peaks to shift to earlier retention times over the course of the analysis batch. The usage of a column heater is optional.

NOTE: During the course of method development, it was discovered that while idle for more than one day, PFAS's built up in the PTFE solvent transfer lines. To prevent long delays in purging high levels of PFAS's from the LC solvent lines, they were replaced with PEEK tubing and the PTFE solvent frits were replaced with stainless steel frits. It is not possible to remove all PFAS background contamination, but these measures help to minimize their background levels.

7.13.2 LC/TANDEM MASS SPECTROMETER – The LC/MS/MS must be capable of negative ion electrospray ionization (ESI) near the suggested LC flow rate of 0.4 mL/min. The system must be capable of performing MS/MS to produce unique product ions for the method analytes within specified retention time segments. A minimum of 10 scans across the chromatographic peak is required to ensure adequate precision.

7.13.3 DATA SYSTEM – An interfaced data system is required to acquire, store, reduce, and output mass spectral data. The computer software should have the capability of processing stored LC/MS/MS data by recognizing an LC peak within any given retention time window. The software must allow integration of the ion abundance of any specific ion within specified time or scan number limits. The software must be able to calculate relative response factors, construct linear regressions or quadratic calibration curves, and calculate analyte concentrations.

7.13.4 ANALYTICAL COLUMN – An LC BEH C_{18} column (2.1 x 50 mm) packed with 1.7 µm d_p C_{18} solid phase particles was used. Any column that provides adequate resolution, peak shape, capacity, accuracy, and precision (Sect. 9) may be used.

8. Reagents and Standards

8.1 GASES, REAGENTS, AND SOLVENTS – Reagent grade or better chemicals should be used.

8.1.1 REAGENT WATER – Purified water which does not contain any measurable quantities of any method analytes or interfering compounds greater than 1/3 the RL for each method analyte of interest. Prior to daily use, at least 3 L of reagent water should be flushed from the purification system to rinse out any build-up of analytes in the system's tubing.

8.1.2 METHANOL (CH_3OH, CAS#: 67-56-1) – High purity, demonstrated to be free of analytes and interferences.

8.1.3 AMMONIUM ACETATE ($NH_4C_2H_3O_2$, CAS#: 631-61-8) – High purity, demonstrated to be free of analytes and interferences.

8.1.4 ACETIC ACID (H_3CCOOH, CAS#: 64-19-7) - High purity, demonstrated to be free of analytes and interferences.

8.1.5 1M AMMONIUM ACETATE/REAGENT WATER – High purity, demonstrated to be free of analytes and interferences.

8.1.6 2mM AMMONIUM ACETATE/METHANOL:WATER (5:95) – To prepare, mix 2 ml of 1M AMMONIUM ACETATE,1 ml ACETIC ACID and 50 ml METHANOL into l Liter of REAGENT WATER.

8.1.7 Methanol/Water (80:20) – To prepare a 1 Liter bottle, mix 200 ml of REAGENT WATER with 800 ml of METHANOL.

8.1.8 AMMONIUM HYDROXIDE (NH_3, CAS#: 1336-21-6) – High purity, demonstrated to be free of analytes and interferences.

8.1.9 Sodium Acetate ($NaOOCCH_3$, CAS#: 127-09-3) – High purity, demonstrated to be free of analytes and interferences.

8.1.10 25 mM Sodium Acetate Buffer – To prepare 250mls, dissolve .625 grams of sodium acetate into 100 mls of reagent water. Add 4 mls Acetic Acid and adjust the final volume to 250 mls with reagent water.

8.1.11 NITROGEN – Used for the following purposes: Nitrogen aids in aerosol generation of the ESI liquid spray and is used as collision gas in some MS/MS instruments. The nitrogen used should meet or exceed instrument manufacturer's specifications. In addition, Nitrogen is used to concentrate sample extracts (Ultra High Purity or equivalent).

8.1.12 ARGON – Used as collision gas in MS/MS instruments. Argon should meet or exceed instrument manufacturer's specifications. Nitrogen gas may be used as the collision gas provided sufficient sensitivity (product ion formation) is achieved.

8.2 STANDARD SOLUTIONS – When a compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. PFAS analyte and IS standards commercially purchased in glass ampoules are acceptable; however, all subsequent transfers or dilutions performed by the analyst must be prepared and stored in polypropylene containers. Standards for sample fortification generally should be prepared in the smallest volume that can be accurately measured to minimize the addition of excess organic solvent to aqueous samples.

NOTE: Stock standards and diluted stock standards are stored at ≤4 °C.

8.2.1 ISOTOPE DILUTION Extracted Internal Standard (ID EIS) STOCK SOLUTIONS - ID EIS stock standard solutions are stable for at least 6 months when stored at 4 °C. The stock solution is purchased at a concentration of 1000 ng/mL.

8.2.2 ISOTOPE DILUTION Extracted Internal Standard PRIMARY DILUTION STANDARD (ID EIS PDS) – Prepare the ID EIS PDS at a concentration of 500 ng/mL. The ID PDS is prepared in 80:20% (vol/vol) methanol:water. The ID PDS is stable for 6 months when stored at ≤4 °C.

Table 2

Isotope Labeled Standard	Conc. of EIS Stock (ng/mL)	Vol. of EIS Stock (mL)	Final Vol. of EIS PDS (mL)	Final Conc. of EIS PDS (ng/mL)
M4PFBA	1000	1.0	2.0	500
M5PFPeA	1000	1.0	2.0	500
M5PFHxA	1000	1.0	2.0	500
M4PFHpA	1000	1.0	2.0	500
M8PFOA	1000	1.0	2.0	500
M9PFNA	1000	1.0	2.0	500
M6PFDA	1000	1.0	2.0	500
M7PFUdA	1000	1.0	2.0	500
MPFDoA	1000	1.0	2.0	500
M2PFTeDA	1000	1.0	2.0	500
M2PFHxDA	50,000	.02	2.0	500
d3-N-MeFOSA	50,000	.02	2.0	500
d5-N-EtFOSA	50,000	.02	2.0	500
d7-N-MeFOSE	50,000	.02	2.0	500
d9-N-EtFOSE	50,000	.02	2.0	500
M8FOSA	1000	1.0	2.0	500
d3-N-MeFOSAA	1000	1.0	2.0	500
d5-N-EtFOSAA	1000	1.0	2.0	500
M3PFBS	929	1.0	2.0	464.5
M3PFHxS	946	1.0	2.0	473
M8PFOS	957	1.0	2.0	478.5
M2-4:2FTS	935	1.0	2.0	467.5
M2-6:2FTS	949	1.0	2.0	474.5
M2-8:2FTS	958	1.0	2.0	479
M3HFPO-DA	50,000	.4	2.0	10,000

8.2.3 ANALYTE STOCK STANDARD SOLUTION – Analyte stock standards are stable for at least 6 months when stored at 4 °C. When using these stock standards to prepare a PDS, care must be taken to ensure that these standards are at room temperature and adequately vortexed.

8.2.4 Analyte Secondary Spiking Standard Prepare the spiking solution of additional add on components for project specific requirements only. ANALYTE PRIMARY SPIKING STANDARD – Prepare the spiking standard at a concentration of 500 ng/mL in methanol. The spiking standard is stable for at least two months when stored in polypropylene centrifuge tubes at room temperature.

Table 3

Analyte	Conc. of IS Stock (ng/mL)	Vol. of IS Stock (mL)	Final Vol. of IS PDS (mL)	Final Conc. of IS PDS (ng/mL)
PFBA	2000	1	4	500
PFPeA	2000	1	4	500
PFHxA	2000	1	4	500
PFHpA	2000	1	4	500
PFOA	2000	1	4	500
PFNA	2000	1	4	500
PFDA	2000	1	4	500
PFUdA	2000	1	4	500
PFDoA	2000	1	4	500
PFTrDA	2000	1	4	500
PFTeDA	2000	1	4	500
FOSA	2000	1	4	500
N-MeFOSAA	2000	1	4	500
N-EtFOSAA	2000	1	4	500
L-PFBS	1770	1	4	442.5
L-PFPeS	1880	1	4	470
L-PFHxSK	1480	1	4	370
Br-PFHxSK	344	1	4	86
L-PFHpS	1900	1	4	475
L-PFOSK	1460	1	4	365
Br-PFOSK	391	1	4	97.75
L-PFNS	1920	1	4	480
L-PFDS	1930	1	4	482.5
4:2FTS	1870	1	4	467.5
6:2FTS	1900	1	4	475
8:2FTS	1920	1	4	480

8.2.5 Analyte Secondary Spiking Standard Prepare the spiking solution of additional add on components for project specific requirements only.

Table 4

Analyte	Conc. of IS Stock (ng/mL)	Vol. of IS Stock (mL)	Final Vol. of IS PDS (mL)	Final Conc. of IS PDS (ng/mL)
ADONA	2000	1	4	500
PFHxDA	2000	1	4	500
PFODA	2000	1	4	500
HFPO-DA	100,000	.4	4	10,000
9ClPF3ONS	50,000	0.04	4	500
11ClPF3OUdS	50,000	0.04	4	500

8.2.6 LOW, MEDIUM AND HIGH LEVEL LCS – The LCS's will be prepared at the following concentrations and rotated per batch; 2 ng/L, 40 ng/L, 500 ng/l for drinking waters. The analyte PDS contains all the method analytes of interest at various concentrations in methanol. The analyte PDS has been shown to be stable for six months when stored at ≤4 °C.

8.2.7 Isotope Dilution Labeled Recovery Stock Solutions (ID REC) – ID REC Stock solutions are stable for at least 6 months when stored at 4 °C. The stock solution is purchased at a concentration of 1000 ng/mL.

8.2.8 Isotope Dilution Labeled Recovery Primary Dilution Standard (ID REC PDS) - Prepare the ID REC PDS at a concentration of 500 ng/mL. The ID REC PDS is prepared in 80:20% (vol/vol) methanol:water. The ID REC PDS is stable for at least six months when stored in polypropylene centrifuge tubes at ≤4 °C.

Table 5

Analyte	Conc. of REC Stock (ng/mL)	Vol. of REC Stock (mL)	Final Vol. of REC PDS (mL)	Final Conc. of REC PDS (ng/mL)
M2PFOA	2000	1	4	500
M2PFDA	2000	1	4	500
M3PFBA	2000	1	4	500
M4PFOS	2000	1	4	500

8.2.9 CALIBRATION STANDARDS (CAL) –

Current Concentrations (ng/mL): 0.5, 1.0, 5.0, 10.0, 50.0, 125, 150, 250, 500

Prepare the CAL standards over the concentration range of interest from dilutions of the analyte PDS in methanol containing 20% reagent water. 20 µl of the EIS PDS and REC PDS are added to the CAL standards to give a constant concentration of 10 ng/ml. The lowest concentration CAL standard must be at or below the RL (2 ng/L), which may depend on system sensitivity. The CAL standards may also be used as CCVs (Sect. 9.8). To make calibration stock standards:

Table 6

Calibration Standard Concentration	Final Aqueous Cal STD Level Concentration	Final Soil Cal STD Level Concentration	24 compound stock added (ul)	PFHxDA Stock added (ul)	500 ng/ml PFHxDA dilution added (ul)	PFODA Stock added (ul)	500 ng/ml PFODA dilution added (ul)	ADONA, HFPO-DA, 11Cl-PF3OUdS, 9Cl-PF3ONS Stock added (ul)	500 ng/ml ADONA dilution added (ul)	Final Volume in MeOH/H_2O (82:20)
.5 ng/ml	2 ng/L	.25 ng/g	6.25		25		25		25	25 mls
1 ng/ml	4 ng/L	.5 ng/g	5		20		20		20	10 mls
5 ng/ml	20 ng/L	1 ng/g	25		100		100		100	10 mls
10 ng/ml	40 ng/L	5 ng/g	125	5		5		5		25 mls

50 ng/ml	200 ng/L	25 ng/g	250	10		10		10		10 mls
125 ng/ml	500 ng/L	62.5 ng/g	625	25		25		25		10 mls
150 ng/ml	600 ng/L	75 ng/g	750	30		30		30		10 mls
250 ng/ml	1000 ng/L	125 ng/g	625							5 mls
500 ng/ml	2000 ng/L	250 ng/g	1250							5 mls

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 MINIMUM REPORTING LIMIT (MRL) CONFIRMATION

9.1.1 Fortify, extract, and analyze seven replicate LCSs at 2 ng/l. Calculate the mean measured concentration (*Mean*) and standard deviation for these replicates. Determine the Half Range for the prediction interval of results (HR_{PIR}) using the equation below

$$HR_{PIR} = 3.963s$$

Where:

s = the standard deviation
3.963 = a constant value for seven replicates.

9.1.2 Confirm that the upper and lower limits for the Prediction Interval of Result (*PIR* = *Mean* ± HR_{PIR}) meet the upper and lower recovery limits as shown below

The Upper PIR Limit must be ≤150% recovery.

$$\frac{Mean + HR_{PIR}}{Fortified\ Concentration} \times 100\% \leq 150\%$$

The Lower PIR Limit must be ≥ 50% recovery.

$$\frac{Mean - HR_{PIR}}{Fortified\ Concentration} \times 100\% \geq 50\%$$

9.1.3 The RL is validated if both the Upper and Lower PIR Limits meet the criteria described above. If these criteria are not met, the RL has been set too low and must be determined again at a higher concentration.

9.2 Blank(s)

9.2.1 **METHOD BLANK (MB) -** A Method Blank (MB) is required with each extraction batch to confirm that potential background contaminants are not interfering with the identification or quantitation of method analytes. Prep and analyze a MB for every 20 samples. If the MB produces a peak within the retention time window of any analyte that would prevent the determination of that analyte, determine the source of contamination and eliminate the interference before processing samples. Background contamination must be reduced to an acceptable level before proceeding. Background from method analytes or other contaminants that

interfere with the measurement of method analytes must be below the RL. If the method analytes are detected in the MB at concentrations equal to or greater than this level, then all data for the problem analyte(s) must be considered invalid for all samples in the extraction batch. Because background contamination is a significant problem for several method analytes, it is highly recommended that the analyst maintain a historical record of MB data.

9.2.2 **FIELD REAGENT BLANK (FRB)** - The purpose of the FRB is to ensure that PFAS's measured in the Field Samples were not inadvertently introduced into the sample during sample collection/handling. Analysis of the FRB is required only if a Field Sample contains a method analyte or analytes at or above the RL. The FRB is processed, extracted and analyzed in exactly the same manner as a Field Sample.

9.3 Laboratory Control Sample (LCS) and Laboratory Control Sample Duplicates (LCSD)

9.3.1 An LCS is required with each extraction batch. The fortified concentration of the LCS may be rotated between low, medium, and high concentrations from batch to batch. Default limits of 50-150% of the true value may be used for analytes until sufficient replicates have been analyzed to generate proper control limits. Calculate the percent recovery (%*R*) for each analyte using the equation

$$\%R = \frac{A \times 100}{B}$$

Where:

A = measured concentration in the fortified sample
B =fortification concentration.

9.3.2 Where applicable, LCSD's are to be extracted and analyzed. The concentration and analyte recovery criteria for the LCSD must be the same as the batch LCS The RSD's must fall within ≤30% of the true value for medium and high level replicates, and ≤50% for low level replicates. Calculate the relative percent difference (RPD) for duplicate MSs (MS and MSD) using the equation

$$RPD = \frac{|LCS - LCSD|}{(LCS + LCSD) / 2} \times 100$$

9.3.3 **If the LCS and or LCSD results do not meet these criteria for method analytes, then all data for the problem analyte(s) must be considered invalid for all samples in the extraction batch.**

9.4 Labeled Recovery Standards (REC)

The analyst must monitor the peak areas of the REC(s) in all injections during each analysis day.

9.5 Extracted Internal Standards (EIS)

9.5.1 The EIS standard is fortified into all samples, CCVs, MBs, LCSs, MSs, MSDs, FD, and FRB prior to extraction. It is also added to the CAL standards. The EIS is a means of assessing method performance from extraction to final

chromatographic measurement. Calculate the recovery (%R) for the EIS using the following equation

$$\%R = (A / B) \times 100$$

Where:

A = calculated EIS concentration for the QC or Field Sample
B = fortified concentration of the EIS.

9.5.2 Default limits of 50-150% may be used for analytes until sufficient replicates have been analyzed to generate proper control limits. A low or high percent recovery for a sample, blank, or CCV does not require discarding the analytical data but it may indicate a potential problem with future analytical data. When EIS recovery from a sample, blank, or CCV are outside control limits, check 1) calculations to locate possible errors, 2) standard solutions for degradation, 3) contamination, and 4) instrument performance. For CCVs and QC elements spiked with all target analytes, if the recovery of the corresponding target analytes meet the acceptance criteria for the EIS in question, the data can be used but all potential biases in the recovery of the EIS must be documented in the sample report. If the associated target analytes do not meet the acceptance criteria,the data must be reanalyzed.

9.6 Matrix Spike (MS)

9.6.1 Analysis of an MS is required in each extraction batch and is used to determine that the sample matrix does not adversely affect method accuracy. Assessment of method precision is accomplished by analysis of a Field Duplicate (FD) (Sect. 9.6); however, infrequent occurrence of method analytes would hinder this assessment. If the occurrence of method analytes in the samples is infrequent, or if historical trends are unavailable, a second MS, or MSD, must be prepared, extracted, and analyzed from a duplicate of the Field Sample. Extraction batches that contain MSDs will not require the extraction of a field sample duplicate. If a variety of different sample matrices are analyzed regularly, for example, drinking water from groundwater and surface water sources, method performance should be established for each. Over time, MS data should be documented by the laboratory for all routine sample sources.

9.6.2 Within each extraction batch, a minimum of one Field Sample is fortified as an MS for every 20 Field Samples analyzed. The MS is prepared by spiking a sample with an appropriate amount of the Analyte Stock Standard (Sect. 8.2.3). Use historical data and rotate through the low, mid and high concentrations when selecting a fortifying concentration. Calculate the percent recovery (%*R*) for each analyte using the equation

$$\%R = \frac{(A - B)}{C} \times 100$$

Where:

A = measured concentration in the fortified sample
B = measured concentration in the unfortified sample
C = fortification concentration.

9.6.3 Analyte recoveries may exhibit matrix bias. For samples fortified at or above their native concentration, recoveries should range between 50-150%. If the accuracy of any analyte falls outside the designated range, and the laboratory performance for that analyte is shown to be in control in the LCS, the recovery is judged to be

matrix biased. The result for that analyte in the unfortified sample is labeled suspect/matrix to inform the data user that the results are suspect due to matrix effects.

9.7 Laboratory Duplicate

9.7.1 FIELD DUPLICATE OR LABORATORY FORTIFIED SAMPLE MATRIX DUPLICATE (FD or MSD) – Within each extraction batch (not to exceed 20 Field Samples), a minimum of one FD or MSD must be analyzed. Duplicates check the precision associated with sample collection, preservation, storage, and laboratory procedures. If method analytes are not routinely observed in Field Samples, an MSD should be analyzed rather than an FD.

9.7.2 Calculate the relative percent difference (*RPD*) for duplicate measurements (*FD1* and *FD2*) using the equation

$$RPD = \frac{|FD1 - FD2|}{(FD1 + FD2) / 2} \times 100$$

9.7.3 RPDs for FDs should be ≤30%. Greater variability may be observed when FDs have analyte concentrations that are within a factor of 2 of the RL. At these concentrations, FDs should have RPDs that are ≤50%. If the RPD of any analyte falls outside the designated range, and the laboratory performance for that analyte is shown to be in control in the CCV, the recovery is judged to be matrix biased. The result for that analyte in the unfortified sample is labeled suspect/matrix to inform the data user that the results are suspect due to matrix effects.

9.7.4 If an MSD is analyzed instead of a FD, calculate the relative percent difference (RPD) for duplicate MSs (MS and MSD) using the equation

$$RPD = \frac{|MS - MSD|}{(MS + MSD) / 2} \times 100$$

9.7.5 RPDs for duplicate MSs should be ≤30% for samples fortified at or above their native concentration. Greater variability may be observed when MSs are fortified at analyte concentrations that are within a factor of 2 of the RL. MSs fortified at these concentrations should have RPDs that are ≤50% for samples fortified at or above their native concentration. If the RPD of any analyte falls outside the designated range, and the laboratory performance for that analyte is shown to be in control in the LCSD where applicable, the result is judged to be matrix biased. If no LCSD is present, the associated MS and MSD are to be re-analyzed to determine if any analytical has occurred. If the resulting RPDs are still outside control limits, the result for that analyte in the unfortified sample is labeled suspect/matrix to inform the data user that the results are suspect due to matrix effects.

9.8 Initial Calibration Verification (ICV)

9.8.1 As part of the IDC (Sect. 13.2), and after each ICAL, analyze a QCS sample from a source different from the source of the CAL standards. If a second vendor is not available, then a different lot of the standard should be used. The QCS should be prepared and analyzed just like a CCV. Acceptance criteria for the QCS are identical to the CCVs; the calculated amount for each analyte must be ±

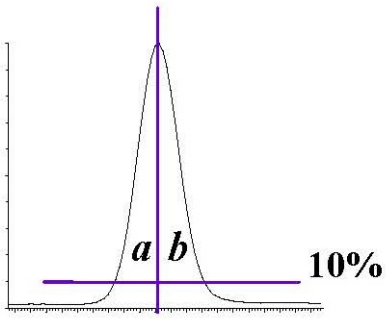
30% of the expected value. If measured analyte concentrations are not of acceptable accuracy, check the entire analytical procedureto locate and correct the problem.

9.9 Continuing Calibration Verification (CCV)

9.9.1 CCV Standards are analyzed at the beginning of each analysis batch, after every 10 Field Samples, and at the end of the analysis batch. See Section 10.7 for concentration requirements and acceptance criteria.

9.10 Method-specific Quality Control Samples

9.10.1 PEAK ASYMMETRY FACTOR – A peak asymmetry factor must be calculated using the equation below during the IDL and every time a calibration curve is generated. The peak asymmetry factor for the first two eluting peaks in a midlevel CAL standard (if only two analytes are being analyzed, both must be evaluated) must fall in the range of 0.8 to 1.5. Modifying the standard or extract composition to more aqueous content to prevent poor shape is not permitted. See guidance in Section 10.6.4.1 if the calculated peak asymmetry factors do not meet the criteria.



$$A_s = b / a$$

Where:

A_s = peak asymmetry factor
b = width of the back half of the peak measured (at 10% peak height) from the trailing edge of the peak to a line dropped perpendicularly from the peak apex
a = the width of the front half of the peak measured (at 10% peak height) from the leading edge of the peak to a line dropped perpendicularly from the apex.

9.11 Method Sequence

- CCV-LOW
- MB
- LCS
- LCSD
- MS
- Duplicate or MSD
- Field Samples (1-10)
- CCV-MID
- Field Samples (11-20)
- CCV-LOW

10. Procedure

10.1 Equipment Set-up

10.1.1 This procedure may be performed manually or in an automated mode using a robotic or automatic sample preparation device. If an automated system is used to prepare samples, follow the manufacturer's operating instructions, but all extraction and elution steps must be the same as in the manual procedure. Extraction and/or elution steps may not be changed or omitted to accommodate the use of an automated system. If an automated system is used, the MBs should be rotated among the ports to ensure that all the valves and tubing meet the MB requirements (Sect. 9.2).

10.1.2 Some of the PFAS's adsorb to surfaces, including polypropylene. Therefore, the aqueous sample bottles must be rinsed with the elution solvent (Sect 10.3.4) whether extractions are performed manually or by automation. The bottle rinse is passed through the cartridge to elute the method analytes and is then collected (Sect. 10.3.4).

10.1.3 **NOTE:** The SPE cartridges and sample bottles described in this section are designed as single use items and should be discarded after use. They may not be refurbished for reuse in subsequent analyses.

10.2 Sample Preparation and Extraction of Aqueous Samples

10.2.1 Samples are preserved, collected and stored as presented in Section 6.

The entire sample that is received must be sent through the SPE cartridge. In addition, the bottle must be solvent rinsed and this rinse must be sent through the SPE cartridge as well. The method blank (MB) and laboratory control sample (LCS) must be extracted in exactly the same manner (i.e., must include the bottle solvent rinse). It should be noted that a water rinse alone is not sufficient. This does not apply to samples with high concentrations of PFAS that are prepared using serial dilution and not SPE.

10.2.2 Determine sample volume. Weigh all samples to the nearest 1g. If visible sediment is present, centrifuge and decant into a new 250mL HDPE bottle and record the weight of the new container.

NOTE: Some of the PFAS's adsorb to surfaces, thus the sample volume may **NOT** be transferred to a graduated cylinder for volume measurement.

10.2.3 The MB, LCS and FRB may be prepared by measuring 250 mL of reagent water with a polypropylene graduated cylinder or filling a 250-mL sample bottle to near the top.

10.2.4 Adjust the QC and sample pH to 3 by adding acetic acid in water dropwise

10.2.5 Add 20 µL of the EIS PDS (Sect. 8.2.2) to each sample and QC, cap and invert to mix.

10.2.6 If the sample is an LCS, LCSD, MS, or MSD, add the necessary amount of analyte PDS (Sect. 8.2.3). Cap and invert each sample to mix.

10.3 Cartridge SPE Procedure

10.3.1 CARTRIDGE CLEAN-UP AND CONDITIONING – DO NOT allow cartridge packing material to go dry during any of the conditioning steps. Rinse each cartridge with 3 X 5 mL of 2% ammonium hydroxide in methanol, followed by 5mls of methanol. Next, rinse each cartridge with 5 mls of the 25 mM acetate buffer, followed by 15 mL of reagent water, without allowing the water to drop below the top edge of the packing. If the cartridge goes dry during the conditioning phase, the conditioning must be started over. Add 4-5 mL of reagent water to each cartridge, attach the sample transfer tubes (Sect. 7.9.3), turn on the vacuum, and begin adding sample to the cartridge.

10.3.2 SAMPLE EXTRACTON – Adjust the vacuum so that the approximate flow rate is approximately 4 mL/min. Do not allow the cartridge to go dry before all the sample has passed through.

10.3.3 SAMPLE BOTTLE AND CARTRIDGE RINSE – After the entire sample has passed through the cartridge, rinse the sample bottles with 4 ml reagent water followed by 4 ml 25 mM acetate buffer at pH 4 and draw the aliquot through the sample transfer tubes and the cartridges. Draw air or nitrogen through the cartridge for 5-10 min at high vacuum (10-15 in. Hg). **NOTE: If empty plastic reservoirs are used in place of the sample transfer tubes to pass the samples through the cartridges, these reservoirs must be treated like the transfer tubes. After the entire sample has passed through the cartridge, the reservoirs must be rinsed to waste with reagent water.**

10.3.4 SAMPLE BOTTLE AND CARTRIDGE ELUTION, Fraction 1 – Turn off and release the vacuum. Lift the extraction manifold top and insert a rack with collection tubes into the extraction tank to collect the extracts as they are eluted from the cartridges. Rinse the sample bottles with 12 mls of methanol and draw the aliquot through the sample transfer tubes and cartridges. Use a low vacuum such that the solvent exits the cartridge in a dropwise fashion.

SAMPLE BOTTLE AND CARTRIDGE ELUTION, Fraction 2 In a separate collection vial, rinse the sample bottles with 12 mL of 2% ammonium hydroxide in methanol and elute the analytes from the cartridges by pulling the 4 mL of methanol through the sample transfer tubes and the cartridges. Use a low vacuum such that the solvent exits the cartridge in a dropwise fashion. To the final extract, add 50 ul of acetic acid.

NOTE: If empty plastic reservoirs are used in place of the sample transfer tubes to pass the samples through the cartridges, these reservoirs must be treated like the transfer tubes. After the reservoirs have been rinsed in Section 10.3.3, the elution solvent used to rinse the sample bottles must be swirled down the sides of the reservoirs while eluting the cartridge to ensure that any method analytes on the surface of the reservoirs are transferred to the extract.

CLEAN-UP CARTRIDGE ELUTION, Elute the clean-up cartridge with 8 additional mls of methanol and draw the aliquot through the cartridge. Use a low vacuum such that the solvent exits the cartridge in a dropwise fashion.

10.3.5 Fractions 1 and 2 are to be combined during the concentration stage (section10.6)

10.4 Sample Prep and Extraction Protocol for Soils

10.4.1 Homogenize and weigh 2 grams of sample (measured to the nearest hundredth of a gram) intoa 50 ml polypropylene centrifuge tube. For laboratory control blanks and spikes, 2 grams of clean sand is used.

10.4.2 Add 20 µL of the EIS PDS (Sect. 8.2.2) to each sample and QC.

10.4.3 If the sample is an LCS, LCSD, MS, or MSD, add the necessary amount of analyte PDS (Sect. 8.2.3). Cap and invert each sample to mix.

10.4.4 To all samples, add 10 mls of methanol, cap, vortex for 25 seconds at 3000RPM and mix for 30 minutes using a shaker table of tumbler at 120RPM.

10.4.5 Following mixing, sonicate each sample for 30 minutes and let samples sit overnight (at least 2 hours is required for RUSH samples).

10.4.6 Centrifuge each sample at 3500RPM for 10 minutes.

10.4.7 Remove supernatant, and reserve for clean-up.

10.5 Extract Clean-up

10.5.1 CARTRIDGE CLEAN-UP AND CONDITIONING –. Rinse each cartridge with 15 mL of methanol and discard. If the cartridge goes dry during the conditioning phase, the conditioning must be started over. Attach the sample transfer tubes (Sect. 7.9.3), turn on the vacuum, and begin adding sample to the cartridge.

10.5.2 Adjust the vacuum so that the approximate flow rate is 1-2 mL/min. Do not allow the cartridge to go dry before all the sample has passed through.

10.5.3 SAMPLE BOTTLE AND CARTRIDGE RINSE – After the entire sample has passed through the cartridge, rinse the sample collection vial with two 1-mL aliquots of methanol and draw each aliquot through the cartridges. Draw air or nitrogen through the cartridge for 5 min at high vacuum (10-15 in. Hg).

10.5.4 If extracts are not to be immediately evaporated, cover collection tubes and store at ambient temperature till concentration.

10.6 Extract Concentration

10.6.1 Concentrate the extract to dryness under a gentle stream of nitrogen in a heated water bath (60-65 °C) to remove all the water/methanol mix. Add the appropriate amount of 80:20% (vol/vol) methanol:water solution and 20 µl of the ID REC PDS (Sect. 8.2.7) to the collection vial to bring the volume to 1 mL and vortex. Transfer two aliquots with a plastic pipet (Sect. 7.6) into 2 polypropylene autosampler vials.

NOTE: It is recommended that the entire 1-mL aliquot not be transferred to the autosampler vial because the polypropylene autosampler caps do not reseal after injection. Therefore, do not store the extracts in the autosampler vials as evaporation losses can occur occasionally in these autosampler vials. Extracts can be split between 2 X 700 µl vials (Sect. 7.4).

10.7 Sample Volume Determination

10.7.1 If the level of the sample was marked on the sample bottle, use a graduated cylinder to measure the volume of water required to fill the original sample bottle to the mark made prior to extraction. Determine to the nearest 10 mL.

10.7.2 If using weight to determine volume, weigh the empty bottle to the nearest 10 g and determine the sample weight by subtraction of the empty bottle weight from the original sample weight (Sect. 10.2.2). Assume a sample density of 1.0 g/mL. In either case, the sample volume will be used in the final calculations of the analyte concentration (Sect. 11.2).

10.8 Initial Calibration - Demonstration and documentation of acceptable initial calibration is required before any samples are analyzed. After the initial calibration is successful, a CCV is required at the beginning and end of each period in which analyses are performed, and after every tenth Field Sample.

10.8.1 ESI-MS/MS TUNE

10.8.1.1 Calibrate the mass scale of the MS with the calibration compounds and procedures prescribed by the manufacturer.

10.8.1.2 Optimize the [M-H]- for each method analyte by infusing approximately 0.5-1.0 µg/mL of each analyte (prepared in the initial mobile phase conditions) directly into the MS at the chosen LC mobile phase flow rate (approximately 0.4 mL/min). This tune can be done on a mix of the method analytes. The MS parameters (voltages, temperatures, gas flows, etc.) are varied until optimal analyte responses are determined. The method analytes may have different optima requiring some compromise between the optima.

10.8.1.3 Optimize the product ion for each analyte by infusing approximately 0.5-1.0 µg/mL of each analyte (prepared in the initial mobile phase conditions) directly into the MS at the chosen LC mobile phase flow rate (approximately 0.4 mL/min). This tune can be done on a mix of the method analytes. The MS/MS parameters (collision gas pressure, collision energy, etc.) are varied until optimal analyte responses are determined. Typically, the carboxylic acids have very similar MS/MS conditions and the sulfonic acids have similar MS/MS conditions.

10.8.2 Establish LC operating parameters that optimize resolution and peak shape. Modifying the standard or extract composition to more aqueous content to prevent poor shape is not permitted.

Cautions: LC system components, as well as the mobile phase constituents, contain many of the method analytes in this method. Thus, these PFAS's will build up on the head of the LC column during mobile phase equilibration. To minimize the background PFAS peaks and to keep background levels constant, the time the LC column sits at initial conditions must be kept constant and as short as possible (while ensuring reproducible retention times). In addition, prior to daily use, flush the column with 100% methanol for at least 20 min before initiating a sequence. It may be necessary on some systems to flush other LC components such as wash syringes, sample needles or any other system components before daily use.

10.8.3 Inject a mid-level CAL standard under LC/MS conditions to obtain the retention times of each method analyte. If analyzing for PFTA, ensure that the LC

conditions are adequate to prevent co-elution of PFTA and the mobile phase interferants. These interferants have the same precursor and products ions as PFTA, and under faster LC conditions may co-elute with PFTA. Divide the chromatogram into retention time windows each of which contains one or more chromatographic peaks. During MS/MS analysis, fragment a small number of selected precursor ions ([M-H]-) for the analytes in each window and choose the most abundant product ion. For maximum sensitivity, small mass windows of ±0.5 daltons around the product ion mass were used for quantitation.

10.8.4 Inject a mid-level CAL standard under optimized LC/MS/MS conditions to ensure that each method analyte is observed in its MS/MS window and that there are at least 10 scans across the peak for optimum precision.

10.8.4.1 If broad, split or fronting peaks are observed for the first two eluting chromatographic peaks (if only two analytes are being analyzed, both must be evaluated), change the initial mobile phase conditions to higher aqueous content until the peak asymmetry ratio for each peak is 0.8 – 1.5. The peak asymmetry factor is calculated as described in Section 9.9.1 on a mid-level CAL standard. The peak asymmetry factor must meet the above criteria for the first two eluting peaks during the IDL and every time a new calibration curve is generated. Modifying the standard or extract composition to more aqueous content to prevent poor shape is not permitted.

NOTE: PFHxS, PFOS, NMeFOSAA, and NEtFOSAA have multiple chromatographic peaks using the LC conditions in Table 5 due to chromatographic resolution of the linear and branched isomers of these compounds. Most PFAS's are produced by two different processes. One process gives rise to linear PFAS's only while the other process produces both linear and branched isomers. Thus, both branched and linear PFAS's can potentially be found in the environment. For the aforementioned compounds that give rise to more than one peak, all the chromatographic peaks observed in the standard must be integrated and the areas totaled. Chromatographic peaks in a sample must be integrated in the same way as the CAL standard.

10.8.5 Prepare a set of CAL standards as described in Section 8.2.5. The lowest concentration CAL standard must be at or below the RL (2 ng/L), which may depend on system sensitivity.

10.8.6 The LC/MS/MS system is calibrated using the IS technique. Use the LC/MS/MS data system software to generate a linear regression or quadratic calibration curve for each of the analytes. This curve **must always** be forced through zero and may be concentration weighted, if necessary. Forcing zero allows for a better estimate of the background levels of method analytes. A minimum of 5 levels are required for a linear calibration model and a minimum of 6 levels are required for a quadratic calibration model.

10.8.7 CALIBRATION ACCEPTANCE CRITERIA – A linear fit is acceptable if the coefficient of determination (r^2) is greater than 0.99. When quantitated using the initial calibration curve, each calibration point, except the lowest point, for each analyte should calculate to be within 70-130% of its true value. The lowest CAL point should calculate to be within 50-150% of its true value. If these criteria cannot be met, the analyst will have difficulty meeting ongoing QC criteria. It is

recommended that corrective action is taken to reanalyze the CAL standards, restrict the range of calibration, or select an alternate method of calibration (forcing the curve through zero is still required).

10.8.7.1 CAUTION: When acquiring MS/MS data, LC operating conditions must be carefully reproduced for each analysis to provide reproducible retention times. If this is not done, the correct ions will not be monitored at the appropriate times. As a precautionary measure, the chromatographic peaks in each window must not elute too close to the edge of the segment time window.

10.9 CONTINUING CALIBRATION CHECK (CCV) – Minimum daily calibration verification is as follows. Verify the initial calibration at the beginning and end of each group of analyses, and after every tenth sample during analyses. In this context, a "sample" is considered to be a Field Sample. MBs, CCVs, LCSs, MSs, FDs FRBs and MSDs are not counted as samples. The beginning CCV of each analysis batch must be at or below the RL in order to verify instrument sensitivity prior to any analyses. If standards have been prepared such that all low CAL points are not in the same CAL solution, it may be necessary to analyze two CAL standards to meet this requirement. Alternatively, the analyte concentrations in the analyte PDS may be customized to meet these criteria. Subsequent CCVs should alternate between a medium and Low concentration CAL standard.

10.9.1 Inject an aliquot of the appropriate concentration CAL standard and analyze with the same conditions used during the initial calibration.

10.9.2 Calculate the concentration of each analyte and EIS in the CCV. The calculated amount for each analyte for medium level CCVs must be within ± 30% of the true value with an allowance of 10% of the reported analytes to be greater than 30%, but less than 40%. The calculated amount for each EIS must be within ± 50% of the true value. The calculated amount for the lowest calibration point for each analyte must be within ± 50%. If these conditions do not exist, then all data for the problem analyte must be considered invalid, and remedial action should be taken (Sect. 10.7.4) which may require recalibration. Any Field or QC Samples that have been analyzed since the last acceptable calibration verification should be reanalyzed after adequate calibration has been restored, with the following exception. **If the CCV fails because the calculated concentration is greater than 130% (150% for the low-level CCV) for a particular method analyte, and Field Sample extracts show no detection for that method analyte, non-detects may be reported without re-analysis.**

10.9.3 REMEDIAL ACTION – Failure to meet CCV QC performance criteria may require remedial action. Major maintenance, such as cleaning the electrospray probe, atmospheric pressure ionization source, cleaning the mass analyzer, replacing the LC column, etc., requires recalibration (Sect 10.6) and verification of sensitivity by analyzing a CCV at or below the RL (Sect 10.7).

10.10 EXTRACT ANALYSIS

10.10.1 Establish operating conditions equivalent to those summarized in Tables 6-8 of Section 16. Instrument conditions and columns should be optimized prior to the initiation of the IDC.

10.10.2 Establish an appropriate retention time window for each analyte. This should be based on measurements of actual retention time variation for each method analyte in CAL standard solutions analyzed on the LC over the course of time. A value of plus or minus three times the standard deviation of the retention time obtained for each method analyte while establishing the initial calibration and completing the IDC can be used to calculate a suggested window size. However, the experience of the analyst should weigh heavily on the determination of the appropriate retention window size.

10.10.3 Calibrate the system by either the analysis of a calibration curve (Sect. 10.6) or by confirming the initial calibration is still valid by analyzing a CCV as described in Section 10.7. If establishing an initial calibration, complete the IDC as described in Section 13.2.

10.10.4 Begin analyzing Field Samples, including QC samples, at their appropriate frequency by injecting the same size aliquots under the same conditions used to analyze the CAL standards.

10.10.5 At the conclusion of data acquisition, use the same software that was used in the calibration procedure to identify peaks of interest in predetermined retention time windows. Use the data system software to examine the ion abundances of the peaks in the chromatogram. Identify an analyte by comparison of its retention time with that of the corresponding method analyte peak in a reference standard.

10.10.6 The analyst must not extrapolate beyond the established calibration range. If an analyte peak area exceeds the range of the initial calibration curve, the sample should be re-extracted with a reduced sample volume in order to bring the out of range target analytes into the calibration range. If a smaller sample size would not be representative of the entire sample, the following options are recommended. Re-extract an additional aliquot of sufficient size to insure that it is representative of the entire sample. Spike it with a higher concentration of internal standard. Prior to LC/MS analysis, dilute the sample so that it has a concentration of internal standard equivalent to that present in the calibration standard. Then, analyze the diluted extract.

11. Data Evaluation, Calculations and Reporting

11.1 Complete chromatographic resolution is not necessary for accurate and precise measurements of analyte concentrations using MS/MS. In validating this method, concentrations were calculated by measuring the product ions listed in Table 7.

11.2 Calculate analyte concentrations using the multipoint calibration established in Section 10.6. Do not use daily calibration verification data to quantitate analytes in samples. Adjust final analyte concentrations to reflect the actual sample volume determined in Section 10.6 where:

C_{ex} = (Area of target analyte * Concentration of Labeled analog) / (area of labeled analog * CF)

C_s = (C_{ex} / sample volume in ml) * 1000

C_{ex} = The concentration of the analyte in the extract
CF = calibration factor from calibration.

11.3 Prior to reporting the data, the chromatogram should be reviewed for any incorrect peak identification or poor integration.

11.4 PFHxS, PFOS, PFOA, NMeFOSAA, and NEtFOSAA have multiple chromatographic peaks using the LC conditions in Table 5 due to the linear and branch isomers of these compounds (Sect. 10.6.4.1). The areas of all the linear and branched isomer peaks observed in the CAL standards for each of these analytes must be summed and the concentrations reported as a total for each of these analytes.

11.5 Calculations must utilize all available digits of precision, but final reported concentrations should be rounded to an appropriate number of significant figures (one digit of uncertainty), typically two, and not more than three significant figures.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

12.1 Section 9.0 outlines sample batch QC acceptance criteria. If non-compliant organic compound results are to be reported, the Organic Section Head and/or the Laboratory Director, and the Operations Manager must approve the reporting of these results. The laboratory Project Manager shall be notified, and may choose to relay the non-compliance to the client, for approval, or other corrective action, such as re-sampling and re-analysis. The analyst, Data Reviewer, or Department Supervisor performing the secondary review initiates the project narrative, and the narrative must clearly document the non-compliance and provide a reason for acceptance of these results.

12.2 All results for the organic compounds of interest are reportable without qualification if extraction and analytical holding times are met, preservation requirements (including cooler temperatures) are met, all QC criteria are met, and matrix interference is not suspected during extraction or analysis of the samples. If any of the below QC parameters are not met, all associated samples must be evaluated for re-extraction and/or re-analysis.

13. Method Performance

13.1 Detection Limit Study (DL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

13.1.1 The laboratory follows the procedure to determine the DL, LOD, and/or LOQ as outlined in Alpha SOP ID 1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

13.2.1 The IDC must be successfully performed prior to analyzing any Field Samples. Prior to conducting the IDC, the analyst must first generate an acceptable Initial Calibration following the procedure outlined in Section 10.6.

13.2.2 INITIAL DEMONSTRATION OF LOW SYSTEM BACKGROUND – Any time a new lot of SPE cartridges, solvents, centrifuge tubes, disposable pipets, and autosampler vials are used, it must be demonstrated that an MB is reasonably free of contamination and that the criteria in Section 9.2.1 are met. If an automated extraction system is used, an MB should be extracted on each port to ensure that all the valves and tubing are free from potential PFAS contamination.

13.2.3 INITIAL DEMONSTRATION OF PRECISION (IDP) – Prepare, extract, and analyze four to seven replicate LCSs fortified near the midrange of the initial calibration curve according to the procedure described in Section 10. Sample preservatives as described in Section 6.2.1 must be added to these samples. The relative standard deviation (RSD) of the results of the replicate analyses must be less than 20%.

13.2.4 INITIAL DEMONSTRATION OF ACCURACY (IDA) – Using the same set of replicate data generated for Section 13.2.3, calculate average recovery. The average recovery of the replicate values must be within ± 30% of the true value.

13.2.5 INITIAL DEMONSTRATION OF PEAK ASYMMETRY FACTOR – Peak asymmetry factors must be calculated using the equation in Section 9.10.1 for the first two eluting peaks (if only two analytes are being analyzed, both must be evaluated) in a mid-level CAL standard. The peak asymmetry factors must fall in the range of 0.8 to 1.5. See guidance in Section 10.6.4.1 if the calculated peak asymmetry factors do not meet the criteria.

13.2.6 Refer to Alpha SOP ID 1739 for further information regarding IDC/DOC Generation.

13.2.7 The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

14.1 Refer to Alpha's Chemical Hygiene Plan and Hazardous Waste Management and Disposal SOP for further pollution prevention and waste management information.

14.2 This method utilizes SPE to extract analytes from water. It requires the use of very small volumes of organic solvent and very small quantities of pure analytes, thereby minimizing the potential hazards to both the analyst and the environment as compared to the use of large volumes of organic solvents in conventional liquid-liquid extractions.

14.3 The analytical procedures described in this method generate relatively small amounts of waste since only small amounts of reagents and solvents are used. The matrices of concern are finished drinking water or source water. However, laboratory waste management practices must be conducted consistent with all applicable rules and regulations, and that laboratories protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Also, compliance is required with any sewage discharge permits and regulations, particularly the hazardous waste identification rules and land disposal restrictions.

15. Referenced Documents

Chemical Hygiene Plan – ID 2124

SOP ID 1732 Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ) SOP

SOP ID 1739 Demonstration of Capability (DOC) Generation SOP

SOP ID 1728 Hazardous Waste Management and Disposal SOP

16. Attachments

Table 7: LC Method Conditions

Time (min)	2 mM Ammonium Acetate (5:95 MeOH/H_2O)	100% Methanol
Initial	100.0	0.0
1.0	100.0	0.0
2.2	85.0	15.0
11	20.0	80.0
11.4	0.0	100.0
12.4	100.0	00.0
15.5	100.0	0.0
Waters Aquity UPLC ® BEHC_{18} 2.1 x 50 mm packed with 1.7 µm BEH C_{18} stationary phase Flow rate of 0.4 mL/min 2-5 µL injection		

Table 8: ESI-MS Method Conditions

ESI Conditions	
Polarity	Negative ion
Capillary needle voltage	.5 kV
Cone Gas Flow	25 L/hr
Nitrogen desolvation gas	1000 L/hr
Desolvation gas temp.	500 ºC

Table 9: Method Analyte Source, Retention Times (RTs), and EIS References

#	Analyte	Transition	RT	IS	Type
1	M3PBA	216>171	2.65		REC
2	PFBA	213 > 169	2.65	2: M4PFBA	
3	M4PFBA	217 > 172	2.65	1: M3PBA	EIS
4	PFPeA	263 > 219	5.67	4: M5PFPEA	
5	M5PFPEA	268 > 223	5.66	1: M3PBA	EIS
6	PFBS	299 > 80	6.35	6: M3PFBS	
7	M3PFBS	302 > 80	6.35	29:M4PFOS	EIS
8	FtS 4:2	327 > 307	7.47	9: M2-4:2FTS	

#	Analyte	Transition	RT	IS	Type
9	M2-4:2FTS	329 > 81	7.47	29:M4PFOS	EIS
10	PFHxA	303 > 269	7.57	10: M5PFHxA	
11	M5PFHxA	318 > 273	7.57	19:M2PFOA	EIS
12	PFPeS	349 > 80	7.88	18: M3PFHxS	
13	PFHpA	363 > 319	8.80	14: M4PFHpA	
14	M4PFHpA	367 > 322	8.80	19:M2PFOA	EIS
15	L-PFHxS	399 > 80	8.94	18: M3PFHxS	
16	br-PFHxS	399 > 80	8.72	18: M3PFHxS	
17	PFHxS Total	399 > 80	8.94	18: M3PFHxS	
18	M3PFHxS	402 > 80	8.94	29:M4PFOS	EIS
19	MPFOA	415 > 370	9.7		REC
20	PFOA	413 > 369	9.7	23: M8PFOA	
21	br-PFOA	413 > 369	9.48	23: M8PFOA	
22	PFOA Total	413 > 369	9.7	23: M8PFOA	
23	M8PFOA	421 > 376	9.7	19: M2PFOA	EIS
24	FtS 6:2	427 > 407	9.66	25: M2-6:2FTS	
25	M2-6:2FTS	429 > 409	9.66	29:M4PFOS	EIS
26	PFHpS	449 > 80	9.78	33: M8PFOS	
27	PFNA	463 > 419	10.41	33: M8PFOS	
28	M9PFNA	472 > 427	10.41	19: M2PFOA	EIS
29	M4PFOS	501 > 80	10.45		REC
30	PFOS	499 > 80	10.45	33: M8PFOS	
31	br-PFOS	499 > 80	10.27	33: M8PFOS	
32	PFOS Total	499 > 80	10.45	33: M8PFOS	
33	M8PFOS	507 > 80	10.45	29: M4PFOS	EIS
34	FtS 8:2	527 > 507	10.99	38: M2-8:2FTS	
35	M2-8:2FTS	529 > 509	10.99	29:M4PFOS	EIS
36	M2PFDA	515 > 470	11.00		REC
37	PFDA	513 > 469	11.00	38: M6PFDA	
38	M6PFDA	519 > 474	11.00	36: M2PFDA	EIS
39	PFNS	549 > 80	11.02	33:M8PFOS	
40	NMeFOSAA	570 > 419	11.41	41: D3-NMeFOSAA	
41	d3-NMeFOSAA	573 > 419	11.41	36: M2PFDA	EIS
42	PFOSA	498 > 78	11.48	29: M8FOSA	
43	M8FOSA	506 > 78	11.48	19: M2PFOA	EIS
44	PFUnDA	563 > 519	11.51	41: M7-PFUDA	
45	M7-PFUDA	570 > 525	11.51	36: M2PFDA	EIS
46	PFDS	599 > 80	11.51	33:M8PFOS	
47	NEtFOSAA	584 > 419	11.68	48: d5-NEtFOSAA	

#	Analyte	Transition	RT	IS	Type
48	d5-NEtFOSAA	589 > 419	11.68	36: M2PFDA	EIS
49	PFDoA	613 > 569	11.96	50: MPFDOA	
50	MPFDOA	615 > 570	11.96	36: M2PFDA	EIS
51	PFTriA	663 > 619	12.34	50: MPFDOA	
52	PFTeA	713 > 669	12.6	53: M2PFTEDA	
53	M2PFTEDA	715 > 670	12.6	36: M2PFDA	EIS
54	M3HFPO-DA	329>285	7.97	19: M2PFOA	EIS
55	HFPO-DA	332>287	7.97	54: M3HFPO-DA	
56	ADONA	377>251	8.00	23: M8PFOA	
57	PFHxDA	813>769	13.20	59: M2PFHxDA	
58	PFODA	913>869	13.50	59: M2PFHxDA	
59	M2PFHxDA	815>770	13.20	36:M2PFDA	EIS
60	NEtFOSA	526>169	11.00	61: NMeFOSA	
61	NMeFOSA	512>169	10.50	63: d3-NMeFOSA	
62	d3-NMeFOSA	515>169	10.50	29: M4PFOS	EIS
63	d5-NEtFOSA	531>169	11.00	29: M4PFOS	EIS
64	NMeFOSE	556>122	11.25	66: d7-NMeFOSE	
65	NEtFOSE	570>136	10.75	67: d9-NEtFOSE	
66	d7-NMeFOSE	563>126	11.25	29: M4PFOS	EIS
67	d9-NEtFOSE	579>142	10.75	29: M4PFOS	EIS
68	FtS 10:2	627>607	11.50	25: M2-6:2FTS	
69	PFDoS	699>99	12.50	33: M8PFOS	

pH, Soil and Waste

References: **Method 9045D, Soil and Waste pH,** Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Revision 4, November 2004.

Method 9040C, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Revision 3, November 2004.

NJDEP Site Remediation Program, Data of Known Quality Protocol, Version 1, April 2014

1. Scope and Application

Matrices: Soils and wastes, including solids, sludges, or non-aqueous liquids.

Definitions: See Alpha Laboratories Quality Manual Appendix A

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the pH meter and in the interpretation of pH data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

The sample is mixed with reagent water, and the pH of the resulting aqueous solution is measured.

2.1 Method Modifications from Reference

The pH meter that is utilized will compensate for the temperature of the sample. Therefore, the temperature is not reported with the data. However, the sample is analyzed at room temperature. Only one aliquot is used for measurement.

3. Reporting Limits

None.

4. Interferences

4.1 Errors will occur when the electrode becomes coated. If an electrode becomes coated with an oily material that will not rinse free, the electrode can (1) be cleaned with an ultrasonic bath, or (2) be washed with detergent, rinsed several times with water, placed in 1:10 HCl so that the lower third of the electrode is submerged, and then thoroughly rinsed with water, or (3) be cleaned per the manufacturer's instructions.

4.2 Samples with a very low or very high pH may give incorrect readings on the meter.

For samples with a true pH of >10, the measured pH may be incorrectly low. This error can be minimized by using a low-sodium-error electrode.
Strong acid solutions, with a true pH of <1, may give incorrectly high pH measurements.

4.3 Coatings of oily material or particulate matter can impair electrode response. These coatings can usually be removed by gentle wiping or detergent washing, followed by rinsing with distilled water. An additional treatment with hydrochloric acid (1:10) may be necessary to remove any remaining film.

4.4 Temperature effects on the electrometric determination of pH arise from two sources. The first is caused by the change in electrode output at various temperatures. This interference should be controlled with instruments having temperature compensation. The second source of temperature effects is the change of pH due to changes in the sample as the temperature changes. This error is sample-dependent and cannot be controlled. However, prior to analysis the samples are brought to room temperature (20 – 25 °C).

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples are collected in 4-ounce glass jars.

6.2 Sample Preservation

None.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

Samples are to be analyzed as soon as possible after sampling. Samples are stored at 4 ± 2°C. Samples that are to be run in conjunction with NJ-Hex DAK acceptance criteria must be analyzed within 24 hours of Hex sample preparation.

7. Equipment and Supplies

7.1 **pH Meter: "Black"** Orion Research, expandable ion analyzer EA 940. Laboratory benchtop model. **"White"** Hanna pH temperature bench meter (HI5521-01). **"White 3"** Hanna pH temperature bench meter (HI2002-01) Or equivalent.

7.2 pH electrode: Accuphast electrode with automatic temperature compensation Fisher Catalog #13-620-296. Incorporates measuring and referenced functions; filled with AgCl solution. Hanna (HI 1131B) Glass body, refillable, combination electrode for "white 3".

7.3 Beakers: 50mL glass or plastic

7.4 Magnetic Stirrer

7.5 Teflon-coated stirring bar

7.6 Analytical Balance: Capable of weighing 0.1g

7.7 Kimwipes

8. Reagents and Standards

8.1 Reagent Water: All references to water in this method refer to reagent water.

8.2 pH Buffers: Commercially available pH 4 (or 4.01), pH 7, pH 10 (or 10.01). In addition, an alternate source of pH 7 Buffer is necessary. All Buffers must have been validated by comparison to NIST standards. Certificate of analysis is required. Buffers are stored at room temperature and expire upon manufacturer's specified date.

9. Quality Control

9.1 Blank(s)

Not applicable.

9.2 Laboratory Control Sample (LCS)

One LCS is analyzed with each batch of 20 samples or less. It is a pH 7 buffer of a different source than the calibration buffer. Results must be within ± 0.05.

9.3 Initial Calibration Verification (ICV)

Refer to LCS Section 9.2.

9.4 Continuing Calibration Verification (CCV)

A CCV of pH 7 is analyzed at the end of the analytical run. Results must be within ± 0.2 units.

9.5 Matrix Spike

Not applicable.

9.6 Laboratory Duplicate

One duplicate sample is analyzed per batch of 20 samples or less

9.7 Method-specific Quality Control Samples

Not applicable.

9.8 Method Sequence

- Calibration

- Calibration Verification (LCS)
- Sample analysis
- Duplicate Analysis
- Calibration Verification (CCV)

10. Procedure

10.1 Equipment Set-up

10.1.1 Sample Preparation

In a 50mL beaker, weigh 20 grams of soil; record the weight. Add 20mL of reagent water, cover and continuously stir the suspension for 30 minutes, using a teflon stir bar on a magnetic stirrer. Let samples stand for 1 hour before measurement.

Additional dilutions are permissible if working with hydroscopic soils, salts or other problematic matrices.

NOTE: If the sample is hygroscopic and absorbs all the reagent water, begin again using 20 grams of sample and 40mL of reagent water.

NOTE: If the supernatant is multiphasic, decant the oily phase and measure the pH of the aqueous base. The electrode may need to be cleaned (Section 4.1) if it becomes coated with an oily material.

10.2 Initial Calibration

The pH meter is calibrated on a daily basis using three pH buffers (Section 8.1). Follow manufacturer's instructions for a 3-point calibration of the pH meter. The results of the calibration must be recorded in the pH Calibration Log.

10.3 Equipment Operation and Sample Processing

10.3.1 Allow the sample suspension to stand for about 30 minutes to allow suspended material to settle out from the suspension.

10.3.2 Immerse the pH electrode just below the suspension and allow pH meter to stabilize. Note and record the sample pH in the Laboratory notebook. Report sampletemperature at the time of measurement.

10.3.3 Rinse the electrode thoroughly between samples, using reagent water.

10.4 Continuing Calibration

Prior to sample analysis, the calibration is initially verified by using a pH 7 buffer from a source other than the source used for calibration. The results must be within ± 0.2. If this criterion is not met, the meter must be re-calibrated before sample analysis can begin.

10.5 Preventative Maintenance

The pH probe is rinsed with DI and gently dried with a KimWipe between sample readings.

11. Data Evaluation, Calculations and Reporting

pH is read directly from the pH meter. No calculations are necessary.

For pH readings of less than 1, the reported result is: pH <1.

For pH readings greater than 10, report result in 3 significant figures.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedence and improper preservation are noted on the nonconformance report form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the maintenance logbook.

The pH electrode is replaced as necessary.

Review of standards for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form.

If the LCS or CCV recovery falls outside the designated acceptance range, the laboratory performance for the parameter is judged to be out of control, and the problem must be immediately identified and corrected. Re-calibration of the meter is necessary prior to sample analysis. All samples analyzed since the last acceptable QC standard must be reanalyzed following re-calibration of the meter.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/1732 MDL/LOD/LOQ Generation

SOP/1739 IDC/DOC Generation

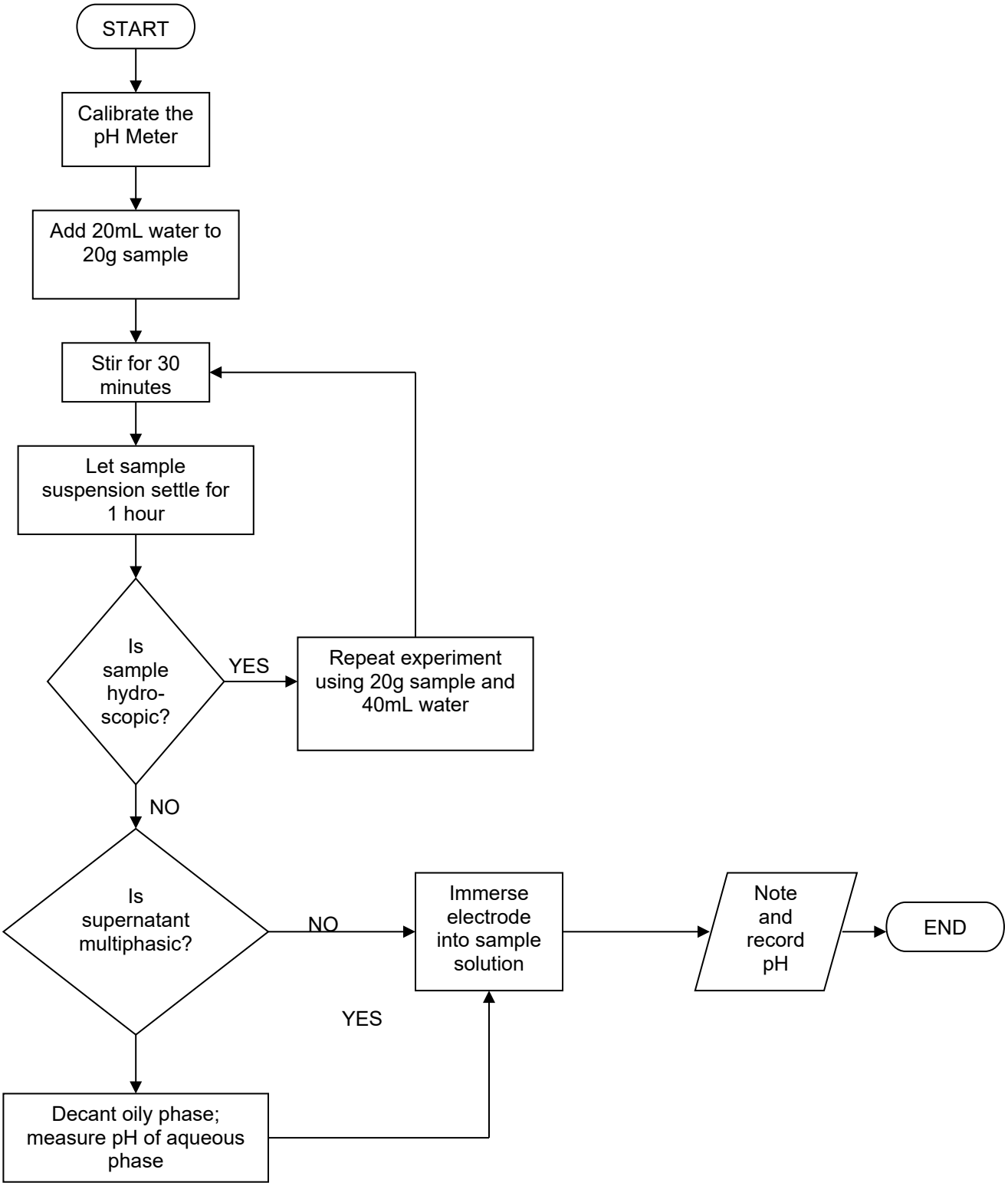
SOP/1728 Waste Management and Disposal SOP

16. Attachments

Flow Chart: Soil and Waste pH

Flow Chart:

Soil and Waste pH



pH, Liquid Samples

Reference Methods: **Method 9040C,** SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update IIIB, 2004.

SM 4500H$^+$-B, Standard Methods for the Examination of Water and Wastewater. APHA-AWWA-WEF. Standard Methods Online.

1. Scope and Application

Matrices: This method is used to measure the pH of aqueous wastes and those multiphase wastes where the aqueous phase constitutes at least 20% of the total volume of the waste.

The corrosivity of concentrated acids and bases, or of concentrated acids and bases mixed with inert substances, cannot be measured. The pH measurement requires some water content.

Definitions: See Alpha Laboratories Quality Manual Appendix A

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the pH Meter and in the interpretation of pH data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

The pH of the sample is determined electrometrically using a temperature compensating combination electrode.

2.1 Method Modifications from Reference

The pH meter that is utilized will compensate for the temperature of the sample. Therefore, the temperature is not reported with the data. However, the sample is analyzed at room temperature.

3. Reporting Limits

None.

4. Interferences

4.1 Errors will occur when the electrode becomes coated. If an electrode becomes coated with an oily material that will not rinse free, the electrode can (1) be cleaned with an ultrasonic bath, or (2) be washed with detergent, rinsed several times with water, placed in 1:10 HCl so that the lower third of the electrode is submerged, and then thoroughly rinsed with water, or (3) be cleaned per the manufacturer's instructions.

4.2 Samples with a very low or very high pH may give incorrect readings on the meter.

For samples with a true pH of >10, the measured pH may be incorrectly low. This error can be minimized by using a low-sodium-error electrode.

Strong acid solutions, with a true pH of <1, may give incorrectly high pH measurements.

4.3 Coatings of oily material or particulate matter can impair electrode response. These coatings can usually be removed by gentle wiping or detergent washing, followed by rinsing with distilled water. An additional treatment with hydrochloric acid (1:10) may be necessary to remove any remaining film.

4.4 Temperature effects on the electrometric determination of pH arise from two sources. The first is caused by the change in electrode output at various temperatures. This interference is controlled with instruments having temperature compensation. The second source of temperature effects is the change of pH due to changes in the sample as the temperature changes. This error is sample-dependent and cannot be controlled. However, prior to analysis the samples are brought to room temperature (20 – 25 °C).

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples are collected in plastic or glass containers. The minimum volume required is 100mL.

6.2 Sample Preservation

None.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

Samples are to be analyzed as soon as possible, within 24 hours of collection. Samples are stored at 4 ±2 °C. However, prior to analysis, samples are allowed to come to room temperature.

7. Equipment and Supplies

7.1 pH Meter: Laboratory benchtop model with automatic temperature compensation.

7.1.1 Combination electrode: Incorporates measuring and referenced functions; solid, gel-type filling material. For use with benchtop pH Meter (Section 7.1).

7.2 Titrator: T70 Mettler Toledo, with Inmontion Flex Autosampler and Computer with LabX software package.

7.2.1 Combination electrode: Mettler Toledo, Type DG111-SC, Part #00089596. For use with Titrator (Section 7.2).

7.3 Plastic Cups: Mettler Toledo brand, 100mL volume.

7.4 Magnetic stirrer

7.5 Teflon-coated stirring bar

7.6 Glass Beakers

7.7 Kimwipes

7.8 Plastic cups

8. Reagents and Standards

8.1 pH Buffers: Commercially available pH 4 (or 4.01), pH 7 (two sources), pH 10 (or 10.01). Buffers must have been validated by comparison to NIST standards. Certificate of analysis is kept on file.

9. Quality Control

9.1 Blank(s)

Not applicable.

9.2 Laboratory Control Sample (LCS)

One LCS is analyzed with each batch 20 of samples or less. It is a pH 7 buffer of a different source than the calibration buffer. Results must be within ±0.05.

When the LCS fails the acceptance criteria, the analyst must locate and correct the source of the problem and repeat the calibration. Repeated failure confirms a general problem with the measurement system or analytical technique of the analyst.

9.3 Initial Calibration Verification (ICV)

Refer to LCS Section 9.2.

9.4 Continuing Calibration Verification (CCV)

The calibration is also verified at the end of the sample sequence and after every 10 samples. The results of the continuing calibration verification must be within ± 2% (6.86-7.14). If this criterion is not met, the titrator or meter must be re-calibrated and any samples analyzed since the last acceptable CCV are reanalyzed

9.5 Matrix Spike

Not applicable.

9.6 Laboratory Duplicate

One sample is analyzed in duplicate per batch of 20 samples or less. %RPD for Duplicates must be within in-house acceptance criteria determined from Control Charts. If the result is outside of the criteria, the duplicate result may be narrated.

9.7 Method-specific Quality Control Samples

Not applicable.

9.8 Method Sequence

- Calibration
- Initial Calibration Verification (LCS)
- Sample analysis, 1-10 samples
- Duplicate analysis
- Continuing Calibration Verification (CCV), analyzed every 10 samples and at the end of analytical run.

10. Procedure

10.1 Equipment Set-Up

Thoroughly rinse and gently wipe the electrode prior to measuring the pH of buffers for calibration.

10.2 Initial Calibration

10.2.1 Benchtop Meter

The benchtop pH meter is calibrated on a daily basis using three pH buffers (Section 8.1). Follow manufacturer's instructions for a 3-point calibration of the pH meter. The results of the calibration must be recorded in the pH Calibration Log including correlation coefficient. Slope of the meter must fall within 95% to 105%.

Initial Calibration Verification: Prior to sample analysis, the calibration is initially verified by using a pH 7 buffer from a source other than the source used for calibration. The results of the initial verification must be within ± 0.05. If this criterion is not met, the meter must be re-calibrated.

10.2.2 Titrator

The pH titrator is calibrated on a daily basis using three pH buffers (Section 8.1).

- Pour 20-25mL of pH 4, 7 and 10 buffers (Section 8.1) into separate plastic cups (Section 7.3). Record the Lot numbers of the buffers into the Laboratory Notebook.
- Place the cups onto the Inmontion Flex Autosampler in that order.
- Ensure the Titrator is in "Remote Control" mode.
- Open LabX software package. Select "Open View" on the Titrator. Choose the appropriate method: Method 1 – PH Calibration.
- With a right-click on the mouse, select "Create Task". Type in the series ID (Cal-MM/DD/YYYY), and the sample sequence: PH 4, PH 7, PH 10. Select the "Save" button.
- The Series ID will appear in the "Samples" column. Click on that Series ID and using a right mouse click, select "Run" from the drop down menu.

After the run is complete, check for printout results. . Slope of the meter must fall within 95% to 105%.

Initial Calibration Verification: Prior to sample analysis, the calibration is initially verified by using a pH 7 buffer from a source other than the source used for calibration. The results of the initial calibration verification (LCS) must be within ±2%. If this criterion is not met, the Titrator must be recalibrated before any samples are analyzed.

10.3 Equipment Operation and Sample Processing

10.3.1 Benchtop Meter

Place the sample in a clean glass beaker or plastic cup using a sufficient volume to cover the sensing elements of the electrode and to give adequate clearance for the magnetic stirring bar.

Thoroughly rinse and gently wipe the electrode prior to measuring the pH of samples. Immerse the electrode into the sample beaker and gently stir at a constant rate to provide homogeneity and suspension of solids. Note and record the sample pH when meter has stabilized. Record temperature at which pH is recorded.

10.3.2 Titrator

- Pour 20-25mL of each sample into a separate, labeled plastic cup (Section 7.3).
- Place the cups onto the Inmontion Flex Autosampler.
- Choose the appropriate method: Method 2 – PH Measurement.
- With a right-click on the mouse, select “Create Task”. Type in the series ID (LCS-MM/DD/YYYY), and the sample sequence. Make sure the order of the samples on the autosampler matches the sample sequence. Select the “Save” button.
- The Series ID will appear in the “Samples” column. Click on that Series ID and using a right mouse click, select “Run” from the drop down menu.
- After the run is complete, check for printout results.

10.4 Continuing Calibration

10.4.1 Benchtop Meter

The meter calibration is also verified at the end of the sample sequence and after every 10 samples. The results of the continuing calibration verification must be within ± 2%. If this criterion is not met, the meter must be re-calibrated and any samples analyzed since the last acceptable CCV are reanalyzed.

10.4.2 Titrator

The titrator calibration is also verified at the end of the sample sequence and after every 10 samples. The results of the continuing calibration verification must be within ± 2%. If this criterion is not met, the titrator must be re-calibrated and any samples analyzed since the last acceptable CCV are reanalyzed.

10.5 Preventative Maintenance

The combination electrode on the benchtop meter is rinsed with DI and gently dried with a KimWipe between sample readings. Both electrodes are stored in buffer 7 when not in use.

11. Data Evaluation, Calculations and Reporting

pH is read directly from the pH benchtop meter or Titrator. No calculations are necessary.

For pH readings of less than 1, the reported result is: pH <1.

For pH readings greater than 10, report result in 3 significant figures.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedence and improper preservation are noted on the nonconformance report form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

The combination electrode is replaced as necessary.

Review of standards for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form.

If the LCS recovery falls outside the designated acceptance range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. Re-calibration of the meter or titrator is necessary prior to sample analysis.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

Not applicable to this method.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1734, 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information

15. Referenced Documents

Chemical Hygiene Plan

SOP/1732 MDL/LOD/LOQ Generation

SOP/1734, 1739 IDC/DOC Generation

SOP/1728 Waste Management and Disposal SOP

16. Attachments

None.

Quality Systems Manual

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D/B/A

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1 Mission Statement

The mission of Alpha Analytical is quite simply to provide our customers with the greatest value in analytical service available. For the 'greatest value' is not only found in the data that is delivered, it is also found in the services provided.

- Data must be of the highest integrity, accuracy and precision.
- Consultation and educational services must be provided to support the customer in establishing data quality objectives and interpretation of the final data package.
- Support services such as sample containers, courier service and electronic data deliverables must be available to the customer.

Alpha's mission continues with an established commitment to our community and environment. We must ensure that we do not produce any additional contamination to our environment or harm our neighbors and community in any way.

The value of Alpha's product is in the honesty and integrity with which each chemist, courier, login staff member, or office staff member performs their tasks. The customer or employee must always feel satisfied that they received the greatest value in their lab experience at Alpha.

Alpha Analytical will vigorously pursue its mission into the next millennium.

Mark Woelfel

President

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3 Introduction

The Quality Systems Manual, referred to as Corporate Quality Systems Manual (CQSM) of Alpha Analytical describes the quality program in use at the laboratory for both Westboro and Mansfield facilities. This Quality Systems Manual provides employees, customers and accrediting agencies with the necessary information to become familiar with how the quality system operates within Alpha Analytical. The quality program includes quality assurance, quality control, and the laboratory systems including feedback mechanisms for the automated continuous improvement of the laboratory operations to meet customer needs.

Implementation of the laboratory operations is by documenting procedures, training personnel and reviewing operations for improvement. Written procedures are maintained as Standard Operating Procedures (SOPs). The SOPs are available to the staff as a controlled, electronic, secure copy. The provisions of the QSM are binding on all temporary and permanent personnel assigned responsibilities. All laboratory personnel must adhere strictly to the QSM and SOPs.

All policies and procedures have been structured in accordance with the NELAC Institute (TNI) Standards), DOD QSM 5.1 and applicable EPA requirements and standards.

Twenty-five (25) sections comprise the QSM. Related quality documentation including the listing of SOPs, forms, floor plan, equipment, personnel and laboratory qualifications are available. The QSM sections provide overview descriptions of objectives, policies, services and operations.

3.1 Scope

The QSM describes the requirements of the Laboratory to demonstrate competency in the operations for performing environmental tests for inorganic, organic, air and microbiological testing. The basis for the environmental tests is the methods found in documents published by the United States Environmental Protection Agency (EPA), ASTM, AOAC, APHA/AWWA/WEF, Standard Methods, and other procedures and techniques supplied by customers.

The QSM includes requirements and information for assessing competence and determining compliance by the laboratory to the quality system. When more stringent standards or requirements are included in a mandated test method, by regulation, or specified in a project plan the laboratory demonstrates achievement of the customer specified requirements through its documented processes.

The QSM is for use by Alpha Analytical for developing and implementing the quality system. Accrediting authorities and customers use the QSM for assessing the competence of Alpha Analytical. Alpha Analytical is committed to continually improving the quality system. Meeting customer needs, operating within regulatory requirements and adhering to Alpha's Data Integrity and Ethics policy are several of the mechanism used to continually improve the quality system.

3.2 Policy Statement

This Quality Systems Manual summarizes the policies, responsibilities and operational procedures associated with Alpha Analytical. This manual applies to all associates of the laboratory and is intended for use in the on-going operations at Alpha Analytical. Specific protocols for sample handling and storage, chain-of-custody, laboratory analyses, data reduction, corrective action, and reporting are described. All policies and procedures have been structured in accordance with the NELAC Institute (TNI) Standards, DOD QSM(which includes 17025 standards), applicable EPA requirements, regulations, guidance, and technical standards. This Quality Systems Manual, laboratory Standard Operating Procedures (SOPs), and related documentation describe the quality systems, policies and procedures for Alpha Analytical.

Alpha Analytical performs chemical analyses for inorganic and organic constituents in water, seawater, soil, sediment, oil, tissue and air matrices. Alpha Analytical's goal is to produce data that is scientifically valid, technically defensible, and of known and documented quality in accordance with standards developed by The NELAC Institute (TNI) Standards and any applicable state or EPA regulations or requirements. It is the commitment of the President, Operations Director, Laboratory Technical Manager and Quality Assurance Officer to work towards continuous improvement of the operation, and towards meeting our customer's needs, requirements, and intended data usage. This continued commitment is built into every activity of the laboratory. It is the responsibility of Senior Management and the Department Managers to ensure that all associates familiarize themselves with, and comply at all times with, the quality systems, procedures and policies set forth in this manual, laboratory SOPs, and related documentation.

Alpha Analytical analyzes Proficiency Test (PT) samples, in accordance with the NELAC Institute (TNI) Standards and other regulatory programs, from a National Institute of Standards and Technology (NIST)-approved PT provider for the analytes established by EPA for water samples, and for other analytes and matrices. The specific analytes and matrices analyzed are based on the current scope of the laboratory services as documented in the laboratory SOPs and state certifications.

The technical and service requirements of all requests to provide analyses are thoroughly evaluated before commitments are made to accept the work. This includes a review of facilities and instrumentation, staffing, and any special QC or reporting requirements to ensure that analyses can be performed correctly and within the expected schedule. All measurements are made using published reference methods or methods developed by Alpha Analytical. Competence with all methods is demonstrated according to the procedure described in SOP/ 1739 prior to use.

Alpha Analytical has developed a proactive program for prevention and detection of improper, unethical or illegal actions. Components of this program include: internal proficiency testing, electronic data audits and post-analysis data review by the QA Officer; a program to improve employee vigilance and co-monitoring; and Ethics Training program identifying appropriate and inappropriate laboratory practices, instrument manipulation practices and consequences. Additionally, all associates are required to sign the Alpha Analytical *Ethics Agreement* form upon commencement of employment and complete annual refresher Ethics Training thereafter. This form clearly outlines the possible consequences of unethical or improper behavior, or data misrepresentation. All staff are required to report any suspected unethical conduct to management. Management will then investigate and determine if the situation was considered unethical and will take appropriate action as described in the Alpha Ethics policy.

It is the policy of the laboratory to discourage and reject all influence or inducements (whether commercial, financial or personal) offered either by customers or suppliers, which might adversely affect results or otherwise compromise the judgment or impartiality of the staff. It is the responsibility of the Operations Director and Laboratory Technical Manager to inform customers and suppliers of this policy when necessary.

In the event that any such influences or inducements are encountered, the staff is instructed to inform management immediately. It is the responsibility of the Operations Director and the Laboratory Technical Manager to take appropriate action to prevent recurrence.

3.3 References

External reference documents are available electronically in the Qualtrax system for staff to access the latest edition or version of the reference methods, regulations or national standards. The Quality Assurance Department maintains the electronic files in the Qualtrax system. Management purchases automated update services, where available, to provide the laboratory with the latest hardcopy edition, where electronic means is not available.

3.4 Definitions

Appendix A lists the definitions as adopted by the laboratory. The definitions are from the 2009 TNI standards.

4 Organization and Management

4.1 Legal Definition of Laboratory

Alpha Analytical is a full service analytical laboratory. Testing services include Drinking Water, Waste Water, Ground Water, Waste material and Air. Alpha Analytical is a privately held corporation incorporated in the state of Massachusetts. Alpha Analytical, Inc. does business as (D/B/A) Alpha Analytical.

Alpha Analytical has been in business since 1985. The types of businesses served include:

Consulting firms,
Engineering firms,
Waste Management Companies,
Industrial sites,
Municipal agencies
Department of Defense projects.

4.2 Organization

The laboratory operates a quality system approach to management in order to produce data of known quality. The laboratory organization provides effective communication and lines of authority to produce analytical data meeting customer specifications. The organizational design provides open communication while ensuring that pressures and day to day operating circumstances do not compromise the integrity of the reporting of the final data. See Appendix B for Organizational Chart.

The President is responsible for directing all areas of the company. The following job functions report to the President:

Operations Manager
Quality Assurance Officer
Marketing / Business Development / Sales
Financial Services
Human Resources

The Operations Manager is responsible for directing all laboratory operational areas of the company. The following job functions report to the Operations Manager:

Laboratory Technical Manager(s)

Customer Services Manager

Department Managers

The Laboratory Technical Manager(s) is(are) responsible for the laboratory data generated by the organics testing, inorganics testing and metals testing areas and the Air Technical Director is responsible for laboratory data generated by air analyses.

The Departmental Managers (Supervisors) have the following responsibilities:

The organics managers direct personnel in the organics extraction and instrumental laboratories.

The wet chemistry manager directs personnel and team leaders in the wet chemistry and/or microbiological testing areas.

The metals manager directs personnel and team leaders in the metals sample preparation and instrumental laboratories.

The Quality Assurance Officer is a member of the staff and reports directly to the President and has defined responsibility and authority for ensuring that the quality system is implemented and adhered to at all times. The Quality Assurance (QA) Officer is responsible for interacting and communicating certification requirements, implementing the Quality Systems Manual and reporting to the Laboratory Technical Manager and Senior Management the status of the quality program. The QAO oversees the Quality Systems Specialists and is responsible for oversight and/or review of quality control data and function independently from laboratory operations.

The Customer Services Manager is responsible for customer interactions, project coordination and laboratory personnel notification of project requirements.

The Marketing, Business Development and Sales personnel are responsible for increasing the volume of work from current customers and adding new customers to the base business of Alpha Analytical. The Marketing and Business Development personnel review all new work with the Laboratory Technical Manager, Operations Manager, President and/or Quality Assurance Officer before contractual commitment.

The CFO is responsible for maintaining and reporting on the financial status of the company. The CFO directs financial personnel on proper accounting procedures and maintaining the list of approved suppliers and subcontractors. The CFO reports directly to the President.

The Human Resource Director is responsible for personnel recruitment, hiring, performance reviews.

Personnel job descriptions define the operational function duties and responsibilities. Administration and Laboratory personnel assignments may include cross-functional training and work performance in multiple areas of the operations. Multiple function training ensures laboratory back up personnel during peak workloads.

During the absence of any staff member, assignment of alternative personnel occurs by memo or e-mail. The Manager or Supervisor authorizes the assignment. The naming of alternative personnel assures the continuing performance of critical tasks during the primary person's absence and ensures that lines of communication remain open for continued decision making. The deputy for the Laboratory Technical Manager is the Quality Assurance (QA) Officer. The deputies for the Quality Assurance (QA) Officer are the Quality Systems Specialists.

For the purposes of the NELAC Institute (TNI) Standards the Lead Laboratory Technical Manager is the Laboratory Technical Manager. The deputies for the Lead Technical Manager are the Quality Assurance (QA) Officer, and the Departmental Managers. The Laboratory Technical Manager meets the requirements specified in the Section 4.1.7.2 Volume 1, Module 2 of the 2009 TNI standards. If the Laboratory Technical Manager is absent for a period of time exceeding 15 consecutive calendar days, a full-time staff member meeting the qualifications of Laboratory Technical Manager will be designated to temporarily perform this function. The primary Accrediting Body shall be notified in writing if the Technical Manager's absence exceeds 35 consecutive calendar days.

4.3 Business Practices

Alpha maintains certification for the programs and analytes required by regulatory programs. The listing of qualifications from the various certifications, registrations and accreditation programs are available upon request. Alpha Analytical operates Monday to Friday from 7:30 a.m. to 5:30 p.m. Management prepares and posts the holiday schedule for the year indicating closed operations. Sample delivery occurs during normal operating hours unless arranged in advance.

Alpha's reputation depends upon timely reporting and quality data. The standard turnaround time for engineering and consulting firms is five business days from time of sample receipt. Standard turnaround for all other customers is ten business days from time of sample receipt. The time of sample receipt is when the verification of the chain of custody and samples meets the laboratory sample acceptance policy. Laboratory management must approve any special arrangements for rush or expedited turnaround time. The basis for data quality depends on customer, regulation and method performance criteria. Accuracy, precision, sensitivity and comparability are expressions of method performance criteria.

All work is performed in the strictest confidence. New and contract employees must review corporate policy and practice requirements for protecting customer confidentiality and proprietary rights. The review occurs during orientation and ethics training. It is the policy of the laboratory to release data to the customer authorized contact. Personnel assigned the duties of interacting with customers review project files and discuss data related only to the project. Personnel whose duties do not include routine customer contact must check with the customer service manager before discussing data with regulators or third parties

5 Quality System

Establishment, Audits, Essential Quality Controls and Data Verification

5.1 Establishment

The Mission Statement presents the policy and objectives for Alpha Analytical. The Quality Systems Manual provides the framework for the processes and operations to implement the Mission. The Quality Systems Manual and documentation controlled by the laboratory system detail the management authorized operations for achieving the objectives of the company.

The laboratory operates a quality system approach to management in order to produce data of known quality. Alpha Analytical is a full service laboratory designed to provide its customers with accurate, precise and reliable data within the best turn-around time and at the most reasonable prices. Alpha employs chemists of the highest training, ethics and caliber in the field of analytical chemistry. This and state-of-the-art instrumentation and automation combine to insure data of known and documented quality.

5.2 Quality Systems Manual

The QA Officer is responsible for the publication and distribution of the Quality Systems Manual and annual review. Management reviews and authorizes the manual. Implementation of major changes in the quality system occurs after revision of the appropriate Quality Systems Manual section and authorization by management.

The authorization of the Quality Systems Manual is documented electronically in Qualtrax. Updates of this manual occur at any time throughout the year. Document control procedures (SOP1729) apply to the distribution of the Quality Systems Manual. Controlled copies of the manual are maintained electronically within Qualtrax. Persons or organizations outside of Alpha Analytical may receive uncontrolled copies. Copies are distinctly indicated “Uncontrolled Documents” within the footer of each page.

5.3 Audits

Laboratory audits, both internal and external, review and examine the operations performed in the laboratory. Internal audits are conducted by qualified QA Specialists and external audits are reviews by external organizations to evaluate the ability of the laboratory to meet regulatory or project requirements. Internal audits are conducted on a frequency of bi-annually, method required and annually for DoD certified methods.

A QA designee schedules internal process audits to ensure the completion of the annual audit of each operational area. The process audits are a more detailed review of the operations. Personnel from areas other than the one audited perform process audits.

The internal system audit is a review of the implementation of the documented quality system. The system audit includes sample tracking from receipt to disposal, a data audit of a completed report, and all operations not audited during the process audit.

The purpose of the internal system audit is:

Verification that adequate written instructions are available for use;

Analytical practices performed in the laboratory are consistent with SOPs;

The quality control practices are applied during production;

Corrective actions are applied as necessary;

Deviations from approved protocols are occurring only with proper authorization and documentation;

Reported data is correct and acceptable for reporting;

SOPs, quality records, analytical records, electronic data files are maintained properly; and

Personnel training files and records are satisfactory and current.

Before a scheduled internal audit, the assigned auditor reviews checklists, if used, and/or the SOP specific to the area. The checklist may be from an external source or prepared by the auditor. . After the audit, the auditor submits a summary or notes from the audit to the Laboratory Technical Manager or QAO as part of the audit report. The summary identifies discrepancies found during the audit. Technical personnel are responsible for the inspection and monitoring of in-process and final data. Personnel independent of those having direct responsibility for the work performed audit the quality system and processes.

Representatives sent by customers and government or accrediting agencies often perform external audits. These audits are most often announced inspections, but sometimes are not announced. The Quality Assurance Officer, Laboratory Technical Manager or assigned deputy, and/or appropriate Department Manager accompany the external audit team through the laboratory. The auditors receive a brief overview of company objectives, activities, and facilities. Interviews with essential supervisory staff and technical staff are arranged, along with retrieval of any documentation pertinent to the audit. Auditors usually provide a report on their findings shortly after the audit. The QA Officer receives the audit report and copies are provided to laboratory personnel for review. Corrective actions are identified and distributed to responsible parties for implementation in response to any cited deficiencies.

5.4 Audit Review

Management reviews internal and external audit reports to evaluate system effectiveness at the annual management review meeting. Tracking of the audit findings occurs through the nonconformance action process. The management and staff work together to establish a time line for resolving the audit findings. The Quality Assurance team tracks the time line and reports to the Laboratory Technical Manager on any outstanding audit findings. Approved corrective actions for DoD that are not implemented or avoided may result in loss of DoD ELAP accreditation and may result in work being discontinued until implementation is verified by DOD ELAP AB.

5.5 Performance Audits

Alpha Analytical participates in inter-laboratory comparisons and proficiency test programs required by customers and certifying agencies. The performance audits provide information on the data comparability of results generated by the laboratory. Test samples received by the laboratory are handled following routine laboratory procedures. Proficiency test samples are unpacked, checked against the packing slip and examined for damage. Reporting requirements and deviations to routine practices are noted as would be required for any project.

Analysts demonstrate proficiency by analyzing either an external proficiency test sample, an internally prepared blind test sample or Initial Demonstration of Capability (IDC) before independent operation of a test method. The results of performance audits serve several purposes. The QA Officer may use performance audits for evaluating analyst proficiency, laboratory performance in a specified area to facilitate laboratory improvement efforts, and/or to provide information to an accrediting agency on correction of past performance of an external performance audit.

5.6 Corrective Actions/Preventative Actions (CAPA)

The corrective action process at Alpha Analytical is detailed in SOP 1736. The corrective action program at Alpha Analytical uses the Nonconformance workflow in Qualtrax to document and follow through the corrective action/preventative action process for three main areas: nonconformance's within the laboratory, customer complaints and failed PT studies. The process ensures continuous improvement of company performance by preventing the recurrence of quality problems.

Nonconformance reports are tracked for closure date and the type. Reports to management include the listing of open nonconformance reports and the frequency of the type of nonconformance occurring. A QA designee monitors the completeness of the forms, as well as verifies the actions are complete and acceptable.

Customers will be notified within 5 days of any question(s) regarding validity of results.

5.7 Managerial Review

The management review occurs at least once per year as part of the strategic planning process. Documentation of the management review meeting is by recording the meeting minutes and listing the attendees. The focus of the quality management review is the frequency of the type of nonconformance, closure status, audit progress and other quality assurance actions. Meetings include discussion and progress on quality system initiatives since the last meeting.

Prior to the meeting, an agenda is distributed to all personnel expected to be in attendance. The meeting is chaired by the President. Minutes are taken and distributed at the conclusion of the meeting by a QA designee. If action is necessary on any issue, a Summary Report is generated and distributed to responsible parties for implementation. Actions are monitored by the QAO or designee until completion.

5.8 Essential Quality Control Procedures

The following general quality control principles apply to all tests. The manner implemented is dependent on the type of test performed. The laboratory SOP presents the specific quality control checks undertaken to ensure precision, accuracy and sensitivity of each test method. Deviations from the existing SOP are allowed only upon approval of the deviation by the department manager and Quality Assurance Officer. This documentation must be either in form of written notice or email.

Alpha Analytical uses quality control samples to evaluate the following:

1. Adequate positive and negative controls to monitor blanks, spikes, reference toxicants, zero blanks;
2. Adequate tests to define the variability and/or reproducibility of laboratory results;
3. Measures to ensure the accuracy of the test data including sufficient calibration and/or continuing calibrations, use of certified reference materials, proficiency test samples;
4. Measures to evaluate test performance, such as detection limits and quantitation limits or range of applicability such as linearity;
5. Selection of appropriate formulae to reduce raw data to final results such as linear regression, internal standards, or statistical packages;
6. Selection and use of reagents and standards of appropriate quality;

7. Measures to assure the selectivity of the test for its intended purpose;
8. Measures to assure constant and consistent test conditions for the method such as temperature, humidity, light, or specific instrument conditions.

   Note: All quality control samples are treated in the same manner as field samples.

All quality control measures are assessed and evaluated on an on-going basis, and quality control acceptance limits are used to determine the usability of the data. Control charts and/or calculated control limits monitor the long-term method performance by analyte, by instrument for water matrices. Routine evaluation and reporting of the control chart performance provides supervisors and management with additional performance measures to ensure data comparability. Control limits are recalculated when trends are observed.

Where no reference method or regulatory criteria exist, the laboratory specifies the acceptance/rejection criteria in the SOP. The test SOP specifies the QC samples performed per batch of samples. The quality control samples are categorized into the following, as appropriate to the method

- Method Blank
- Laboratory Duplicate
- Laboratory Control Sample (LCS)
- Laboratory Control Sample Duplicate (LCSD)
- Matrix Spike (MS)
- Matrix Spike Duplicate (MSD)

Selection of samples for Duplicate, Matrix Spike (MS) & Matrix Spike Duplicate (MSD)

2. Duplicate samples
   a. Samples will be selected if identified and requested by customer
   b. If no samples are identified by the customer then random samples will be analyzed within the batch as defined by the method, program or at a minimum batch of 20 samples.

3. Matrix Spike (MS) / Matrix Spike Duplicate (MSD) samples
   a. Samples will be selected if identified and requested by customer
   b. If no samples are identified by the customer then random samples will be selected and analyzed within the batch as defined by the method, program or at a minimum batch of 20 samples.
   c. If MS/MSD is not required, LCS/LCSD may be substituted for

precision and accuracy evaluation.
All DOD projects require MS/MSD.

The frequency is dependent on the reference method and test protocol. The following is the default requirement for quality control checks in lieu of any other guidance. The frequency for each quality control sample is generally one (1) per every 20 samples.

5.9 Data Reduction

After completion of the test procedure, the data reduction process begins.

Chromatography data may require the manual integration of peak areas or heights before reporting of results. The analyst must perform manual integration when software does not properly integrate or identify the peak. Manual integration must not occur for the purpose of achieving acceptable quality control or calibration. Signatures of analyst performing manual integrations can be found by electronic entry of analysts initials that can be traced to original signatures in the "Employee Signature Register". The analyst notes the rationale for performing the manual integration using the M-Codes listed in the manual integration SOP 1731 and ensures the "TIC" marks from the software represent the integration area used for reporting the results. The analyst must minimize and avoid manual integration. The establishment of the proper integration parameters in the software reduces the number of manual integration occurrences.

The SOP for each test presents the formulas used for the specific test method. The formulas for the data calculations used throughout the laboratory are the following:

% Recovery (LCS)

$$\frac{MV}{TV} * 100 = \%R_{LCS}$$

where:
MV = Measured Value
TV = True Value

% Recovery (MS or MSD)

$$\frac{MV - SV}{TV} * 100 = \%R_{MS}$$

where:
MV = Measured Value
TV = True Value
SV = Amount found in sample

Average ($\overline{X}$)

$$\sum_{i-1}^{n} X_i \Big/ n = \overline{X}$$

where:
$\overline{X}$ = Average of all values
X = Result of each measurement
n = Number of values

Relative Percent Difference (% RPD)

$$\frac{R_1 - R_2}{\left(R_1 + R_2\right)/2} * 100 = \%RPD$$

where: R_1 = Larger of two observed values
R_2 = Smaller of two observed values

% Difference (%D)

$$\frac{X - \overline{X}}{\overline{X}} * 100 = \%D$$

where: $\overline{X}$ = Average of all values
X = Result of measurement

Standard Deviation of the sample (S_x)

$$\sqrt{\frac{\sum\left(X - \overline{X}\right)^2}{n-1}} = S_x$$

where: $\overline{X}$ = Average of all values
X = Result of each measurement
n = Number of values

Relative Standard Deviation (%RSD)

$$\frac{S_x}{\overline{X}} * 100 = \%RSD$$

where: $\overline{X}$ = Average of all values
Sx = Standard Deviation (n - 1)

Range of Logs (for microbiological enumeration analysis)

10% of routine samples are analyzed in duplicate and the range of logs is determined.

MDL (See 40CFR Part 136 for details)

where: *MDL* = The method detection limit

$$\left[\sqrt{\frac{\sum_{i=1}^{n} x_i^2 - \left(\sum_{i=1}^{n} x_i\right)^2 \Big/ n}{n-1}}\right] * t_{0.99} = MDL$$

X = Result of each measurement
n = Number of values
t(n-1,1 = .99) = The students' T value appropriate for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom. (See Students t Test Table)

Reporting Limit (RL)

Lowest calibration standard or greater

Control Limits

Upper Control Limit: $\overline{X} + 3 * S_x = UCL$
Lower Control Limit: $\overline{X} - 3 * S_x = LCL$

Warning Limits

$\overline{X} + 2 * S_x = UWL$

Upper Warning Limit:
Lower Warning Limit: $\overline{X} - 2 * S_x = UWL$

Method of Standard Additions (MSA): (See EPA 7000A for details)

The simplest version of this technique is the single-addition method, in which two identical aliquots of the sample solution, each of volume Vx, are taken. To the first (labeled A) is added a known volume Vs of a standard analyte solution of concentration Cs. To the second aliquot (labeled B) is added the same volume Vs of the solvent. The analytical signals of A and B are measured and corrected for non-analyte signals. The unknown sample concentration Cx is calculated:

$$C_x = \frac{SB\ V_S\ C_s}{(SA - SB)\ V_X}$$

where SA and SB are the analytical signals (corrected for the blank) of solutions A and B, respectively. V_S and C_s should be chosen so that SA is roughly twice SB on the average, avoiding excess dilution of the sample. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure.

Improved results can be obtained by employing a series of standard additions. To equal volumes of the sample are added a series of standard solutions containing different known quantities of the analyte, and all solutions are diluted to the same final volume.

For example, addition 1 should be prepared so that the resulting concentration is approximately 50 percent of the expected absorbance from the endogenous analyte in the sample. Additions 2 and 3 should be prepared so that the concentrations are approximately 100 and 150 percent of the expected endogenous sample absorbance.

The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated to zero absorbance, the point of interception of the abscissa is the endogenous concentration of the analyte in the sample. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate. A linear regression program may be used to obtain the intercept concentration.

5.10 Document Control

The Document Control Procedure (SOP/1729) describes the process for controlled and uncontrolled documents. The use of the revision number allows for the retention of a previous document for historical information purposes.

Every document is assigned a unique identification number, which is present on each page of the document. A master list of documents includes the unique identification. Each controlled copy includes the revision number, published date and page number.

Full document control includes the status of each document: active, inactive or superseded/archived. Inactive documents are procedures not currently requested, but may be in the future. Archived documents are procedures replaced with a later revision. Authorized personnel must review and approve each document and any subsequent revisions before use in the laboratory. Personnel authorized to review and approve a document have access to all necessary information on which to base their review and approval. The history section of the document in Qualtrax includes a description of the nature of the document change.

Standard Operating Procedures (SOPs) are instructions for repetitive or standard operations performed by the laboratory. The SOP author is the person familiar with the topic. The standard format for writing SOPs is set-up as a template for administration and technical SOPs. Each SOP is peer reviewed, authorized by management, and QA before final publication and implementation. Authorized signatories for controlled documentation include one or more of the following personnel: Company President, Quality Assurance Officer, Laboratory Technical Manager, Department Manager, Department Team Leader. Personnel acknowledge approved documents as read, understood and agreed to through electronic attestation forms associated with each document as SOP Attestation Tests which reside in Qualtrax.

SOPs must receive evaluation and input by laboratory supervisors and key technical personnel. The content of each SOP must conform to applicable requirements of analytical methods and certification agencies. Within these constraints, the content of a SOP meets the needs of a particular area of the laboratory. A new or revised SOP is needed when regulatory programs update or add methods, the scope of the existing method is extended, or when activities are being performed without adequate documentation.

Updating, modifying and changing SOPs, forms and the contents of this QSM are prompt and part of the routine practices. The prompt modification of these documents ensures the documents reflect the current practices and operations of the laboratory. During annual review of a document, (including but not limited to: SOPs, Ethics Policy, Quality Systems Manual), requested changes are reviewed and the document reissued using the information and a new revision number is assigned and published in Qualtrax.

The laboratory maintains control over the possession and distribution of all documents that directly affect the quality of data. This includes, but is not limited to, documents such as the Quality Systems Manual, Standard Operating Procedures, customer instructions, Laboratory Work Instructions, data sheets, check lists and forms.

5.11 Detection Limits

Detection Limits (DLs), previously referred to as Method Detection Limits (MDLs), are determined for all analytes as specified in the Institute (TNI) Standards. DLs are determined for all new instrumentation, whenever there is a change in the test method or instrumentation that affects performance or sensitivity of the analysis. From these, detection limits, Reporting Limits (RLs), are established. The RL is the minimum concentration of an analyte that can be identified and quantified within specified limits of precision and bias during routine and analytical operating conditions.

Method Blanks are evaluated to determine an MDLb when performing an initial MDL study and annually thereafter.

Laboratory reporting limits lie within the calibration range, at or above the RL. For methods that require only one standard, the reporting limit is no lower than the low-level check standard, which is designed to verify the integrity of the curve at lower levels. If reporting limits are required below the lower level of the calibration curve, RL, or low-level check standard, method modifications are required. Refer to DL/LOQ SOP/1732. Note: ”J” Estimated value: Upon customer request, the Target analyte concentration can be reported below the quantitation limit (RL), but above the Detection Limit (DL) with a “J” qualifier.

5.12 LOD/LOQ Studies

A. LOD (Limit of Detection) Verification - DOD only

1. LOD is required quarterly for all DOD projects. If there are no DOD projects for a particular quarter than LOD is not required for that quarter.

2. All sample-processing steps of the analytical method shall be included in the determination of the LOD.

3. The validity of the LOD shall be confirmed by ***qualitative*** identification of the analyte(s) in a QC sample in each quality system matrix containing the analyte at no more than 2-3X the LOD for single analyte tests, and > 1X up to 4X the LOD for multiple analyte tests. This verification must be performed on every instrument that is to be used for analysis of samples and reporting of data.

4. An LOD study is not required for any component for which spiking solutions or quality control samples are not available such as temperature. Where an LOD study is not performed, the laboratory may not report a value below the limit of quantitation.

B. LOQ (Limit of Quantitation) Verification

1. LOQ (Limit of Quantitation) verification is required quarterly for each target analyte. The validity of the LOQ shall be confirmed by successful analysis of a QC sample containing the analytes of concern in each quality system matrix. A successful analysis is one where the recovery of each analyte is within the established test

method acceptance criteria for accuracy

The LOQ study is not required for any component or property for which spiking solutions or quality control samples are not commercially available or otherwise inappropriate (e.g., pH)..

Refer to DL/LOQ SOP/1732

5.13 Range of Logs – Precision of Quantitative Methods - Microbiology

A. Precision of duplicate analyses is calculated for samples examined by enumerative microbiological methods according to the following procedure:

   a. Perform duplicate analyses on first 15 positive samples.

   b. Record duplicate analyses as D1 and D2 and calculate the logarithm of each result.

   c. If either of a set of duplicate results is <1, add 1 to both values before calculating the logarithms.

   d. Calculate the range (R) for each pair of transformed duplicates as the mean of these ranges.

6 Personnel

6.1 Laboratory Management Responsibilities

Management is responsible for communicating the requirements of the quality system, customer specifications and regulatory needs to all personnel. Management job descriptions detail the responsibilities of each position.

The H.R. Director has job descriptions for all positions in the laboratory defining the level of qualifications, training, and experience and laboratory skills. During initial training, management provides access to documented operations procedures, observes personnel performance, and

evaluates personnel proficiency. Management documents technical laboratory staff's proficiency initially and on a continuing basis through use of laboratory control samples and purchased proficiency evaluation standards.

Management is responsible for verification of proper sample management and all aspects of data reporting. The communication of the operating practices of the laboratory is through the document control and attestation process.

Either the Quality Assurance Officer, Operations Director and/or Technical Managers have the authority to stop work due to non-conformances and have the authority to resume work after it has been stopped.

6.2 Laboratory Staff Requirements

Recruitment is the responsibility of the Operations Manager and HR Department, with input from other personnel as required. The Training Program procedure SOP/1565 details the process for completing requirements and training to ensure personnel have adequate skills and competence for the job function. Initial training includes ethics training, Qualtrax Training, QA Basics, IT/LIMs including computer security.

A job description details the necessary requirements for each job and includes position title, minimum educational requirements, skills, responsibilities and reporting relationships and any supervisory responsibility.

Initial training of new employees and contract staff includes laboratory ethics and quality policies, signing the Employee Signature Log, as well as execution of an Ethics Agreement. Any employee found to knowingly violate the Ethics Policy Agreement, report data values, that are not actual values obtained or improperly manipulated, or intentionally report dates and times of data analyses that are not the actual dates and times of analysis, will lead to disciplinary action, including termination, as outlined in Section V.K of the Employee Handbook. Each employee must report personally or anonymously to the Laboratory Technical Manager, QA Officer and/or Ethics Team Member any accidental or suspected intentional reporting of non-authentic data by others for follow up action. The review of the laboratory ethics and ethics training occurs annually with all personnel.

(DOD) All inappropriate and prohibited laboratory practices, as detailed in the DOD QSM 5.2, will be reported to the appropriate accrediting body within 15 business days of discovery. Records of corrective actions or proposed will be submitted within 30 business days. Failure to notify the AB within 15 business days will result in suspension of the DOD ELAP accreditation.

The Ethics program consists of the following key components:

- Ethics Policy /Agreement (Appendix F)
- Initial and annual ethics training
- Internal audits conducted annually
- Adherence to Manual Integration SOP/1731
- Ethical or Data Integrity issues reported to Lab Managers, QAO or HR Director
- Anonymous reporting to HR Director - This is accomplished by writing a detailed description of the suspected ethics breach and submitting the information, anonymously, to the Human Resource Director.

- "No-fault" policy encouraging reporting of incidences without fear of retribution

- Electronic tracking and audit trails through LIMs and instruments enabled where available.

6.3 Training

The Quality Systems Manual and related documentation is available to all employees. Cross training, supervisory training and other related training takes place on a scheduled and as-needed basis. Training ensures the communication and understanding of all personnel in the laboratory-documented procedures and practices.

All personnel undertake orientation-training sessions upon initial employment. Orientation training includes laboratory business practices, employment specifications, Ethics Policy, Quality Systems Manual, Chemical Hygiene Plan, and all SOPs required for the job function.

Managers ensure the training for new employees and review the continuing training for current employees. Training includes on-site and off-site programs presented by staff members, contractors, equipment manufacturers, and institutions of higher learning.

Training of new personnel to any job assignment takes place on-site according to the Training Program procedure. Laboratory personnel may perform their assigned methods/protocols without supervision only after documentation of acceptable proficiency. Training records lists the current training status.

On-the-job training includes demonstration of skills during job performance, initial demonstration of proficiency, and review of SOPs. Health and Safety training takes place on an annual basis with careful introduction to new principles. Personnel have access to the Chemical Hygiene Plan and Safety Data Sheets. On-site training includes side-by-side hands-on training, formal classroom type instruction on the SOP or a meeting to discuss procedural changes or to address questions related to the laboratory operation. All training is documented via the Training Attestation Form, which is signed by all in attendance that they understood and will implement what was presented to them.

Training is an on-going opportunity to evaluate the laboratory operations. The updating of SOPs, Quality Systems Manual and other related information documents all changes to the quality system. Training is documented via the Training Attestation Form or in Qualtrax with training test records.

Off-site training takes place on an as-needed basis. Recommendations and suggestions regarding educational programs come from all levels of staff. It is the employee's responsibility to present a copy of any certificates or attendance information to the HR Director. The information is added to the individual's training record.

6.4 Records

The QA Department is responsible for maintaining training records. Certificates, demonstration of capability forms and other records of training are placed in the individual's training file.

Appropriate personnel are notified through email and/or Qualtrax or by the QA department when a revision is complete for the controlled version of a document. The manager of the area determines when a change is significant to require training.

Job descriptions are included in the training record files. The Human Resources Department reviews the job descriptions, Resumes and/or biosketches are kept on file with the Human Resources Department and the QA Department.

7 Physical Facilities – Accommodation and Environment

This laboratory facility has a total area of 25,000 square feet for each of the Westboro and Mansfield Facilities

The laboratory functional areas include:

Administration and offices

Sample receiving

Sample management
Air analysis (Mansfield Facility only)
Microbiological (Westboro Facility only)
General analytical chemistry
Metals sample preparation (Mansfield Facility only)
Organic sample preparation
Metals analysis (Mansfield Facility only)
Volatiles gas chromatography (GC)
Volatiles gas chromatography/mass spectrometry (GC/MS)
Volatiles air analysis (Mansfield Facility only)
Semivolatiles gas chromatography/mass spectrometry (GC/MS)
Semivolatiles gas chromatography (GC)
Miscellaneous facility mechanical and storage areas.

All chemicals are stored in appropriate cabinets and properly disposed of as required. All flammable solvents are stored in OSHA and NFPA approved cabinets. Acids are stored in OSHA acid cabinets. Separate waste areas houses the sample and chemical waste before pickup by a licensed waste hauler.

7.1 Environment

Lighting, noise, humidity, heating, ventilation and air conditioning satisfy the needs of the testing performed on the premises. The laboratory building design ensures regulated temperature control for analytical equipment. Air-handling systems minimize airborne contaminants that may jeopardize sample integrity or analytical performance.

The analytical instrumentation is in separate rooms from laboratory activities that involve the use of large quantities of organic solvents or inorganic acids. A separate room, in the Westboro facility, provides the facilities for the microbiological testing.

Standards and other materials requiring below 0°C storage temperatures are placed in freezers and separated from samples or potential contaminating materials. Refrigerators provide cooling needs for samples and materials with temperature requirements of below room temperature and greater than freezing. Sample and standard storage areas are monitored and controlled for temperature and recorded in the data logger system. Sample storage areas for volatiles are separated from other samples and monitored for any effects due to cross contamination.

Bulk hazardous waste containers are located away from the testing activities. Waste disposal uses lab pack procedures and those designated by the regulatory authorities. The Chemical Hygiene Plan and the Waste Management and Disposal SOPs (Westboro: SOP/1728 and Mansfield SOP/1797)) include the procedures for handling and disposing of chemicals used in the laboratory.

The working and storage environments are maintained in a safe and appropriate manner. A Chemical Hygiene Plan details the requirements for safety and chemical handling. Safety measures that protect property and personnel from injury or illness include: fume hoods, fire extinguishers, fire blankets, alarm systems, safety training, protective clothing, emergency showers, eyewashes, and spill control kits.

7.2 Work Areas

Good housekeeping is the responsibility of all personnel. Each person is responsible for assuring clean and uncluttered work areas. The job descriptions list specific housekeeping duties. Records, samples and waste materials are the common cause for clutter in the laboratory.

. Removal of administration and laboratory records to the record storage area occurs to reduce clutter and ensure traceability. The individual filling the laboratory record box, labels the box with a number, the contents, date and laboratory area. Authorized personnel assign and record into a permanent record the box number, discard date and box contents. Authorized personnel review the box label for number, discard date and contents. Boxes are stored onsite and off-site for the record retention period identified in the NELAC Institute (TNI) Standards and EPA regulations, whichever is more stringent.

Sample management personnel remove samples to the sample storage area after all data is correct and complete. Sample coolers are removed to a designated storage area for recycling. Samples are stored in the designated process storage areas until testing is complete. Sample removal from the process storage occurs after mailing of the final report. The sample management staff places the samples in the archive storage area for thirty days after report release. The archive sample storage area is not controlled or monitored. Based on customer specifications, samples are properly disposed or returned to the customer.

Waste materials, expired reagents, expired standards and materials are disposed of and not stored in the laboratory. Hazardous waste labeled accumulation containers in the laboratory collect designated waste streams for later bulk disposal. Laboratory personnel remove the less than five-gallon accumulation containers when full from the laboratory and place the containers in the bulk hazardous waste area. Refer to the Waste Management and Disposal SOPS for Westboro: SOP/1728 and Mansfield SOP/1797. Personnel identifying out of date reagents and standards remove the materials to the proper disposal area.

7.3 Security

Alpha Analytical provides a secure environment for our employees, guests, customers, samples and analytical data. Security procedures require that all exterior doors remain locked unless manned. Access to the laboratory is limited to employees and contractors. Visitors not under signed contract are required to sign the Visitors Log and must be accompanied by a laboratory employee at all times within the testing areas.

The defined high security area is the sample management area. Identification card locks on the internal doors control entry into the laboratory area.

All doors are locked after hours and require a key for entry. The security alarm continuously monitors for smoke and fire related heat. When the alarm is activated, the appropriate emergency response officers are notified. The local emergency offices have the emergency contact list for the laboratory.

8 Equipment and Reference Materials

8.1 Maintenance

The laboratory has a proactive equipment maintenance program. The laboratory maintains service contracts for most major equipment, which include routine preventative maintenance visits by the service provider. Technical personnel perform manufacturer's specified maintenance on a routine basis to ensure equipment operates at peak performance.

A brief summary of some common preventive maintenance procedures is provided in Appendix D. All instrument preventative and corrective maintenance is recorded in the maintenance logbook assigned to the equipment. After maintenance or repair, the instrument must successfully calibrate following the method SOP. Laboratory personnel must demonstrate quality control performance before sample analysis.

The laboratory maintains a stock of spare parts and consumables for analytical equipment. Backup instrumentation for some analytical equipment is available on site for use in case of major equipment failure. The person discovering or suspecting an equipment maintenance problem or failure tags the equipment with 'out of service' tag. If routine maintenance measures do not eliminate the problem, the Laboratory Technical Manager or Operations Director is notified and the appropriate equipment service provider is contacted.

All major laboratory equipment has individual and traceable maintenance logbooks in which to document manufacturer's recommended maintenance procedures, specific cleaning procedures, comments on calibration, replacement of small worn or damaged parts, and any work by outside contractors. The person performing routine or non-routine maintenance signs and dates the maintenance logbook. If an instrument is down for maintenance, a complete record of all steps taken to put it back into service is recorded including reference to the new calibration and quality control checks. Any equipment service providers working on the equipment are recorded in the logbook.

Record repetitive or on-going equipment problems other than normal maintenance requirements on nonconformance action forms. The nonconformance action form notifies management and the Quality Assurance Officer of a problem affecting the performance and data quality.

The laboratory groups some equipment into a single laboratory equipment maintenance logbook. Examples include: autopipets, thermometer calibration. The identity of each item is by serial number or a laboratory-designated item number. The same data recorded for major equipment applies to this documentation.

The maintenance records shall include:

Equipment name;

Manufacturer's name, type identification, serial number or other unique identification;

Date received, date put into service, condition when received;

Current location;

Details of past maintenance and future schedule;

A history of any damage, malfunction, modification or repair;

Dates and results of calibration or verification.

The maintenance logbook may include the reference to the location of the equipment operational and maintenance manuals. The logbook may include the reference to laboratory run logbook or data files for the calibration and quality checks of daily or frequent calibrations.

The Courier Supervisor ensures that maintenance and records for transportation vehicles are complete. The purchasing process is used for ordering garage maintenance, the garage work order is reviewed, and the vehicle checked for condition. The Controller receives all paperwork for completion of the maintenance process.

8.1.1 Microbiology General Equipment Maintenance

Optics of the Quebec colony counter and microscope are cleaned prior to each use. The stage of the microscope is also cleaned and the microscope is kept covered when not in use.

Glassware is checked for residual alkaline or acid residue utilizing bromothymol blue (BTB) on each day of media preparation.

8.2 Equipment Listing

A listing of the major equipment used for testing is available upon request. The equipment list details the unique identification number, equipment location, serial number, model number, and purchase date. The unique identification number is attached to the piece of equipment.

The laboratory performs analyses using state of the art equipment. In addition to the major equipment, the most common equipment used in the laboratory are: thermometers, balances, autopipets, water baths, hot plates, autoclaves, pH meters, conductivity meters and a variety of labware. The SOPs list the calibration and verification requirements for all laboratory equipment used in measurements.

8.3 Laboratory Water

Laboratory water is purified from central DI and RO water systems and piped to all laboratory areas. The QA Department samples the laboratory grade water and submits the samples for analysis by the lab to document the water meets the drinking water certification criteria. The Laboratory Water Logbook lists the daily conductivity checks and acceptance criteria for the laboratory water. The laboratory documents the daily, monthly and annual water quality checks. Please refer to Table 8-1 for tested parameters, monitoring frequency and control limits for each parameter (SOP/1738). Additional parameters may be tested for at the laboratory's discretion.

When additional treatment occurs in the test area, that test area records the water quality checks from the most frequently used tap. At a minimum the quality of the laboratory grade water is monitored daily by conductivity measurements. Records of the daily checks are found in the Laboratory Water Logbook. If out of specification results occur, a nonconformance action form is submitted.

TABLE 8-1

Parameter	Monitoring Frequency	Control Limits
Conductivity	Daily	<2 µmhos/cm @ 25°C
pH	Daily	5.5 - 7.5
Total Organic Carbon	Monthly	< 1.0 mg/L
Total Residual Chlorine	Monthly	< detection limit
Ammonia	Monthly	< 0.1 mg/L
Metals: Cd, Cr, Cu, Pb, Ni and Zn	Monthly (Required Annually)	< 0.05 mg/L
Total Metals	Monthly (Required Annually)	< 0.1 mg/L

Heterotrophic Plate Count (Westboro only)	Monthly	< 500 CFU/mL
Water Quality Test (Biosuitability) (Westboro only)	Annually	0.8 – 3.0 ratio

8.4 Reference Materials

Reference materials include: Class 1 weights, NIST thermometers and reference standards. Timers used for DOD projects are NIST-certified. Logbooks record the reference materials used for calibration and verification. The Department Manager or QA Department maintains any certificates received with the reference materials. Laboratory personnel record in the standards logbook the reference standards date received, unique identification number, expiration date and number of containers. Each laboratory area records the unique identifier on the reference standard certificate and the Department Manager maintains the certificate. The identifier allows traceability from the certificate to the analytical data.

9 Measurement Traceability and Calibration

9.1 General Requirements

All measuring operations and testing equipment having an effect on the accuracy or validity of tests are calibrated and/or verified before put into service and on a continuing basis. The results are recorded in the instrument specific logbook. The laboratory has a program for the calibration and verification of its measuring and test equipment. The program includes all major equipment and minor equipment such as balances, thermometers and control standards. The Quality Systems Manual and method SOP describe the calibration records, frequency and personnel responsibilities.

9.2 Traceability of Calibration

The program of calibration and/or verification and validation of equipment is such that measurements are traceable to national standards, where available. Calibration certificates indicate the traceability to national standards, provide the results, and associated uncertainty of measurement and/or a statement of compliance with identified metrological specifications. A body that provides traceability to a national standard calibrates reference standards. The laboratory maintains a permanent file of all such certifications.

9.3 Reference Standards and Materials

Alpha Analytical has a program for calibration and verification of reference standards. The results and program are recorded in the appropriate instrument logbook. Required in-service checks between calibrations and verifications are described in method SOPs and are recorded in the appropriate instrument logbook.

Calibration standards are maintained within the area of consumption. A logbook of use is maintained and use is limited strictly to method required calibrations. Each calibration standard is identified as to test method used, date received, date opened, and expiration date. Calibrations are verified by using a second source or lot number of the calibration standard. Calibration check procedures are stated in applicable test method SOPs.

Preparation of standards must be performed using Class A glassware. Class A glassware must be used for all processes involving quantitative analyses.

Reference standards of measurement in the laboratory's possession (such as calibration weights or traceable thermometers) are used for calibration only and for no other purpose.

Standards and reagents are uniquely identified as outlined in Westboro SOP 1745 and Mansfield SOP 1816.

9.4 Calibration General Requirements

Each calibration record is dated and labeled with method, instrument, analysis date, analyst(s) and each analyte name, concentration and response. For electronic processing systems that compute the calibration curve, the equation for the curve and the correlation coefficient are recorded in the appropriate instrument logbook. This is also true for manually prepared curves. Calibrations are tagged to the specific instrument through use of the instrument logbook and or sequence file documentation.

Initial calibration requires a standard curve that brackets the expected sample concentration. Initial calibration generally uses three to five standards depending on the equipment and reference method specifications. Before the start of each analytical sequence, initial calibration is

verified by using a continuing calibration standard. Calibration verification or continuing calibration uses the same standard as the ICAL unless method specifies otherwise. The ICV is from a second source or lot number than that used for initial calibration. The acceptance criteria for the continuing calibration standard must meet acceptance criteria before analysis of any samples. When the acceptance criteria is not within limits, review maintenance protocols and perform any necessary maintenance before starting the initial calibration sequence.

9.5 Equipment Calibration

The SOP used for the analysis defines the instrument and equipment calibration required. The following defines the general practices for equipment calibration of selected equipment.

9.5.1 Gas Chromatography/Mass Spectrometry (GC/MS)

The GC/MS is hardware tuned before performing the initial and continuing calibrations. Results must meet the peak ratio specifications of the analytical methods. For volatiles analyses, bromofluorobenzene (BFB) is used, and for semivolatiles analyses, decafluorotriphenylphosphine (DFTPP) is used for instrument tuning.

The mass spectrometer response is calibrated by analyzing a set of five or more initial calibration solutions, as appropriate, for each GC/MS method. Each solution is analyzed once, unless the method or the customer requires multiple analyses. The relative response factor for each analyte is calculated for internal standard calibration. The calibration factor for external standard calibration is calculated using the expressions found in the laboratory method SOP. Calibration is acceptable when all acceptance criteria are within method criteria.

The initial calibration is verified through the analysis of a continuing calibration standard every 12 hours. The concentration of the continuing calibration standard is dependent on the requirements of the specific method. The relative response factors for all analytes of interest are calculated and verified against the initial calibration mean relative response factors. The percent difference (%D) for each analyte is calculated and must be less than the acceptance criteria stated in the method.

An acceptable continuing calibration run must have measured percent differences for the analytes within method specified ranges. If any criteria for an acceptable calibration are not met, either instrument maintenance must be performed until the continuing calibration analysis meets all criteria or a new initial calibration is established before any samples are analyzed. No samples may be analyzed unless the acceptance criteria are met for the initial and continuing calibration.

Additional quality control samples are part of the GC/MS analysis. These include internal standards, surrogates, method blanks, instrument blanks, laboratory control samples, matrix spikes and matrix spike duplicates. The frequency and control criteria are defined in the laboratory SOP.

9.5.2 Gas Chromatography (GC)

Internal standard calibration or external standard calibration is utilized for analysis by GC. The method-specified number of calibration standards is used. Each solution is analyzed once and the analyte relative response factors or calibration factors are calculated. The mean relative response factor for each analyte is then obtained by using the expression in the formula listed in the SOP. Integrated areas are utilized for these expressions.

For multiple response pesticides, PCBs or hydrocarbons the quantitation consists of the average of selected peaks or the integration of the area defined by a reference standard. The SOP details the integration criteria for each compound.

The initial calibration is verified through the analysis of a continuing calibration standard every 12 hours or 20 samples. The concentration of the continuing calibration standard is dependent on

the requirements of the specific method. The relative response factors for all analytes of interest are calculated and verified against the initial calibration mean relative response factors. The percent difference (%D) for each analyte is calculated. The percent drift (%d) may be calculated when calibration factors are used for quantitation.

An acceptable continuing calibration must have measured percent differences or percent drift for the analytes within method specified ranges. Should any criteria for an acceptable calibration not be met, either instrument maintenance is performed until the continuing calibration analysis meets all criteria, or a new calibration is established before any samples are analyzed. No samples may be analyzed unless the acceptance criteria are met for the initial and continuing calibration.

Other standard checks may be required for a specified reference method. Instrument performance checks specified in the reference method must be performed and be within the acceptance limits stated in the reference method. Additional quality control samples are part of the GC analysis. These include internal standards, surrogates, method blanks, instrument blanks, laboratory control samples, matrix spikes and matrix spike duplicates. The frequency and control criteria are defined in the laboratory SOP.

9.5.3 Cold Vapor Atomic Absorption Spectrophotometry (CVAA)

An initial calibration is performed daily with freshly prepared working standards that bracket the expected concentration range of the sample. A minimum of a three-point calibration curve is acquired which must have a correlation coefficient of 0.995 or better. The initial calibration is verified every 10 samples. The continuing calibration is required to be within method-defined criteria, depending on the analytical method employed. Continuing calibration blanks are run at the same frequency. Analysis of samples cannot begin until an initial calibration verification has been performed and is found to be within ± 10% of the true value.

9.5.4 Inductively Coupled Plasma Emission Spectrophotometry-Mass Spectrometry (ICP-MS)

Initial calibration and instrument tune is performed daily, not to exceed 24 hours, and continuing calibrations are performed every 10 samples. Initial calibration consists of a minimum of three standards and a Blank that bracket the expected concentration range of the samples. Analysis of samples cannot begin until an initial calibration verification has been performed and is found to be within method-defined criteria. The continuing calibration is required to be within method-defined criteria. Interference check standards are performed at the beginning of the sequence. Acceptance criteria are stated in the SOP.

9.5.5 Inductively Coupled Plasma Emission Spectrophotometry (ICP)

Initial calibration is performed daily, not to exceed 24 hours, and continuing calibrations are performed every 10 samples. Initial calibration consists of one standard and a Blank that bracket the expected concentration range of the samples. Analysis of samples cannot begin until an initial calibration verification has been performed and is found to be within 5% of the true value for EPA Method 200.7 and 10% for SW846 6010 methods. The continuing calibration is required to be within 10% of the true value. Interference check standards are performed at the beginning and end of the sequence. Acceptance criteria are stated in the SOP.

9.5.6 Thermometers

Laboratory thermometers are checked annually for accuracy against certified, NIST traceable thermometers. Correction factors derived from the annual calibrations are applied to temperature readings where applicable. The analyst records the corrected temperature for all observations.

NIST traceable thermometers are calibrated professionally and re-certified every year. Records of thermometer calibrations are retained by the QA Department. All thermometers are tagged with the ID number, correction factor to be applied and the expiration of the calibration check.

NOTE: Electronic-based thermometers are calibrated on an annual basis. Thermometers are tagged with calibration information by the vendor, including the ID number, correction factor to be applied and the expiration of the calibration check. Certificates are kept on file in the QA Department.

Thermometers are not used past the calibration expiration date or if the thermometer is not reading properly. Replacement thermometers are calibrated and the maintenance logbook is updated when a change in the thermometer is required due to breakage, damage or expired calibration.

9.5.7 Balances

Calibration checks are performed for each day of use, for each balance. The calibration consists of a minimum of two weights, which bracket the weight to be measured. Additional calibration check procedures are performed on balances utilized in Microbiology laboratory. This additional procedure consists of a deflection test, which is performed to ensure that 100mg is detectable at a weight of 150 grams.

The balance logbook lists the acceptance criteria and performance criteria for the various balances used in the laboratory. Calibration weight measurements must meet the acceptance criteria listed on the record form.

Each balance is serviced and calibrated by a professional semi-annually. Balances are labeled with the balance number, date of service and the expiration date for the annual service check. The balance number used for any measurements requiring traceability is recorded with measurement data. Balances are not used past the expiration date or when the weight check is not within acceptable criteria. The accuracy of the calibration weights used by Alpha Analytical is verified annually by an accredited calibration service.

9.5.8 Mechanical volumetric pipettes

Delivery volumes for the mechanical volumetric pipettes (i.e. Eppendorf) are checked and recorded gravimetrically before use and on a quarterly basis. The verification is performed at the volume of use or bracketing the volume range of use. The check must be within the criteria stated in the laboratory logbook. Pipettes failing acceptance criteria are tagged and removed from service until repaired and the criteria are met, or discarded and replaced. Automatic pipettes are labeled with a unique ID number, volumes verified and expiration date.

9.5.9 Ion Chromatography

The ion chromatograph calibration is by analyzing a set of five or more initial calibration solutions, with concentrations of analytes appropriate to the analytical methods. The concentrations must bracket the expected concentration range of the samples analyzed. Procedures for verifying the calibration curve are method specific. The initial calibration is performed at the start of each day. The calibration curve is verified at least after every 20 samples.

9.5.10 pH Meters

pH meters are calibrated prior to use for each day of use. The meter is calibrated following the procedure for pH analysis. The records of the calibration are recorded in an instrument logbook or in the raw data for the analysis being performed. At least two buffer solutions that bracket the measurement range for the analysis are used for calibration. A second source check standard is used at the end of a run to verify meter stability. Buffer solutions used for calibration are NIST

certified. Standard buffer solutions are not retained or re-used. The lot number of the buffer solutions is recorded in the data record to ensure traceability of the measurement to NIST.

9.5.11 Conductivity Meters

Three calibration standards of potassium chloride (KCL) solutions are analyzed annually on each instrument range. The calibration standards are used to verify instrument performance. The acceptance criteria are defined in the test SOP. If unacceptable performance is found, the cell is cleaned and rechecked. The cell is not used until satisfactory performance is achieved.

A single KCL standard solution is used to calibrate each range of the instrument. A second standard is used to check the calibration each day the meter is used. The check standard is near the measurement range for the samples to be analyzed. The acceptance criterion is ± 20% of the true value. The meter is labeled with expiration date for the annual calibration. A check standard that is NIST traceable is used to allow traceability. The check standard is performed at the end of the analysis run or at least after every 20 samples.

9.5.12 Autoclave

The date, contents, sterilization time and temperature, total cycle time and analyst's initials are recorded each time the autoclave is used. Autoclave cycles must be completed within 45 minutes when a 15 minute sterilization time is used. Autoclave timing mechanisms are checked quarterly with a stopwatch to verify timing controls. A maximum temperature thermometer is used with each cycle to ensure the sterilization temperature is reached.

Spore strips or ampoules are used weekly to confirm sterilization. BTSure ampoules are utilized as follows: An indicator ampoule is placed in most challenging area of sterilizer. Load is processed according to standard operating instructions. Remove from sterilizer and allow to cool for a minimum of 10 minutes. (Chemical indicator on label changes from green to black when processed.) Place the autoclaved indicator and un-autoclaved control indicator in an upright position in the plastic crusher provided. Gently squeeze crusher to break glass ampoules. Incubate both indicators at 55-60^{o}C for 24 hours. Examine appearance for color change. Yellow color indicates bacterial growth. No color change indicates adequate sterilization.

Calibration is conducted and certified annually by an outside service provider and recorded. Certificates are kept on file. Routine maintenance includes cleaning the autoclave seal to ensure freedom of caramelized media and cleaning drain screens to remove any debris buildup. For the efficient operation of the unit, overcrowding is avoided.

10 Test Methods and Standard Operating Procedures

10.1 Methods Documentation

Analysis consists of setting up proper instrument operating conditions, executing acceptable calibrations, monitoring instrument performance tests, analyzing prepared samples, and collecting data from the analyses. The test method SOP describes the instrumental analysis procedures, quality control frequencies and acceptance criteria. EPA accepted methods, national recognized methods or customer-specified methods are the basis for performance criteria, instrument conditions and the steps of the procedure. The method performance requirements of the published methods are followed unless otherwise specified by the customer.

The reference methods define the instrument operating conditions. In many of the reference methods, a range or general guidance on the operating conditions is defined. Documented modifications to the operating conditions clarify the reference methods or improve the quality of the results. In all cases where the method modifications are adopted, the performance criteria from the reference method must be met. Modifications to the operating conditions are stated in the SOP. Changes in the operating conditions made at the time of the analysis are documented in the appropriate laboratory or sequence log. A revision to the SOP takes place, when a day to day change in the operating condition improves performance for all matrices.

The laboratory SOPs include the operation of measurement equipment. The SOPs contain the - following information, as applicable:

- The equipment used in the procedure, including equipment type
- Equipment calibration and process for obtaining the measurement from the calibration
- The step by step instructions to perform the measurement
- Acceptance criteria for the calibrations
- Corrective action for failed acceptance criteria, including assessment of previous calibration results
- The basis used for the calibration standards such as traceability to NIST or EPA or demonstration of comparability
- Frequency at which the equipment will be calibrated, adjusted and checked
- The records maintained to document the calibration and use of measurement equipment
- The calibration status for the equipment
- The environmental conditions necessary before measurement equipment may be calibrated or used for measurement
- Allowed adjustments to measurement equipment, including software, which will not invalidate the laboratory analysis
- Maintenance of the equipment and record keeping to track performance before and after maintenance is completed
- Define the standards, reagents and sample handling, interferences, preservation, and storage in order to assure measurement performance

10.2 Standard Operating Procedures (SOPs)

Alpha Analytical maintains SOPs that accurately reflect all phases of current laboratory activities such as assessing data integrity, nonconformance actions, handling customer complaints, sample receipt and storage, purchasing of all materials, and all test methods. These documents include equipment manuals provided by the manufacturer, internally written documents, and published methods with documented changes or modifications.

Copies of all SOPs are accessible to all personnel in electronic form through Qualtrax. Each SOP clearly indicates the published date of the document and the revision number.

10.3 Laboratory Method Manual (s)

All SOPs are posted as secure documents in the Alpha Qualtrax system. Directories are available for each laboratory area and administrative area in appropriate subfolders. Each SOP includes or references where applicable:

1) identification of the test method and where applicable;
2) applicable matrix or matrices;
3) method detection limit;
4) scope and application;
5) summary of method;
6) definitions;
7) interferences;
8) safety;
9) equipment and supplies
10) reagents and standards
11) sample collection, preservation, shipment and storage;
12) quality control;
13) calibration and standardization;
14) procedure;
15) calculations;
16) method performance;
17) pollution prevention;
18) data assessment and acceptance criteria for quality control measurements;
19) corrective actions for out-of-control data;
20) contingencies for handling out-of-control or unacceptable data;
21) waste management;
22) references; and
23) any tables, diagrams, flowcharts and validation data.

In cases where modifications to the published method have been made by the laboratory or where the referenced method is ambiguous or provides insufficient detail, these changes or clarifications are clearly described in the SOP.

10.4 Test Methods

The laboratory uses appropriate methods and procedures for all tests and related activities within its responsibility (including sampling, handling, transport and storage, preparation of items, estimation of uncertainty of measurement and analysis of test data). The method and procedures are consistent with the accuracy required, and with any standard specification relevant to the calibrations or tests concerned. When the use of mandated methods for a sample matrix is required, only those methods are used. Where methods are employed that are not required, the methods are fully documented and validated and are available to the customer and other recipients of the relevant reports.

The customer requests the reference method for sample analysis usually based on the regulatory program. The customer services staff may assist the customer with method selection when the customer specifies the regulatory program, but is unsure of the correct method required. The Laboratory Technical Manager or Quality Assurance Officer recommends methods for non-regulatory programs. In all cases, recommendation of methods is based on customer-defined method performance criteria. Customer services may recommend a procedure that meets the customer method performance criteria.

10.5 Method Validation/Initial Demonstration of Method Performance

Before acceptance and use of any method, satisfactory initial demonstration of method performance is required. In all cases, appropriate forms are completed and retained by the laboratory and made available upon request. All associated supporting data necessary to reproduce the analytical results is retained. Initial demonstration of method performance is completed each time there is a significant change in instrument type, personnel or method.

10.6 Sample Aliquots

The aliquot sampling process from a submitted sample is part of a test method. The laboratory uses documented and appropriate procedures and techniques to obtain representative sub-samples. Sample aliquots removed for analysis are homogenized and representative portions removed from the sample container. Personnel record observations made during aliquot sampling in the test method logbooks.

10.7 Data Verification

Calculations and data transfers are subject to appropriate checks which is a 3 tier approach. The initial analyst verifies all of his work, a secondary review of 100% of the initial is conducted by a an independent qualified analyst. A Customer Services representative reviews data for project and method performance requirements where applicable. A QA representative reviews data for project and method performance requirements when requested by a Customer. Final report review is performed by an authorized company signatory.

For drinking water suppliers, every effort is made to notify the Customer within 24-hours of obtaining valid data of any results that exceed any established maximum contaminant level or reportable concentration. Analyst or Department Supervisor notifies the Customer Services Department of the sample number(s), Customer name, analysis and sample results (preliminary or confirmed). The Customer Services Department notifies the customer.

The laboratory Report Generation and Approval SOP describes the practices to ensure that the reported data is free of transcription errors and calculation errors. Manually entered data into the LIMS is dual entered and checked by the LIMS to minimize transcription errors. The laboratory test method SOP describes the quality control measures used to assure method performance before reporting data.

10.8 Labeling of Standards and Reagents

The purchase, receipt and storage of consumable materials used for the technical operations of the laboratory include the following:

a) The laboratory retains records of manufacturer’s statement of purity, of the origin, purity and traceability of all chemical and physical standards.
b) Original reagent containers are labeled with the date opened and the expiration date.
c) Detailed records are maintained on reagent and standards preparation. These records indicate traceability to purchased stocks or neat compounds and include the date of preparation and preparer's initials.

d) Where calibrations do not include the generation of a calibration curve, records show the calibration date and type of calibration standard used.
e) All prepared reagents and standards are uniquely identified and the contents are clearly identified with preparation date, concentration and preparer's initials. These procedures are outlined in Westboro SOP/1745 and Mansfield SOP/1816.

10.9 Computers and Electronic Data Related Requirements

Computers or automated equipment are used for the capture, processing, manipulation, recording, reporting, storage or retrieval of test data. The laboratory ensures that computer software and firmware is documented and adequate. The goals of the software development methodology, existing system validations and the change control system are to ensure that:

the software systems perform the required functions accurately,

the users understand how to use the system, and

auditors can assure themselves of the validity of the analytical data.

The computer systems used at Alpha Analytical are purchased. A coordinated effort is made with the supplier to assure the computer operations meet the laboratory requirements for data integrity. Alpha Analytical has a formal validation program of its computer systems. The validation program is a comprehensive program to ensure data transmitted, reported or manipulated by electronic means is correct and free of errors. The validation and verification approach is separated into three areas.

1. New software is developed and validated using test data. Records of validation include the test data report, date and initials. Where formulas are part of the program, documentation includes manual verification of the final calculated values. New software includes the development of macros for spreadsheets and other tools using commercial software packages.

2. Reasons for changes to software are identified through flaws in existing documentation or the need to improve system processes and are documented on the Nonconformance Report. Final implementation of the change is documented on the nonconformance action form. The tracking and timelines of making the change is readily available. This process also provides the complete documentation of all software and electronic data reporting problems. All nonconformance identified with electronic data process result in corrective action that are reported to management before or at the bi-weekly executive meeting. Customers will be notified prior to any changes to software or hardware that will adversely affect customer electronic data. This information is provided by IT department to QA and Project Managers to be communicated to appropriate customers.

Verification of system integrity is through routine maintenance, protection from unauthorized access and electronic verification programs. Routine maintenance including system backups are performed on a scheduled basis. The backup process and password and access protections are defined in the Computer System Backup Control SOP/1562 and Computer Security SOP/1563. Electronic verification may be used to assure the commercially purchased software is performing at its original specifications. This includes virus checking of all network operation at least once per week. Documentation of all verification and maintenance operations is retained.

11 Sample Handling, Sample Acceptance Policy and Sample Receipt

The Sample Login and Custody procedures define the process for sample management from sample receipt through analysis and to disposal. These procedures detail the process for sample receipt, records and storage pending analysis.

Customers or Alpha's Couriers deliver samples to the laboratory during normal business hours. Sample receiving occurs in the sample management area.

Customer service personnel place bottle orders. The orders are filled following the bottle order instruction form. Blanks are prepared as needed with minimal storage. All glass containers are packed to minimize or prevent breakage. The containers are placed in plastic coolers or shipping packages and Chain-of Custody forms, seals (if requested) and labels enclosed. The bottle order is shipped by third party, picked up by the customer or customer representative or delivered by Alpha courier to the customer.

11.1 Sampling Supplies

11.1.1 Sample Containers

Sample containers provided by Alpha Analytical include labels, preservatives and a blank chain of custody form. Preservatives and containers are lot controlled and verified as appropriate for the indicated type of analysis.

Each lot of containers used for the collection of samples for microbiological analysis is checked for sterility prior to distribution. Sterility checks are performed by Microbiology staff and results recorded in Microbiology Sample Container Sterility Log.

Sample Containers for collecting Air samples (TO-15) are cleaned and prepared according to SOP 2190 "Cleaning and Preparation Procedures for Equipment used to collect Air sample for analytis of Volatile Organic Compounds".

11.1.2 Chain of Custody

Chain of custody forms must accompany all samples received by Alpha personnel. The chain of custody form indicates the sample origin and arrival at the laboratory and identifies the analyses requested.

11.1.3 Reagent Water

Alpha Analytical supplies laboratory pure water for field QC blanks. Water used for volatile organics must be free of volatile compounds below the method detection limit. The quality of the laboratory water is monitored for conductivity once per day. Additional water quality criteria may be monitored based on customer specific requests. The water quality in the laboratory is monitored for chemical parameters as required by the EPA certification manual for drinking water (Water Quality Monitoring SOP/1738).

11.2 Sample Tracking

Alpha Analytical uses an internal chain-of-custody in LIMs for sample tracking control purposes. When requested or required by regulation a legal custody program is used in addition to the routine laboratory practices. Legal custody practices must be arranged at the time of contractual commitment.

For legal custody the process must include complete and continuous records of the physical possession, storage, and disposal of sample containers, collected samples, sample aliquots, and sample extracts or digestates. For legal custody a sample is in someone's custody if:

1. It is in one's actual physical possession;
2. It is in one's view, after being in one's physical possession;
3. It is in one's physical possession and then locked up so that no one can tamper with it;
4. It is kept in a secured area, restricted to authorized personnel only.

The routine sample handling and tracking process includes unique identification of all sample containers, initials of the person removing the sample from the sample management area and documentation of the date of sample removal for disposal.

Samples are assigned a unique identification number from the LIMS program. Each sample container label includes a unique identifier for the container. The person handling the sample is recorded along with the unique identifier in the container tracking records in LIMS.

ALPHA ANALYTICAL utilizes a custom designed Laboratory Information Management System (LIMS) to uniquely identify and track samples and analytical data throughout the facility. The LIMS log-in, is initiated by the Sample Custodian when the following information is entered into the computer:

- Quote number (unique to the project if requested)
- Project name or description
- Analyses requested (per matrices received)
- Sample number (unique to this sample)
- Sample descriptions (customer ID, including number of received containers)
- Date received
- Date(s) and time(s) collected
- Date analytical results are due

11.2.1 Chain of Custody

Chain of custody forms must accompany all samples received by Alpha personnel. The chain of custody form indicates the sample origin and arrival at the laboratory and identifies the analyses requested.

- Customer's name and address
- Notation of special handling instructions
- Additional comments or instruction for the laboratory
- Purchase order number(s), if applicable

Alpha Job Numbers (Process for assigning numbers)

Alpha Job Numbers are unique #'s automatically designated by our LIMS computer system for every individual customer project.

There are 3 parts to this number:

- All numbers start with the letter "L"
- The next two numbers are the last two numbers of the current year.
- The last five numbers are pulled sequentially by the LIMS as each Login personnel requests a new number for a job.

For example…. L0904165 ---- Year 2009 and 4,165th job to be logged in this year.

The Alpha Job Number then may contain as many extensions as there are individual samples in a job. L0904165-01 is the first sample, L0904165-02 is the second and so on. Each sample may contain as many as 26 containers as the containers are designated with the letters of the Alphabet, and each container receives its own bar-coded label. For example, L0904165-09A is the first container of the 9th sample listed on a customer's Chain of Custody.

Each container is labeled with a unique identifier, a label with a unique identifier number is placed on each sample container. Once labeled, the sample containers are placed in the appropriate storage area.

11.3 Sample Acceptance Policy

The sample management personnel check for proper sample labeling, preservation and handling at the time of arrival at the laboratory. The customer and customer services manager specifies the proper sample preservation, containers, cooling and other criteria on the project review form and in the LIMS. Sample management staff record all observations and immediate notify customer services of any discrepancies or questions arising during sample receipt.

It is possible for samples or sample containers to be lost, damaged, or determined to be unsuitable, for whatever reason, after initial receipt at Alpha Analytical. The problem is brought to the attention of a customer services manager who reports it to the customer. Plans for disposition of the affected samples or container are agreed upon with the customer, carried out, and recorded in the project records. Sample hold times and preservations are listed on the Alpha website (www.alphalab.com) under Support Services "Sampling Reference Guide".

11.4 Sample Receipt Protocols

The sample management staff receives all samples. A unique job number is assigned to each shipment of samples received from a customer. The in-house records for the incoming job, including the internal Chain-of -Custody, are initiated with a Sample Delivery Group (SDG) form. The customer, and Alpha courier and/or the sample management personnel sign the sample custody form at the time of receipt at the laboratory. Samples received via overnight courier are signed on the bill of lading. The bill of lading, SDG form and the sample custody form are completed for external courier delivered samples.

The sample management staff examines the shipping containers, their contents, and accompanying customer documentation. Information about the sample identification, the location, date and time of collection, collector's name, preservation type, sample type, presence and condition of custody seals, the state of preservation of the samples and other required information is noted on the SDG form. Any discrepancies in documentation or problems with sample condition such as appropriate sample containers, thermal preservation variation, holding times and adequate sample volumes are noted and brought to the attention of the customer via the nonconformance action form, The login staff or project manager contacts the client via email or or by phone. The Customer Services Manager provides clarification or further instruction to the sample management staff on the processing of the samples that are incomplete or missing required information.

The sample management staff logs the samples in the LIMs and a durable label for each container is printed. The custodian attaches each label to the appropriate sample container. The following information is recorded for tracking internal custody: laboratory sample ID, customer sample ID, sample matrix and storage location. Sample receipt and log-in specifically requires: date and time of laboratory receipt of sample(s); sample collection date; unique laboratory ID code; field ID code supplied by sample submitter; requested analyses; signature or initials of data logger; comments from inspection for sample acceptance or rejection and in some cases, sample bottle codes.

11.5 Storage Conditions

Alpha Analytical stores samples under proper environmental conditions to ensure their integrity and security. Samples are stored at temperatures that meet specifications of the methodology, regulatory agencies and customer directives. Refrigerators are monitored and controlled to be within 4 ± 2°C. Chemical, temperature, holding times and container storage requirements are listed in the LIMS project database.

Customer Quality Assurance Project Plans may list preservation requirements differing from the laboratory. The sample management staff reviews project information for projects specific handling. Addition of chemical preservative to sample containers normally is done in the field at the time of sampling. Chemical preservation and temperature preservation checks at the time of receipt are recorded except for volatile organic compounds, bacteria, sulfite, and dissolved oxygen preservation. Any differences from laboratory or customer specific requirements are recorded on nonconformance action forms and contact made with the customer by the Customer Services Manager or designee.

Sample storage facilities are located within the sample management area, walk-in custody refrigerator or in designated sample storage areas within the analytical departments. Internal chain-of-custody procedures and documentation pertaining to sample possession, removal from storage, and transfer are outlined in the sample custody procedure. Samples are returned to the sample storage area after the sample portion is removed for analysis. Extracts and digestates are tracked and follow the same internal custody operation. Extracts and digestates are removed to the waste disposal area after analysis for proper disposal.

Sample storage precautions are used to ensure that cross contamination does not occur during sample storage. Refrigerator storage blanks are monitored bi-weekly for volatile compounds.. The storage blank information allows the assessment of potential cross contamination in the sample storage refrigerator.

Temperatures of cold storage areas are recorded continuously in the data logger system. Corrective action is done as necessary when temperatures are not within the control criteria. In both the Westboro and Mansfield facilities, Automated Data loggers are linked to thermocouples in custody refrigerators and freezers in the Sample Storage areas as well as department standards/storage refrigerators and freezers. The Data logger is calibrated and certified by an outside vendor annually and on a quarterly basis for DOD standards/storage refrigerators and freezers. If there is a catastrophic failure of custody refrigerators, a record of all samples affected and customers associated with such samples are notified of any samples affected by the failure. Refrigerators and/or freezers not connected to the Data Logger system have temperatures measured with NIST traceable thermometers. Temperature records indicate the thermometer or sensor (Data logger) used for obtaining the measurement.

11.6 Sample Disposal

Samples are held for 21 calendar days after the report is released to the customer. Upon written customer request samples may be held longer in an uncontrolled area. Requests for controlled

sample storage must be arranged at the time of contractual commitment. Air canister samples are held for 3 days after the report is released to the customer.

An authorized waste carrier is contracted to pick up waste as needed and dispose of it, in accordance with all regulatory requirements. Post-analysis disposition of samples is dependent upon project specific requests. Remaining sample material may be returned to the customer, safely discarded, or archived for a specific time prior to disposal. The waste disposal SOP 1797 defines the specific requirements for sample disposal and other waste disposal operations.

The sample management staff are responsible for the archival and disposal of raw samples, extracts and digestates. Raw and prepared samples may not be archived or disposed until all of the designated analyses are complete and resultant analytical data is sent to customers. Samples in storage are retained a minimum of 21 calendar days after reporting the results to the customer. Any samples requiring more than 21 calendar days are archived. Air canister samples requiring storage more than 3 business days require prior approval.

When a customer has requested the return of samples, the sample management staff prepares and ships the samples according to the same custody procedures in which the samples were received and following any customer specified requirements. Protection of the samples during delivery is ensured by the implementation of special packaging procedures. Packages are delivered by a commercial carrier whose procedures for protecting the samples are not within the control of this laboratory. Customers are informed that a commercial carrier will deliver their samples if required**.**

12 Records

Alpha Analytical has a record system that produces accurate records, which document all laboratory activities. The laboratory retains records of all original observations, calculations and derived data, calibration records and a copy of the test for ten years minimum. The system retains records longer than the minimum upon the request of authorized customers, agencies or another regulator. Note: Ohio VAP requires notification before disposal of any VAP records.

12.1 Record Keeping System and Design

The record keeping system allows reconstruction of laboratory processes that produced the analytical data of the sample.

a) The records include the names of personnel involved in sampling, preparation, calibration or testing.

b) Information relating to laboratory facilities equipment, analytical methods, and activities such as sample receipt, preparation, or data verification are documented.

c) The record keeping system provides retrieval of working files and archived records for inspection and verification purposes.

d) Documentation entries are signed or initialed by responsible staff.

e) Generated data requiring operator logging on appropriate logsheets or logbooks are recorded directly and legibly in permanent ink

f) Entries in records are not obliterated by any method. Corrections to errors are made by one line marked through the error. The person making the correction signs and dates the correction.

g) Data entry is minimized by electronic data transfer and ensuring the number of manual data transcriptions is reduced.

12.2 Records Management and Storage

1. Records including calibration and test equipment, certificates and reports are safely stored, held secure and in confidence to the customer.
2. The laboratory maintains hardware and software necessary for reconstruction of data.
3. Records that are stored or generated by computers have hard copy or write-protected backup copies.
4. Alpha Analytical has established a record management system, for control of hard copy laboratory notebooks.
5. Access to archived information is carefully controlled and is limited to authorized personnel. These records are protected against fire, theft, loss, environmental deterioration, vermin, and in the case of electronic records, electronic or magnetic sources.
6. In the event that Alpha Analytical transfers ownership or goes out of business, there is a plan to ensure that the records are maintained or transferred according to the customer's instructions. A plan will be

developed to maintain continuity of our record keeping systems as requested and/or required by both state and federal laws.

Alpha Analytical retains all original hard copy or electronic raw data for calibrations, samples, and quality control measures for ten years, including:

1. Analysts work sheets and data output records,
2. Reference to the specific method,
3. Calculation steps including definition of symbols to reduce observations to a reportable value,
4. Copies of all final reports
5. Archived SOPs,
6. Correspondence relating to laboratory activities for a specific project,
7. All nonconformance action reports, audits and audit responses,
8. Proficiency test results and raw data,
9. Data review and cross checking.

The basic information to tie together analysis and peripherals such as strip charts, printouts, computer files, analytical notebooks and run logs for Alpha Analytical includes:

1. Unique ID code for each Laboratory sample or QC sample;
2. Date of analysis;
3. Instrument identification and operating conditions;
4. SOP reference and version;
5. Calculations;
6. Analyst or operator's initials/signature.

In addition, Alpha Analytical maintains records of:

1. Personnel qualifications, experience and training
2. Initial and continuing demonstration of proficiency for each analyst
3. A log of names, initials and signatures for all individuals who are responsible for signing or initialing any laboratory records. Use of electronic signatures has been approved by regulatory agencies.

12.3 Laboratory Sample Tracking

A record of all procedures to which a sample is subjected while in the possession of the laboratory is maintained. These include but are not limited to records pertaining to:

a) Sample preservation including appropriate sample container and compliance with holding time requirement; If the time of the sample collection is not provided, the laboratory must assume the most conservative time of day (i.e., earliest).

b) Sample identification, receipt, acceptance or rejection and log-in;

c) Sample storage and tracking including shipping receipts, transmittal forms, and internal routing and assignment records; this includes inter-laboratory transfers of samples, extracts and digestates.

d) Sample preparation including cleanup and separation protocols, ID codes, volumes, weights, instrument printouts, meter readings, calculations, reagents;

e) Sample analysis;

f) Standard and reagent origin, receipt, preparation, and use;

g) Equipment receipt, use, specification, operating conditions and preventative maintenance;

h) Calibration criteria, frequency and acceptance criteria;

i) Data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions;

j) Method performance criteria including expected quality control requirements;

k) Quality control protocols and assessment;

l) Electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries;

m) Automated sample handling systems;

n) Records storage and retention; and

o) Disposal of hazardous samples including the date of sample or sub-sample disposal and the name of the responsible person.

p) The COC records account for all time periods associated with the samples.

q) The COC records include signatures of all individuals who had access to individual samples. Signatures (written or electronic) of all personnel who physically handle the samples. Time of day and calendar date of each transfer or handling procedure.

r) Common carrier documents.

13 Laboratory Report Format and Contents

The Process Planning and Control Procedure details the recording and reporting of data as required by the customer and in accordance with relevant environmental regulations.

Customers specify the report delivery and deliverables required for the work submitted. Report delivery includes standard turnaround and rush turnaround. Customers specify the delivery address or multiple addresses and method of delivery such as U.S. Mail, facsimile or electronic at the start of the project. Alpha Analytical provides data deliverables in hardcopy or electronic format. At the start of any project, the electronic deliverable formats required must be received before sample arrival. Affidavits are required with each report or series of reports generated for a particular project for Ohio VAP reports.

Reporting packages are available for routine regulatory reporting requirements. Regulatory reporting packages include only the information requested by the regulatory agency. In addition to regulatory report packages, Alpha Analytical prepares a standard report format. The standard report format includes:

1. Title: “Certification of Analysis”
2. Name and address of the laboratory
3. Laboratory Job Number, page number and total number of pages included in the report.
4. Name and address of the customer
5. Alpha sample number, Customer identification, Sample location
6. Samples identified that do not meet the sample acceptance requirements for project.
7. Date of sample receipt, sample collection, preparation or extraction date and time (if applicable), analysis date and time, report date and analyst
8. Identification of data reported by subcontractors
9. Test name and reference method number
10. Delivery method and sampling procedures when collected by lab personnel
11. Deviations or modifications that affect data quality and/or data integrity. These deviations or modifications are included in narrative statements and/or data merger files.
12. Statement that results relate only to the sample tested
13. Statement that report must be copied in full unless the laboratory provides written permission for partial copies
14. Glossary, References and limits of liability
15. Units of measure and reporting detection limit
16. Quality control data for: % Recovery surrogates, % Recovery of LCS, % RPD of LCSD, Blank analysis, % Recovery Matrix Spike, %RPD of Laboratory Duplicates, as applicable
17. Signature, title and date of report

18. A “Certificate/Approval Program Summary” page is included at the end of the report that identifies analytes for which Alpha Analytical holds certification and for those analytes reported that it does not. This summary also includes the certification numbers for either NELAP certified states, State certifications (e.g. Massachusetts laboratory certification identification number)..
19. Alpha Analytical does not accept samples from private residents for drinking water analysis and therefore maximum contaminant levels are not necessary. If Alpha were to change its policy and report drinking water samples, MCLs would be included with the report.

Results transmitted by facsimile or other electronic means include a statement of confidentiality and return of the materials at the laboratory’s expense.

The laboratory notifies the customer in writing of any circumstance that causes doubt on the validity of the results. The amended or modified report lists the change, reason for the change, affected page numbers, date of the amendment and authorized signature. The customer will be notified prior to changes in LIMs software or hardware configurations that will adversely affect customer electronic data.

13.1 Data Qualifiers

The following data qualifiers are used in conjunction with analytical results depending on the definition, state or regulatory program and report type.

Note: ”J” Estimated value: Upon customer request, the Target analyte concentration can be reported below the quantitation limit (RL), but above the Method Detection Limit (DL) with a “J” qualifier as long as there is a LOD study on file. (See section 5.11)

Data Qualifier	Qualifier Information	Regulatory Requirement
A	Spectra identified as "Aldol Condensation Product".	CT RCP, NC
B	The analyte was detected above the reporting limit in the associated method blank. Flag only applies to associated field samples that have detectable concentrations of the analyte at <5x the concentration found in the blank. For MCP-related projects, flag only applies to associated field samples that have detectable concentrations of the analyte at less than 10x the concentration found in the blank. For NJ-Air-related projects, flag only applies to associated field samples that have detectable concentrations of the analyte above the reporting limit. For NJ-related projects (excluding Air), flag only applies to associated field samples that have detectable concentrations of the analyte, which was detected above the reporting limit in the associated method blank or above five times the reporting limit for common lab contaminants (Phthalates, Acetone, Methylene Chloride, 2-Butanone) For DOD related projects, flag applies to detectable concentration of target analyte in the blank that exceeds ½ the LOG or is greater than 1/10 the concentration in the field sample	EPA Functional Guidelines 'MassDEP MCP, CT RCP, NJ-TO15/LL-TO15; NJ Tech Guidance 2014, DOD QSM 5.1
C	Co-elution: target analyte co-elutes with a known lab standard (i.e. surrogates, internal standards, etc.) for co-extracted analyses.	
D	Concentration of analyte was quantified from diluted analysis. Flag only applies to field samples that have detectable concentrations of the analyte.	NJ-TO15/LL-TO15 - Air only EPA Functional Guidelines; EPA Region 2,5
DL	Same was re-analyzed at a dilution. Qualifier applied to sample number.	

E		Concentration of analyte exceeds the range of the calibration curve and/or linear range of the instrument.	EPA Region 2,5 CT RCP, NJ-TO15/LL-TO15
G		The concentration may be biased high due to matrix interferences (i.e. co-elution) with non-target compound(s). The result should be considered estimated.	In-house/Forensics.
H		The analysis of pH was performed beyond the regulatory-required holding time of 15 minutes from the time of sample collection.	THE NELAC INSTITUTE (TNI) STANDARDS
I		The lower value for the two columns has been reported due to obvious interference.	In-house.
J		Estimated value. This represents an estimated concentration for Tentatively Identified Compounds (TICs).	CT RCP (for TICs),
JN (NJ)		Presumptive evidence of compound. This represents an estimated concentration for Tentatively Identified Compounds (TICs), where the identification is based on a mass spectral library search.	EPA Functional Guidelines 'NJ-TO15-LL
ND	DU-J	Not detected at the method detection limit (MDL) for the sample, or estimated detection limit (EDL) for same-related analysis	In-house
P	All DU	The RPD between the results for the two columns exceeds the method-specified criteria.	MassDEP MCP, CT RCP
Q	All DU	The quality control sample exceeds the associated acceptance criteria. Note: This flag is not applicable for matrix spike recoveries when the sample concentration is greater than 4x the spike added or for batch duplicate RPD when the sample concentrations are less than 5x the RL. (Metals only.)	
R	All DU	Analytical results are from sample re-analysis	Customer-specific

RE	All DU	Analytical results are from sample re-extraction.	Customer-specific
S		Analytical results are from modified screening analysis	

13.2 Compound Summation for Organic Analyses

In order to be compliant with regulations from certain states, Alpha Analytical has created the following Summation Rules to cover reporting “Total Analytes”. The following are an example of several compounds that can be reported as “Totals”:

Volatiles:	
1,3-Dichloropropene, Total	cis + trans isomers
Xylenes, Total	m/p + o isomers
1,2-Dichloroethene, Total	cis + trans isomers
Trihalomethanes, Total	Chloroform + Bromoform +
	Dibromochloromethane +
	Dichlorobromomethane
PCBs:	
PCBs, Total	Sum of reportable Aroclors
	(all Aroclors reported for the project)

The following are the summation rules that the LIMs uses to calculate the Total values:

Summation Rules:		
H + H = H		Key:
H + J = J		H = Hit (above RL)
J + J = J		J = J-flagged value
H + ND = H		ND = U-flagged value
J + ND = J		
ND + ND = ND		

The ND values are considered “0” during the calculations.
The “E” flagged values (over the calibration) are ignored and not utilized during the calculations.
Any “N” flagged values (do not report) are ignored and not utilized during the calculations.
For dual-column analysis, the Total is reported as part of column “A” data, unless all individuals are reported from “B” column.

For analytical group summations, the Total is reported based on the associated "Reporting List". For example, if only 7 Aroclors are requested, then the Total is based on 7 Aroclors, not 9.

The RL and MDL for Totals will always be the lowest of the individual compounds used in the summation.

For each Total summation, two values are calculated: TOTALH (calculated from all associated hits above the R L– used in DU reporting formats) and TOTALJ (calculated from all associated hits and J flagged values – used in DJQL reporting formats). Total concentrations are calculated for all samples and QC samples (however, recoveries are not calculated since they are only calculated for the compounds spiked)

If a Total summation is requested, the individual compounds must also be reported.

14 Outside Support Services and Supplies

When Alpha Analytical purchases outside services and supplies in support of tests, the laboratory uses only those outside services and supplies that are of adequate quality to maintain confidence in the tests. Differences between Request/Tender and Contracts must be resolved before work commences.

The Purchasing SOP/1726 describes approval and monitoring of all suppliers and subcontractors used by the laboratory. Where no independent assurance of the quality of outside support services or supplies is available, the laboratory ensures that purchased equipment, materials, and services comply with specifications by evaluating method performance before routine use.

The laboratory checks shipments upon receipt as complying with purchase specifications. The use of purchased equipment and consumables is only after the evaluation and compliance to the specifications is complete. The Purchasing SOP/1726 describes the details for receipt and inspection of purchased product.

The Purchasing SOP describes the process for raising, review and placement of purchase orders. It is company policy to purchase from third party certified suppliers and subcontractors wherever possible. Purchases must be from suppliers approved by the Laboratory. Laboratory or sampling subcontractors specified by the customer are noted as “Trial” on the purchase order. This identifies the subcontractor as a non-approved subcontractor. All DoD work that is subcontracted must comply with Alpha’s management system and must comply with the QSM standard and is subject to DoD customer approval.

The laboratory maintains list of approved vendors (Form 18302) and subcontractors from whom it obtains support services or supplies required for tests.

14.1 Subcontracting Analytical Samples

Customers are advised, verbally and/or in writing, if any analyses will be subcontracted to another laboratory. Any testing covered under the NELAC Institute (TNI) Standards that requires subcontracting, will be subcontracted to another THE NELAC Institute (TNI) Standard accredited laboratory for the tests to be performed. The laboratory approves testing and sampling subcontractors by review of current state, national or other external parties’ certifications or approvals. This document must indicate current approval for the subcontracted work. Any sample(s) needing special reports (*i.e.*, MCL exceedance) will be identified on the chain of custody when the laboratory subcontracts with another laboratory. Subcontractor Laboratory Certifications are located in Qualtrax under Customer Services folder

The Sample Receipt and Login Procedure describes the process for sample handling when subcontracting samples. The quotation form lists the subcontractor in order to notify the customer of any subcontracted work. Customer notification of subcontracted work is in writing before releasing samples to the subcontractor.

The review of subcontractor documents for completeness and meeting the specifications defined for the project follows the laboratory process for reporting and verification of process data. The person responsible for receiving the order reviews the information supplied by the subcontractor instead of the Department Supervisor.

15 Customer Relations

15.1 Customer Service

The majority of the customer services occur from personnel in the administration, sample receiving and sampling areas. Customer service involves inquiries into services offered, technical consulting, placing orders, and receiving orders, providing updates on the status of orders and completing orders. Personnel interacting with customers must document and review customer specific project requirements. Call Tracker is used to document communications with customers (SOP/1723). Personnel must document customer interactions following the appropriate laboratory procedures. Each person must communicate deviations, modifications and customer requests following the laboratory defined procedures.

15.2 Project Management

During staff meetings the laboratory management reviews requests for new work. The Operations Director and/or Laboratory Technical Manager address all capacity and capability issues. Where conflicts in workload arise, customer notification is immediate. The Project Communication Form (PCF) contains the documentation of all project information. Cooperation between laboratory and customer services staff allows direct communication and scheduling. Management arranges complex scheduling and coordination between departmental areas. Documentation of approval for waivers from the DoD QSM requirements must be documented on a project specific waiver. This documentation needs to be in writing and readily available for review.

15.3 Complaint Processing

The laboratory staff documents all customers or other parties' complaints or concerns regarding the data quality or laboratory operations. The Nonconformance Report records complaints, correcting the concern, and resolving the concern with the customer or other party. The process uses the same form and process as the nonconformance action process. Where repetitive corrective actions indicate a problem, an audit of the area, Customer Inquiry and Complaint SOP/1722 is immediate to ensure the corrective action has effectively solved the concern.

16 Appendix A – Definitions/References

The following definitions are from Section 3.0 of the 2009 TNI Standard. The laboratory adopts these definitions for all work performed in the laboratory.

Acceptance Criteria: specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

Accreditation: the process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. (TNI)

Accuracy: the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (TNI)

Aliquot: A discrete, measured, representative portion of a sample taken for analysis. (EPA QAD glossary)

Analyst: The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality. (TNI)

Analyte: The specific chemicals or components for which a sample is analyzed; it may be a group of chemicals that belong to the same chemical family, and which are analyzed together. (EPA Risk Assessment Guide for Superfund; OSHA Glossary)

Analytical Uncertainty: A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis. (TNI)

Assessment: The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of laboratory accreditation. (TNI)

Assessment (Clarification): The evaluation process used to measure the performance or effectiveness of a system and its elements against specific criteria.

Assessment Criteria: the measures established by The NELAC Institute (TNI) Standards and applied in establishing the extent to which an applicant is in conformance with the NELAC Institute (TNI) Standards requirements.

Audit: A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives. (TNI).

Batch: Environmental samples, which are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A

preparation batch is composed of one (1) to twenty (20) environmental samples of the same quality systems matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates), which are analyzed together as a group. An analytical batch can include prepared samples originating from various quality system matrices and can exceed 20 samples. (TNI)

Bias: The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value). (TNI)

Blank: a sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. (TNI)

Blanks include:

Equipment Blank: a sample of analyte-free media, which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures.

Field Blank: blank prepared in the field by filling a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA OSWER)

Instrument Blank: a clean sample (e.g. distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

Method Blank: A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses, (TNI)

Reagent Blank: (method reagent blank): a sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps. (QAMS)

Blind Sample: a sub-sample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst or laboratory's proficiency in the execution of the measurement process.

Calibration: set of operations which establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or

measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards. (TNI)

1) In calibration of support equipment the values realized by standards are established through the use of Reference Standards that are traceable to the International System of Units (SI).

2) In calibration according to test methods, the values realized by standards are typically established through the use of Reference Materials that are either purchased by the Laboratory with a certificate of analysis or purity, or prepared by the Laboratory using support equipment that has been calibrated verified to meet specifications.

Calibration Range: The range of values (concentrations) between the lowest and highest calibration standards of a multi-level calibration curve. For metals analysis with a single-point calibration, the low-level calibration check standard and the high standard establish the linear calibration range, which lies within the linear dynamic range.

Calibration Curve: the graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (TNI)

Calibration Method: A defined technical procedure for performing a calibration.

Calibration Standard: A substance or reference material used to calibrate an instrument. (TNI)

Certified Reference Material (CRM): Reference material, accompanied by a certificate, having a value, measurement uncertainty, and stated metrological traceability chain to a national metrology institute. (TNI)

Chain of Custody Form: Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and types of containers; the mode of collection; collector; time of collection; preservation; and requested analyses. See also Legal Chain of Custody Protocols (TNI)

Clean Air Act: the enabling legislation in 42 U.S.C. 7401 *et seq*., Public Law 91-604, 84 Stat. 1676 Pub.L. 95-95, 91 Stat., 685 and Pub. L. 95-190, 91 Stat., 1399, as amended, empowering EPA to promulgate air quality standards, monitor and to enforce them.

Confirmation: Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to: Second column confirmation, Alternate wavelength, Derivatization, Mass spectral interpretation, Alternative detectors, or Additional cleanup procedures (TNI)

Customer: Any individual or organization for which items or services are furnished or work performed in response to defined requirements and expectations. (ANSI/ASQ E4-2004)

Congener: A member of a class of related chemical compounds (e.g., PCBs, PCDDs)

Comprehensive Environmental Response, Compensation and Liability Act (CERCLA/Superfund): the enabling legislation in 42 U.S.C. 9601-9675 et seq., as amended by the Superfund Amendments and Reauthorization Act of 1986 (SARA), 42 U.S.C. 9601 et seq., to eliminate the health and environmental threats posed by hazardous waste sites.

Conformance: an affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ASQC E4-1994)

Consensus Standard: A standard established by a group representing a cross-section of a particular industry or trade, or a part thereof. (ANSI/ASQ ANSI/ASQ E4-2004)

Continuing calibration verification: The verification of the initial calibration that is required during the course of analysis at periodic intervals. Continuing calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models. (IDQTF)

Corrective Action: the action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

Completeness: the percentage of measurements judged to be valid compared to the total number of measurements made for a specific sample matrix and analysis.

Data Quality Objectives (DQO):

Data Reduction: the process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form. (TNI)

Definitive Data: Analytical data of known quality, concentration, and level of uncertainty. The levels of quality and uncertainty of the analytical data are consistent with the requirements for the decision to be made. Suitable for final decision-making. (UFP-QAPP)

Demonstration of Capability: a procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision. (TNI)

Detection Limit: (previously referred to as Method Detection Limit –MDL) the lowest concentration or amount of the target analyte that can be identified, measured, and reported with confidence that the analyte concentration is not a false positive value. See Method Detection Limit.

Detection Limit (DL) (Clarification): The smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration at the 99% level of confidence. At the DL, the false positive rate (Type I error) is 1%.

Document Control: the act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed. (ASQC)

Environmental Data: Any measurements or information that describe environmental processes, locations, or conditions; ecological or health effects and consequences; or the performance of environmental technology. (ANSI/ASQ E4-2004)

False Negative: An analyte incorrectly reported as absent from the sample, resulting in potential risks from their presence.

False Positive: An item incorrectly identified as present in the sample, resulting in a high reporting value for the analyte of concern.

Federal Insecticide, Fungicide and Rodenticide Act (FIFRA): the enabling legislation under 7 U.S.C. 135 *et seq*., as amended, that empowers the EPA to register insecticides, fungicides, and rodenticides.

Federal Water Pollution Control Act (Clean Water Act, CWA): the enabling legislation under 33 U.S.C 1251 et seq., Public Law 92-50086 Stat. 8.16, that empowers EPA to set discharge limitations, write discharge permits, monitor, and bring enforcement action for non-compliance.

Field Measurement: The determination of physical, biological, or radiological properties, or chemical constituents; that are measured on-site, close in time and space to the matrices being sampled/measured, following accepted test methods. This testing is performed in the field outside of a fixed-laboratory or outside of an enclosed structure that meets the requirements of a mobile laboratory.

Field of Accreditation: Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation. (TNI)

Finding: an assessment conclusion, referenced to a laboratory accreditation standard and supported by objective evidence that identifies a deviation from a laboratory accreditation standard requirement. (TNI)

Finding (Clarification): An assessment conclusion that identifies a condition having a significant effect on an item or activity. An assessment finding may be positive or negative and is normally accompanied by specific examples of the observed condition (ANSI/ASQ E4-2004).

Holding Times: The maximum time that can elapse between two (2) specified activities. (TNI)

The maximum times that samples may be held prior to analysis and still be considered valid or not compromised. (40 CFR part 136)

Inspection: An activity such as measuring, examining, testing, or gauging one or more characteristics of an entity and comparing the results with specified

requirements in order to establish whether conformance is achieved for each characteristic. (ANSI/ASQC E4-1994)

Internal Standard: A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical method. (TNI)

Isomer: One of two or more compounds, radicals, or ions that contain the same number of atoms of the same elements but differ in structural arrangement and properties. For example, hexane (C6H14) could be n-hexane, 2-methylpentane, 3-methylpentane, 2,3-dimethylbutane, 2,2-dimethylbutane.

Laboratory: Body that calibrates and/or tests. (ISO 25)

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank or QC check sample): a sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system. (TNI).

Laboratory Duplicate: aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently.

Legal Chain of Custody Protocols: procedures employed to record the possession of samples from the time of sampling until analysis and are performed at the special request of the customer. These protocols include the use of a Chain of Custody Form that documents the collection, transport, and receipt of compliance samples by the laboratory. In addition, these protocols document all handling of the samples within the laboratory. (TNI)

Limit of Detection (LOD): A laboratory's estimate of the minimum amount of an analyte in a given matrix that an analytical process can reliably detect in their facility. (TNI)

Limit of Detection (Clarification): The smallest amount or concentration of a substance that must be present in a sample in order to be detected at a high level of confidence (99%). At the LOD, the false negative rate (Type II error) is 1%.

Limits of Quantitation (LOQ): The minimum levels, concentrations, or quantities of a target variable (e.g. target analyte) that can be reported with a specified degree of confidence. (TNI) For DOD projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard and within the calibration range.

Limit of Quantitation (Clarification): The lowest concentration that produces a quantitative result within specified limits of precision and bias.

Management: Those individuals directly responsible and accountable for planning, implementing, and assessing work. (ANSI/ASQ E4-2004)

Management System: System to establish policy and objectives and to achieve those objectives (ISO 9000).

Matrix: The substrate of a test sample. (TNI)

Matrix Spike (spiked sample, fortified sample): A sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of Target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency. (TNI).

Matrix Spike Duplicate (spiked sample or fortified sample duplicate): a second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte. (TNI).

Measurement System: A test method, as implemented at a particular laboratory, and which includes the equipment used to perform the test and the operator(s). (TNI)

Method: A body of procedures and techniques for performing an activity (e.g., sampling, chemical analysis, quantification), systematically presented in the order in which they are to be executed. (TNI)

Method Detection Limit: (now referred to as Detection Limit) one way to establish a Detection Limit, defined as the minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.

Method Detection Limit (MDL) (Clarification): The MDL is one way to establish a Detection Limit, not a Limit of Detection.

Method of Standard Additions: A set of procedures adding one or more increments of a standard solution to sample aliquots of the same size in order to overcome inherent matrix effects. The procedures encompass the extrapolation back to obtain the sample concentration. (This process is often called spiking the sample.) (Modified Skoog, Holler, and Nieman. Principles of Instrumental Analysis. 1998)

Mobile Laboratory: A portable enclosed structure with necessary and appropriate accommodation and environmental conditions for a laboratory, within which testing is performed by analysts. Examples include but are not limited to trailers, vans and skid-mounted structures configured to house testing equipment and personnel. (TNI)

National Institute of Standards and Technology (NIST): A federal agency of the US Department of Commerce's Technology Administration that is designed as the United States national metrology institute. (NMI). (TNI)

National Environmental Laboratory Accreditation Program (NELAP): The overall National Environmental Laboratory Accreditation Program of which TNI is a part.

Negative Control: Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.

Positive Control: Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects.

Precision: The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (TNI).

Preservation: Any conditions under which a sample must be kept in order to maintain chemical and/or biological integrity prior to analysis. (TNI)

Procedure: A specified way to carry out an activity or a process. Procedures can be documented or not. (TNI)

Proficiency Testing: A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source. (TNI)

Proficiency Testing Program: The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories. (TNI)

Proficiency Test Sample (PT): A sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria. (TNI).

Protocol: A detailed written procedure for field and/or laboratory operation (e.g., sampling, analysis) which must be strictly followed. (TNI)

Quality Assurance: An integrated system of management activities involving planning, implementation, assessment, reporting and quality improvement to ensure that a process, item, or service is the type and quality needed and expected by the customer. (TNI)

Quality Assurance [Project] Plan (QAPP): A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EPA-QAD)

Quality Control: The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements or quality; also the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against "out of control" conditions and ensuring that the results are of acceptable quality. (TNI)

Quality Control Sample: A sample used to assess the performance of all or a portion of the measurement system. One of any number of samples, such as Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking intended to demonstrate that a measurement system or activity is in control. (TNI)

Quality Manual: A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, the ensure the quality of its product and the utility of its product to the users. (TNI)

Quality Manual Clarification: Alpha Analytical refers to Quality Manual as Corporate Quality Systems Manual (CQSM). (Alpha)

Quality System: A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required quality assurance (QA) and quality control (QC) activities. (TNI)

Quality System Matrix: These matrix definitions are to be used for purposes of batch and quality control requirements: (TNI)

Air and Emissions: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbent tube, impinger solution, filter, or other device.

Aqueous: Any aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine. Includes surface water, ground water effluents, and TCLP or other extracts.

Biological Tissue: Any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Chemical Waste: A product or by-product of an industrial process that results in a matrix not previously defined.

Drinking Water: Any aqueous sample that has been designated a potable or potential potable water source.

Non-Aqueous Liquid: Any organic liquid with <15% settleable solids.

Saline/Estuarine: Any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.

Solids: Includes soils, sediments, sludges and other matrices with >15% settleable solids.

Raw Data: The documentation generated during sampling and analysis. This documentation includes, but is not limited to, field notes, electronic data, magnetic tapes, untabulated sample results, QC sample results, print outs of chromatograms, instrument outputs, and handwritten records. (TNI)

Reference Material: Material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (TNI)

Reference Standard: Standard used for the calibration of working measurement standards in a given organization or at a given location. (TNI)

Representativeness: the degree to which the sample represents the properties of the particular sample being analyzed.

Resource Conservation and Recovery Act (RCRA): the enabling legislation under 42 USC 321 *et seq.* (1976), that gives EPA the authority to control hazardous waste from the "cradle-to-grave", including its generation, transportation, treatment, storage and disposal.

Safe Drinking Water Act (SDWA): the enabling legislation, 42 USC 300f *et seq.* (1974), (Public Law 93-523), that requires the EPA to protect the quality of drinking water in the U.S. by setting maximum allowable contaminant levels, monitoring, and enforcing violations.

Sample Tracking: procedures employed to record the possession of the samples from the time of sampling until analysis, reporting and archiving. These procedures include the use of a Chain of Custody Form that documents the collection, transport, and receipt of compliance samples to the laboratory. In addition, access to the laboratory is limited and controlled to protect the integrity of the samples.

Sampling: Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure. (TNI)**Second source calibration verification (ICV):** A standard obtained or prepared from a source independent of the source of standards for the initial calibration. Its concentration should be at or near the middle of the calibration range. It is done after the initial calibration.

Selectivity: The ability to analyze, distinguish, and determine a specific analyte or parameter from another component that may be a potential interferent. (TNI)

Sensitivity: The capability of a test method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (TNI)

Signal to Noise Ratio: The signal carries information about the analyte, while noise is made up of extraneous information that is unwanted because it degrades the accuracy and precision of an analysis and also places a lower limit on the amount of analyte that can be detected. In most measurements, the average strength of the noise is constant and independent of the magnitude of the signal. Thus, the effect of noise on the relative error of a measurement becomes greater and greater as the quantity being measured (producing the signal) decreases in magnitude. (Skoog, Holler, and Nieman. Principles of Instrumental Analysis. 1998)

Signatures, Electronic: A technology that allows a person to electronically affix a signature or its equivalent to an electronic document. The electronic signature links the signature to the signer's identity and to the time the document was signed. Alpha approves the use of electronic signatures for signing and initializing any laboratory record including, by not limited to: analytical reports, controlled documents, workflows and purchasing requests.

Standard: The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of standard setting and meets the approval requirements of standard adoption organizations procedures and policies. (TNI)

Standard Operating Procedures (SOPs): A written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks. (TNI)

Standard Method: a test method issued by an organization generally recognized as competent to do so.

Standardized Reference Material (SRM): a certified reference material produced by the U.S. National Institute of Standards and Technology or other equivalent organization and characterized for absolute content, independent of analytical method.

Surrogate: a substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.

Technology: a specific arrangement of analytical instruments, detection systems, and/or preparation techniques. (TNI)

Test: A technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate. (ISO/IEC Guide 2 - 12.1, amended)

Tentatively Identified Compound (TIC): A compound that has been identified to be present and is not part of the target compound list (TCL) for the method and/or program. All TICs are qualitatively identified and reported as estimated concentrations. Tentatively Identified Compounds, if requested, are reported for compounds identified to be present and are not part of the method/program Target Compound List, even if only a subset of the TCL are being reported.

Test Method: An adoption of a scientific technique for performing a specific measurement, as documented in a laboratory SOP or as published by a recognized authority.

Toxic Substances Control Act (TSCA): the enabling legislation in 15 USC 2601 et seq. (1976), the provides for testing, regulating, and screening all chemicals

produced or imported into the United States for possible toxic effects prior to commercial manufacture.

Traceability: The ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical constants or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project. (TNI)

Tuning: A check and/or adjustment of instrument performance for mass spectrometry as required by the method.

United States Environmental Protection Agency (EPA): the federal governmental agency with responsibility for protecting public health and safeguarding and improving the natural environment (i.e. the air, water and land) upon which human life depends. (US-EPA)

Validation: the confirmation by examination and provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

Verification: confirmation by examination and provision of evidence that specified requirements have been met. (TNI)

NOTE - In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment.

The result of verification leads to a decision either to restore in service, to perform adjustments, or to repair, or to downgrade, or to declare obsolete. In all cases, it is required that a written trace of the verification performed shall be kept on the measuring

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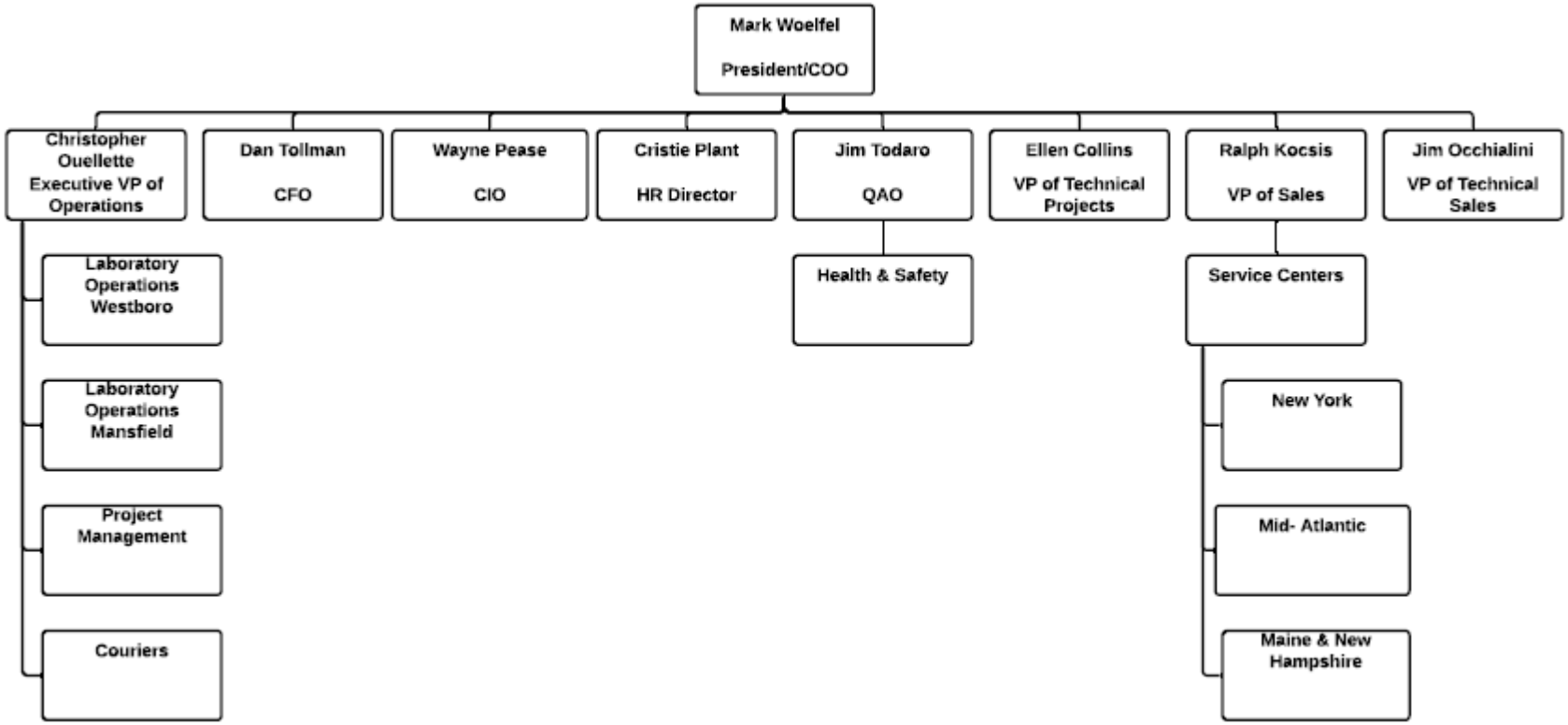
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17 Appendix B – Organization Charts

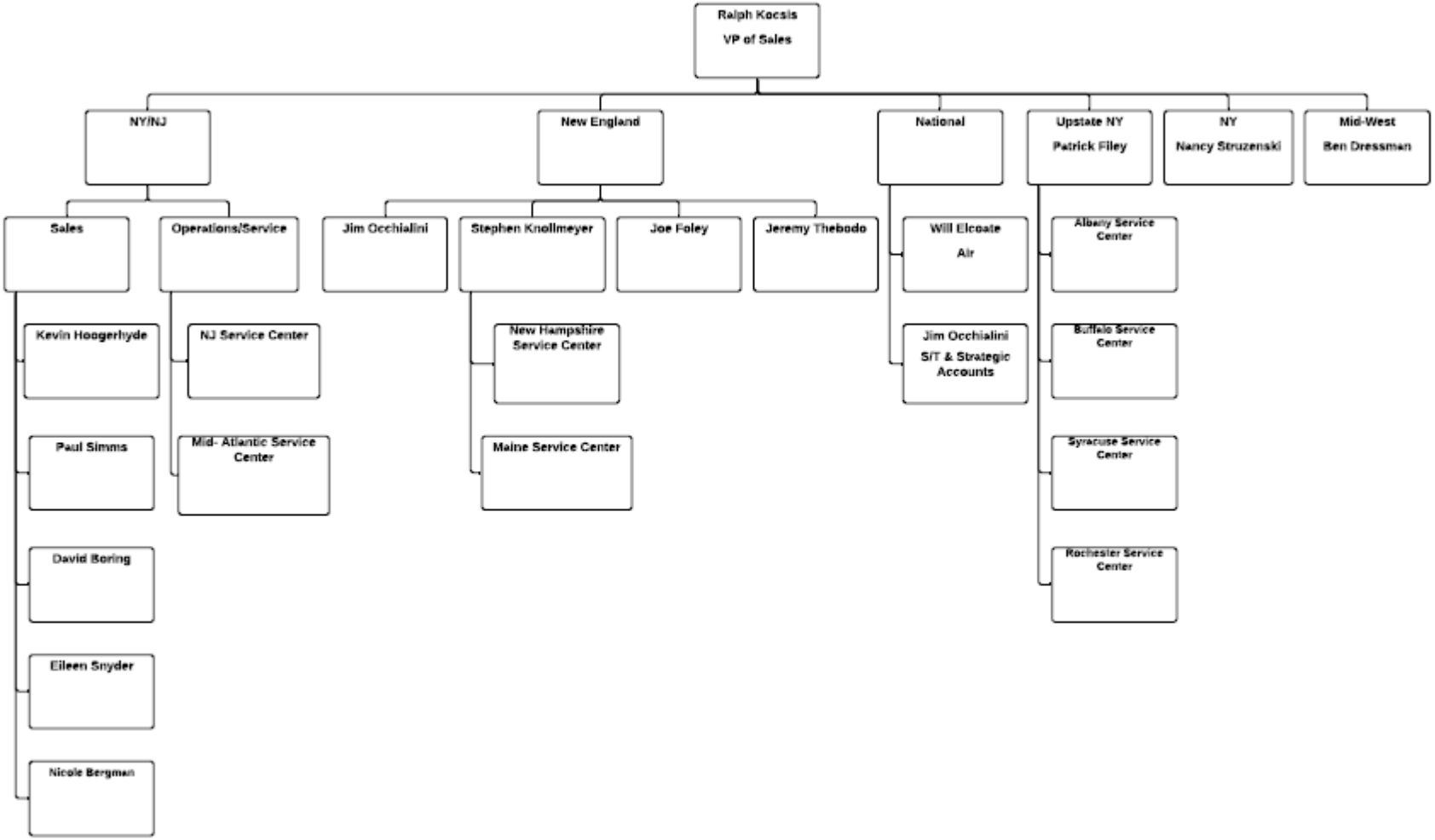
The following charts provide an overview of the organizational structure of Alpha Analytical. The chart also identifies the key personnel responsible for the listed positions. For the various laboratory areas, the individual departmental supervisors are noted. For a listing of all current key personnel, please refer to Section 18, Appendix C.

Updated 01/08/2020

Alpha Analytical
Company Organizational Chart

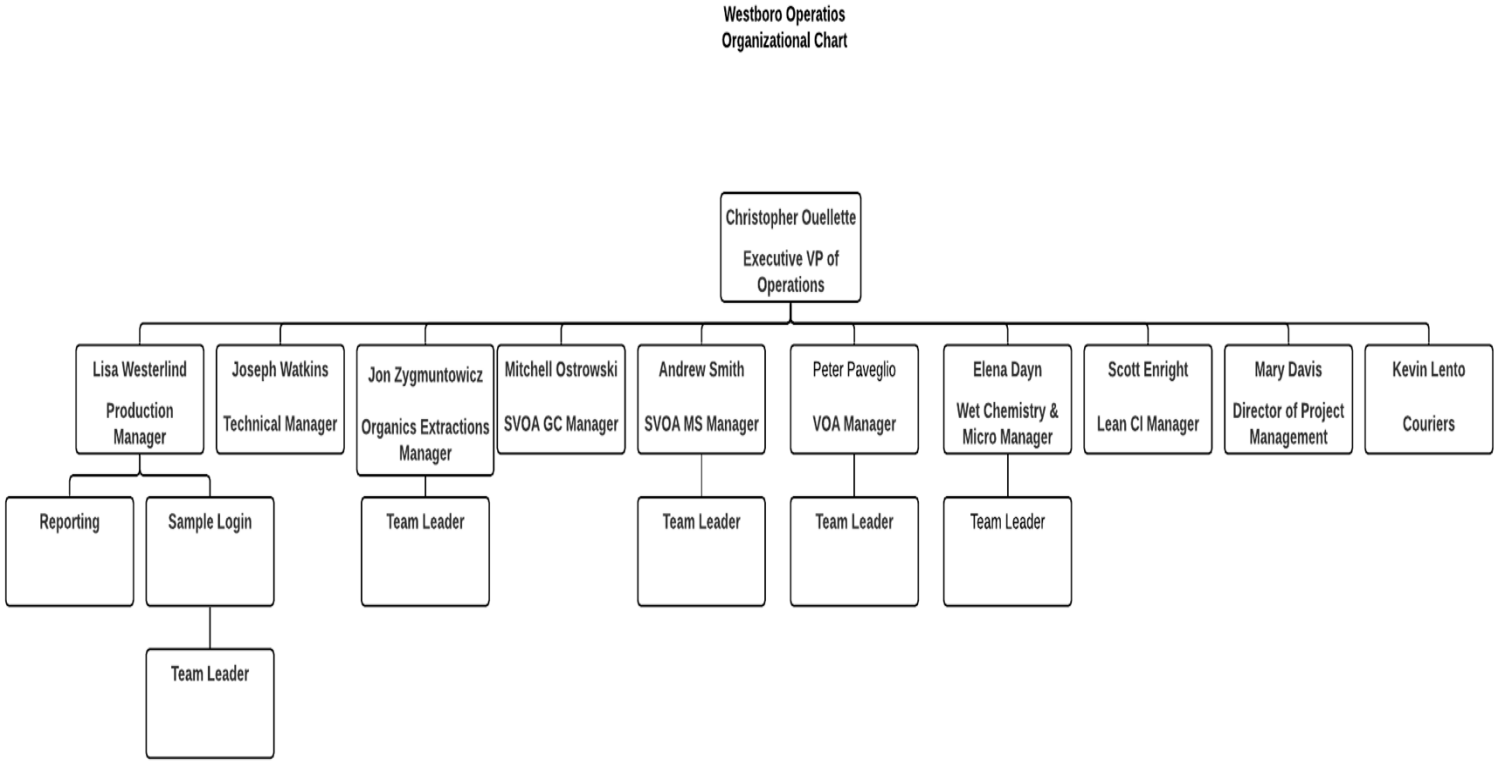


Updated 01/08/2020
Alpha Analytical
Sales Organizational Chart



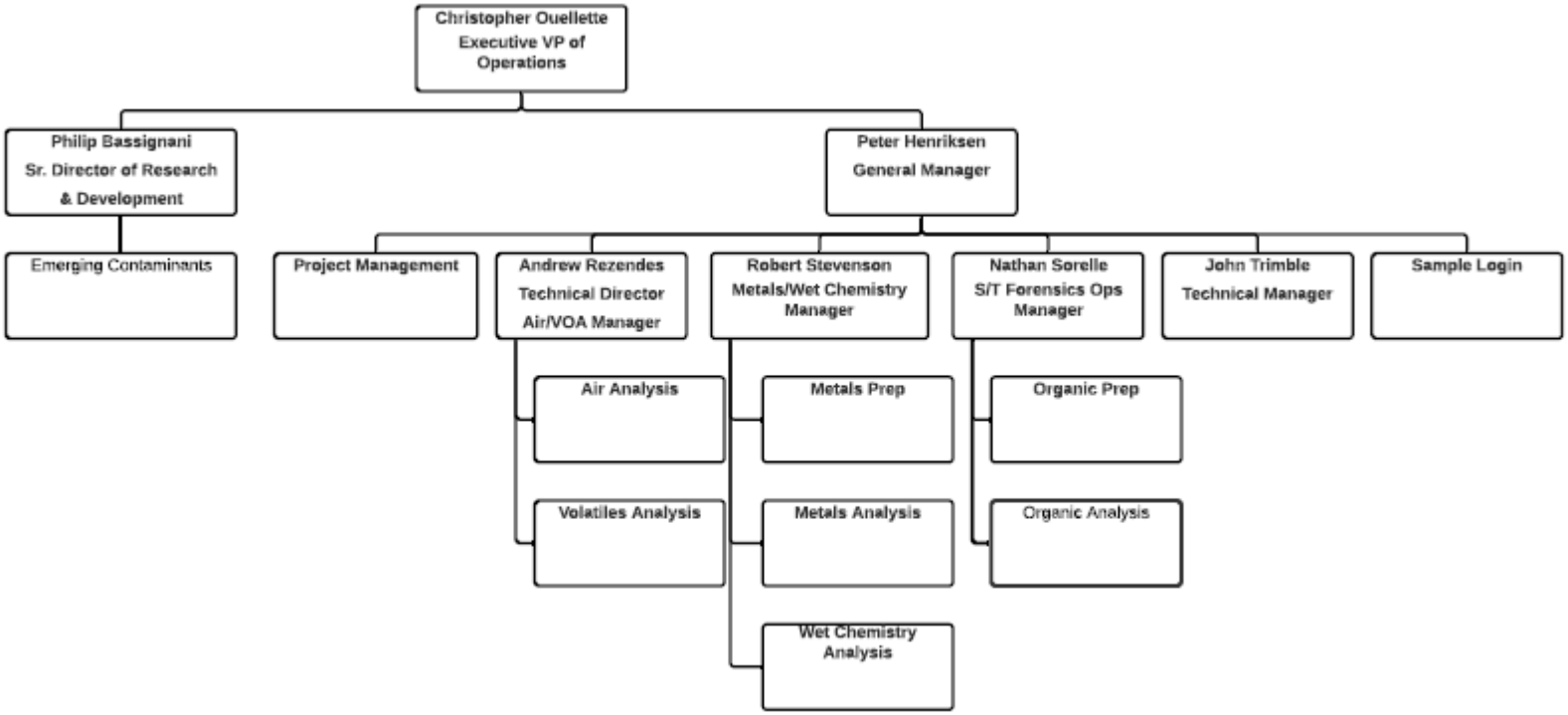
Updated 06/13/2019

Alpha Analytical
Westboro Facility Organizational Chart



Updated 01/09/2020

Alpha Analytical
Mansfield Facility Organizational Chart



18 Appendix C – List of Key Personnel

The following is a listing of all current key personnel. If role is specific to a facility it is denoted by either Westboro or Mansfield following the position title. **Updated 01/2019.**

President / COO: Mark Woelfel

Executive VP of Operations: Christopher Ouellette

CFO: Dan Tollman

CIO: Wayne Pease

Laboratory Technical Manager - Westboro: Joseph Watkins

Laboratory Technical Manager - Mansfield: John Trimble

Laboratory Technical Manager- Air, Volatiles Manager - Mansfield: Andy Rezendes

Quality Assurance Officer/Health & Safety Manager: James C. Todaro

Senior Director of Research & Development: Philip Bassignani

VP, Technical Projects: Ellen Collins

VP of Sales: Ralph Kocsis

VP, Technical Sales: James Occhialini, Pat Filey, Kevin Hoogerhyde, Steven Knollmeyer, Nancy Struzenski

Technical Sales Reps: Paul Simms, David Boring, Joe Foley, Jeremy Thebodo, Ben Dressman

General Manager, Mansfield: Peter Henriksen

Director of Project Management: Mary Davis

National Air Account Manager: Will Elcoate

Information Technology Manager: Glenn Fitzgibbons

Human Resources Director: Cristie Plant

Health & Safety Officer: Melissa White

Forensic & S/T Operations Manager, Mansfield: Nathan Sorelle

SVOA GC Manager, Westboro: Mitchell Ostrowski

SVOA GC/MS Manager, Westboro: Andrew Smith

Extractions Manager, Westboro: John Zygmuntowicz

VOA Department Manager, Westboro: Peter Paveglio

Wet Chemistry Department Manager, Westboro: Elena Dayn

Metals Department Manager, Mansfield: Robert Stevenson

Login Manager/ Reporting Manager, Westboro Lisa Westerlind

Quality Systems Specialists: Amy Rice, Rene Bennett, Jason Hebert, Michael Selling, Kirols Andrawis

Purchasing: David Peak

Logistics Manager: Kevin Lento

Equipment Technical Specialists: Patrick Sullivan, Szymon Sus, Kimberly Rivera

19 Appendix D – Preventive Maintenance Procedures

Optimized Service-Calibration Intervals		
Equipment	**Frequency**	**Type of Calibration or Maintenance**
Balances	semiannually daily	cleaning & operations check by service technician (external) calibration verification using Class S-1 certified weights
COD Reactor	annually	reaction temperature verification
Conductivity Bridge	annually annually each use	verification of cell constant complete operations check by service technician (external) calibration verification
DI Water System	as needed monthly annually daily	complete operations check by service technician (external) Residual Chlorine check Biosuitability testing (external) pH and Conductivity check
DO Meter	annually each use	complete operations check by service technician (external) calibration against air as specified by manufacturer
Emergency/Safety Equipment	annually monthly	fire extinguishers and emergency exit lighting check eye washes, showers, fire blanket and first aid kits checked
Freezers	daily	temperature verification
Gas Chromatographs	as needed as needed beginning and end of batch and 10 to 20 samples as per method	injection port preparation; cleaning of detectors initial multi-point calibration continuing calibration verification (CCV) against initial calibration
ICP	Every other day Daily Annually Annually As needed	Change pump tubing Calibration, profile Complete operations check by service technician (external), Linear Dynamic Range determination Clean torch, clean nebulizer, clean spray chamber
Lachat analyzer	Daily As needed	Calibration, clean lines Change tubing, change O-rings
Mass Spectrometers (GC & ICP)	bi-annually as needed 12 hour or daily	change of mechanical pump oil by service technician (external) cleaning of source BFB, DFTPP or ICP-MS tune analysis followed by ICAL or CCV
Mercury Analyzer	monthly each use	clean cell and change pump windings calibration using multi-point curve
Auto-pipettes	Quarterly Daily	verification of accuracy and precision verification of precision for DOD method auto-pipettes
Microwave	Quarterly Annually	power and temperature verification RPM verification
Ovens	Annually	temperature verification
pH Meters	Annually each use	complete operations check by service technician (external) calibration using certified buffers
Refrigerators (General Use)	daily	temperature verification
Refrigerators (Sample Management)	daily	temperature verification
Spectrophotometer	Semi-annually Semi-annually daily	cleaning & operations check by service technician (external) wavelength verification (external) continuing calibration verification (CCV) against initial calibration
TCLP/ZHE Rotator	Quarterly	RPM verification
Thermometers (Mercury/Alcohol) Thermometers (Bimetal/mechanical)	Annually Quarterly	calibration against NIST traceable thermometer (internal) calibration against NIST traceable thermometer (internal)
Thermometers (digital/IR)	Quarterly	calibration against NIST traceable thermometer (external)
Thermometer (NIST Traceable)	Annually	calibration and certification of conformance (external)
Turbidity meter	Annually each use	cleaning & operations check by service technician (external) calibration using formazin
Weights (Class S-1)	Annually Triennially	Working weights verified against reference weights Reference weights calibrated for conformance (external)

20 Appendix E – Alpha Code of Ethics Agreement

Alpha Analytical, Inc.
Ethical Conduct and Data Integrity Agreement

A. ***Personal Pledge:*** I understand that I am charged with meeting the highest degree of ethical standards in performing all of my duties and responsibilities and pledge to only report data, test results and conclusions that are accurate, precise and of the highest quality.

B. ***Protocol Pledges:*** I agree to adhere to the following protocols and principles of ethical conduct in fulfilling my work assignments at Alpha:

1. All work assigned to me will be performed using Standard Operating Procedures (SOPs) that are based on EPA approved methods or Alpha methods.

2. I will only report results or data that match the actual results observed or measured.

3. I will not intentionally nor improperly manipulate or falsify data in any manner, including both sample and QC data. Furthermore, I will not modify data values unless the modification can be technically justified through a measurable analytical process or method acceptable to Alpha. All such modifications will be clearly and thoroughly documented in the appropriate laboratory notebooks and raw data and include my initials or signature and date.

4. I will not intentionally report dates and times of analyses that are not the actual dates and times the analyses were conducted.

5. I will not intentionally represent another individual's work as my own or represent my work as someone else's.

6. I will not make false statements to, or seek to otherwise deceive Alpha staff, leaders or customers. I will not, through acts of commission, omission, erasure or destruction, improperly report measurements, standards results, data, test results or conclusions.

C. ***Guardian Pledge:***

1. I will not condone any accidental or intentional reporting of unauthentic data by other Alpha staff and will immediately report such occurrences to my supervisor, the QA Officer, the Laboratory Technical Manager or corporate leadership. I understand that failure to report such occurrences may subject me to immediate discipline, including termination.

2. If a supervisor or other member of the Alpha leadership group requests me to engage in, or perform an activity that I feel is compromising data validity or quality, I have the right to not comply with the request and appeal this action through Alpha's QA Officer, senior leadership or corporate officers, including the President of the company.

3. I understand that, if my job includes supervisory responsibilities, then I will not instruct, request or direct any subordinate to perform any laboratory practice that is unethical or

improper. Also, I will not discourage, intimidate or inhibit a staff member who may choose to appropriately appeal my supervisory instruction, request or directive that may be perceived to be improper, nor retaliate against those who do so.

D. ***Agreement Signature:*** I have read and fully understand all provisions of the *Alpha Analytical Ethical Conduct and Data Integrity Agreement*. I further realize and acknowledge my responsibility as an Alpha staff member to follow these standards. I clearly understand that adherence to these standards is a requirement of continued employment at Alpha.

_____
Employee Signature

_____
Printed Name

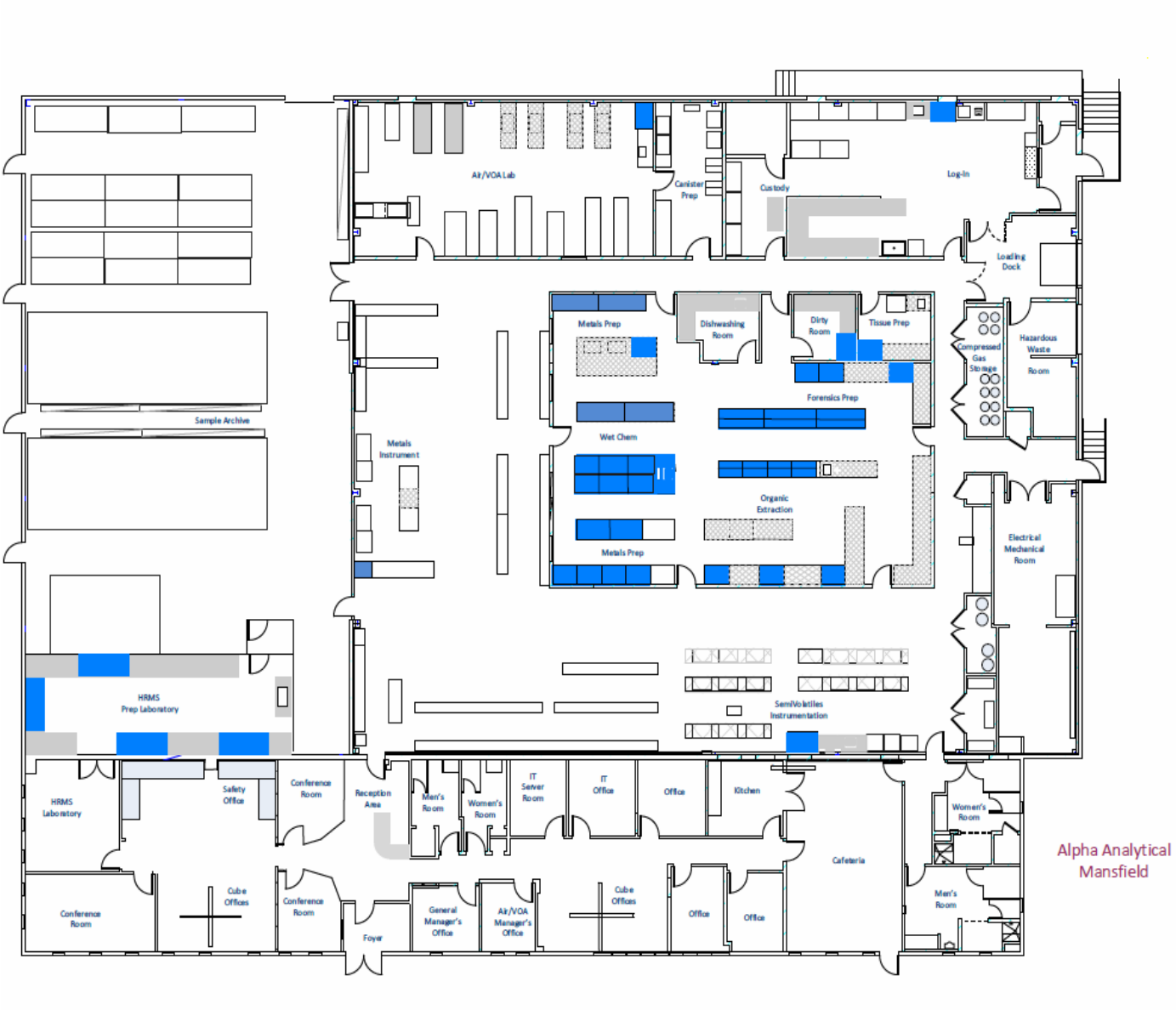
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Date

Review Requirements
The *Ethical Conduct and Data Integrity Agreement* must be signed at the time of hire (or within 2 weeks of a staff member's receipt of this policy). Such signature is a condition of continued employment at Alpha. Failure to comply with these requirements will result in immediate discharge from Alpha employment. This agreement is not an employment contract and does not modify in any manner the company's *Employment-at-Will* Agreement.

21 Appendix F– Floor Plan Westboro Facility



22 Appendix G– Floor Plan Mansfield Facility



23 Appendix H – Job Titles and Requirements

TITLE*	REQUIRED EDUCATION**	MINIMUM REQUIRED ENVIRONMENTAL LAB EXPERIENCE	MINIMUM REQUIRED SKILLS***
Technical Manager (Director) Organic Laboratory	BS or BA in Chemical, Environmental, or Biological Science; including minimum 24 credit hours in Chemistry. Masters or Doctoral degree in one of above disciplines may be substituted for 1 year of experience.	Two (2) years with the analysis of organic analytes in an environmental laboratory	1. Advanced technical knowledge of all analytical methods performed by the lab 2. Advanced technical instrumentation/lab systems knowledge 3. Knowledge of safe laboratory practices, OSHA regs and emergency protocols 4. Experience with and understanding of LIMS 5. Experience with method development and implementation 6. Experience monitoring standards of performance in Quality Control and Quality Assurance
Technical Manager (Director) Inorganic Laboratory	BS or BA in Chemical, Environmental, or Biological Science; including minimum 16 credit hours in Chemistry. Masters or Doctoral degree in one of above disciplines may be substituted for 1 year of experience.	Two (2) years with the analysis of inorganic analytes in an environmental laboratory	1. Advanced technical knowledge of all analytical methods performed by the lab 2. Advanced technical instrumentation/lab systems knowledge 3. Knowledge of safe laboratory practices, OSHA regs and emergency protocols 4. Experience with and understanding of LIMS 5. Experience with method development and implementation 6. Experience monitoring standards of performance in Quality Control and Quality Assurance
Technical Manager (Director) Microbiology Laboratory	BS or BA in Chemical, Environmental, or Biological Science; including minimum 16 credit hours in the Biological Sciences, including at least one course having microbiology as a major component. Masters or Doctoral degree in one of above disciplines may be substituted for 1 year of experience.	Two (2) years with the analysis of microbiological analytes in an environmental laboratory	1. Advanced technical knowledge of all analytical methods performed by the lab 2. Advanced technical instrumentation/lab systems knowledge 3. Knowledge of safe laboratory practices, OSHA regs and emergency protocols 4. Experience with and understanding of LIMS 5. Experience with method development and implementation 6. Experience monitoring standards of performance in Quality Control and Quality Assurance
Quality Assurance Officer	BS/BA in Chemistry, Biology, Environmental or related Science	Two (2) years Environmental Laboratory Experience	1. Advanced technical knowledge of all analytical methods performed by the lab 2. Knowledgeable in Federal, State Programs (THE NELAC INSTITUTE (TNI) STANDARDS, etc.) 3. Able to develop QA/QC policies and certification requirements 4. Able to develop training programs for quality procedures 5. Documented training and/or experience in QA and QA procedures 6. Knowledge of safe laboratory practices and emergency protocols

TITLE*	REQUIRED EDUCATION**	MINIMUM REQUIRED ENVIRONMENTAL LAB EXPERIENCE	MINIMUM REQUIRED SKILLS***
Laboratory Coordinator	High School Diploma; Associates or BS/BA in Chemistry, Biology or Environmental or related Science preferred	1 year +	1. Knowledge of safe laboratory practices and emergency protocols 2. Proficient in all methods and SOP's within their department 3. Experience with and understanding of LIMS 4. Proven ability to meet TAT (turnaround times)
Quality Systems Specialist	BS/BA Chemistry, Biology, Environmental or related Science	2 years +	1. General knowledge of laboratory methods 2. Experience with and understanding of LIMS 3. Strong attention to detail 4. Strong oral/written communication and organizational skills 5. Knowledge of QA/QC policies and certification requirements
EH&S Coordinator	High School or Equivalent	2 years +	1. General knowledge of lab operations 2. Detailed knowledge of safe lab practices and emergency protocols 3. Hazardous Waste Management and RCRA Regulation Training 4. DOT Hazardous Materials Regulations Training 5. OSHA Compliance Training 6. Able to develop and deliver new hire and ongoing safety training programs
Lab Technician I	HS or Equivalent	0-1 years. 1+ years preferred.	1. Knowledge of safe laboratory practices 2. Able to follow direction and Standard Operating Procedures (SOP's) 3. Familiarity with standard and reagent preparation 4. Knowledgeable in using volumetric pipettes and glassware 5. Strong oral/written communication and organizational skills
Lab Technician II	HS or Equivalent	2-4 years	1. All skills of Lab Technician I 2. Trained in majority of technician skills relative to department
Lab Technician III	HS or Equivalent	5 years +	1. All skills of Lab Technician II 2. Experienced in training staff
Lab Technician/Chemist I	BS/BA in Chemistry, Biology, Environmental or related Science	0-1 years	1. Knowledge of safe laboratory practices 2. Able to follow direction and Standard Operating Procedures (SOP's) 3. Familiarity with standard and reagent preparation 4. Knowledgeable in using volumetric pipettes and glassware 5. Strong oral/written communication and organizational skills
Lab Technician/Chemist II	BS/BA in Chemistry, Biology, Environmental or related Science	2-4 years	1. All skills of Chemist I 2. Trained in majority of department methods

TITLE*	REQUIRED EDUCATION**	MINIMUM REQUIRED ENVIRONMENTAL LAB EXPERIENCE	MINIMUM REQUIRED SKILLS***
Lab Technician/Chemist III	BS/BA in Chemistry, Biology, Environmental or related Science	5 years +	1. All skills of Chemist II 2. Experienced in training staff
Analyst I	HS or Equivalent	0-1 years	1. Knowledge of safe laboratory practices 2. Able to follow direction and Standard Operating Procedures (SOP's) 3. Experienced with sample handling, preparation and/or extraction
Analyst II	HS or Equivalent	2-4 years	1. All skills of Analyst I 2. Experienced in machine operation, maintenance and troubleshooting
Analyst III	HS or Equivalent	5 years +	1. All skills of Analyst II 2. Experienced in data review and reporting 3. Experienced in training staff
Analytical Chemist I	BS/BA in Chemistry, Biology, Environmental or related Science	6 mos-1 year	1. Knowledge of safe laboratory practices 2. Able to follow direction and Standard Operating Procedures (SOP's) 3. Experienced with sample handling, preparation and/or extraction
Analytical Chemist II	BS/BA in Chemistry, Biology, Environmental or related Science	2-4 years	1. All skills of Analytical Chemist I 2. Experienced in machine operation, maintenance and troubleshooting
Analytical Chemist III	BS/BA in Chemistry, Biology, or Environmental or related Science	5 years +	1. All skills of Analytical Chemist II 2. Experienced in data review and reporting 3. Experienced in training staff
Data Deliverable Specialist I	HS Diploma, BS/BA or Associates preferred	0-1 years	1. Introductory knowledge of laboratory methods 2.Able to follow direction and Standard Operating Procedures (SOP's) 3. Working knowledge of Adobe Acrobat, Microsoft Word, Excel 4. Good writing and typing skills
Data Deliverable Specialist II	HS Diploma, BS/BA or Associates preferred	2-4 years	1. All skills of Data Deliverable Specialist I 2. General knowledge of laboratory methods 3. Understanding of data review/ data reporting process 4. Experience with and understanding of LIMS and electronic data deliverables

TITLE*	REQUIRED EDUCATION**	MINIMUM REQUIRED ENVIRONMENTAL LAB EXPERIENCE	MINIMUM REQUIRED SKILLS***
Data Deliverable Specialist III	HS Diploma, BS/BA or Associates preferred	5 years +	1. All skills of Data Deliverable Specialist II 2. Intermediate/advanced knowledge of laboratory methods 3. Able to perform report review 4. Experience with and understanding of LIMS and electronic data deliverables 5. Able to initiate re-work where necessary
Laboratory Intern	2 Semesters of Chemistry, Biology or Environmental Science	None; Lab work study experience preferred	1. Knowledge of safe laboratory practices 2. Able to follow direction and Standard Operating Procedures

KEY

* Internal terms only. Full title would have "Environmental Laboratory" and specific department preceding it.

** Substitutions: Equivalent knowledge may be substituted for a degree in some instances.

*** Not meant to be an exhaustive list of skill requirements. For full list of skills consult the "Laboratory Skills" list. Actual Job Duties and Responsibilities can be found within job descriptions for each position.

24 Appendix I – Standard Operating Procedures

WESTBORO SOP #	Title
1728	Waste Management and Disposal
1730	Balance Calibration Check
1733	Thermometer Calibration
1737	Inorganics Glassware Cleaning and Handling
1738	Water Quality Monitoring
1745	Reagent, Solvent and Standard Control
1948	Separatory Funnel Liquid-Liquid Extraction – EPA 3510C
1953	Organic Extraction Glassware Cleaning & Handling
1954	Soxhlet Extraction – EPA 3540C
1955	Sulfur Cleanup – EPA 3660A
1956	Oil and Waste Dilution – EPA 3580A
1959	Microwave Extraction – EPA 3546
1960	Sulfuric Acid Cleanup – EPA 3665A
1962	Florisil Cleanup
1963	Fractionation Cleanup
1964	Preparation of Samples for Chlorinated Herbicides
2107	Volatile Organic Compounds – EPA 524.2
2108	Volatile Organic Compounds – EPA 8260C
2109	Polynuclear Aromatic Hydrocarbons (PAHs) by SIM – EPA 8270D (modified)
2111	Semivolatile Organics by GC/MS – EPA 8270D
2112	TCLP/SPLP Extraction - Volatile Organics SW-846 Method 1311/1312
2113	EDB, DBCP & TCP in Water by Microextraction & Gas Chromatography – EPA 504.1, 8011
2116	Organochlorine Pesticides by Capillary Column GC – EPA 8081B
2119	Extractable Petroleum Hydrocarbons – MADEP
2120	Volatile Petroleum Hydrocarbons – MADEP
2123	Polychlorinated Biphenyls in Oil – EPA 600/4-81-045
2125	TPH-Diesel Range Organics, Maine 4.1.25, EPA 8015C (Modified)
2126	TPH- Gasoline Range Organics, Maine 4.2.17, EPA 8015C (Modified)
2127	CT-ETPH
2128	Herbicides by 8151A
2129	PCBs by Capillary Column Gas Chromatography - EPA 8082A
2131	New Jersey EPH Method
2133	TCLP Extraction Metals and Semi-Volatile Organics – SW-846 Method 1311
2135	SPLP Extraction Inorganics and Semivolatile Organics, EPA 1312

WESTBORO SOP #	Title
2161	Fecal Coliform by Membrane Filtration – SM 9222D
2163	Fecal Coliform by Multiple Tube Fermentation – SM 9221E
2191	Heterotrophic Plate Count – SM 9215B
2192	Total Coliform/E.Coli – Presence/Absence (Colilert) – SM 9223B
2193	Total Coliform by Membrane Filtration – SM 9222B
2194	Total Coliform by Multiple Tube Fermentation – SM 9221B
2195	Chlorophyll A – SM 10200H
2196	E. Coli – Membrane Filtration
2197	Chlorophyll A – EPA 446
2198	Air Density Monitoring
2199	Inhibitory Residue Test
2200	Enterococcus – MF
2201	Total Coliform, E.Coli & Enterococcus by Quantification Methods (Quanti Tray)
2202	pH, Liquid Samples
2203	pH, Soil & Waste Samples
2204	Hexavalent Chromium
2205	Biological Oxygen Demand
2206	Ammonia Nitrogen
2207	Total Kjeldahl Nitrogen
2208	Chemical Oxygen Demand
2209	Oil & Grease by n-Hexane Extraction Method & Gravimetry
2210	Cyanide, Total
2211	Phenol, Total
2212	Sulfate, Turbidimetric Method
2213	Alkalinity, Titration Method –SM 2320B
2214	Determination of Inorganic Anions by Ion Chromatography – EPA 300.0
2215	Total Organic Carbon/Dissolved Organic Carbon
2216	Chloride – SM 4500Cl-E, EPA 9251
2217	Nitrate, Nitrite and Nitrate/Nitrite Nitrogen – EPA 353.2, SM 4500NO_3-F
2218	Total Solids (Dried @ 103-105^o) and TVS – SM 2540B, SM 2540E
2219	Total Dissolved Solids – SM 2540C
2220	Total Suspended Solids – SM 2540D
2221	Total Sulfide – SM 4500S2-AD, EPA 9030B
2222	MBAS, Anionic Surfactants – SM 5540C
2223	Fluoride, Electrode Method – SM 4500F-BC
2224	Turbidity, Nephelometric Method – EPA 180.1, SM 2130B
2225	Orthophosphate, Colorimetric Single Reagent Method – SM 4500P-E
2226	Total Phosphorous, Colorimetric Combined Reagent Method – SM 4500P-E
2227	Flashpoint – EPA 1010

WESTBORO SOP #	Title
2228	Reactivity – EPA Chapter 7.3
2229	Total Solids (Dried @ 103-105°) – SM 2540G
2230	Specific Conductance and Salinity
2231	True and Apparent Color, Visual Comparison Method
2232	Acidity, Titration Method
2233	Determination of Formaldehyde by HPLC, EPA 8315A
2234	Sulfite, Iodometric
2235	Ferrous Iron
2236	Residual Chlorine
2237	ORP
2238	Ignitability of Solids EPA 1030
2239	Physiologically Available Cyanide (PAC)
2240	Total Settleable Solids SM 2540 F
2241	Fixed and Volatile Solids in Solid and Semisolid Samples – SM 2540G
2242	Tannin & Lignin
2243	Nitrite - Manual Colorimetric Method
2244	Paint Filter Liquids Test
2245	Odor, Threshold Odor Test
2249	Dissolved Oxygen
2251	Perchlorate by IC/MS/MS
3743	Free Cyanide
9177	Total Phenol - SEAL Method
9733	Oil & Grease and TPH in Soil
10807	Percent Organic Matter in Soil
12838	Buchi Concentration
14751	Determination of UV-Absorbing Organic Constituents at 254nm
17972	Extractable Organic Halides (EOX)
18236	Chloropicrin and Carbon Tetrachloride by EPA 8011
19332	DI Water Extraction ASTM D3987
21994	Nonfractionated EPH
23148	Gilson EPH Fractionation
23561	Volatile Petroleum Hydrocarbons (VPH) by GC/MS
25691	Semivolatile Organic Compounds by GC/MS EPA 625.1
25693	Volatile Organic Compounds by EPA 624.1
26801	TPH - Gasoline Range Organics Maine 4.2.17, EPA 8015D
27634	True and Apparent Color, Single Wavelength Method
28200	PCBs by EPA 608.3
28201	Pesticides by EPA 608.3

MANSFIELD SOP #	Title
1753	Glassware Cleaning
1754	Balance Calibration
1755	Pipette Checks
1797	Haz Waste
1816	Reagent Solvent Standard Control
2134	Hot Block Digestion for Aqueous Samples EPA 3005A
2138	Mercury Aqueous 7470A
2139	Mercury Soil 7471B
2140	AVS SEM
2141	Hydride Generation
2142	Mercury Aqueous 1631E
2143	Mercury Soil 7474
2148	Metals Soil Digestion 3050
2150	Metals Microwave 3015
2152	Seawater Extraction of Metals
2155	EPA 8270D
2157	PAH by SIM
2158	EPA 8081B
2160	EPA 8082A Aroclors/Congeners by GC and TO-10A
2162	Pesticides/PCB Aroclors/Congeners by GC/MS SIM
2164	1,4-Dioxane GC/MS SIM
2165	Separatory Funnel Extraction EPA 3510C
2166	Tissue Prep
2167	GPC
2168	Sulfur Cleanup 3660
2169	Sulfuric Acid Cleanup 3665
2170	Silica Gel Cleanup
2171	% Lipids
2172	Microscale Solvent Extraction EPA 3570
2173	Soxhlet Extraction EPA 3540C
2174	Soxhlet Extraction of PUFs
2175	% Total Solids
2182	TOC by Lloyd Kahn
2183	Particle Size Determination
2184	Particulates in Air PM-10
2186	EPA TO-15
2187	APH
2188	Air PIANO
2189	Dissolved Gases

MANSFIELD SOP #	Title
2190	Cleaning & Preparation of Air Sampling Equipment
2246	TPH and SHC
2247	Alkylated PAH
2248	Organic Lead
2252	Fixed Gases
2255	PIANO Volatiles
2256	Ethanol in Oil
2257	Whole Oil Analysis
2259	Density Determination of Oils
2260	Alumina Cleanup
2261	Shaker Table
2263	Gravimetric Determination
2264	Tissue Extraction
2265	Organic Waste Dilution
2267	Client SOP: SGC - Manual Method
2268	Client SOP: DCM Extractable Method
4246	PAHs by SPME
6438	Mercury in Sorbent Tubes by CVAA
7900	Mercury 1631E Using Cetac-M-8000 Analyzer
9077	Porewater Generation
9480	EPA-TO-12
12863	EPA 8270D GC/MS Full Scan TO-13A
13091	HPAH
13406	Particulate Organic Carbon
14500	Lead in Particulate Matter
17452	TOC by EPA 9060A
17456	Moisture, Ash and Organic Matter
17829	Specific Gravity of Soil
17830	Liquid Limit, Plastic Limit and Plasticity Index of Soils
17940	1,4-Dioxane in Drinking Water by EPA 522
18086	Total Suspended Solids (TSS) SM 2540D
18705	PCB Congeners by GC/MS-SIM EPA 8270D
18710	Trace Elements in Waters and Wastes by ICP-MS EPA 200.8
18711	Metals by ICP EPA 200.7
18715	Mercury in Water (CVAA) EPA 245.1
18716	Hot Block Digestion for Aqueous Samples EPA 3005A
18717	Microwave Assisted Acid Digestion of TCLP Extracts EPA 3015
18718	Microwave Assisted Acid Digestion for Metals EPA 3015A/3051A
18817	Alcohols by FID- Aqueous Direct Injection EPA 8015D

MANSFIELD SOP #	Title
19625	Glycols by GC-FID EPA 8015D
19971	Air Drying Samples for PCBs and Metals Analysis
19978	Density of Soil
22132	Data Review – Ohio VAP
23511	PFAS by LC/MS/MS by EPA 537
23528	PFAS by LC/MS/MS Isotope Dilution by EPA 537(M)
24426	Preparation of EPA 537 by SPE for Analysis via LC/MS/MS
24454	Acetonitrile Extraction for Unknown Compounds via GCFID
25896	EPA 8290A Dioxins and Furans by Hi-Res MS
25900	EPA 1613B Dioxins and Furans by Hi-Res MS
25923	Mercury in Liquid Waste (Automated Cold-Vapor Technique) EPA 7470A
25924	Mercury in Solid/Semisolid Waste (Manual Cold-Vapor Technique) EPA 7471B
26026	PCB Congeners by GC/ECD 8082A
26796	Metals by ICP EPA 6010D
26797	Metals by ICP-MS EPA 6020B
27322	In Vitro Accessibility Assay for Lead in Soil EPA 1340
27360	PFAS in Cranberry Matrix by EPA 537 (M) LC/MS/MS Isotope Dilution
27485	Total Petroleum Hydrocarbons Screen by GC/FID 8015D
27897	PCB Congeners by High Resolution GC/MS
29033	PFAS by LC/MS/MS in Non-Potable Water
27056	HiRes Laboratory Glassware Cleaning
29139	Biomimetic Extraction Using SPME
32082	MADEP PFAS by SPE & LC/MS/MS Isotope

CORPORATE SOP #	Title
1559	Sample Receipt and Login
1560	Sample Custody and Tracking
1561	Bottle Order Preparation
1562	Computer System Backup/Control
1563	Computer and Network Security
1564	Software Validation and Control
1565	Training Program
1566	Report Generation and Approval
1567	Organics Data Deliverable Package Review
1722	Customer Inquiry and Complaint Procedures
1723	Project Management
1724	Quote/Contract Procedure
1725	Project Communication Form Generation

CORPORATE SOP #	Title
1726	Purchasing Procedure
1727	Accounts Payable Invoice Processing
1729	Document Control
1731	Manual Integration and Compound Rejection
1732	DL LOD LOQ Generation
1734	Control Limit Generation
1735	Analytical Guidelines for Method Validation
1736	Corrective and Preventative Actions
1739	Demonstration of Capability (DOC) Generation
1740	Internal Audit Procedure
1741	Data Review – Organics
1742	Calculating Measurement Uncertainty
1743	Annual Management Review
1744	Sample Compositing Procedure
1746	Nonconformance Planning/Procedures
1747	Temperature Datalogger Operation
2274	Data Validation Package
17553	Lab Supply Transfer Procedure
18821	Weights Verification
18909	PT Corrective and Preventive Action Process

25 Appendix J– Report Signing - List of Authorized Personnel

All final reports are reviewed and signed by authorized personnel, who have been designated to perform such review.

The following is the listing of all authorized representatives of the company who have been authorized by the Laboratory Technical Manager to perform final report review.

Name	Title
Peter Henriksen	General Manager
Susan O'Neil	Project Manager
Christopher Anderson	Project Manager
Elizabeth Porta	Project Manager
Andrew Rezendes	Volatiles Manager and Air Technical Manager
Lisa S. Westerlind	Technical Representative
Ellen M. Collins	Technical Representative
Michelle M. Morris	Technical Representative
Kelly Stenstrom	Technical Representative
Cristin Walker	Technical Representative
Cynthia McQueen	Technical Representative
Melissa Sturgis	Technical Representative
Amita Naik	Technical Representative
Caitlin Walukevich	Technical Representative
Tiffani Morrissey	Technical Representative
Jennifer Clements	Technical Representative
Kelly O'Neill	Technical Representative
Alycia Mogayzel	Technical Representative
James C. Todaro	Quality Assurance Officer

VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE FOR THE DETERMINATION OF GAMMA ISOTOPES

(GL-RAD-A-013 REVISION 27)

APPLICABLE TO METHODS:
EPA 600/4-80-032 Method 901.1
DOE EML HASL-300 Section 4.5.2.3
DOE EML HASL-300 Ga-01-R

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1.0 STANDARD OPERATING PROCEDURE FOR THE DETERMINATION OF GAMMA ISOTOPES

2.0 METHOD OBJECTIVE, PURPOSE, AND SUMMARY

2.1 This standard operating procedure (SOP) provides the necessary instructions to conduct the analysis for gamma isotopes in water, soil, urine, filters, drinking water and miscellaneous matrices.

2.2 Water samples are typically counted in Marinelli beakers. Soil samples are typically sealed in aluminum cans, which can be counted immediately if Ra-226 is not desired. If Ra-226 is desired, the sealed can is set aside for minimum of 20 days to allow equilibrium between Rn-222 and Bi-214 to become re-established. Ra-226 is then quantified using the 609 keV line of Bi-214.

2.3 This method is based on the source method EPA 600/4-80-032 "Prescribed Procedures for Measurement of Radioactivity in Drinking Water," August 1980, Method 901.1, and the Department of Energy (DOE) EML Procedures Manual source method for Gamma PHA in environmental samples, HASL-300 Section 4.5.2.3 and Ga-01-R, Gamma Radioassay.

2.4 This SOP is applicable for analyzing samples that contain radionuclides emitting gamma photons with energies ranging from about 5 to 2000 keV (including I-131).

3.0 METHOD SCOPE, APPLICABILITY, AND DETECTION LIMIT

3.1 Minimum Detectable Activity (MDA): The MDA is based upon sample volume, Compton background, instrument efficiency, count time, and other statistical factors, as well as specific isotopic values such as abundance and half-life. A typical detection limit is 10 pCi/L or 0.1 pCi/g (based on Cs-137). The MDA for drinking water samples is 10 pCi/L (based on Cs-137). Typical aliquot for drinking waters is 2 liters. Typical count time for drinking waters is 2-4 hours, to ensure the required sensitivity (specified by the National Primary Drinking Water Regulations) is achieved.

3.2 Method Precision: Typical Relative Percent Difference (RPD) is 20% or less or 100% or less if the activity is less than five times the MDA.

3.3 Method Bias (Accuracy): The method accuracy requirement for gamma, measured by running a Laboratory Control Sample (LCS) with each batch, is 25% of the true value. For drinking water samples, laboratory fortified blanks (LFB, equivalent to LCS) recoveries should be between 90-110% of the known value.

3.4 Procedures contained in this SOP may be used to analyze REMP samples.

3.5 Analysts training records are maintained as quality records as outlined in GL-QS-E-008. Analysts training and proficiency in the method is outlined in the Employee Training SOP GL-HR-E-002.

3.6 For drinking water samples, analyst initial and ongoing demonstrations of proficiency will follow critical elements for radiochemisrtry, chapter VI, section 1.5, of The Manual for the Certification of Laboratories Analyzing Drinking Water (reference 20.5).

3.7 Sensitivity studies will follow critical elements for radiochemistry, chapter VI, section 7.3 of The Manual for the Certification of Laboratories Analyzing Drinking Water (reference 20.5).

4.0 METHOD VARIATIONS

4.1 Some variations may be necessary due to special matrices encountered in the lab. These variations may be used with approval from a Group Leader or Team Leader. Variations to a method will be documented with the analytical raw data.

4.2 Filter samples can either be counted directly, or digested prior to counting. If filters are digested, they are digested in accordance with GL-RAD-A-026.

4.3 No method modifications are permitted for drinking water samples.

5.0 DEFINITIONS

5.1 National Institute of Standards and Technology (NIST): For the purpose of this method, the national scientific body responsible for the standardization and acceptability of analyte solutions.

5.2 Deionized (DI) water: Type I water.Refer to GL-LB-E-016.

5.3 AlphaLIMS: GEL's Laboratory Information Management System.

5.4 Batch: Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents.

5.5 Method Blank (MB): A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples containing an analyte of interest through all steps of the analytical procedures.

5.6 Laboratory Duplicate (DUP): For soils, when sufficient sample is available, a separate duplicate will be prepared. For liquid samples and when sufficient sample is not available for solids, an independent count of the sample container will be performed to show precision.

5.7 Laboratory Control Sample (LCS): A sample matrix, similar to the batch of associated samples (when available) that is free from the analytes of interest, spiked with verified known amounts of analytes from a source independent of the calibration standards or a material containing known and verified amounts of analytes. The LCS is equivalent to a Fortified Blank in the EPA drinking water compliance manual (See to section 20.5).

5.8 Refer to SOP GL-QS-B-001 the Quality Assurance Plan for additional lab-wide used definitions.

6.0 INTERFERENCES

6.1 Some gamma isotopes emit gamma lines that may overlap with other isotopes. If the energies of the two isotopes are within the energy tolerance setting, the peaks may not be resolvable and may give a positive bias to the result. This problem is minimized by careful review of the peak search.

6.2 Soil samples may vary in density from the standard used for calibration. A density correction is applied to the "CAN" geometry. This correction was determined using solids with weights varying between 54 g and 192 g.

7.0 SAFETY PRECAUTIONS AND WARNINGS

7.1 Keep hands free from moving parts of canning device and gamma shields.

7.2 Personnel performing this analytical procedure are trained in and follow the safe laboratory practices outlined in the Safety, Health and Chemical Hygiene Plan, GL-LB-N-001.

7.3 Personnel handling radioactive materials are trained in and follow the procedures outlined in GL-RAD-S-004 for Radioactive Material Handling.

7.4 Personnel handling biological materials are trained in and follow the procedures outlined in GL-RAD-S-010 for The Handling of Biological Materials.

7.5 If there is any question regarding the safety of any laboratory practice, **stop immediately**, and consult qualified senior personnel such as a Group or Team Leader.

8.0 APPARATUS, EQUIPMENT, AND INSTRUMENTATION

8.1 Ancillary Equipment

8.1.1 100 cc aluminum cans with lids for soil and miscellaneous samples

8.1.2 10 cc Gelman Sciences Petri dish for soil, filters and miscellaneous samples

8.1.3 2 L and 500 mL Marinelli beakers for water samples

8.1.4 Air displacement pipettes

8.1.5 Can sealing tool

8.1.6 Graduated cylinder

8.1.7 25 cc VWR Petri for soil and miscellaneous samples

8.1.8 250 mL plastic jar for filters, soil, and miscellaneous samples

8.1.9 Hot plate

8.1.10 Teflon beakers and lids

8.1.11 1 L Marinelli beaker for soil samples

8.2 Instrumentation

8.2.1 High purity germanium detector, with associated electronics and data reduction software

8.2.2 Top loader balance

9.0 REAGENTS, CHEMICALS, AND STANDARDS

9.1 NIST traceable mixed gamma standard in 100 cc aluminum can

9.2 NIST traceable mixed gamma standard in 2.0 L Marinelli beaker

9.3 NIST traceable mixed gamma standard in 0.5 L Marinelli beaker

9.4 NIST traceable mixed gamma standard in Gelman Sciences 10 cc Petri dish

9.5 NIST traceable mixed gamma standard in 13, 47 mm glass fiber filter composites in Gelman Sciences Petri dish.

9.6 NIST traceable mixed gamma standard in 0.4 L jar

9.7 NIST traceable mixed gamma standard in 0.25 L jar

9.8 NIST traceable mixed gamma standard in 1, 47 mm glass fiber filter

9.9 NIST traceable mixed gamma standard in Impregnated Charcoal Sample Cartridge.

9.10 NIST traceable mixed gamma standard in VWR (53 mm x 15 mm) Petri dish (approximately 25 cc)

9.11 NIST traceable mixed gamma standard in aqueous solution

9.12 NIST traceable mixed gamma standard in 1.0 L Marinelli beaker

9.13 NIST traceable mixed gamma standard in 20 mL liquid scintillation vial

9.14 16 M Nitric acid, reagent grade (HNO_3)

10.0 SAMPLE HANDLING AND PRESERVATION

10.1 For soil samples, 500 g of sample should be collected, preferably in a plastic container to avoid breakage.

10.2 For water samples, 2 L of sample should be collected in a plastic container and preserved to a pH < 2 with nitric acid.

10.2.1 Before beginning an analysis, the analyst should check the sample pH by removing a minimal amount of sample with a transfer pipette and placing it on a pH strip. DO NOT insert pH strip into sample container. If the sample is received with a pH greater than 2, the analyst should contact the Group Leader or Team Leader.

NOTE: If the analysis is requesting I-131 (or any other iodine isotopes) Analysis without preserving is acceptable. If a sample is preserved with acid without stabilizing the iodine, Iodine may volatilize and escape the solution as a gas.

10.2.2 If approved by the client, the analyst should adjust the pH with nitric acid to a pH < 2. If the sample pH is adjusted, let the sample sit in the original container for a minimum of 24 hours before analysis. This

acidification should be documented on a batch history sheet and attached to the batch paperwork.

10.3 For filters no preservation is necessary.

11.0 SAMPLE PREPARATION

11.1 Solid Sample Preparation.

11.1.1 Prepare the sample for gamma counting in accordance with SOP GL-RAD-A-021, Soil Sample Preparation for the Determination of Radionuclides.

11.1.2 Fill the appropriate container with sample prepared from step 11.1.1 using the following steps as a guideline:

11.1.2.1 If Ra-226 analysis is required, the sample is placed in a 100 cc can for in-growth.

NOTE: It is recommended that in-growth be allowed 20 days to quantify Ra-226. Shorter ingrowth periods can be used at the request of the client. However, shorter in-growth periods may decrease the accuracy of the data. If there is insufficient mass of sample to fill the 100 cc can, contact the Team or Group Leader.

11.1.2.2 If sufficient mass is available, homogenized samples should be placed in the 100 cc can. Determine the net weight of the sample. If the net weight is less than 54 g or greater than 192 g, contact the Team or Group Leader to determine the appropriate counting container. Record sample weight and date in AlphaLIMS and on sample container.

11.2 Water Sample Preparation

11.2.1 Place the appropriate labeled Marinelli beaker (typically 500 mL or 2 L) on a balance and tare the balance.

11.2.2 If less than approximately 1.1 L is available, sample should be poured into a 500 mL Marinelli beaker.

11.2.3 Transfer the appropriate volume to the tared container and record the volume of the sample on the Queue sheet.

NOTE: If there is insufficient sample to fill the Marinelli, record the exact amount of sample volume on the container and on the Queue sheet. Dilute the sample to the appropriate volume to maintain the calibration geometry. Record the volume the sample was diluted to on the sample container, also.

11.2.4 The MB should be recorded on the Queue sheet to be the same aliquot as the largest sample in the batch. An empty Marinelli beaker should be labeled as the MB and submitted with each batch of samples.

11.2.5 Submit the Marinellis and completed paperwork to the count room for gamma counting analysis.

11.3 Urine Sample Preparation

11.3.1 Refer to GL-RAD-B-030.

11.4 Preparation of Miscellaneous Matrices

11.4.1 Prepare the sample in accordance with GL-RAD-A-026 for The Preparation of Special Matrices for the Determination of Radionuclides.

11.4.2 If sample(s) was (were) received from the client in a container that matches a calibrated geometry, a direct count of the sample can be performed.

12.0 QUALITY CONTROL SAMPLES AND REQUIREMENTS

Refer to GL-RAD-D-003.

13.0 INSTRUMENT CALIBRATION, STANDARDIZATION, AND PERFORMANCE

Refer to GL-RAD-I-001.

14.0 ANALYSIS AND INSTRUMENT OPERATION

Refer to GL-RAD-I-001.

15.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

Refer to GL-RAD-I-010.

16.0 DATA RECORDING, CALCULATION, AND REDUCTION METHODS

Data recording, calculation and reduction take place in accordance with SOP GL-RAD-D-003 and GL-RAD-D-006.

17.0 DATA REVIEW, APPROVAL, AND TRANSMITTAL

Data are reviewed and packaged in accordance with GL-RAD-D-003 for Data Review, Validation, and Data Package Assembly.

18.0 RECORDS MANAGEMENT

Records generated as a result of this procedure are maintained as Quality Documents in accordance with GL-QS-E-008 for Quality Records Management and Disposition.

19.0 LABORATORY WASTE HANDLING AND DISPOSAL

Radioactive samples and material shall be handled and disposed of as outlined in the Laboratory Waste Management Plan, GL-LB-G-001.

20.0 REFERENCES

20.1 USEPA. Prescribed Procedures for Measurement of Radioactivity in Drinking Water, Method 901.1, August 1980.

20.2 Canberra Nuclear Genie System Spectroscopy, Applications and Display User's Guide, Vol. I and II, May 1991.

20.3 DOE EML Procedures Manual, HASL-300, 27th Edition. (Section 4.5.2.3)

20.4 DOE EML Procedures Manual, HASL-300, 28th Edition. (Ga-01-R)

20.5 Manual for the Certification of Laboratories Analyzing Drinking Water. Criteria and Procedures Quality Assurance. Fifth Edition EPA 815-R-05-004 January 2005.

20.6 Dept. of Defense (DOD), Dept. of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories DOD QSM Version 5.0, July 2013 and Version 5.1, January 2017; DOE QSAS 3.0, July 2013 and Version 3.1 January 2017.

21.0 HISTORY

Revision 23: Procedure updated to include requirements for drinking water samples.

Revision 24: Changed recovery limit for laboratory fortified blank from 90-100% to 90-110% in section 3.3.

Revision 25: Type II to type I water.

Revision 26: Removed reference to obsolete software. Updated the reagents and standards section. Updated sample prep section to current practices.

Revision 27: Added clarification of aliquots volume and count times for samples analyzed for drinking water methods.

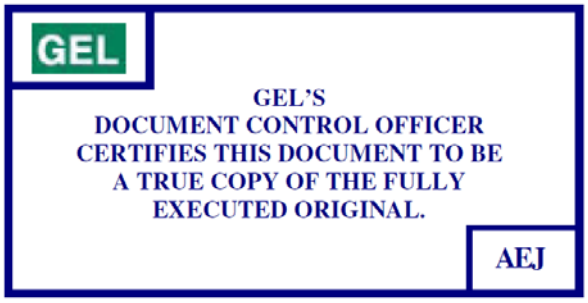
VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE FOR DIGESTION FOR SOIL

(GL-RAD-A-015 REVISION 18)

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1.0 STANDARD OPERATING PROCEDURE FOR DIGESTION FOR SOIL

2.0 METHOD OBJECTIVE, PURPOSE, CODE, AND SUMMARY

2.1 This standard operating procedure provides the necessary instructions to conduct digestion on soil type matrices. An appropriate amount of sample is aliquoted into a Teflon, glass or polypropylene container. Concentrated acids and heat are applied until a homogenous aqueous solution is produced. This is accomplished by an acid dissolution or a fusion technique.

2.2 This procedure is applicable to soils for any analysis requiring complete dissolution. Sample aliquots of one gram or less can be accommodated by the equipment and amount of reagents specified. Sequential analysis of more than one radionuclide may be accomplished by the addition of carriers and tracers for each applicable analysis prior to digestion.

2.3 To minimize errors due to non-homogeneity of a sample, it is always desirable to use the largest possible aliquot of sample. The existence of "hot particles" may lead to questionable data when very small sample aliquots are used. In cases where radionuclide concentrations are "higher than normal environmental levels" and smaller aliquots are necessary, it is preferable to dilute the digested 1 gram sample rather than select a smaller aliquot. The analyst should consult the group leader for guidance in these situations.

3.0 METHOD SCOPE, APPLICABILITY, AND DETECTION LIMIT

Method Detection Limit (MDL): Refer to the specific analytical SOP for minimum detectable activity (MDA) values.

4.0 METHOD VARIATIONS

Some variations may be necessary due to special matrices encountered in the lab. These variations may be used with approval from a group leader or senior technical specialist. Variations to a method will be documented with the analytical raw data.

5.0 DEFINITIONS

5.1 Batch: Environmental samples that are prepared and/or analyzed together with the same process and personnel using the same lot(s) of reagents.

5.2 Laboratory Control Sample (LCS): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes from a source independent of the calibration standards or a material containing known and verified amounts of analytes.

5.3 Laboratory Duplicate (DUP): Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently.

5.4 Matrix Spike (MS): Prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available.

5.5 Matrix Spike Duplicate (MSD): A second replicate matrix spike is prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

5.6 Method Blank (MB): A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples containing an analyte of interest through all steps of the analytical procedures.

5.7 National Institute of Standards and Technology (NIST): For the purpose of this method, the national scientific body responsible for the standardization and acceptability of analyte solutions.

5.8 Deionized (DI) water: Type I DI water, Refer to GL-LB-E-016.

5.9 Refer to GL-QS-B-001 the Quality Assurance Plan for additional lab-wide used definitions.

6.0 INTERFERENCES

Not Applicable

7.0 SAFETY PRECAUTIONS AND WARNINGS

7.1 Personnel performing this analytical procedure are trained to the safe laboratory practices outlined in the Safety, Health, and Chemical Hygiene Plan, GL-LB-N-001.

7.2 Personnel handling radioactive materials are trained in and follow the procedures outlined in GL-RAD-S-004 for Radioactive Material Handling.

7.3 Personnel handling biological materials are trained in and follow the procedures outlined in GL-RAD-S-010 for The Handling of Biological Materials.

7.4 If there is any question regarding the safety of any laboratory practice, **stop immediately**, and consult qualified senior personnel such as a group or team leader.

8.0 APPARATUS, EQUIPMENT, AND INSTRUMENTATION

8.1 Equipment

8.1.1 Beakers (Glass and Teflon of various sizes)

8.1.2 Platinum crucibles

8.1.3 Hot plate

8.1.4 Blast burner

8.1.5 PTFE spatula

8.1.6 High temperature resistant gloves

8.1.7 Tongs

8.1.8 Segmented muffle furnace

8.1.9 Watch glasses (various sizes)

8.1.10 Zirconium crucibles and lids

8.1.11 Aluminum Foil

8.1.12 Analytical balance

8.1.13 Polypropylene centrifuge tube (50 mL)

8.1.14 Hotblock

9.0 REAGEANTS AND STANDARDS

9.1 Reagents

9.1.1 Concentrated Nitric Acid, reagent grade (16 M HNO_3)

9.1.2 Concentrated Hydrofluoric Acid, (49% HF).

9.1.3 Concentrated Hydrochloric Acid, reagent grade (12 M HCl)

9.1.4 Deionized (DI) Water

9.1.5 Saturated Boric Acid, 5%: Dissolve 50 g of H_3BO_3 per liter of DI water.

9.1.6 Ammonium Hydroxide (28-30% NH_4OH)

9.1.7 Potassium Fluoride, anhydrous (KF)

9.1.8 Sodium Sulfate (Na_2SO_4)

9.1.9 Concentrated Sulfuric Acid, (18 M H_2SO_4)

9.1.10 Sodium Hydroxide pellets

9.1.11 1.25 M Calcium Nitrate: Dissolve 205 g of anhydrous Calcium Nitrate or 295 g hydrated Calcium Nitrate, $Ca(NO_3)_2 \cdot 4H_2O$, in 500 mL of DI water. Dilute to 1 L with DI water.

9.1.12 Iron Carrier (10 mg/mL): Dissolve 62.7 g of Fe(NO3)3 • 6H2O or 72.3 g of Fe(NO3)3 • 9H2O in 800 mL DI water and dilute to 1 L with DI water.

9.2 Standards

9.2.1 See applicable analytical SOP for the appropriate standards.

10.0 SAMPLE HANDLING AND PRESERVATION

Soil and sand require no preservation and may be shipped in any suitable container.

11.0 SAMPLE PREPARATION

11.1 For instructions on drying, grinding, homogenizing, and blending soil samples, refer to GL-RAD-A-021 for Soil Sample Preparation for the Determination of Radionuclides.

11.2 Alpha Spec soil digestion procedure (also to be used for total Uranium digestions).

11.2.1 If samples are to be ashed, weigh an appropriate aliquot (0.1 to 1.0 g) into a glass container. If dried sample used, the sample aliquot should be placed in teflon beaker. If required, the DUP, MS, and MSD should be the same aliquot as the appropriate sample referenced. Record all aliquots. For alpha spec, add approximately 4 to 6 drops of Iron Carrier to the blank and LCS beakers. The blank and LCS aliquot should be recorded to be the same aliquot as the largest sample in the batch. Iron Carrier may also be added to all of the samples in the batch, if needed, based on the appearance and iron content of each sample. If Solid Reference Material is required, weigh out approximately 0.1 g into the LCS beaker, and record the exact weight.

11.2.2 Add appropriate spikes and tracers according to the analytical SOP you are performing. Record tracer and spike IDs and volumes. If dried samples used, proceed to step 11.2.5.

11.2.3 Place each glass container in the furnace with even spacing. A removable shelf is provided that can be used if necessary. Cover glass containers prior to closing door to furnace to prevent cross-contamination. Cover with a material appropriate for the sample, such as aluminum foil or glass. If aluminum foil is used, poke holes in the foil so that pressure does not build up inside the container. Muffle for approximately 2 to 4 hours. Use tongs and/or wear protective gloves to remove the containers from the furnace when the furnace temperature has decreased below 100° C.

NOTE: Determining ashing routine is based on analyst experience with the matrix. If flammability of the matrix is in question, such as vegetation, tissues, and paper, use the slow ramp procedure. (Consult operation method to set up procedure.) The time required to obtain an ash sample will vary depending on the type of material, size of sample, oven type, and capacity, and other factors. The influence of these factors generally can be established by good judgment, and experience with the materials being tested and the apparatus being used.

11.2.4 Add 5 to 10 mL concentrated Nitric Acid and 5 to 10 mL concentrated Hydrochloric Acid to each container and cover with a watch glass. Reflux for approximately 30 minutes. Transfer each sample into a previously labeled, clean teflon beaker. Rinse any remaining soil from the glass container into the teflon beaker using approximately 5 to 10 mL of concentrated Hydrochloric Acid. Dry on a hot plate using medium heat.

NOTE: All glass containers should be disposed of after muffling.

11.2.5 Add 10 mL of 49% Hydrofluoric Acid to each teflon beaker. Cover beakers with a teflon cover and place on hot plate on medium heat for approximately 30 minutes. Remove lids and evaporate solution to dryness on medium heat.

11.2.6 Add 10 mL of concentrated Nitric Acid and 10 mL of 49% Hydrofluoric Acid to each teflon beaker. Cover beakers with a teflon cover and place on medium heat for approximately 30 minutes. Remove lids and evaporate the solution to dryness on medium heat.

11.2.7 Add 10 mL of concentrated Hydrochloric Acid and 5 mL of 49% Hydrofluoric Acid to each teflon beaker. Cover beakers with a teflon cover and place on medium heat for approximately 30 minutes. Remove lids and evaporate the solution to dryness on medium heat.

11.2.8 Add 10 mL concentrated Hydrochloric Acid and 10 mL concentrated Nitric Acid to each teflon beaker and evaporate to dryness on medium heat.

11.2.9 Add 10 mL concentrated Hydrochloric Acid and 1 mL of saturated Boric Acid to each sample and evaporate to dryness on medium heat.

11.2.10 Add 5 mL concentrated Nitric Acid and evaporate to dryness on medium heat (Th, U by ICPMS and Ra samples only).

11.2.11 Digestion should be complete at this point. Proceed to appropriate analytical procedure for analysis. For alpha spec analyses**,** if further clean up of sample is deemed necessary by your team leader/group leader, continue with step 11.2.12.

11.2.12 Dissolve the sample residue in 10 to 15 mL of concentrated Hydrochloric Acid and transfer to a disposable centrifuge tube using DI water as a rinse. Add Ammonium Hydroxide until Iron Hydroxide precipitates, then add 2 mL excess Ammonium Hydroxide. Centrifuge sample and discard supernate.

11.2.13 Wash precipitate with 20 mL of DI water that has been adjusted to pH 10 with Ammonium Hydroxide. Centrifuge sample and discard supernate.

11.2.14 For any combination of alpha spec analyses, dissolve the Iron precipitate in the required reagent listed in the appropriate analytical procedure being used and proceed per SOP.

11.3 Gross Alpha/Beta Soil Digest Procedure in Teflon Beakers

11.3.1 Weigh out an appropriate aliquot (normally 0.1 g) into a teflon beaker. If required, the DUP, MS, and MSD should be the same aliquot as the appropriate sample referenced. Record all aliquots.

11.3.2 Add spike solutions to the applicable samples and record volumes.

11.3.3 Add 10 mL of concentrated Nitric Acid to each sample.

11.3.4 Place samples on medium heat and cover each sample with a teflon lid. Reflux all samples for approximately 30 minutes.

11.3.5 Remove teflon lids and add 5 mL concentrated Hydrochloric Acid and 10 mL 49% Hydrofluoric Acid to each sample. Cover samples and reflux for approximately 120 minutes.

11.3.6 Remove teflon lids and allow samples to evaporate to dryness on medium heat.

11.3.7 Add 5 mL of concentrated Nitric Acid and evaporate to dryness on medium heat.

11.3.8 Repeat Step 11.3.7.

11.3.9 Add 5 mL of concentrated Nitric Acid to the dry samples. Place the samples back on the hot plate long enough so that the dried sample dissolves into the acid.

11.3.10 Samples are now ready for planchetting in accordance with GL-RAD-A-001B.

11.4 Gross Alph/Beta Soil Digest Procedure in polypropylene Tubes

11.4.1 Weigh out an appropriate aliquot (normally 0.1 g) into a 50 mL polypropylene tube. If required, the DUP, MS and MSD should be the same aliquot as the appropriate sample referenced. Record all aliquots.

11.4.2 Add spike solutions to the applicable samples and record volumes.

11.4.3 Add 3 mL of concentrated Nitric Acid, 3 mL concentrated Hydrochloric Acid, and 5 mL 49% Hydrofluoric Acid to each sample. Place tubes into a hotblock. Cover samples and reflux for approximately 120 minutes.

11.4.4 Remove polypropylene lids and evaporate to dryness.

11.4.5 Add 1-2 mL of concentrated Nitric Acid and evaporate to dryness.

11.4.6 Repeat Step 11.4.5.

11.4.7 Add 5 mL of concentrated Nitric Acid to the dry samples. Place a watchglass on the tubes and reflux for approximately 15 minutes.

11.4.8 Samples are now ready for planchetting in accordance with GL-RAD-A-001B.

11.5 Pyrosulfate Fusion Dissolution Procedure

NOTE: Leather gloves and a leather apron should be worn when working in the fusion hood. All equipment should be checked prior to fusing samples. Extreme caution should be taken when working with open flames.

11.5.1 Add appropriate tracers to each sample as well as to control samples according to the analytical SOP you are performing. Prepare the laboratory control samples and spikes in the same manner as the other samples in the batch.

11.5.2 Transfer the dried, tared sample into a Platinum dish. Wet the sample with approximately 1 mL concentrated Nitric Acid in order to eliminate spattering from Carbonates.

11.5.3 When the bubbling has ceased, add about 1 mL 49% Hydrofluoric Acid and heat to dryness on a medium hot plate.

11.5.4 Once the sample is dry, add approximately 3 g of Potassium Fluoride; using a PTFE spatula, stir the dried sample and Potassium Fluoride together. Fuse the sample over a blast burner until all bubbling has ceased, sample has visibly dissolved, and clear fusion flux is observed.

11.5.5 Remove from the burner, place on a hot plate using medium heat, and let equilibrate to the hot plate temperature.

11.5.6 Slowly add about 4 mL concentrated Sulfuric Acid rinsing down the walls of the Platinum dish to the extent possible. Let the sample set on the hot plate until bubbling has ceased.

11.5.7 Add approximately 2 g of Sodium Sulfate to the sample. Heat over the blast burner again in order to transpose the Fluoride cake into a Pyrosulfate cake. Heat until bubbling ceases and most of the liquid is gone. Place on the hot plate to cool to the hot plate's temperature.

11.5.8 Add approximately 5 mL concentrated Hydrochloric Acid and approximately 35 mL DI water to the fusion cake while heating to effect dissolution. Take volume up to about 100 mL with DI water.

11.5.9 Proceed to the applicable SOP for analysis.

11.6 Sodium Hydroxide Fusion Dissolution Procedure

11.6.1 Add appropriate tracers to each sample as well as to control samples according to the analytical SOP you are performing. Prepare the laboratory control samples and spikes in the same manner as the other samples in the batch.

NOTE: For Ra analyses, if an empty crucible is analyzed as a reagent blank, 100 mg Ca (by evaporating 2 mL of 1.25 M Calcium Nitrate) should be added to the Zirconium crucible.

11.6.2 Place crucibles on a hot plate and heat to dryness on medium heat.

11.6.3 Remove crucibles from hot plate and allow to cool.

11.6.4 Add 10-15 grams of Sodium Hydroxide to the crucibles.

11.6.5 Place the crucibles with lids in the 600 °C furnace using tongs.

11.6.6 Fuse crucibles for 15-20 minutes.

NOTE: Longer times may be needed for larger particle sizes.

11.6.7 Remove hot crucibles from furnace very carefully using tongs, and transfer to hood after crucibles have cooled.

11.6.8 Add approximately 25-50 mL of water to each and heat on hotplate to loosen/dissolve solids.

11.6.9 If necessary for dissolution, add more water and warm as needed on a hotplate.

11.6.10 Proceed to applicable SOP for analysis.

12.0 QUALITY CONTROL SAMPLES AND REQUIREMENTS

12.1 Method Verification

Refer to GL-RAD-D-002 for instructions concerning the validation of analytical methods.

12.2 Method Specific Quality Control Requirements

See specific SOP for specific method quality control requirements.

13.0 INSTRUMENT CALIBRATION, STANDARDIZATION, AND PERFORMANCE

Refer to GL-LB-E-002 and GL-LB-E-004.

14.0 ANALYSIS AND INSTRUMENT OPERATION

Refer to GL-LB-E-002 for Balances.

15.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

Refer to GL-LB-E-002 for Balances.

16.0 DATA RECORDING, CALCULATION, AND REDUCTION METHODS

Refer to GL-LB-E-008 for Basic Requirements for the Use and Maintenance of Laboratory Notebooks, Logbooks, Forms, and Other Recordkeeping Devices.

17.0 DATA REVIEW, APPROVAL, AND TRANSMITTAL

Not Applicable

18.0 RECORDS MANAGEMENT

Records generated as a result of this procedure are maintained as quality documents in accordance with GL-QS-E-008 for Quality Records Management and Disposition.

19.0 LABORATORY WASTE HANDLING AND WASTE DISPOSAL

Radioactive samples and material are be handled and disposed in accordance with the Laboratory Waste Management Plan, GL-LB-G-001.

20.0 REFERENCES

None

21.0 HISTORY

Revision 13: Technical: Updated section 11.2.10. Updated Appendix I to reflect change to section 11.2.10.

Revision 14: Updated sections 8.0 and 9.0. Added Section 11.5.

Revision 15: Technical updates for SOP consistency as part of annual review.

Revision 16: Updated Sample preparation section with current volume of Acid used in process.

Revision 17: Revised to remove reference to queue sheets.

APPENDIX 1
ALPHA SPECTROSCOPY SOIL DIGESTION

____ Weigh appropriate amount of dry soil to meet MDA

____ Add appropriate tracers and spikes to samples

____ Muffle samples for approximately 2-4 hours

____ Add ~10 mL concentrated HNO_3 and ~10 concentrated HCl

____ Reflux for approximately 30 minutes

____ Transfer to teflon

____ Evaporate to dryness on medium heat

____ Add 10 mL concentrated HF

____ Reflux for approximately 30 minutes

____ Evaporate to dryness on medium heat

____ 10 mL concentrated HNO_3 and 10 mL concentrated HF

____ Reflux for approximately 30 minutes

____ Evaporate to dryness on medium heat

____ 10 mL concentrated HCl and 5 mL concentrated HF

____ Reflux for approximately 30 minutes

____ Evaporate to dryness on medium heat

____ 10 mL concentrated HNO_3 and 10 mL concentrated HCl

____ Evaporate to dryness on medium heat

____ 10 mL concentrated HCl and 1 mL boric acid

____ Evaporate to dryness on medium heat

____ For Thorium, Uranium by ICPMS or Radium samples add 5 mL concentrated HNO_3 and evaporate to dryness on medium heat

____ Proceed to the appropriate procedure for completion of analysis

APPENDIX 2

GROSS ALPHA/BETA SOIL DIGESTION
Teflon beakers

____ Weigh appropriate amount of dry soil to meet MDA

____ Add appropriate spikes to samples

____ 10 mL concentrated HNO_3

____ Reflux for approximately 30 minutes

____ 5 mL concentrated HCl and 10 mL concentrated HF

____ Reflux for approximately 120 minutes

____ Evaporate to dryness on medium heat

____ 5 mL concentrated HNO_3

____ Evaporate to dryness on medium heat

____ 5 mL concentrated HNO_3

____ Evaporate to dryness on medium heat

____ Dissolve sample with 5 mL concentrated HNO_3

____ Proceed to GL-RAD-A-001B for completion of analysis

APPENDIX 3

GROSS ALPHA/BETA SOIL DIGESTION
Polypropylene Tubes

____ Weigh appropriate amount of dry soil to meet MDA

____ Add appropriate spikes to samples

____ 3 mL concentrated HNO_3, 3 mL concentrated HCl, and 5 mL concentrated HF

____ Reflux for approximately 120 minutes

____ Evaporate to dryness

____ 1-2 mL concentrated HNO_3

____ Evaporate to dryness

____ 1-2 mL concentrated HNO_3

____ Evaporate to dryness

____ Dissolve sample with 5 mL concentrated HNO_3

____ Proceed to GL-RAD-A-001B for completion of analysis

VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

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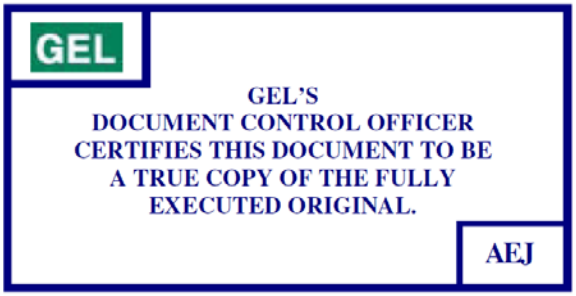
THE DETERMINATION OF

RADIOMETRIC POLONIUM

(GL-RAD-A-016 REVISION 19)

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1.0 STANDARD OPERATING PROCEDURE FOR THE DETERMINATION OF RADIOMETRIC POLONIUM

2.0 METHOD OBJECTIVE, PURPOSE AND SUMMARY

2.1 This standard operating procedure provides the necessary instructions to conduct the analysis for isotopic alpha emitting polonium in liquids, solids, and filters.

2.2 A solid sample is leached in concentrated nitric acid for a minimum of 2 hours. After centrifuging to remove the solids, the sample is diluted with DI water and treated as a liquid sample. Transuranic elements are scavenged by coprecipitation with calcium phosphate. This precipitate is dissolved and polonium is plated onto a metal disk (Ni, Ag, or Cu plated). This Ni, Ag, or Cu plated disk is then mounted onto a metal disk and placed in a partially evacuated chamber for measurement of isotopic alpha emission.

3.0 METHOD SCOPE, APPLICABILITY, AND DETECTION LIMIT

3.1 Method Detection Limit: Typical minimum detectable activity (MDA) for samples analyzed for polonium is 1 pCi/L or 1 pCi/g.

3.2 Analysts training records are maintained as quality records. Refer to GL-QS-E-008 for Quality Records Management and Disposition.

4.0 METHOD VARIATIONS

Some variation may be necessary due to special matrices encountered in the laboratory. These variations may be used with approval from a Group or Team Leader. Variations to a method will be documented with the analytical raw data.

5.0 DEFINITIONS

5.1 AlphaLIMS: GEL's Laboratory Information Management System.

5.2 Batch: Environmental samples prepared and/or analyzed together with the same process and personnel using the same lot(s) of reagents.

5.3 Laboratory Control Sample (LCS): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes from a source independent of the calibration standards or a material containing known and verified amounts of analytes.

5.4 Laboratory Duplicate (DUP): Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently.

5.5 Matrix Spike (MS): Prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available.

5.6 Matrix Spike Duplicate (MSD): A second replicate matrix spike is prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

5.7 National Institute of Standards and Technology (NIST): For the purpose of this method, the national scientific body responsible for the standardization and acceptability of analyte solutions.

5.8 Deionized (DI) Water: Type I water (Refer to GL-LB-E-016).

5.9 Refer to GL-QS-B-001 the Quality Assurance Plan for additional lab-wide used definitions.

6.0 INTERFERENCES

Not applicable.

7.0 SAFETY PRECAUTIONS AND WARNINGS

7.1 Personnel performing this analytical procedure are trained in and follow the safe laboratory practices outlined in the Safety, Health and Chemical Hygiene Plan, GL-LB-N-001.

7.2 Personnel handling Radioactive Materials are trained in and follow the procedures outlined in GL-RAD-S-004 for Radioactive Material Handling.

7.3 Personnel handling biological materials are trained in and follow the procedures outlined in GL-RAD-S-010 for The Handling of Biological Materials.

7.4 If there is any question regarding the safety of any laboratory practice, **stop immediately**, and consult senior qualified personnel such as a Group or Team Leader.

8.0 APPARATUS, EQUIPMENT, AND INSTRUMENTATION

8.1 Apparatus and Equipment

8.1.1 Polypropylene centrifuge tubes (50 mL)

8.1.2 Beakers (Glass and Teflon of various sizes)

8.1.3 Black electrical tape, 1” in width

8.1.4 Ni, Ag, or Cu plated disk, 7/8” diameter

8.1.5 Magnetic stir bars, 1” in length

8.1.6 Magnetic stir plates

8.1.7 Stainless steel tweezers

8.1.8 29 mm flat metal planchet

8.1.9 Sample drying apparatus

8.1.10 Sample homogenizing apparatus

8.1.11 Watch glasses (various sizes)

8.1.12 Small plastic cups

8.1.13 Petri dishes

9.0 REAGENTS AND STANDARDS

9.1 Reagents

9.1.1 Hydrochloric acid, concentrated (12 M HCl)

9.1.2 Nitric acid, concentrated (16 M HNO_3)

9.1.3 Ammonium hydroxide, concentrated (14 N NH_4OH)

9.1.4 Ascorbic acid powder

9.1.5 Phosphoric acid, concentrated (H_3PO_4)

9.1.6 Hydrogen peroxide (30% H_2O_2)

9.1.7 1.25 M Calcium nitrate carrier: Dissolve 205 g of anhydrous calcium nitrate or 295 g hydrated calcium nitrate, $Ca(NO_3)_2 \bullet 4H_2O$, in 500 mL of DI water. Dilute to 1L with DI water.

9.1.8 1 M Hydrochloric acid: Add 84 mL of concentrated hydrochloric acid to 500 mL of DI water. Allow to cool and dilute to 1 L with DI water.

9.1.9 6 M Hydrochloric Acid: Add 500 mL of concentrated hydrochloric acid to 500 mL of DI water.

9.2 Standards

9.2.1 NIST traceable standards: Po-209 and Po-210

9.2.2 Refer to GL-RAD-M-001.

10.0 SAMPLE HANDLING AND PRESERVATION

10.1 Water samples should be collected in plastic bottles and preserved with concentrated nitric acid to pH < 2.

10.2 Before beginning an analysis, the analyst should check the sample pH by removing a minimal amount of sample with a transfer pipette and placing it on a pH strip. DO NOT insert pH strip into sample container. If the sample is received with a pH greater than 2, the analyst should contact the Group Leader or Team Leader. If approved by the client, the analyst should adjust the pH with nitric acid to a pH < 2. If the sample pH is adjusted, let the sample sit in the original container for a minimum of 24 hours before analysis. This acidification should be documented on a batch history sheet and attached to the batch paperwork.

10.3 If the sample has exceeded the hold time, the analyst should contact the Group Leader before continuing with the batch.

10.4 Solid samples require no preservation and may be shipped in any suitable container.

11.0 SAMPLE PREPARATION

NOTE: Aliquots may be estimated by using the count time estimator spreadsheet.

11.1 Aqueous Sample Preparation

11.1.1 Add an appropriate aliquot of sample to a labeled beaker. Prepare a Blank and LCS using DI water and a small amount of concentrated nitric acid to a pH < 2. The volume of DI water used should be the same as the largest volume of sample in the batch. If required, the DUP, MS and MSD should be the same aliquot as the appropriate sample referenced. Record all aliquots.

11.1.2 Add a certified dpm of the appropriate tracer to each of the samples (usually between 5 to 10 dpm). Add a certified dpm (usually between 5 to 10 dpm) of the appropriate spike to the MS, MSD, LCS and LCSD as applicable. Reference batch pull sheet for client requirements to determine appropriate tracer and spike.

11.1.2.1 For the determination of isotopic polonium, Po-209 is typically used as the tracer and Po-210 is typically used as the spike.

NOTE: The addition of tracers and spikes should be witnessed by either another analyst qualified on this procedure, a Team Leader, or a Group Leader. After adding the tracers and spikes, the witness must initial and record the date of witnessing.

11.1.3 Add 2 mL of 1.25 M calcium carrier, 1 mL of phosphoric acid, and 20 mL of 30% hydrogen peroxide to each sample. Stir thoroughly. Reflux on low setting for approximately 90 minutes.

11.1.4 Add concentrated ammonium hydroxide in a drop wise manner until visible white precipitate forms and the pH is between 8 and 9. Check the pH using a pH strip while precipitating.

11.1.5 Cover and reflux samples on low setting for approximately 90 minutes. Allow the sample to stand until precipitate settles.

11.1.6 Decant excess supernate and discard. Collect the remaining precipitate by centrifugation in a 50 mL centrifuge tube and discard the supernate.

11.1.7 Rinse the beaker with approximately 25 mL 1 M hydrochloric acid, and cover and heat on a hot plate for several minutes to dissolve any residual calcium phosphate. Cool slightly and transfer rinse to the centrifuge tube. Cap and shake to dissolve the precipitate. Add 2 g of ascorbic acid powder to each sample. Cap and shake each centrifuge tube until the ascorbic acid dissolves. The sample should appear clear or slightly opaque.

NOTE: If any samples appear to contain iron, additional ascorbic acid should be added in 0.5 g increments. Cap and shake the centrifuge tube well after each addition. Continue until the sample has a clear or slightly opaque hue.

11.1.8 Transfer each sample to a clean, labeled Teflon beaker or new plastic cup. Rinse the centrifuge tube with approximately 25 mL of 1 M hydrochloric acid. Transfer rinse to the Teflon beaker or plastic cup. (Final volume should be approximately 50 mL.)

11.1.9 Proceed to step 11.3.

11.2 Solid or Filter Preparation

11.2.1 If not already done, dry and homogenize the solid samples by performing GL-RAD-A-021. This step is not performed when analyzing filter media.

11.2.2 Measure an appropriate aliquot of solid (usually 0.2 g to 1.0 g) in a glass container. If required, the DUP, MS and MSD should be the same aliquot as the appropriate sample referenced. Record all aliquots. The Blank and LCS aliquots should be recorded to be the same aliquot as the largest sample in the batch.

NOTE: Ashing of soils in a muffle furnace will result in significant polonium loss prior to analysis.

11.2.3 Add a certified dpm of the appropriate tracer to each of the samples (usually between 5 to 10 dpm). Add a certified dpm (usually between 5 to 10 dpm) of the appropriate spike to the MS, MSD, LCS and LCSD as applicable. Reference batch pull sheet for client requirements to determine appropriate tracer and spike.

11.2.3.1 For the determination of isotopic polonium, Po-209 is typically used as the tracer, and Po-210 is typically used as the spike.

NOTE: The addition of tracers and spikes should be witnessed by either another analyst qualified on this procedure, a Team Leader, or a Group Leader. After

adding the tracers and spikes, the witness must initial and record the date of witnessing.

11.2.4 Leach the samples in approximately 20 mL of concentrated nitric acid. More acid may be required for larger aliquots. Slowly add 5-10 mL of 30% hydrogen peroxide. Cover the samples and reflux on a low to medium setting for approximately 2 hours. Agitate the sample periodically to enhance the leaching process. Larger aliquots may need to reflux for a longer period of time. Do not allow the samples to go dry; add more acid if necessary.

11.2.5 Allow the samples to cool. Transfer each solid sample to a labeled centrifuge tube. Make sure to rinse the entire sample into the centrifuge tube using DI water. Centrifuge to separate the solid and leached portions of the sample. Decant the leachate back into a clean beaker. Rinse the solid phase with DI water. Centrifuge the sample and decant rinse into the beaker.

11.2.6 Dilute the leachate with DI water to a total volume of approximately 150 mL.

11.2.7 Proceed to step 11.1.3.

11.3 Metal Disk (Ni, Ag, Cu plated 7/8" diameter) preparation

11.3.1 If the metal disk provided does not have an insulated back, prepare the disk by taping one side with black electrical tape. Degrease the disk by first suspending each disk, one at a time, in concentrated nitric acid for approximately 5 seconds, followed by immediately suspending the disk in concentrated hydrochloric acid for approximately 5 seconds. Rinse each disk by suspending it in DI water. Blot the disk dry with a clean paper towel. Do not allow the disk to sit exposed to air for more than one hour.

NOTE: Excessive degreasing may result in low yields due to removal of the metal plating.

11.3.2 Tape a magnetic stir bar to the back of each metal disk. Place a disk in each Teflon beaker or new plastic cup from step 11.1.8. Make sure the metal side of the disk is facing up. Using a stir plate, stir the samples for at least 4 hours and no more than 16 hours at a low speed. DO NOT USE HEAT. Periodically check the samples to see if the disks are metal side up and submerged in sample.

11.3.3 After plating is complete, remove each disk, one at a time, and thoroughly rinse with 6 M hydrochloric acid. Rinse the disc with DI

water. Dry the disk by carefully blotting with a clean paper towel. Center each disk onto a labeled 29 mm flat planchet and place in a labeled Petri dish. Dry samples under heat lamp for approximately 10 minutes and let cool. Submit the samples for alpha spectroscopy counting.

12.0 QUALITY CONTROL SAMPLES AND REQUIREMENTS

NOTE: Client contractual QC requirements override the requirements in this section.

12.1 Method Verification Requirements

Refer to GL-RAD-D-002 for instructions concerning the validation of analytical methods.

12.2 Method Specific Quality Requirements

12.2.1 A method blank should accompany each batch of 20 or less samples. The reported value of the blank should be less than or equal to the CRDL (contract required detection limit).

12.2.2 The tracer added to all samples is used to calculate the method recovery. The method recovery of all samples should be between 15-125% when compared to the reference standard.

12.2.3 A Duplicate (DUP) sample should be run with every batch. The Relative Percent Difference (RPD) between the actual sample and the DUP should be less than or equal to 20% if both the sample and the DUP results are greater than 5 times MDC, or up to 100% if they are both less than 5 times the MDC. If both results are less than MDC, then limits are not applicable.

12.2.4 A laboratory control sample (LCS) should be run with each batch of 20 or less samples. The recovery of the LCS should fall between 75-125%.

12.3 Actions Required if the Quality Control Requirements Are Not Met

If any of the QC criteria from 12.2.1 through 12.2.4 cannot be satisfied, the analyst should inform the Group Leader and initiate a Data Exception Report as outlined in GL-QS-E-004.

13.0 INSTRUMENT CALIBRATION, STANDARDIZATION, AND PERFORMANCE

For direction on calibration and instrument performance refer to GL-RAD-I-009.

14.0 PROCEDURE FOR ANALYSIS AND INSTRUMENTATION OPERATION

For analysis and instrument operation refer to GL-RAD-I-009.

15.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

For maintenance of system refer to GL-RAD-I-010.

16.0 DATA RECORDING, CALCULATION, AND REDUCTION METHODS

Data recording, calculation and reduction take place in accordance with GL-RAD-D-003 and GL-RAD-D-006.

17.0 DATA REVIEW, APPROVAL, AND TRANSMITTAL

Data are reviewed and packaged in accordance with GL-RAD-D-003 for Data Review, Validation and Data Package Assembly.

18.0 RECORDS MANAGEMENT

Data generated as a result of this procedure are maintained as quality documents in accordance with GL-QS-E-008 for Quality Records Management and Disposition.

19.0 LABORATORY WASTE HANDLING AND WASTE DISPOSAL

Laboratory waste is disposed in accordance with the Laboratory Waste Management Plan, GL-LB-G-001.

20.0 REFERENCES

20.1 Pb-210 Dating of Sediment Samples via Po-210 Alpha Spectrometry, Dr. Bill Burnett: Florida State University, 1992.

20.2 HASL-300 28th Edition. Polonium in Water and Urine. PO-01-RC.

21.0 HISTORY

Revision 19: Removed the reference to the use of Queue Sheets.

Revision 18: Updated for minor edits from Annual Review.

Revision 17: Minor edits to reflect current processes.

Revision 16: Replaced nickel disk with metal disk (Ni, Ag, or Cu plated), updated method specific quality requirements (section 12.2.3), and referenced GL-RAD-D-003 to data recording, calculation, and reduction methods.

Revision 15: Made technical changes to section 5.8, 9.1.7, 11.3.2, and Appendix 1. Removed MS requirement from section 12.2. Updated section 16.0.

APPENDIX 1

Polonium

_____ Add 2 mL 1.25 M calcium carrier, 1 mL phosphoric acid, and 20 mL 30% H_2O_2

_____ Cover and reflux 90 minutes

_____ Add concentrated NH_4OH dropwise until pH is between 8 and 9

_____ Cover and reflux 90 minutes (allow to settle)

_____ Decant supernate, collect precipitate in c-tube and centrifuge

_____ Rinse beaker with approximately 25 mL 1 M HCl, cover and reflux

_____ Transfer to c-tube, Add 2 g ascorbic acid powder

_____ Transfer to plastic cup

_____ Rinse c-tube with 25 mL 1 M HCl

_____ Prepare metal disk with black electrical tape (if necessary)

_____ Suspend disk in concentrated HNO_3, concentrated HCl, and DI water for 5 seconds each.

_____ Tape stir bar to disk and place in plastic cup

_____ Spin minimum 4 hours, maximum 16 hours

_____ Rinse with 6 M HCl and DI water

VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE FOR THE DETERMINATION OF LEAD-210

IN LIQUID AND SOLID MATRICES

(GL-RAD-A-018 REVISION 15)

APPLICABLE TO METHODS
DOE RP280 (MODIFIED)

PROPRIETARY INFORMATION

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APPENDIX 1....12

1.0 STANDARD OPERATING PROCEDURE FOR THE DETERMINATION OF LEAD-210 IN LIQUID AND SOLID MATRICES

2.0 METHOD OBJECTIVE, PURPOSE AND SUMMARY

2.1 This standard operating procedure provides the necessary instructions to conduct the analysis for lead-210 in water and soil.

2.2 Soil samples are leached in concentrated nitric acid and separated via lead resin. Liquid samples are precipitated with ammonium hydroxide and iron before being separated via lead resin. Acetic acid and potassium dichromate are then added to all samples to form a precipitate that is filtered, weighed and submitted for gas flow proportional counting.

3.0 METHOD SCOPE, APPLICABILITY AND DETECTION LIMIT

3.1 Method Detection Limit: Typical minimum detectable activity (MDA) for samples analyzed for lead-210 is 5 pCi/L for liquids and 5 pCi/g for soils.

3.2 Analyst training records are maintained as quality records. Refer to GL-QS-E-008.

4.0 METHOD VARIATIONS

Some variations in procedure may be necessary due to special matrices encountered in the lab. These variations may be used with approval from a Group Leader or Team Leader. Variations to a method will be documented with the analytical raw data.

5.0 DEFINITIONS

5.1 AlphaLIMS: GEL's Laboratory Information Management System.

5.2 Batch: Environmental samples that are prepared and/or analyzed together with the same process and personnel using the same lot(s) of reagents.

5.3 Laboratory Control Sample (LCS): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes from a source independent of the calibration standards or a material containing known and verified amounts of analytes.

5.4 Laboratory Duplicate (DUP): Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently.

5.5 Matrix Spike (MS): Prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available.

5.6 Matrix Spike Duplicate (MSD): A second replicate matrix spike is prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

5.7 Method Blank (MB): A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed

simultaneously with and under the same conditions as samples containing an analyte of interest through all steps of the analytical procedures.

5.8 National Institute of Standards and Technology (NIST): For the purpose of this method, the national scientific body responsible for the standardization and acceptability of analyte solutions.

5.9 Type I water: Deionized (DI) water. Refer to GL-LB-E-016.

6.0 INTERFERENCES

No interferences have been encountered.

7.0 SAFETY PRECAUTIONS AND WARNINGS

7.1 Lead is a cumulative poison that can cause brain damage; therefore, proper laboratory techniques are essential.

7.2 Personnel performing this analytical procedure are trained in and follow the safe laboratory practices outlined in the Safety, Health and Chemical Hygiene Plan, GL-LB-N-001.

7.3 Personnel handling radioactive materials are trained in and follow the procedures outlined in GL-RAD-S-004 for Radioactive Material Handling.

7.4 Personnel handling biological materials are trained in and follow the procedures outlined in GL-RAD-S-010 for The Handling of Biological Materials.

7.5 If there is any question regarding the safety of any laboratory practice, **stop immediately**, and consult qualified senior personnel such as a Group or Team Leader.

8.0 APPARATUS, EQUIPMENT AND INSTRUMENTATION

8.1 Apparatus and Equipment

8.1.1 Aluminum foil

8.1.2 Filtration apparatus

8.1.3 Centrifuge

8.1.4 Centrifuge tubes

8.1.5 Beakers

8.1.6 Stirring rods

8.1.7 Hot plates

8.1.8 47 mm PES (or equivalent)

8.1.9 2" stainless steel planchettes

8.1.10 Drying oven

8.1.11 Microwave oven or hotbath

8.2 Instrumentation

8.2.1 Gas flow proportional counter

9.0 REAGENTS, CHEMICALS AND STANDARDS

9.1 Acetic acid, concentrated (glacial, CH_3COOH)

9.2 6 M Acetic acid: Add 344.83 mL of concentrated acetic acid to 500 mL of DI water. Dilute to 1000 mL with DI water.

9.3 Ammonium hydroxide, concentrated (28-30% NH_4OH)

9.4 DI Water

9.5 Hydrogen Peroxide (30% H_2O_2)

9.6 Iron Carrier (10mg/L): Dissovle 62.7g of $Fe(NO_3)_3 \cdot 6H_2O$ or 72.3g of $Fe(NO_3)_3 \cdot 9H_2O$ in 800 mL DI Water and dilute to 1 L with DI Water.

9.7 Lead carrier, standardized ($Pb(NO_3)_2$ F.W.= 331.21 g), 10 mg/mL Pb: Preferably use Pb 10,000 mg/L o2si standard. Alternatively dissolve 15.985 g lead II nitrate in 900 mL of DI water then dilute to 1 L with DI water. See section 13.4 for standardization of lead carrier.

9.8 Nitric acid, concentrated (16 M HNO_3)

9.9 0.1 M Nitric acid: Carefully add 6.25 mL concentrated nitric acid (16 M) to 500 mL DI water in a graduated cylinder. Dilute to 1000 mL.

9.10 1 M Nitric acid: Carefully add 62.5 mL concentrated nitric acid (16 M) to 500 mL DI water in a graduated cylinder. Dilute to 1000 mL.

9.11 8 M Nitric acid: Carefully add 500 mL concentrated nitric acid (16 M) to 500 mL DI water.

9.12 NIST traceable Pb-210 standard

9.13 Pb resin Eichrom Technologies (Alternatively, Sr resin from Eichrom Technologies may be used).

9.14 Potassium dichromate ($K_2Cr_2O_7$): Add 80 g of potassium dichromate to 200 mL DI water. Dilute to 500 mL with DI water.

10.0 SAMPLE HANDLING AND PRESERVATION

10.1 Water samples must be collected in plastic bottles and preserved with concentrated nitric acid to pH < 2.

10.2 Before beginning an analysis, the analyst should check the sample pH by removing a minimal amount of the sample with a transfer pipette and placing it on a pH strip. DO NOT insert pH strip into sample container. If the sample is received with a pH greater than 2, the analyst should contact the Group Leader or Team Leader. If approved by the client, the analyst should adjust the pH with nitric acid to a pH < 2. If the sample pH is adjusted, let the sample sit in the original container for a minimum of 24 hours before analysis. This acidification should be documented on a batch history sheet and attached to the batch paper

work.

10.3 If the sample has exceeded the hold time, the analyst should contact the Group Leader before continuing with the batch.

10.4 Soil samples require no preservation and may be shipped in any suitable container.

11.0 SAMPLE PREPARATION

11.1 Preparation of Solid Samples

11.1.1 If not already done, dry and homogenize the sample by performing GL-RAD-A-021.

11.1.2 Measure an appropriate aliquot of soil in a glass container. If required, the DUP, MS and MSD should be the same aliquot as the appropriate sample referenced. Record all aliquots. The Blank and LCS aliquot should be recorded to be the same aliquot as the largest sample in the batch.

11.1.3 Add a certified dpm (typically 0.1 mL) of Pb-210 working standard to the MS, MSD, LCS and LCSD as applicable.

11.1.4 Add 1 mL lead and 1 mL iron carrier to each beaker.

NOTE: The addition of tracers and spikes should be witnessed by either another analyst qualified on this procedure, a Team Leader or a Group Leader. After adding the tracers and spikes, the witness must initial and record the date of witnessing.

11.1.5 It is recommended that the samples be ashed in a muffle furnace as specified in GL-RAD-A-021B.

11.1.6 Add 10-25 mL of concentrated nitric acid to each beaker. Place a watch glass on each beaker and heat the beakers on low heat for approximately 1 to 2 hours.

11.1.7 Stir or swirl the solution frequently during the leaching process.

11.1.8 Remove the beakers from the hot plate and allow to cool.

11.1.9 Transfer the leachate and the residue from the beaker with 8M HNO_3 into a centrifuge tube and centrifuge.

11.1.10 Decant the supernatant into a clean labeled beaker.

11.1.11 Wash the residue in the centrifuge tube with 10-25 mL of DI water. (Vortex may be used to wash the residue.)

11.1.12 Centrifuge and decant the supernatant into the beaker from step 11.1.10.

11.1.13 Evaporate the solution to dryness on medium to low heat. Add hydrogen Peroxide while cooking down if necessary.

11.1.14 Dissolve the residue with 10 to 15 mL of 8 M nitric acid.

11.1.15 Proceed to step 11.3.1

11.2 Preparation of Liquid Samples

11.2.1 Measure an appropriate aliquot of sample into a glass beaker. If required, the DUP, MS and MSD should be the same aliquot as the appropriate sample referenced. Record all aliquots. The Blank and LCS aliquot should be recorded to be the same aliquot as the largest sample in the batch.

11.2.2 Add a certified dpm (typically 0.1 mL) of Pb-210 working standard to the MS, MSD, LCS and LCSD as applicable.

11.2.3 Add 1 mL of lead carrier and 1 mL iron carrier to each beaker.

NOTE: The addition of tracers and spikes should be witnessed by either another analyst qualified on this procedure, a Team Leader or a Group Leader. After adding the tracers and spikes, the witness must initial and record the date of witnessing.

11.2.4 Carefully add 28-30% ammonium hydroxide until turbidity persists (this is orange to brown in color), or pH > 9. Then add approximately 2 mL in excess. Cover with a watch glass and heat to boiling for approximately 10 minutes or until precipitate breaks into fine particles.

11.2.5 Remove from heat and allow to settle and cool.

11.2.6 Decant the supernatant and transfer the precipitate into a 50 mL plastic centrifuge tube.

11.2.7 Centrifuge and discard the supernatant.

11.2.8 Wash the precipitate with 10 mL water. Centrifuge and discard the supernatant. (A vortex may be used to wash the residue.)

11.2.9 Repeat step 11.2.9.

11.2.10 Add 10 to 15 mL of 8 M nitric acid to dissolve the precipitate.

11.3 Preparation of Lead Columns and Column Work for Soil and Water Samples

11.3.1 Condition an Eichrom Pb resin column with 5 mL of 8 M nitric acid.

11.3.2 Load the dissolved precipitate (from step 11.1.14 or 11.2.11) onto the column. Discard eluent.

11.3.3 Add 10 mL 1 M nitric acid to the column. Record the time and date of this addition. This will be used to calculate the ingrowth of bismuth-210. Discard the eluent.

NOTE: This will remove bismuth and iron, if present.

11.3.4 Add 10 mL 0.1 M nitric acid to the column and discard the eluent.

NOTE: This will remove any polonium-210, if present in the sample.

11.3.5 Place clean and labeled glass centrifuge tubes under the column. Add 20 mL of water to the column to elute lead.

NOTE: Samples should be precipitated as quickly as possible after eluting from resin as Bi may not co-precipitate with Pb.

11.3.6 Add 1 mL 6 M acetic acid and 1 mL saturated potassium dichromate to the glass centrifuge tube. An orange precipitate should be formed.

11.3.7 Preweigh a 47 mm PES filter (or equivalent) and record weights.

11.3.8 Microwave the centrifuge tubes from step 11.3.6 for approximately 30 seconds or heat in hot bath for approximately 2 to 5 minutes.

11.3.9 Filter the precipitate immediately.

NOTE: Rinse the centrifuge tube with enough DI water to remove the precipitate from the centrifuge tube. Then add 20-50 mL of DI water to filtration cup.

11.3.10 Dry the filter in an oven at approximately 110° F.

11.3.11 Weigh the dry filters to obtain the precipitate weight and record the results.

11.3.12 Place the filter in a 2” stainless steel planchette and cover the filter with aluminum foil. The aluminum foil must be the same brand and thickness that was used for the calibration.

11.3.13 Submit the samples and completed paperwork into the count room for gas flow proportional analysis.

12.0 QUALITY CONTROL SAMPLES AND REQUIREMENTS

NOTE: Client contractual QC requirements override the requirements in this section.

12.1 Method Verification Requirements

Refer to GL-RAD-D-002 for instructions concerning the validation of analytical methods.

12.2 Method Specific Quality Requirements

12.2.1 A method blank (MB) should accompany each batch of 20 or less samples. The reported value of the blank should be less than or equal to the CRDL (contract required detection limit).

12.2.2 The tracer added to all samples is used to calculate the method recovery. The method recovery of all samples should be between 15-125% when compared to the reference standard.

12.2.3 A matrix spike (MS) should be run with each batch of 20 or less samples. The recovery of the MS should be between 75-125%.

12.2.4 A duplicate (DUP) sample should be run with each batch of 20 or less samples. The relative percent difference (RPD) between the actual sample and the DUP should be less than or equal to 20% if both the sample and DUP results are greater than 5 times the LLD or 100% if either result is less than 5 times the LLD.

12.2.5 A laboratory control sample (LCS) should be run with each batch of 20 or less samples. The recovery of the LCS should fall between 75-125%.

12.3 Actions Required if the Quality Control Requirements Are Not Met

If any of the QC criteria from 12.2.1 through 12.2.5 cannot be satisfied, the analyst should inform the Group Leader and initiate a Data Exception Report as outlined in GL-QS-E-004.

13.0 INSTRUMENT CALIBRATION, STANDARDIZATION AND PERFORMANCE

13.1 Refer to the appropriate counting procedures, GL-RAD-I-006, GL-RAD-I-015, and GL-RAD-I-016 for instrument calibration and performance.

NOTE: Source preparation is only performed under guidance of a Group Leader or a Team Leader.

13.2 Instrument Calibration Preparation for Gas Flow Proportional Counters for Pb-210.

13.2.1 Add a known dpm of Pb-210 NIST traceable standard, with Bi-210 at equilibrium, to 8 separate centrifuge tubes. Record the information.

13.2.2 Add an increasing amount of lead carrier to each of the tubes.

13.2.3 Add 10 mL DI water to each of the tubes.

13.2.4 Add 4 mL 6 M acetic acid and 4 mL saturated potassium dichromate.

13.2.5 Preweigh a 47 mm PES filter and record weights.

13.2.6 Microwave the centrifuge tubes for approximately 30 seconds, or place in a hotbath for approximately 2-5 minutes.

13.2.7 Filter the precipitate immediately.

13.2.8 Dry the filters in an oven at approximately 110° F.

13.2.9 Weigh the filters to obtain the precipitate weight and record the information.

13.2.10 Place the filter in a 2” stainless steel planchette and cover with aluminum foil.

13.2.11 Submit sources and paperwork to count room for gas flow proportional counting.

13.3 Verification Source Preparation

Verification sources are prepared as in section 13.2 using an alternate source than used in the calibration.

13.4 Standardization of Lead Carrier

NOTE: Standardize any new lead carrier.

13.4.1 Pipet four aliquots (usually 1.0 mL) of lead carrier into tared centrifuge tubes using a pipet. Record weight of each carrier aliquot. Dilute to 20 mL with DI water.

13.4.2 Add 1 mL of 6 M acetic acid and 1 mL saturated potassium dichromate to the glass centrifuge tube. An orange precipitate should be formed.

13.4.3 Microwave the centrifuge tubes for approximately 30 seconds, or place in a hotbath for approximately 2-5 minutes.

13.4.4 Filter each sample through a labeled, tares and preweighed PES filter.

13.4.5 Dry in oven for approximately 30 minutes. Cool and weigh.

13.4.6 Enter data into the appropriate spreadsheet and calculate the standard weight of lead carrier in mg/g of solution. Acceptable precision is a standard deviation of less than 1% of the mean value.

13.4.7 Give the calculation spreadsheet to the Team Leader or Group Leader for review and label the carrier with the standardized results.

14.0 ANALYSIS AND INSTRUMENT OPERATION

Refer to the appropriate counting procedure in GL-RAD-I-006, GL-RAD-I-015, and GL-RAD-I-016 instrument operation.

15.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

Refer to the appropriate counting procedure in GL-RAD-I-006 , GL-RAD-I-015, and GL-RAD-I-016 for instrument maintenance.

16.0 DATA RECORDING, CALCULATIONS, AND REDUCTION METHODS

Data recording, calculation and reduction take place in accordance with GL-RAD-D-003.

17.0 DATA REVIEW, APPROVAL AND TRANSMITTAL

Refer to GL-RAD-D-003 for instructions concerning the data review process, approval, and transmittal.

18.0 RECORDS MANAGEMENT

Records generated as a result of this procedure are maintained as quality documents in accordance with GL-QS-E-008 for Quality Records Management and Disposition.

19.0 LABORATORY WASTE HANDLING AND DISPOSAL

Radioactive samples and material shall be handled and disposed of as outlined in the Laboratory Waste Management Plan, GL-LB-G-001.

20.0 REFERENCES

20.1 Radiochemistry Methods in DOE Methods for Evaluating Environmental and Waste Management Samples, RP280, Lead-210 in Water Using Extraction Chromatography, 1994.

20.2 E. Philip Horwitz, Renato Chiarizia, and Mark L. Dietz. " A Novel Strontium-Selective Extraction Chromatograhic Resin."

20.3 Special thanks to Dr. Bill Burnett and his associates for their development of this method at Florida State University.

21.0 HISTORY

Revision 15: Replaced Tuffryn Filters with PES filters. Updated minor

Revision 14: Removed reference to Queue sheets.

Revision 13: Added note about speed of precipitation, revised ASTM DI water type.

Revision 12: Updated section 13.4.6 to read acceptable precision is a standard deviation of less than 1% of the mean value.

Revision 11:Updated section 9.2 on lead carrier.

APPENDIX 1:

Use a 2.5 cm^3 LEAD column

COLUMN WORK

_____ 5 mL 8 M HNO_3 (conditioning)

_____ Load solution: 10 to 15 mL 8 M HNO_3

_____ 10 mL 1 M HNO_3 (Rinse): Record as bismuth separation date and time.

_____ 10 mL 0.1 M HNO_3 (Rinse)

_____ ELUTE: 20 mL DI water (Catch in glass centrifuge tube)

PRECIPITATE AND FILTER

_____ Add 1 ml 6 M acetic acid and 1 mL saturated $K_2Cr_2O_7$

_____ Preweigh a 47 mm PES filter (or equivalent)

_____ Microwave centrifuge tubes for approximately 30 seconds or place in hot bath for Approximately 5 minutes

_____ Filter immediately

_____ Dry in oven at approximately 110˚ F

_____ Reweigh and record results

_____ Place filter in 2” planchette and cover with aluminum foil

_____ Submit to count room

Reactive Sulfide and Reactive Cyanide

Reference Method: This standard operating procedure (SOP) is a performance-based method. This SOP describes the procedure as developed by Alpha Analytical.

1. Scope and Application

Matrices: This method is applicable to water, wastewater, seawater, soil and solid samples with the condition that the samples combined with acids do not form explosive mixtures.

Definitions: See Alpha Analytical Quality Manual

The regulation in 40CFR 261.23 defines reactive wastes to include wastes that have any of the following properties:

(1) readily undergo violent chemical change;

(2) react violently or form potentially explosive mixtures with water;

(3) generate toxic fumes when mixed with water or, in the case of cyanide- or sulfide-bearing wastes, when exposed to mild acidic or basic conditions;

(4) explode when subjected to a strong initiating force;

(5) explode at normal temperatures and pressures; or

(6) fit within the Department of Transportation's forbidden explosives, Class A explosives, or Class B explosives classifications.

This definition is intended to identify wastes that, because of their extreme instability and tendency to react violently or explode, pose a problem at all stages of the waste management process. The definition is to a large extent a paraphrase of the narrative definition employed by the National Fire Protection Association. The Agency chose to rely almost entirely on a descriptive, prose definition of reactivity because most of the available tests for measuring the variegated class of effects embraced by the reactivity definition suffer from a number of deficiencies.

This procedure releases only the hydrogen sulfide evolved at the test conditions. It is not intended to measure forms of sulfide other than those that are evolvable under the test conditions.

This test measures only the hydrocyanic acid evolved at the test conditions. It is not intended to measure forms of cyanide other than those that are evolvable under the test conditions.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Laboratory Services Manager, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

An aliquot of acid is added to a fixed volume or weight of sample in a closed system. The generated gas is swept into a scrubber. The analyte is quantitated.

2.1 Method Modifications from Reference

None.

3. Reporting Limits

The reported reactive sulfide detection limit is 1.0 mg/L (aqueous samples) and 10mg/kg (solid samples).

The reported reactive cyanide detection limit is 1.0 mg/L (aqueous samples) and 10mg/kg (solid samples).

4. Interferences

4.1 Reactive Sulfide Interferences

4.1.1 Strong reducing agents interfere in the methylene blue method by preventing formation of the blue color.

4.1.2 Thiosulfate at concentrations about 10mg/L may retard color formation or completely prevent it.

4.1.3 High Concentration of sulfide itself may inhibit blue color development. To avoid the possibility of reporting false negative results, test a 1000x dilution of the scrubber solution to see if blue color develops. (Refer to Section 10.3.1).

4.1.4 Ferrocyanide is a positive interference since it produces a blue color; distillation process will minimize interference.

4.2 Reactive Cyanide Interferences

Reactive Cyanide interferences are undetermined.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. Therefore, analysts should take every precaution by utilizing latex/nitrile gloves, safety glasses and a lab coat to minimize or eliminate exposure. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples are collected with minimum aeration in either a glass or plastic bottle or jar. Fill the container completely and cover, ensuring no head space is present.

6.2 Sample Preservation

Samples are stored at 4 ± 2° C.

6.3 Sample Shipping
No special shipping requirements.

6.4 Sample Handling
Aqueous samples are analyzed with 7 days of collection. Soil samples are analyzed within 14 days of collection.

7. Equipment and Supplies

7.1 Spectrophotometer: Genesys 10vis (or equivalent); for use at a wavelength of 664nm and 578nm with cells providing light paths of 1cm.

7.2 Centrifuge Tubes: 50mL capacity

7.3 Pipets: Class A, glass, volumetric, various volumes

7.4 Volumetric Flasks: Class A, various volumes

7.5 Non-rubber Flexible Tubing: For connection from pump supply to apparatus.

7.6 Analytical Balance: Capable of weighing to 0.001g.

7.7 Midi Distillation System: Lab-Crest (or equivalent); 10-position distillation unit equipped with a full set of glassware including 10 each of the following:

7.7.1 Reaction Flask: Flat-bottom flask with sufficient volume (over 100mL) to contain violent reactions; has permanent 50mL indicator.

7.7.2 Absorption Flask: Identical to Reaction Flask (Section 7.7.1).

7.7.3 Reflux Impinger: Fits the reaction flask; has air and reagent intake, full-length impinger, and hose barb.

7.7.4 Cold Finger: Fits the Reflux Impinger; has two hose barbs for water circulation, which is not utilized for this test. It can therefore be substituted with a glass or plastic stopper of the same size.

7.7.5 Absorption Impinger: Full-length impinger with 13mm, medium porosity glass filter, and two hose barbs.

8. Reagents and Standards

8.1 Reagent water: Deionized water (DI) produced by Alpha's water treatment system.

8.2 Sulfide Calibration Stock Standard: Weigh 1.1045 gram of $Na_2S \cdot 9H_2O$ in a 140mL amber bottle and fill to top with DI water. Mix by swirling gently to prevent oxygen entrapment. Store at room temperature. Solution expires one month from the date of preparation. Final concentration is 1097 mg/L

8.3 Sulfide 1 Reagent: Available commercially from HACH Company. Store at 4 ± 2°C. Reagent expires on manufacturer's specified expiration date.

8.4 Sulfide 2 Reagent: Available commercially from HACH Company. Store at 4 ± 2°C. Reagent expires on manufacturer's specified expiration date.

8.5 Sulfide Calibration Standards: These standards are used to generate the calibration curve in Section 9.1.1. Prepare a series of sulfide solutions by diluting the Sulfide Calibration Stock Standard (Section 8.2) with 0.1N NaOH (Section 8.6.2).

Into seven 100mL volumetric flasks, pipet 1mL, 2mL, 5mL, 10mL, 15mL, 30mL and 0mL of the sulfide stock standard (Section 8.2) Bring each flask to volume using 0.1N NaOH solution (Section 8.6.2).

The concentration of the calibration standards is determined by the results of the standardization of the stock sulfide standard, as described in Section 8.2.1. The lowest calibration standard must be 0.1mg/L or less. If necessary, add an additional point to the curve by pipetting 0.5mL of Stock Sulfide Standard (Section 8.2) into an eighth 100mL volumetric flask.

Note: This calibration curve is identical to calibration curve, used for Total Sulfide.

8.6 Sodium Hydroxide, NaOH: In pellet form.

8.6.1 Sodium Hydroxide Solution, NaOH, 1N: Dissolve 40g NaOH (Section 8.11) in 1L of DI water. Store at room temperature. Expires three months from the date of preparation.

8.6.2 Sodium Hydroxide Solution, NaOH, 0.1N: In a 100mL volumetric flask, pipet 10mL of 1N NaOH (Section 8.11.1). Bring to volume with DI water. Store at room temperature. Expires three months from the date of preparation.

8.7 Sulfide LCS Stock Standard: Using a different source than was used for the Calibration Stock Standard. (8.2)

8.8 Sulfide LCS Standard: Pipet 0.5mL of LCS Sulfide Stock Standard (Section 8.7) into a 25mL volumetric flask. Bring to volume with DI water. True value: 22 mg/l.

8.8.1 Sulfide ICV / CCV Standard: Pipet 1.0 mL of LCS Sulfide Standard (Section 8.7) into a 50mL volumetric flask, add 5 ml of 1N NAOH. Bring to volume with DI water. Acceptance criteria +/- 25%; if failed outside of acceptance criteria, data is invalid.

8.9 Cyanide LCS/ICV/CCV Stock Standard, 1000ppm: Commercially available with a certificate of analysis. This must be from a different source than the Calibration Standard Stock. Store at 4 ± 2 °C. Expires on manufacturer's specified expiration date.

8.9.1 Cyanide LCS/ICV/CCV Working Standard, 40ppm: To a 25mL volumetric flask, add 1mL of 1000ppm cyanide LCS Stock Standard (Section 8.9). Bring to volume with DI water. Prepare fresh on each day of use.

8.9.1.1 Cyanide ICV/CCV 0.8ppm Standard: Into a 25mL volumetric flask, pipet 0.5mL of the 40ppm cyanide working calibration standard (Section 8.9.1). Bring to volume using 0.1N NaOH (Section 8.6.2) (5 ml of 1N NaOH can be used and volume brought up to 50 ml with DI. This standard is prepared fresh at the time of use.

8.10 Cyanide 1000ppm Stock Calibration Standard Stock Standard: Commercially purchased with a certificate of analysis. The ICV stock must be a different lot than the calibration standards.

8.10.1 Cyanide 10ppm Working Calibration Standard: Into a 100mL volumetric flask, pipet 1mL of 1000ppm cyanide stock calibration standard (Section 8.10) and bring to volume with 0.1N NaOH (Section 8.6.2). This standard expires after one week. Store in a brown bottle at room temperature, out of direct light.

8.10.1.1 0.0ppm Cyanide Calibration Standard: To a 100mL volumetric flask, add 0.0mL of Cyanide 10ppm Working Calibration Standard (Section 8.10.1). Bring to volume with 0.1N NaOH (Section 8.6.2). This standard is prepared fresh at the time of calibration.

8.10.1.2 0.05ppm Cyanide Calibration Standard: To a 100mL volumetric flask, add 0.5mL of Cyanide 10ppm Working Calibration Standard (Section 8.10.1). Bring to volume with 0.1N NaOH (Section 8.6.2). This standard is prepared fresh at the time of calibration.

8.10.1.3 0.1ppm Cyanide Calibration Standard: To a 100mL volumetric flask, add 1.0mL of Cyanide 10ppm Working Calibration Standard (Section 8.10.1). Bring to volume with 0.1N NaOH (Section 8.6.2). This standard is prepared fresh at the time of calibration.

8.10.1.4 0.2ppm Cyanide Calibration Standard: To a 100mL volumetric flask, add 2.0mL of Cyanide 10ppm Working Calibration Standard (Section 8.10.1). Bring to volume with 0.1N NaOH (Section 8.6.2). This standard is prepared fresh at the time of calibration.

8.10.1.5 0.4ppm Cyanide Calibration Standard: To a 100mL volumetric flask, add 4.0mL of Cyanide 10ppm Working Calibration Standard (Section 8.10.1). Bring to volume with 0.1N NaOH (Section 8.6.2). This standard is prepared fresh at the time of calibration.

8.10.1.6 0.5ppm Cyanide Calibration Standard: To a 100mL volumetric flask, add 5.0mL of Cyanide 10ppm Working Calibration Standard (Section 8.10.1). Bring to volume with 0.1N NaOH (Section 8.6.2). This standard is prepared fresh at the time of calibration.

8.11 Chloramine-T Solution: Dissolve 1.0g of Chloramine-T in 100mL DI water. Prepare fresh on each day of use.

8.12 Sodium dihydrogen phosphate monohydrate, $NaH_2PO_4 \cdot H_2O$

8.12.1 Phosphate Buffer: Dissolve 138g sodium dihydrogen phosphate monohydrate (Section 8.12) in DI water and dilute to 1L. Store at room temperature. Expires one month from the date of preparation.

8.13 Barbituric acid: Store at room temperature. No expiration date.

8.14 Pyridine: Store in Flammables cabinet. Expires on manufacturer's specified expiration date.

8.15 Concentrated Hydrochloric Acid, HCl: Store in Acids cabinet. No expiration date.

8.16 6M Hydrochloric Acid (HCl): Dilute 500mL of concentrated HCl (Section 8.20) into 500mL DI water. Store at room temperature. No expiration date.

8.17 Pyridine-Barbituric Acid Reagent: Place 60g barbituric acid (Section 8.13) in a 1000mL volumetric flask and add just enough DI water to wash sides of flask and wet barbituric acid. Add 300mL pyridine (Section 8.14) and mix. Add 60mL conc HCl (Section 8.15), mix and cool to room temperature. Dilute to volume and mix until barbituric acid is dissolved. The solution is stable for one month if stored in an amber bottle at 4 ± 2 °C. Discard if precipitate develops.

8.18 Concentrated Sulfuric Acid, H_2SO_4: Store in Acids cabinet. Expires on manufacturer's specified expiration date.

8.19 Reactivity Stock Solution: Add 2.8mL of conc H_2SO_4 (Section 8.18) to a 1L volumetric flask and bring to volume with DI. Prepare fresh on each day of use.

8.19.1 Reactivity Working Solution, 0.025M: Add 100mL of reactivity stock solution (Section 8.19) to a 1L volumetric flask and bring to volume DI.

8.20 Ottawa sand

8.21 Boiling chips

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

Prepare and analyze one Method Blank per batch of 20 samples or less. Blank results must be less than the RL. If this criterion is not met, a batch narrative is included with the Client Report and/or the sample batch is reanalyzed. Refer to Section 12 for corrective actions.

For soils: weigh 1.0 g of Ottawa sand, for water- use 10 ml if DI and analyze with batch.

9.2 Laboratory Control Sample (LCS)

9.2.1 Reactive Sulfide

Run one LCS using the LCS Working Solution per batch of 20 samples or less. .

For waters: to 10 ml of DI add 2.0 ml of LCS Standard (8.8); final concentration is 4.4 mg/l

For soils: to 1.0 g of Ottawa sand add 2.0 ml of LCS Standard (8.8); final concentration 44 mg/kg

The LCS must be recovered within 60 – 125%, otherwise the entire sample batch is reanalyzed. Refer to Section 12 for corrective actions.

9.2.2 Reactive Cyanide

Run one LCS using the LCS Working Solution per batch of 20 samples or less.

For waters: to 10 ml of DI add 1.0 ml of LCS Standard (8.9.1); final concentration is 4.0 mg/l

For soils: to 1.0 g of Ottawa sand add 1.0 ml of LCS Standard (8.9.1); final concentration 40 mg/kg

The LCS must be recovered within 30 – 125%, otherwise the entire sample batch is reanalyzed. Refer to Section 12 for corrective actions.

9.3 Initial Calibration Verification (ICV)

9.3.1 Reactive Sulfide

The ICV is analyzed at the beginning of each sample batch. The ICV must be recovered within 75 – 125%, otherwise the entire sample batch is reanalyzed. Refer to Section 12 for corrective actions.

9.3.2 Reactive Cyanide

An ICV is analyzed at the beginning of each sample batch. The ICV must be recovered within 75 – 125%, otherwise the entire sample batch is reanalyzed. Refer to Section 12 for corrective actions.

9.4 Continuing Calibration Verification (CCV)

9.4.1 Reactive Sulfide

A CCV is read on the spectrophotometer after every 10 samples and at the end of each sample batch. The CCV must be recovered within 75 – 125%, otherwise the entire sample batch is reanalyzed. Refer to Section 12 for corrective actions.

9.4.2 Reactive Cyanide

A CCV is read on the spectrophotometer after every 10 samples and at the end of each sample batch. The CCV must be recovered within 75 – 125%, otherwise the entire sample batch is reanalyzed. Refer to Section 12 for corrective actions.

9.5 Matrix Spike

Not applicable.

9.6 Laboratory Duplicate

Prepare and analyze one sample in duplicate per batch of 20 samples or less. The %RPD for liquid samples must be $\leq$ 25%. For solid/soil samples, the %RPD must be $\leq$ 40%. If %RPD is not within the specified range, the sample and its duplicate must be narrated and/or reanalyzed. Refer to Section 12 for corrective actions.

9.7 Method-specific Quality Control Samples

Not applicable.

9.8 Method Sequence

- Generate calibration curves.
- Prepare samples and QC samples.
- Analyze samples and QC samples.
- Calculate results.

10. Procedure

10.1 Equipment Set-up

10.1.1 Calibration Curve Generation

Calibration curves are generated yearly or when ICV/CCV are not passing acceptance criteria.

10.1.1.1 Reactive Sulfide

Prepare a calibration curve using the standards in Section 8.5. Analyze the standards as described in Section 10.3.1. Plot the concentration vs. absorbance. Determine the slope and the y-intercept. The correlation coefficient must be $\geq$ 0.995.

10.1.1.2 Reactive Cyanide

Prepare a calibration curve using the standards in Section 8.10. Analyze the standards as described in Section 10.3.2. Plot the concentration vs. absorbance. Determine the slope and the y-intercept. The correlation coefficient must be $\geq$ 0.995.

10.1.2 Sample Preparation

10.1.2.1 Add 10mL of 1N NaOH solution to the absorption flask, and bring to a volume of 50mL with DI.

10.1.2.2 Weigh 1g of solid sample or 10mL of liquid sample into a 50mL centrifuge tube and record the weight/volume in the laboratory notebook. Label the tube and secure the cap. For solid Blank and LCS, use PTFE boiling stones or Ottawa sand. Add the contents of the tube to the reaction vessel. Record the tube number and corresponding sample number in the laboratory notebook. Assemble the Midi Distillation Unit.

10.1.2.2.1 Ensure the pump is plugged in and connected to the Midi unit via a Teflon hose. After adding the sample to the reaction vessel (large tube in the back of the unit), place the absorption flask in the front of the unit ; flask should be filled with NaOH. Place the reflux impinger in the reaction flask and place the cold finger (or stopper) into the impinger. Make sure that all of the glass connections are tight. Place the absorption impinger into the absorption flask and make sure that the glass seal is tight. Connect the absorption impinger to the reaction impinger by connecting the front hose of the absorption impinger to the Midi Distillation unit (where the air comes out) and the rear hose to the reaction impinger. Make sure that all glass connections are tight and that all of the hoses are snug.

10.1.2.3 Turn on the pump and adjust the air flow rate of air so that the gas scrubbers produce small bubbles (approx. 60mL/minute).

10.1.2.4 Add the appropriate LCS to their respective reaction vessels. Then add the reactivity solution (Section 8.19.1) to each reaction vessel so that the final volume is about 50mL.

10.1.2.5 Make sure all of the glassware is connected and all of the tubes are connected. All of the reflux impingers should now be producing air bubbles in the reaction (rear) vessels. For any are not, the glassware is not air tight. Run the pump for 30 minutes, and then shut the pump off. Record in the laboratory notebook the time started and the time ended.

10.1.2.6 Disconect the apparatus and bring the volume of the absorption tube to 100mL in DI with a volumetric flask. The normality of this hydroxide solution will be 0.1N.

10.1.2.7 Pour off 25mL into a 50mL centrifuge tube for Sulfide analysis. Pour off 20mL into a 50mL centrifuge tube for Cyanide analysis.

10.2 Initial Calibration

Refer to Sections 10.1.1 and 9.3.

10.3 Equipment Operation and Sample Processing

10.3.1 Reactive Sulfide Analysis

Take a 25mL aliquot of the calibration standards (Section 8.5) or of the volumized scrubber solution for each sample and color as follows:

Add 1.0mL Sulfide 1 reagent (Section 8.3) and swirl to mix. Add 1mL of Sulfide 2 reagent (Section 8.4). Let the reaction continue for 5 minutes. Excessive time between the addition of the "1" and "2" Sulfide coloring reagents causes low results by loss of H_2S as a gas before it has had time to react. The presence of sulfide will be indicated by the appearance of blue color. Wait 5 minutes and read the absorbance on the spectrophotometer (below).

Absorbance Determination: Set the spectrophotometer to a wavelength of 664nm. Zero the instrument with a portion of the blank sample. Read the absorbances of the samples and QC samples and record this information in the laboratory notebook.

If the absorbance of a sample is greater than the highest standard in the calibration curve, the sample must be diluted with 0.1N NaOH and reanalyzed as in Section 10.3.1 above. Record in the laboratory notebook any dilutions that are prepared.

NOTE: Very high sulfide concentrations interfere with blue color formation. For a sample in which high sulfide is suspected, but no blue color develops, make a 1000x dilution of the scrubber solution with 0.1N Color with Reagent 1 (Section 8.3) and Reagent 2 (Section 8.4). Experiment with various dilutions until the absorbance falls within the calibration curve.

10.3.2 Reactive Cyanide Analysis

Take a 20mL aliquot of the calibration standards (Section 8.10.1) or of the volumized scrubber solution and color as follows:

Add 5mL of phosphate buffer solution (Section 8.12.1), 2mL of chloramine-T solution (Section 8.11), 5mL of pyridine (Section 8.14). Bring final volume to 50mL with DI. The presence of a pink color indicates cyanide.

Absorbance Determination: Set the spectrophotometer to a wavelength of 578nm. Zero the instrument with a portion of the blank sample. Read the absorbances of the samples and QC samples and record this information in the laboratory notebook.

If the absorbance is greater than the highest standard in the calibration curve, the sample must be diluted with 0.1N NaOH and reanalyzed as in Section 10.3.2 above. Record in the laboratory notebook any dilutions that are prepared.

10.4 Continuing Calibration

Refer to Section 9.4.

10.5 Preventive Maintenance

The spectrophotometers are calibrated on a semi-annual basis by an instrument service company. Certificates are kept on file.

11. Data Evaluation, Calculations and Reporting

11.1 Reactive Sulfide

Calculate reactive sulfide concentration using the calibration curve generated in Section 10.1.1.1:

$$\text{Reactive Sulfide (mg/L)} = \frac{\text{Absorbance} - \text{y intercept}}{\text{Slope}} \times \text{Dilution factor}$$

$$\text{Reactive Sulfide (mg/kg)} = \frac{\text{Absorbance} - \text{y intercept}}{\text{Slope}} \times \frac{\text{Dilution factor}}{\text{Weight (g)}}$$

11.2 Reactive Cyanide

Calculate reactive cyanide concentration using the calibration curve generated in Section 10.1.1.2:

$$\text{Reactive CN (mg/L)} = \frac{\text{Absorbance} - \text{y intercept}}{\text{Slope}} \times \text{Dilution factor}$$

$$\text{Reactive CN (mg/kg)} = \frac{\text{Absorbance} - \text{y intercept}}{\text{Slope}} \times \frac{\text{Dilution factor}}{\text{Weight (g)}}$$

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedence, improper preservation and sample headspace (liquid samples) are noted on the nonconformance report form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

Review of standards, blanks and standard response for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form.

If the method blank contains a reportable amount of cyanide of Sulfide, evaluate the method blank as follows. If the samples associated with the method blank are non-detect, or if a sample has an analyte concentration that is greater than 10X the concentration of the contaminated method blank, the data may be reported and the method blank results narrated on the final report. If sample results are positive, but at a concentration less than 10X the concentration of the method blank, then those samples should be re-digested and re-analyzed.

If the LCS fails acceptance criteria, then the entire batch must be re-digested and re-analyzed. If there is insufficient sample volume to re-digest, then the Client Services Department is notified, and the batch results are narrated on the final report as estimated.

If the ICV fails acceptance criteria, a fresh aliquot is poured and re-analyzed. If the standard still fails, prepare a fresh ICV and begin analysis again. If the ICV fails again, and the spectrophotometer is working properly, prepare a new calibration curve.

If the ICB fails acceptance criteria, a fresh aliquot is poured and re-analyzed. If the blank still fails, ensure that the spectrophotometer is “blanked” correctly. If the ICB failure continues, and the spectrophotometer working properly, prepare a new calibration curve.

If the CCV fails acceptance criteria, a fresh aliquot is poured and re-analyzed. If the standard still fails, prepare and analyze a fresh CCV and re-analyze the last 10 samples up to the last passing CCV. If the CCV recovery is greater than 125%, and all associated samples are non-detect, then analysis may be continued. If the CCV recovery is greater than 125%, any associated positive samples are re-prepared and re-analyzed.

If the CCB fails acceptance criteria, a fresh aliquot is poured and re-analyzed. If the CCB still fails, ensure that the spectrophotometer is working properly. Also ensure that the cell is clean and has no smudges, and is indexed correctly. When the problem has been corrected, all samples analyzed since the last passing CCB must be re-analyzed.

If %RPD of duplicates fails acceptance criteria, the non-conformance is narrated and the information is included on the final report.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1734, 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/1732 MDL/LOD/LOQ Generation

SOP/1734, 1739 IDC/DOC Generation

SOP/1728 Waste Management and Disposal SOP

16. Attachments

None.

Semivolatile Organic Compounds by Gas Chromatography/ Mass Spectrometry (GC/MS)

Reference Method No.: **EPA 8270 D**

Reference: SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update V, February 2007.

1. Scope and Application

Matrices: This method is used to determine the concentration of semivolatile organic compounds in extracts prepared from many types of solid waste matrices, soils, and wastewater samples.

This method is used to quantitate most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and capable of being eluted, without derivatization, as sharp peaks from a gas chromatographic fused-silica capillary column coated with a slightly polar silicone.

Table 9 lists “difficult” compounds that may require special treatment when being determined by this method.

Approval of any method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of a gas chromatograph/mass spectrometer and in the interpretation of mass spectra. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability (Section 13.2).

2. Summary of Method

The samples are introduced into the GC/MS by injecting 1µL of the sample extract into a gas chromatograph (GC) with a narrow-bore fused-silica capillary column. The GC column is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS) connected to the gas chromatograph.

Analytes eluted from the capillary column are introduced into the mass spectrometer via direct connection. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact spectra of standards run on the same GC/MS system. Quantitation is accomplished by comparing the response of quantitation ion relative to an internal standard using a calibration curve.

2.1 Method Modifications from Reference

None.

3. Reporting Limits

Table 6 lists our routine reporting limits.

4. Interferences

4.1 Only high purity helium is used in the GC system to eliminate this source of possible contamination. The helium (carrier gas) is certified by the gas supplier.

4.2 Preventive instrument maintenance is performed routinely. Section 10.5 details the maintenance steps.

4.3 Glassware must be scrupulously cleaned. This procedure is detailed in the Organic Extraction Glassware Cleaning & Handling SOP/1953.

4.4 Contaminated solvents or reagents are also possible sources of contamination. All solvents used are pesticide grade or equivalent, and reagents are purchased as certified contaminant free.

4.5 Contamination by carry-over can occur whenever high-concentration and low-concentration samples are sequentially analyzed. Whenever an unusually concentrated sample is encountered (concentrations greater than 2x the highest concentration) and the next sample has reportable hits this sample should to be re-analyzed for confirmation based on analyst discretion.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound must be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

5.1 Lab coats, safety glasses, and gloves must be worn when handling samples, extracts, standards or solvents.

5.2 All solvent and extract transfers must be handled in the vented bench area in the GC/MS laboratory.

5.3 All stock standards, working standards, and vialed sample extracts must be placed into the waste bucket in the lab for future disposal by the Health and Safety Officer. The container must be labeled properly with hazard warning labels indicating the container contents.

5.4 Flammable solvent bottles must be stored in the flammables cabinet.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Aqueous samples are collected in two 1L amber glass jars with teflon-lined lids. For LVI, aqueous samples are collected in two 275mL amber glass jars with teflon-lined lids. Solid samples are collected in 250mL wide-mouth glass jars with teflon-lined lids. All containers are purchased pre-cleaned and certified from commercial vendors.

6.2 Sample Preservation

Both aqueous and solid samples are then preserved by packing in coolers with ice or ice packs, to maintain a temperature of 4 ± 2ºC. Upon receipt at the laboratory, the samples are transferred into sample storage refrigerators to maintain at a temperature of 4 ± 2ºC.

6.3 Sample Handling

Aqueous samples must be extracted within 7 days of sample collection, solid samples within 14 days of collection. Once extracted, the samples must be analyzed within 40 days of the extraction date.

7. Equipment and Supplies

7.1 Gas Chromatograph/Mass Spectrometer System:

7.1.1 Gas Chromatograph, Hewlett Packard 6890 (or equivalent): An analytical system complete with a temperature-programmable gas chromatograph configured for split/splitless-injection and all required accessories, including syringes, analytical columns, and gases. The capillary column is directly coupled to the source.

7.1.2 Column: Rxi-5Sil MS30m x 0.32mm ID, 0.25µm film thickness or column of similar configuration.

7.1.3 Mass Spectrometer, Hewlett Packard 5973 (or equivalent): Scanning from 35 to 500 amu every 1 second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer is capable of producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) which meets the criteria in Table 1 when 1 µL of the GC/MS tuning standard is injected through the GC (50ng of DFTPP).

7.1.4 Data System: A computer system is interfaced to the Mass Spectrometer. The system allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer software allows the analyst to search for any GC/MS data file for ions of specific mass and plot such ion abundances versus time or scan number. *HP ChemServer* software is used for data acquisition and *MSD Chemstation/Enviroquant version E.02.02* is used for data reduction.

7.2 Syringe: 10 µL.

7.3 Volumetric Flasks, Class A: Appropriate sizes with ground-glass stoppers.

7.4 Vials: Glass autosampler vials with polytetrafluoroethylene (PTFE)-lined crimp top caps.

8. Reagents and Standards

8.1 Stock Standard Solutions

Certified stock standard solutions, traceable to NIST, when available, are purchased from commercial vendors. They can be replaced with different standards as long as they contain all target analytes.

All stock standards, lot number, catalog number, expiration date, preparation date and initials are recorded in a logbook. Standards are stored in the refrigerator or freezer.

Stock standard expire 6 months from the date of preparation or on the earliest expiration date of any of the stock solution used to prepare it.

Please note that the following preparation instructions and stock standards are included for illustration purposes and may be modified as needed (ex. to accommodate standard availability or client requests), however final concentrations for the initial calibration levels shall always follow the example in 8.1.4.

Vendor	Standard	Catalog#	Concentration
Restek			
	8270 Mega Mix	31850	500-1000ug/mL
	Benzoic Acid Mix	31879	2000ug/mL
	Acid Surrogate Mix	31087	10000ug/mL
	B/N Surrogate Mix	31086	5000ug/mL
	Benzaldehyde Standard	33017	2000ug/mL
	Custom AP9 ICAL Standard	571813-FL	2000ug/mL
	Custom ADP Standard	572745-FL	2000ug/mL
	Benzidine Mix	31834	2000ug/mL
	SV Internal Standard Mix	31206	2000ug/mL
	1,4-Dioxane	30287	2000ug/mL
	Custom CLP 04.1 BNA Surrogate Mix	571320-FL	1000ug/mL
Absolute			
	Aromatic Amines Mix	99410	2000ug/ml
Ultra			
	Semi-Volatiles GC/MS Tuning Standard	GCM-150-1	1000ug/mL

8.1.1 ABN Stock Standard, 200ug/mL

Use 5mL of each of the following:
- Benzoic Acid Mix
- Benzidine Mix

and use 10mL of each of the following:
- 8270 Mega Mix
- Custom CLP 04.1 BNA Surrogate Mix

Bring up to 50mL volume with DCM.

8.1.2 AP9 Additional Compounds Stock Standard, 200ug/mL

Use 5mL of each of the following:
- Custom AP9 ICAL Standard
- Benzaldehyde Standard

Bring up to 50mL volume with DCM.

8.1.3 ADP Stock Standard, 200ug/ml

Use 5ml of:
Custom ADP Standard

Bring up to 50mL volume with DCM.

8.1.4 Calibration Standard

A minimum of 5 calibration standards must be included for each analyte:

Calibration Curve Levels	
Level	Concentration ug/mL
1	1.0
2	2.0
3	3.0
4	5.0
5	10
6	20
7	50
8	100
9	150
10	200

LVI Calibration Curve Levels	
Level	Concentration ug/mL
1	0.2
2	0.4
3	1.0
4	2.0
5	3.0
6	5.0
7	10
8	15
9	35
10	50

*LVI- Low Volume Initiative

8.2 Internal Standard Solution

The internal standards are:
1,4-dichlorobenzene-d_4
naphthalene-d_8
acenaphthene-d_{10}
phenanthrene-d_{10}
chrysene-d_{12}
perylene-d_{12}

Each 500µL of standards, blank and sample extracts are spiked with 10µL of SV Internal Standard Mix, resulting in a concentration of 40ng/ µL.

For the LVI method, a 1:10 dilution is made of the Internal Standard Stock Solution. 500µL of standards, blank and sample extracts are spiked with 10µL of this preparation, resulting in a concentration of 4ng/ µL.

8.3 GC/MS Tuning Standard

The tuning standard is a methylene chloride solution containing 50ng/µL of decafluorotriphenylphosphine (DFTPP). The standard also contains 50ng/µL each of 4,4'DDT, pentachlorophenol, and benzidine to verify injection port inertness and GC column performance.

Prepare the GC/MS Tuning Standard with 25µL GCM-150 and 475µL Dichloromethane.

8.4 Surrogate Spiking Solution

8.4.1 Extraction Surrogate Preparation

In a 1000mL volumetric flask, add 5ml of 31086 and 31087. Bring up to volume with Acetone. The final concentration is 50µg/mL for the acid surrogates and 25µg/mL for the B/N surrogates.

8.4.2 LVI Extraction Surrogate Preparation

The LVI surrogate is a 10 fold dilution of the surrogate solution prepared in 8.4.1. For example, to make 200mL of LVI surrogate, add 20mL of 8.4.1 to a 200mL volumetric flask and fill to volume with Acetone. The resulting surrogate concentration is 5µg/mL for the acid surrogates and 2.5µg/mL for the B/N surrogates.

8.5 Spike Solution (LCS, MS, MSD)

Spike Solution Preparation

ABN SPK1:
In a 500ml volumetric flask, add 20ml of 8270 Mega Mix #31850, 10ml of Benzoic Acid Mix #31879, 10ml Custom AP9 ICAL Standard #571813-FL and 10ml Benzaldehyde Standard #33017. Bring up to volume with Acetone. The final concentration is 40µg/ml.

Note: the LVI ABN SPK1 is prepared by making an 8 fold dilution of the 40µg/ml ABN SPK (in acetone), resulting in a 5µg/ml LVI ABN SPK1.

ABN SPK2:
In a 500ml volumetric flask, add 10ml Benzidine Mix #31834 and 10mL Custom ADP Standard #572945-FL. Bring up to volume with Acetone. The final concentration is 40µg/ml.

Note: the LVI ABN SPK2 is prepared by making an 8 fold dilution of the 40µg/ml ABN SPK (in acetone), resulting in a 5µg/ml LVI ABN SPK2.

8.6 Dichloromethane (DCM): Pesticide quality.

8.7 Acetone: Pesticide quality.

9. Quality Control

9.1 Blank(s)

Extraction blanks are performed with each extraction batch of 20 or less samples. The extraction blank must not contain any of the reportable analytes above the reporting limit. Corrective actions:

- No corrective action required if concentration of contaminant in sample is >10x concentration in blank or if contaminant not detected in sample

- If the blank have reportable hits and re-extraction could not be performed due to lack of additional sample volume, the sample results are reported and qualified with “B” flag for any associated samples that concentration is less than 10x the blank concentration

For NJ regulatory work the method blank must have all the target analytes less than RL except for Phthalates which must be less than 5x of the RL. Sample results are qualified with

"B" flag for analytes observed in the blank greater than RL and the Phthalates observed in the blank greater than 5x RL

The surrogate recoveries must also be within the acceptance criteria listed in Table 2. If surrogate acceptance criteria are exceeded, the extraction batch must be evaluated to determine if re-extraction or re-analysis is necessary.

9.2 Laboratory Control Sample and Laboratory Control Sample Duplicate (LCS / LCSD)

A Laboratory Control Sample/Laboratory Control Sample Duplicate pair (LCS/LCSD) are extracted and analyzed with each analytical batch of 20 or fewer samples.

The LCS/LCSD acceptance criteria are based on in-house control limits. Less than 10% of total compounds may be outside of control limits provided that recoveries are >10%. Note: this does not apply to difficult analytes listed in Table 9 which may be accepted at recoveries <10. If >10% of analytes are recovered above control limits, this is deemed acceptable as long as the analytes in question are not detected in associated samples.

If these criteria are not met, the entire batch is re-extracted. If re-extraction is not possible, due to insufficient sample or holding time exceedance, the analyst must write up the failure on a narrative sheet for inclusion in the client report.

9.3 Initial Calibration Verification (ICV)

Refer to Section 10.2.7.

9.4 Continuing Calibration Verification (CCV)

Refer to Section 10.4.

9.5 Matrix Spike and Matrix Spike Duplicate (MS / MSD)

A matrix spike/matrix spike duplicate pair is extracted and analyzed for each batch of 20 or fewer samples per client request. The MS/MSD acceptance criteria are based on in-house control limits. If the recovery criteria are not met, but are met in the LCS/LCSD, this is noted on a narrative sheet for inclusion in the client report.

9.6 Laboratory Duplicate

Not applicable.

9.7 Method-specific Quality Control Samples

9.7.1 Surrogates

All extracted samples and associated QC are spiked with surrogates. The acceptable surrogate recovery limits are listed in Table 2.

Corrective action: Up to one surrogate can be out in each fraction (Acid and Base/Neutral) but not less than 10% recovery, before any corrective action is necessary. Otherwise, analysis must be repeated once to see if an analytical error has occurred. If the % recovery still exceeds the control limits the sample must be re-extracted and re-analyzed to confirm sample matrix. If matrix effect is confirmed, this must be noted on a narrative sheet for inclusion in the client report.

Re-extraction is not required if surrogate recoveries are high and target analytes are not detected in the sample.

9.7.2 Internal Standards

If the area for any of the internal standards in the samples changes by a factor of two (-50% to +100%) from that in the CCV, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

9.8 Method Sequence

In a 12-hour period, the typical analytical sequence is:

- Degradation Check
- DFTPP
- Continuing or Daily Standards (1 – 3)*
  (1) ABN 50 ppm
  (2) AP9 50 ppm
  (3) ADP 50 ppm
- Method Blank
- Samples
- QC (as required)

***Additional Continuing standards may be run at the analyst's discretion or by client request.**

10. Procedure

10.1 Equipment Set-up

10.1.1 GC/MS Operating Conditions:

Typical GC/MS operating conditions are listed below, but may be altered as long as method performance criteria are met.

Mass range:	35 – 500 amu
Scan time:	3.15 second / scan
Initial temperature:	50°C, hold for 1.5 minutes
Temperature program:	28°C/minute to 250°C then 9°C/minute to 320°C
Final temperature:	320°C for 1.50 min
Injector temperature:	300°C
Transfer line temperature:	280°C
Source temperature:	230ºC
Injector:	split ratio 5:1; 11.7mL/min
Injection volume:	1µL
Carrier gas:	helium at 523 cm/second (2.0 mL/min) constant flow

GC/MS Operating Conditions for LVI method:

Mass range:	35 – 550 amu
Scan time:	3.15 second / scan
Initial temperature:	45°C, hold for 4 minutes
Temperature program:	25°C/minute to 250°C then 20°C/minute to 320°C
Final temperature:	320°C for 4.3 min
Injector temperature:	270°C
Transfer line temperature:	280°C
Source temperature:	320ºC
Injector:	split ratio 5:1; 8.57mL/min

Injection volume:	2µL
Carrier gas:	helium at 1.7148mL/min) constant flow

10.1.2 GC/MS Tune:

At the beginning of every 12 hour sequence, analyze DFTPP tuning solution (Section 8.3).

The resultant mass spectrum for DFTPP must meet the criteria given in Table 1 before sample analysis begins. The mass spectrum of DFTPP should be acquired in the following manner:

(1) Three scans (the peak apex scan, the scan immediately preceding the apex and the scan immediately following the apex) are acquired and averaged.
(2) Background subtraction is performed using a single scan of no more than 20 scans prior to the elution of DFTPP.

The GC/MS tuning standard is also used to assess GC column performance and injection port inertness. Degradation of DDT to DDE and DDD must not exceed 20%. Benzidine and pentachlorophenol must be present at their normal responses and no peak tailing must be visible.

The tailing factor for benzidine and pentachlorophenol must be calculated in every DFTPP run. (See Table 4)

If degradation is excessive and/or poor chromatography is noted, the system needs maintenance (see Section 10.5).

10.2 Initial Calibration

10.2.1 Prepare calibration standards for all target analytes at a minimum of five concentration levels as specified in Section 8.1.4.

10.2.2 Add 10µL of Internal Standard to each calibration standard directly into the autosampler vial containing 500µL of standard. Analyze each calibration standard under the conditions specified in Section 10.1.1.

10.2.3 Record the calibration standard, unique lab identifier code (lot), concentration, and analyst's initials in the analytical sequence list.

10.2.4 In each standard, calculate the response factor (RF) for each analyte, the average RF, and the relative standard deviation (RSD) of the RFs, using the Enviroquant data processing software. The calculations are performed automatically, using the formulae listed in Alpha's Quality Manual.

It is recommended that a minimum response factor for the most common target analytes, as noted in Table 8, be demonstrated for each individual calibration level as a means to ensure that these compounds are behaving as expected. In addition, meeting the minimum response factor criteria for the lowest calibration standard is critical in establishing and demonstrating the desired sensitivity.

10.2.5 Initial Calibration %RSD Criteria:

For all analytes, the RSD must be ≤ 20% for the mean response factor to be used for sample quantitation.

For RCP, the RSD must be ≤ 15% for the mean response factor to be used for sample quantitation.

An alternate calculation fits may be performed provided that the minimum correlation coefficient ≥ 0.99 is met.

When linear regression model is used a minimum quantitation check of the lowest calibration point is performed. The recalculated concentration of the low calibration point should be within ± 30% of the standard's true concentration.

10.2.6 Evaluation of Retention Times:

The relative retention time (RRT) of each target analyte in each calibration standard should agree within 0.06 RRT units.

10.2.7 Initial Calibration Verification (Second Source Verification)

10.2.7.1 The initial calibration (Section 10.2) for each compound of interest must be verified prior to sample analysis. This is accomplished by analyzing second source calibration standards at a concentration near the midpoint concentration for the calibrating range of the GC/MS.

10.2.7.2 Analyze the standards and calculate the % Difference for each analyte according to the formula in Alpha's Quality Manual.

If the % Difference for each analyte is ± 30%, then the calibration is assumed to be valid. If this criterion is not met, then corrective action must be taken prior to the analysis.

For RCP, if the % Difference for each analyte is ± 20%, then the calibration is assumed to be valid. If this criterion is not met, then corrective action must be taken prior to the analysis.

10.2.7.3 In cases where compounds fail (greater than 30% difference), they may still be reported as non-detects.

10.3 Equipment Operation and Sample Processing

GC/MS Analysis of Samples

10.3.1.1 Allow the sample extracts to warm to room temperature.

10.3.1.2 Transfer all of the sample extract to a 1.5mL vial. Remove 500µL of sample extract to another vial, and add 10µL of the internal standard solution (Section 8.2).

10.3.1.3 The autosampler is programmed to inject 1µL aliquot of the sample extract into the GC/MS system, using the same instrument conditions that were used for calibration. The injection volume of the sample must be the same as the volume used for the calibration standard.

10.3.1.4 If the response of any quantitation ion exceeds the initial calibration range of the GC/MS system, the sample extract must be diluted and reanalyzed.

10.3.2 Qualitative Identification

Perform first level data review. Obtain the primary m/z (Table 5) masses for each parameter of interest. The following criteria must be met to make qualitative identification:

- Compare the background subtracted mass spectra for the sample to the reference spectra. The characteristic masses of each parameter of interest must maximize in the same or within one scan of each other.
- The retention time must fall within ± 0.1 minutes of the retention time of the compound in the analytical standard. However, analyst experience must be used in making the qualitative identification.
- The relative peak height of the one characteristic mass must fall within 30% of the relative intensity of the mass in a reference mass spectrum. The reference spectrum is obtained from a standard analyzed on the GC/MS system.

Structural isomers that have very similar mass spectra are identified only if the resolution between authentic isomers in a standard mix is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

10.4 Continuing Calibration

10.4.1 The initial calibration (Section 10.2) for each compound of interest must be verified once every 12 hours prior to sample analysis. This is accomplished by analyzing calibration standards at a concentration near the midpoint concentration for the calibrating range of the GC/MS.

10.4.2 Analyze the standards and calculate the % Difference for each analyte according to the formula in Alpha's Quality Manual.

If the % Difference for each CCV analyte is $\leq$ 20%, then the calibration is assumed to be valid. If the criterion is not met for more than 20% of the compounds then corrective action must be taken.

Due to the large number of analytes present, allowances may be made for a RF that drifts out high, as long as there are no positive hits for that particular analyte in any of the associated samples.

10.4.3 If this criterion is exceeded, inspect the gas chromatographic system to determine the cause and perform whatever maintenance is necessary before verifying calibration and proceeding with sample analysis.

10.4.4 If routine maintenance does not return the instrument performance to meet the QC requirements based on the last initial calibration, then a new initial calibration must be performed.

10.4.5 Internal Standard Retention Time

The retention times of the internal standards in the calibration verification standard is evaluated after data acquisition. If the retention time for any internal standard changes by more than 30 seconds from that in the mid-point standard of the most recent initial calibration, then the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

10.4.6 Internal Standard Response

Refer to section 9.7.2

10.5 Preventive Maintenance

When poor sensitivity is observed, replacement of the injector liner and seal may solve the problem. If not, clip approximately 3 – 6 inches from the injector end of the GC column. If the sensitivity does not improve it may be necessary to replace the split line or the injector weldment assembly. If the problem persists, it may be necessary to replace the GC column.

Periodic cleaning (typically twice per year) of the mass spectrometer ion source is required. More frequent source cleaning may be needed, especially if dirty samples are analyzed.

11. Data Evaluation, Calculations and Reporting

When a parameter is identified, the quantitation of that parameter must be based on the integrated abundance of the quantitation characteristic m/z given in Table 5

Calculate the concentration in the sample using the average response factor (RF) from the initial calibration curve according to the formula in Alpha's Quality Manual.

After performing technical data review, validating that all QC criteria have been met and confirming all positive hits, the data report is sent electronically to the LIMS computer for generation of the client report. There are two levels of review of the data in the LIMS system prior to release of data. These reviews must be done by two separate individuals.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedence and improper preservation are noted on the nonconformance report form.

Perform instrument maintenance as described throughout this SOP as needed when instrument calibration criteria are not met. Record all maintenance in the instrument logbook.

All batch and sample specific QC criteria outlined in Section 9 are evaluated by the analyst prior to approval of the data. When any QC criteria fail, the cause for the failure must be identified and corrected. This may include instrument recalibration followed by sample reanalysis, sample cleanup, or sample re-extraction. If it is determined that the failure is due to sample matrix effects, a project narrative report is written by the analyst for inclusion in the data report. If there is insufficient sample volume to perform the re-analysis for confirmation, this is also noted in the narrative and included in the client report.

13. Method Performance

13.1 Detection Limit Study (DL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the DL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

Alpha SOP/1732 DL/LOD/LOQ Generation

Alpha SOP/1739 IDC/DOC Generation

Alpha SOP/1729 Waste Management and Disposal SOP

16. Attachments

Table 1: DFTPP Key Ions and Ion Abundance Criteria

Table 2: Acceptable Surrogate Spike Recovery Limits

Table 3A: Acceptable Aqueous QC Limits

Table 3B: Acceptable Soil QC Limits

Table 4: Tailing Factor Calculation

Table 5: Characteristic Ions for Semivolatile Compounds

Table 6: Reported Detection Limits

Table 7: Semivolatile Internal Standards with Corresponding Target Compounds and Surrogates Assigned for Quantitation

Table 8: Recommended Minimum Response Factor Criteria

Table 9: Difficult analytes

TABLE 1

DFTPP KEY IONS AND ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
51	10-80% of mass 198
68	< 2% of mass 69
70	< 2% of mass 69
127	10-80% of mass 198
197	< 2% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
275	10-60% of mass 198
365	> 1% of mass 198
441	Present but less than 24% mass 442
442	> 50% of mass 198
443	15-24% of mass 442

TABLE 2

ACCEPTABLE SURROGATE SPIKE RECOVERY LIMITS

Analytical Fraction	Surrogate Compound	Water	Soil/Sediment
BN-8270D	Nitrobenzene-d_5	23-120%	23-120%
BN-8270D	2-Fluorobiphenyl	15-120%	30-120%
BN-8270D	p-Terphenyl-d_{14}	41-149%	18-120%
Acid-8270D	Phenol-d_6	10-120%	10-120%
Acid-8270D	2-Fluorophenol	21-120%	25-120%
Acid-8270D	2,4,6-Tribromophenol	10-120%	10-136%

It is allowable for one surrogate from each fraction be outside acceptance criteria, provided a minimum recovery of 10% has been achieved.

TABLE 3A

ACCEPTABLE AQUEOUS QC LIMITS

	STANDARD TARGET COMPOUND LIST (Aqueous)		NEW JERSEY TARGET COMPOUND LIST (Aqueous)		CT TARGET COMPOUND LIST (Aqueous)	
Analyte	**Acceptance Criteria**	**Duplicate RPD**	**Acceptance Criteria**	**Duplicate RPD**	**Acceptance Criteria**	**Duplicate RPD**
1,2,4,5-Tetrachlorobenzene			70-130	20	40-140	20
1,2,4-Trichlorobenzene	39-98	30	70-130	20	40-140	20
1,2-Dichlorobenzene	40-140	30	70-130	20		
1,3-Dichlorobenzene	40-140	30	70-130	20		
1,3-Dinitrobenzene	15-130	30				
1,4-Dichlorobenzene	36-97	30	70-130	20		
1-Methylnaphthalene	41-103	30				
2,3,4,6-Tetrachlorophenol			70-130	20		
2,4,5-Trichlorophenol	30-130	30	70-130	20	30-130	20
2,4,6-Trichlorophenol	30-130	30	70-130	20	30-130	20
2,4-Dichlorophenol	30-130	30	70-130	20	30-130	20
2,4-Dimethylphenol	30-130	30	70-130	20	30-130	20
2,4- Dimethylaniline	40-140	30	70-130	20		
3,4- Dimethylaniline	40-140	30	70-130	20		
2,3- Dimethylaniline	40-140	30	70-130	20		
2,4,5-Dimethylaniline	40-140	30	70-130	20		
4-Chlorotoluidine	40-140	30	70-130	20		
2-Ethylaniline	40-140	30	70-130	20		
O-toluidine	40-140	30	70-130	20		
2-Napthylamine	40-140	30	70-130	20		
2,4-Dinitrophenol	20-130	30	20-130	20	30-130	20
2,4-Dinitrotoluene	24-96	30	70-130	20	40-140	20
2,6-Dinitrotoluene	40-140	30	70-130	20	40-140	20
2-Chloronaphthalene	40-140	30	70-130	20	40-140	20
2-Chlorophenol	27-123	30	70-130	20	30-130	20
2-Methylnaphthalene	40-140	30	70-130	20	40-140	20
2-Methylphenol	30-130	30	70-130	20	30-130	20
2-Nitroaniline	52-143	30	70-130	20	40-140	20
2-Nitrophenol	30-130	30	70-130	20	30-130	20
3,3'-Dichlorobenzidine	40-140	30	70-130	20	40-140	20
3,3'-Dimethylbenzidine			20-160	20		
3-Methylphenol/4-Methylphenol	30-130	30	20-160	20	30-130	20
3-Nitroaniline	25-145	30	70-130	20	40-140	20
4,6-Dinitro-o-cresol	20-164	30	70-130	20	30-130	20
4-Bromophenyl phenyl ether	40-140	30	70-130	20	40-140	20
4-Chloroaniline	40-140	30	20-160	20	40-140	20
4-Chlorophenyl phenyl ether	40-140	30	70-130	20	40-140	20
4-Nitroaniline	51-143	30	70-130	20	40-140	20
4-Nitrophenol	10-80	30	20-160	20	30-130	20
Acenaphthene	37-111	30	70-130	20	40-140	20
Acenaphthylene	45-123	30	70-130	20	40-140	20

	STANDARD TARGET COMPOUND LIST (Aqueous)		NEW JERSEY TARGET COMPOUND LIST (Aqueous)		CT TARGET COMPOUND LIST (Aqueous)	
Analyte	**Acceptance Criteria**	**Duplicate RPD**	**Acceptance Criteria**	**Duplicate RPD**	**Acceptance Criteria**	**Duplicate RPD**
Acetophenone	39-129	30	70-130	20		
Aniline	40-140	30	20-160	20	40-140	20
Anthracene	40-140	30	70-130	20	40-140	20
Atrazine			70-130	20		
Azobenzene	40-140	30	70-130	20		
Benzaldehyde			20-160	20		
Benzidine	10-75	30	20-160	20		
Benzo(a)anthracene	40-140	30	70-130	20	40-140	20
Benzo(a)pyrene	40-140	30	70-130	20	40-140	20
Benzo(b)fluoranthene	40-140	30	70-130	20	40-140	20
Benzo(ghi)perylene	40-140	30	70-130	20	40-140	20
Benzo(k)fluoranthene	40-140	30	70-130	20	40-140	20
Benzoic Acid	10-164	30	20-160	20		
Benzyl Alcohol	26-116	30	20-160	20		
Biphenyl	40-140	30	70-130	20		
Bis(2-chloroethoxy)methane	40-140	30	70-130	20	40-140	20
Bis(2-chloroethyl)ether	40-140	30	70-130	20	40-140	20
Bis(2-chloroisopropyl)ether	40-140	30	70-130	20	40-140	20
Bis(2-Ethylhexyl)phthalate	40-140	30	70-130	20	40-140	20
Butyl benzyl phthalate	40-140	30	70-130	20	40-140	20
Caprolactam			20-160	20		
Carbazole	55-144	30	70-130	20	40-140	20
Chrysene	40-140	30	70-130	20	40-140	20
Dibenzo(a,h)anthracene	40-140	30	70-130	20	40-140	20
Dibenzofuran	40-140	30	70-130	20	40-140	20
Diethyl phthalate	40-140	30	70-130	20	40-140	20
Dimethyl phthalate	40-140	30	70-130	20	40-140	20
Di-n-butylphthalate	40-140	30	70-130	20	40-140	20
Di-n-octylphthalate	40-140	30	70-130	20	40-140	20
Fluoranthene	40-140	30	70-130	20	40-140	20
Fluorene	40-140	30	70-130	20	40-140	20
Hexachlorobenzene	40-140	30	70-130	20	40-140	20
Hexachlorobutadiene	40-140	30	70-130	20	40-140	20
Hexachlorocyclopentadiene	40-140	30	20-160	20	40-140	20
Hexachloroethane	40-140	30	20-160	20	40-140	20
Indeno(1,2,3-cd)Pyrene	40-140	30	70-130	20	40-140	20
Isophorone	40-140	30	70-130	20	40-140	20
Naphthalene	40-140	30	70-130	20	40-140	20
Nitrobenzene	40-140	30	70-130	20	40-140	20
NitrosoDiPhenylAmine(NDPA)/ Diphenylamine (DPA)	40-140	30	70-130	20	40-140	20
n-Nitrosodimethylamine	22-74	30	20-160	20		
n-Nitrosodi-n-propylamine	29-132	30	70-130	20	40-140	20
P-Chloro-M-Cresol	23-97	30	70-130	20	30-130	20
Pentachlorophenol	9-103	30	20-160	20	30-130	20
Pentachloronitrobenzene					40-140	20

	STANDARD TARGET COMPOUND LIST (Aqueous)			NEW JERSEY TARGET COMPOUND LIST (Aqueous)		CT TARGET COMPOUND LIST (Aqueous)	
Analyte	**Acceptance Criteria**	**Duplicate RPD**		**Acceptance Criteria**	**Duplicate RPD**	**Acceptance Criteria**	**Duplicate RPD**
Phenanthrene	40-140	30		70-130	20	40-140	20
Phenol	12-110	30		20-160	20	30-130	20
Pyrene	26-127	30		70-130	20	40-140	20
Pyridine	10-66	30				40-140	20
2-Fluorophenol	*21-120*			*15-110*		*15-110*	
Phenol-d6	*10-120*			*15-110*		*15-110*	
Nitrobenzene-d5	*23-120*			*30-130*		*30-130*	
2-Fluorobiphenyl	*15-120*			*30-130*		*30-130*	
2,4,6-Tribromophenol	*10-120*			*15-110*		*15-110*	
4-Terphenyl-d14	*41-149*			*30-130*		*30-130*	

TABLE 3B

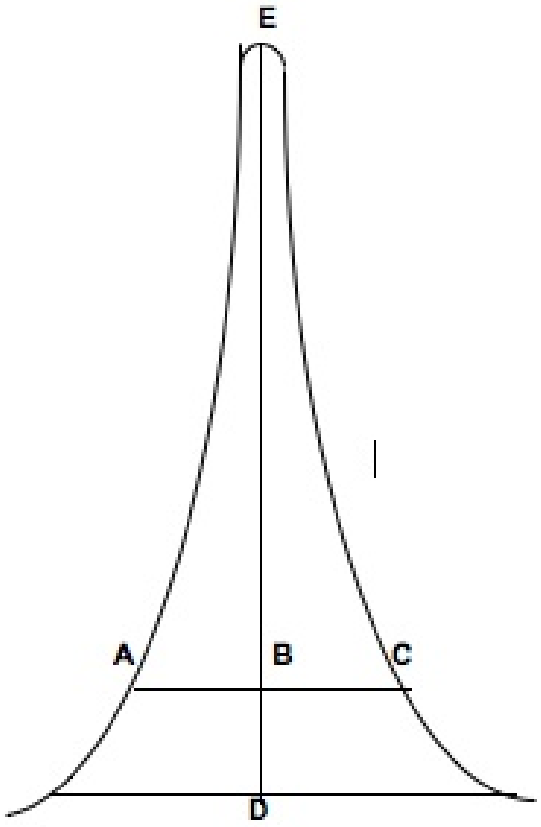
ACCEPTABLE SOIL QC LIMITS

	STANDARD TARGET COMPOUND LIST (Soil)		NEW JERSEY TARGET COMPOUND LIST (Soil)		CT TARGET COMPOUND LIST (Soil)	
Analyte	**Acceptance Criteria**	**Duplicate RPD**	**Acceptance Criteria**	**Duplicate RPD**	**Acceptance Criteria**	**Duplicate RPD**
1,2,4,5-Tetrachlorobenzene	40-117	50	70-130	30	40-140	30
1,2,4-Trichlorobenzene	38-107	50	70-130	30	40-140	30
1,2-Dichlorobenzene	40-140	50	70-130	30		
1,3-Dichlorobenzene	40-140	50	70-130	30		
1,3-Dinitrobenzene	40-140	50				
1,4-Dichlorobenzene	28-104	50	70-130	30		
1-Methylnaphthalene	26-130	50				
2,3,4,6-Tetrachlorophenol	40-140	50	70-130	30		
2,4,5-Trichlorophenol	30-130	50	70-130	30	30-130	30
2,4,6-Trichlorophenol	30-130	50	70-130	30	30-130	30
2,4-Dichlorophenol	30-130	50	70-130	30	30-130	30
2,4-Dimethylphenol	30-130	50	70-130	30	30-130	30
2,4-Dinitrophenol	4-130	50	20-160	30	30-130	30
2,4-Dinitrotoluene	28-89	50	70-130	30	40-140	30
2,6-Dinitrotoluene	40-140	50	70-130	30	40-140	30
2-Chloroaniline	30-130	50				
2-Chloronaphthalene	40-140	50	70-130	30	40-140	30
2-Chlorophenol	25-102	50	70-130	30	30-130	30
2-Methylnaphthalene	40-140	50	70-130	30	40-140	30
2-Methylphenol	30-130.	50	70-130	30	30-130	30
2-Nitroaniline	47-134	50	70-130	30	40-140	30
2-Nitrophenol	30-130	50	70-130	30	30-130	30
3,3'-Dichlorobenzidine	40-140	50	70-130	30	40-140	30
3,3'-Dimethylbenzidine	15-115	50				
3-Methylphenol/4-Methylphenol	30-130	50	20-160	30	30-130	30
3-Nitroaniline	26-129	50	70-130	30	40-140	30
4,6-Dinitro-o-cresol	10-130	50	70-130	30	30-130	30
4-Bromophenyl phenyl ether	40-140	50	70-130	30	40-140	30
4-Chloroaniline	40-140	50	20-160	30	40-140	30
4-Chlorophenyl phenyl ether	40-140	50	70-130	30	40-140	30
4-Nitroaniline	41-125	50	70-130	30	40-140	30
4-Nitrophenol	11-114	50	20-160	30	30-130	30
Acenaphthene	31-137	50	70-130	30	40-140	30

	STANDARD TARGET COMPOUND LIST (Soil)		NEW JERSEY TARGET COMPOUND LIST (Soil)		CT TARGET COMPOUND LIST (Soil)	
Analyte	**Acceptance Criteria**	**Duplicate RPD**	**Acceptance Criteria**	**Duplicate RPD**	**Acceptance Criteria**	**Duplicate RPD**
Acenaphthylene	40-140	50	70-130	30	40-140	30
Acetophenone	14-144	50	70-130	30	40-140	30
Aniline	40-140	50	20-160	30	40-140	30
Anthracene	40-140	50	70-130	30	40-140	30
Atrazine	40-140	50	70-130	30		
Azobenzene	40-140	50	70-130	30		
Benzaldehyde	40-140	50	20-160	30		
Benzidine	10-66	50	20-160	30		
Benzo(a)anthracene	40-140	50	70-130	30	40-140	30
Benzo(a)pyrene	40-140	50	70-130	30	40-140	30
Benzo(b)fluoranthene	40-140	50	70-130	30	40-140	30
Benzo(e)pyrene	40-140	50				
Benzo(ghi)perylene	40-140	50	70-130	30	40-140	30
Benzo(k)fluoranthene	40-140	50	70-130	30	40-140	30
Benzoic Acid	10-110	50	20-160	30		
Benzyl Alcohol	40-140	50	20-160	30		
Biphenyl	37-127	50	70-130	30		
Bis(2-chloroethoxy)methane	40-117	50	70-130	30	40-140	30
Bis(2-chloroethyl)ether	40-140	50	70-130	30	40-140	30
Bis(2-chloroisopropyl)ether	40-140	50	70-130	30	40-140	30
Bis(2-Ethylhexyl)phthalate	40-140	50	70-130	30	40-140	30
Butyl benzyl phthalate	40-140	50	70-130	30	40-140	30
Caprolactam	15-130	50	20-160	30		
Carbazole	54-128	50	70-130	30	40-140	30
Chrysene	40-140	50	70-130	30	40-140	30
Dibenzo(a,h)anthracene	40-140	50	70-130	30	40-140	30
Dibenzofuran	40-140	50	70-130	30	40-140	30
Diethyl phthalate	40-140	50	70-130	30	40-140	30
Dimethyl phthalate	40-140	50	70-130	30	40-140	30
Di-n-butylphthalate	40-140	50	70-130	30	40-140	30
Di-n-octylphthalate	40-140	50	70-130	30	40-140	30
Diphenamid	40-140	50				
Fluoranthene	40-140	50	70-130	30	40-140	30
Fluorene	40-140	50	70-130	30	40-140	30
Hexachlorobenzene	40-140	50	70-130	30	40-140	30
Hexachlorobutadiene	40-140	50	70-130	30	40-140	30
Hexachlorocyclopentadiene	40-140	50	20-160	30	40-140	30
Hexachloroethane	40-140	50	20-160	30	40-140	30
Indeno(1,2,3-cd)Pyrene	40-140	50	70-130	30	40-140	30
Isophorone	40-140	50	70-130	30	40-140	30

	STANDARD TARGET COMPOUND LIST (Soil)		NEW JERSEY TARGET COMPOUND LIST (Soil)		CT TARGET COMPOUND LIST (Soil)	
Analyte	**Acceptance Criteria**	**Duplicate RPD**	**Acceptance Criteria**	**Duplicate RPD**	**Acceptance Criteria**	**Duplicate RPD**
Naphthalene	40-140	50	70-130	30	40-140	30
Nitrobenzene	40-140	50	70-130	30	40-140	30
NitrosoDiPhenylAmine(NDPA)/ Diphenylamine (DPA)	36-157	50	70-130	30	40-140	30
n-Nitrosodimethylamine	22-100	50	20-160	30		
n-Nitrosodi-n-propylamine	32-121	50	70-130	30	40-140	30
Parathion, ethyl	40-140	50	20-160	30		
P-Chloro-M-Cresol	26-103	50	70-130	30	30-130	30
Pentachloronitrobenzene	42-153	50			40-140	30
Pentachlorophenol	17-109	50	20-160	30	30-130	30
Phenanthrene	40-140	50	70-130	30	40-140	30
Phenol	26-90	50	20-160	30	30-130	30
Pyrene	35-142	50	70-130	30	40-140	30
Pyridine	10-93	50	20-160	30	40-140	30
Thionazin	40-140	50				
2-Fluorophenol	*25-120*		*30-130*		*30-130*	
Phenol-d6	*10-120*		*30-130*		*30-130*	
Nitrobenzene-d5	*23-120*		*30-130*		*30-130*	
2-Fluorobiphenyl	*30-120*		*30-130*		*30-130*	
2,4,6-Tribromophenol	*10-136*		*30-130*		*30-130*	
4-Terphenyl-d14	*18-120*		*30-130*		*30-130*	

TABLE 4 – Tailing Factor Calculation



Tailing Factor = $\frac{BC}{AB}$

Example calculation:

Peak Height = DE = 100mm
10% Peak Height = BD = 10mm
Peak Width at 10% Peak Height = AC = 23mm

AB = 11mm
BC = 12mm

Therefore: Tailing Factor = $\frac{12}{11}$ = 1.1

Tailing factor for benzidine < 2.0

Tailing factor for pentachlorophenol <2.0

TABLE 5

CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

Compound	Primary Ion	Secondary Ion(s)
Acenaphthene	154	153, 152
Acenaphthylene	152	151, 153
Acetophenone	105	71, 51, 120
Aniline	93	66, 65
Anthracene	178	176, 179
Atrazine	200	202, 215
Azobenzene	77	182, 105
Benzaldehyde	105	77
Benzidine	184	92, 185
Benzo(a)anthracene	228	229, 226
Benzo(a)pyrene	252	253, 125
Benzo(b)fluoranthene	252	253, 125
Benzo(g,h,i)perylene	276	138, 277
Benzo(k)fluoranthene	252	253, 125
Benzoic acid	105	122, 77
Benzyl alcohol	79	77,108
Biphenyl	154	153,152
Bis (2-chloroethoxy) methane	93	95, 123
Bis (2-chloroethyl) ether	93	63, 95
Bis (2-chloroisopropyl) ether	45	77, 121
Bis (2-ethylhexyl) phthalate	149	167, 279
4-Bromophenyl phenyl ether	248	250, 141
Butyl Benzyl phthalate	149	91, 206
Caprolactam	55	85, 113
Carbazole	167	168, 166
4-Chloro-3-methylphenol	107	144, 142
2-Chloroaniline	127	129, 65
3-Chloroaniline	65	127, 129
4-Chloroaniline	65	127,129
2-Chloronaphthalene	162	127, 164
4-Chlorophenyl phenyl ether	204	206, 141
2-Chlorophenol	128	64,130
Chrysene	228	226, 229
Dibenzo(a,h)anthracene	278	139, 279
Dibenzofuran	168	139
1,2-Dichlorobenzene	146	148, 111

1,3-Dichlorobenzene	**146**	**148, 111**
1,4-Dichlorobenzene	**146**	**148, 111**
3,3'-Dichlorobenzidine	**252**	**254, 126**
2,4-Dichlorophenol	**162**	**164, 98**
Diethyl phthalate	**149**	**177, 150**

TABLE 5 (continued)

CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

Compound	Primary Ion	Secondary Ion(s)
3,3-Dimethylbenzidine	**212**	**211, 213**
Dimethyl phthalate	**163**	**194, 164**
2,4-Dimethylphenol	**107**	**121,122**
Di-n-butyl phthalate	**149**	**150, 104**
Di-n-octyl phthalate	**149**	**167, 43**
4,6-Dinitro-2-methylphenol	**198**	**51, 105**
O-Toluidine	**106**	**107, 77**
2-Ethylaniline	**106**	**121, 77**
2,4-Dimethylaniline	**121**	**120, 106**
2,3-Dimethylaniline	**106**	**121, 120**
3,4- Dimethylaniline	**121**	**120,106**
2,4,5-Trimethylaniline	**120**	**135, 134**
4-Chlorotoluidine	**106**	**141, 140**
2-Napthylamine	**143**	**115, 116**
2,4-Dinitrophenol	**184**	**107,91**
2,4-Dinitrotoluene	**165**	**63, 89**
2,6-Dinitrotoluene	**165**	**63, 89**
Diphenamide	**167**	**72, 165**
1,4-Dioxane	**88**	**58,43**
Ethyl parathion	**109**	**97, 291**
Fluoranthene	**202**	**101, 203**
Fluorene	**166**	**165, 167**
Hexachlorobenzene	**284**	**142, 249**
Hexachlorobutadiene	**225**	**223, 227**
Hexachlorocyclopentadiene	**237**	**235, 272**
Hexachloroethane	**117**	**201, 199**
Indeno(1,2,3-cd)pyrene	**276**	**138, 227**
Isophorone	**82**	**95, 138**
1-Methylnaphthalene	**115**	**141, 142**
2-Methylnaphthalene	**142**	**141**
2-Methylphenol	**108**	**107,90**
3/4-Methylphenol	**108**	**107,90**

Naphthalene	**128**	**129, 127**
2-Nitroaniline	**65**	**92, 138**
3-Nitroaniline	**138**	**92,65**
4-Nitroaniline	**138**	**65, 108, 92, 80, 39**
Nitrobenzene	**77**	**123, 65**

TABLE 5 (continued)

CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

Compound	Primary Ion	Secondary Ion(s)
2-Nitrophenol	**139**	**109, 65**
4-Nitrophenol	**65**	**109, 139**
n-Nitrosodimethylamine	**74**	**42,44**
n-Nitrosodi-n-butylamine	**84**	**57, 41, 116, 158**
n-Nitrosodi-n-propylamine	**70**	**42, 101, 130**
n-Nitrosodiphenylamine/Diphenylamine	**169**	**168, 167**
Pentachlorobenzene	**250**	**252, 108, 248, 215, 254**
Pentachloronitrobenzene	**237**	**142, 214, 249, 295, 265**
Pentachlorophenol	**266**	**264, 268**
Phenanthrene	**178**	**179, 176**
Phenol	**94**	**65, 66**
Pyrene	**202**	**200, 203**
Pyridine	**79**	**52**
1,2,4,5-Tetrachlorobenzene	**216**	**214, 179, 108, 143, 218**
2,3,4,6-Tetrachlorophenol	**232**	**131, 230, 166, 234, 168**
m-Toluidine	**106**	**107, 79**
1,2,4-Trichlorobenzene	**180**	**182, 145**
2,4,5-Trichlorophenol	**196**	**200,198**
2,4,6-Trichlorophenol	**196**	**198, 200**
Acenaphthene-d_{10} (IS)	**164**	**162, 160**
Chrysene-d_{12} (IS)	**240**	**120, 236**
1,4-Dichlorobenzene-d_4 (IS)	**152**	**150, 115**
Naphthalene-d_8 (IS)	**136**	**68**
Perylene-d_{12} (IS)	**264**	**260, 265**
Phenanthrene-d_{10} (IS)	**188**	**94, 80**
2-Fluorobiphenyl (Surrogate)	**172**	**171**
2-Fluoroophenol (Surrogate)	**112**	**64**
Nitrobenzene-d_5 (Surrogate)	**82**	**128, 54**
Phenol-d_6 (Surrogate)	**99**	**42, 71**
Terphenyl-d_{14} (Surrogate)	**244**	**122, 212**
2,4,6-Tribromophenol (Surrogate)	**330**	**62,141**

TABLE 6

REPORTED DETECTION LIMITS FOR SEMIVOLATILE ORGANIC COMPOUNDS*

Analyte	RDL (µg/L)	RDL (µg/Kg)
Acenaphthene	2	133.34
Acenaphthylene	2	133.34
Acetophenone	5	333.34
Aniline	2	133.34
Anthracene	2	133.34
Atrazine	10	666.67
Azobenzene	2	500
Benzaldehyde	5	333.34
Benzidine	20	1333.34
Benzo(a)anthracene	2	133.34
Benzo(b)fluoranthene	2	133.34
Benzo(k)fluoranthene	2	133.34
Benzo(ghi)perylene	2	133.34
Benzo(a)pyrene	2	133.34
Benzoic acid	50.0	3333.34
Benzyl alcohol	2	133.34
Biphenyl	2	366.67
Bis(2-chloroethyl)ether	2	133.34
Bis(2-chloroisopropyl)ether	2	133.34
Bis(2-chloroethoxy)methane	5.0	333.34
Bis(2-ethylhexyl)phthalate	3	200
4-Bromophenyl phenyl ether	2	133.34
Butyl benzyl phthalate	5.0	333.34
Caprolactam	10	666.67
Carbazole	2	166.67
2-Chloroaniline	2	na
3-Chloroaniline	10	na
4-Chloroaniline	5	333.34
p-Chloro-m-cresol (4-chloro-3-cresol)	2	133.34
2-Chloronaphthalene	2	133.34
2-Chlorophenol	2	133.34
4-Chlorophenyl phenyl ether	2	133.34
Chrysene	2	133.34
m/p-Methylphenol (3/4-methylphenol)	5.0	333.34
o-Methylphenol (2-methylphenol)	5.0	333.34

Dibenzo(a,h)anthracene	2	133.34
Dibenzofuran	2	133.34
Di-n-butylphthalate	5.0	333.34
1,2-Dichlorobenzene	2	133.34

TABLE 6 (continued)

REPORTED DETECTION LIMITS FOR SEMIVOLATILE ORGANIC COMPOUNDS*

Analyte	RDL (µg/L)	RDL (µg/Kg)
1,3-Dichlorobenzene	2	133.34
1,3-Dinitrobenzene	2	N/A
1,4-Dichlorobenzene	2	133.34
3,3-Dichlorobenzidine	5	333.34
2,4-Dichlorophenol	5	333.34
O-Toluidine	2	N/A
2-Ethylaniline	2	N/A
2,4-Dimethylaniline	2	N/A
2,3-Dimethylaniline	2	N/A
3,4-Dimetylaniline	2	N/A
2,4,5-Trimethylaniline	2	N/A
4-Chlorotoluidine	2	N/A
2-Napthylamine	2	N/A
2,6-Dichlorophenol	10.0	666.67
Diethyl phthalate	5.0	333.34
3,3-Dimethylbenzidine	4	500
2,4-Dimethylphenol	5	333.34
Dimethyl phthalate	5.0	333.34
4,6-Dinitro-o-cresol	10	666.67
2,4-Dinitrophenol	20	1333.4
2,4-Dinitrotoluene	5.0	333.34
2,6-Dinitrotoluene	5.0	333.34
Di-n-octylphthalate	5.0	333.34
Diphenamide	5	N/A
1,4-Dioxane	5	166.67
Ethyl Parathion	N/A	166.67
Fluoranthene	2	133.34
Fluorene	2	133.34
Hexachlorobenzene	2	133.34
Hexachlorobutadiene	2	133.34
Hexachlorocyclopentadiene	20	1333.34
Hexachloroethane	2	133.34
Indeno(1,2,3-cd)pyrene	2	133.34
Isophorone	5.0	333.34

1-Methylnaphthalene	2	166.67
2-Methylnaphthalene	2	133.34
Naphthalene	2	133.34
2-Nitroaniline	5.0	333.34

TABLE 6 (continued)

REPORTED DETECTION LIMITS FOR SEMIVOLATILE ORGANIC COMPOUNDS*

Analyte	RDL (µg/L)	RDL (µg/Kg)
3-Nitroaniline	5.0	333.34
4-Nitroaniline	5.0	333.34
Nitrobenzene	2	133.34
2-Nitrophenol	10.0	666.67
4-Nitrophenol	10.0	666.67
Nitrosodi-n-butylamine	10.0	666.67
n-Nitrosodimethylamine	2	133.34
n-Nitrosodiphenylamine/Diphenylamine	2	133.34
Nitrosodipiperidine	20.0	2000
n-Nitrosodi-n-propylamine	5.0	333.34
Pentachlorobenzene	20.0	1333.34
Pentachloronitrobenzene	10.0	150
Pentachlorophenol	10.0	666.67
Phenanthrene	2	133.34
Phenol	5.0	333.34
Pyrene	2	133.34
Piridine	5	666.67
1,2,4,5-Tetrachlorobenzene	10	666.67
1,2,4-Trichlorobenzene	5.0	333.34
2,4,5-Trichlorophenol	5.0	333.34
2,4,6-Trichlorophenol	5.0	333.34
2,3,4,6-Tetrachlorophenol	5.0	166.66
m-Toluidine	5	300

*** Note**: Reporting Limits are based on standard 8270 reporting list. RLs may vary for other reporting lists.

Table 7
Semivolatile Internal Standards with Corresponding
Target Compounds and Surrogates Assigned for Quantitation

1,4-dichlorobenzene-d4	Naphthalene-d8	Acenaphthene-d10	Phenanthrene-d10	Chrysene-d12	Perylene-d12
O-Toluidine	2-Ethylaniline	2-Naphthylamine	3,3-Dimethylbenzidine	3,3'-Dichlorobenzidine	Benzo(g,h,i)perylene
1,2,4-Trichlorobenzene	2,4-Dimethylaniline	2,3,4,6-Tetrachlorophenol	Anthracene	Benzo(a)Anthracene	Dibenzo(a,h)anthracene
1,2-Dichlorobenzene	3,4-Dimethylaniline	2,3,5,6-Tetrachlorophenol	Benzidine	Benzo(a)pyrene	Indeno(1,2,3-cd)pyrene
1,3-Dichlorobenzene	2,3-Dimethylaniline	2,4,6-Tribromophenol, surr	Benzyl butyl phthalate	Benzo(b)fluoranthene	
1,4-Dichlorobenezne	2,4,5-Trimethylaniline	2,4-Dinitrophenol	Carbazole	Benzo(k)fluoranthene	
2,4-Dichlorophenol	4-Chlorotoludine	2,4-Dinitrotoluene	Di-n-Butylphthalate	Bis(2-ethylhexyl) phthalate	
2,4-Dimethylphenol	1,2,4,5-Tetrachlorobenzene	3-Nitroaniline	Diphenamid	Chrysene	
2-Chloroaniline	1,2-Dichlorobenzene	4,6-Dinitro-2-methylphenol	Fluoranthene	Di-n-octylphthalate	
2-Chlorophenol	1,3-Dichlorobenzene	4-Bromophenyl-phenyl ether	n-Octadecane		
2-Fluorophenol, surr	1,4-Diclorobenzene	4-Chlorophenyl-phenyl ether	Parathion		
2-Methylphenol	1-chloror-2-nitrobenzene	4-Nitroaniline	Phenanthrene		
2-Nitrophenol	1-Methylnapthalene	4-Nitrophenol	Pyrene		
3-Methylphenol / 4-Methylphenol	2,4,5-Trichlorphenol	Acenaphthene	Terphenyl-d14, surr		
Acetophenone	2,4,6-Trichlorophenol	Atrazine			
Aniline	2,6-Dichlorophenol	Azobenzene			
Benzaldehyde	2,6-Dinitrotoluene	Dibenzofuran			
Benzyl Alcohol	2-Chloronaphthalene	Dichloran			
Bis(2-chloroethoxy)methane	2-Fluorobiphenyl, surr	Diethyl phthalate			
Bis(2-chloroethyl)ether	2-Methylnaphthalene	Fluorene			

Table 7 (cont.)
Semivolatile Internal Standards with Corresponding
Target Compounds and Surrogates Assigned for Quantitation

1,4-dichlorobenzene-d4	Naphthalene-d8	Acenaphthene-d10	Phenanthrene-d10	Chrysene-d12	Perylene-d12
bis(2-Chloroisopropyl)ether	2-Nitroaniline	Hexachlorobenzene			
Hexachloroethane	3-Choloroaniline	NDPA/DPA			
Isophorone	4-Chloro-3-Methylphenol	Pentachloronitrobenzene			
m-Toluidine	4-Chloroaniline	Pentachlorophenol			
n-Decane	Acenaphthylene				
Nitrobenzene	a-Terpineol				
Nitrobenzene-d5, surr	Benzoic Acid				
N-Nitrosodimethylamine	Biphenyl				
N-Nitrosodi-n-propylamine	Caprolactam				
Phenol	Dimethyl Phthalate				
Phenol-d6, surr	Hexachlorobutadiene				
Pyridine 1,4-Dioxane	Hexachlorocyclopentadiene				
Phenol-d6, surr	Naphthalene				

Table 8

Recommended Minimum Response Factor Criteria from Initial and Continuing Calibration Verification Using the Suggested Ions in Table 5

Analyte	MRF
Benzaldehyde	0.010
Phenol	0.800
Bis(2-chloroethyl)ether	0.700
2-Chlorophenol	0.800
2-Methylphenol	0.700
2,2'-Oxybis-(1-chloropropane)	0.010
Acetophenone	0.010
4-Methylphenol	0.600
N-Nitroso-di-n-propylamine	0.500
Hexachloroethane	0.300
Nitrobenzene	0.200
Isophorone	0.400
2-Nitrophenol	0.100
2,4-Dimethylphenol	0.200
Bis(2-chloroethoxy)methane	0.300
2,4-Dichlorophenol	0.200
Naphthalene	0.700
4-Chloroaniline	0.010
Hexachlorobutadiene	0.010
Caprolactam	0.010
4-Chloro-3-methylphenol	0.200
2-Methylnaphthalene	0.400
Hexachlorocyclopentadiene	0.050
2,4,6-Trichlorophenol	0.200
2,4,5-Trichlorophenol	0.200
1,1'-Biphenyl	0.010
2-Chloronaphthalene	0.800
2-Nitroaniline	0.010
Dimethyl phthalate	0.010
2,6-Dinitrotoluene	0.200
Acenaphthylene	0.900
3-Nitroaniline	0.010
Acenaphthene	0.900
2,4-Dinitrophenol	0.010
4-Nitrophenol	0.010
Dibenzofuran	0.800
2,4-Dinitrotoluene	0.200
Diethyl phthalate	0.010
1,2,4,5-Tetrachlorobenzene	0.010

Table 8 (cont.)

Recommended Minimum Response Factor Criteria from Initial and Continuing Calibration Verification Using the Suggested Ions in Table 5

Analyte	MRF
4-Chlorophenyl-phenyl ether	0.400
Fluorene	0.900
4-Nitroaniline	0.010
4,6-Dinitro-2-methylphenol	0.010
4-Bromophenyl-phenyl ether	0.100
N-Nitrosodiphenylamine	0.010
Hexachlorobenzene	0.100
Atrazine	0.010
Pentachlorophenol	0.050
Phenanthrene	0.700
Anthracene	0.700
Carbazole	0.010
Di-n-butyl phthalate	0.010
Fluoranthene	0.600
Pyrene	0.600
Butyl benzyl phthalate	0.010
3,3'-Dichlorobenzidine	0.010
Benzo(a)anthracene	0.800
Chrysene	0.700
Bis-(2-ethylhexyl)phthalate	0.010
Di-n-octyl phthalate	0.010
Benzo(b)fluoranthene	0.700
Benzo(k)fluoranthene	0.700
Benzo(a)pyrene	0.700
Indeno(1,2,3-cd)pyrene	0.500
Dibenz(a,h)anthracene	0.400
Benzo(g,h,i)perylene	0.500
2,3,4,6-Tetrachlorophenol	0.010

Table 9
Difficult analytes

Aniline

Benzaldehyde
Benzidine
Benzoic acid
Benzyl alcohol

Caprolactam
4-Chloroaniline
4-chloro-3-methylphenol (p-chloro-m-cresol)

3,3-Dimethylbenzidine
Dimethylphthalate
2,4 Dinitrophenol
4,6-dinitro-2-methylphenol (4,6-dinitro-o-cresol)

Hexachlorocyclopentadiene
Hexachloroethane

2-Methylphenol
3-Methylphenol/4-Methylphenol

2-nitroaniline
3-nitroaniline
4-nitroaniline
4-Nitrophenol
Nitrosodiphenylamine and diphenylamine (NDPA/DPA)
n-Nitrosodimethylamine

Parathion
Pentachloronitrobenzene
Pentachlorophenol
Phenol
Pyridine

Separatory Funnel Extraction of Liquid Samples EPA 3510C

Reference Method: **EPA 3510C (EPH 608, EPA 625, EPA 608.3, EPA 625.1, and MA-DEP EPH)** SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update III, December 1996.

1. Scope and Application

Matrices: This method is applicable to aqueous samples.

Definitions: Refer to Alpha Analytical Quality Manual.

This method describes the procedure for extracting water-insoluble and lightly water-soluble organic compounds from aqueous samples. The method also describes concentration techniques suitable for preparing the extract for the various determinative methods listed in Table 1.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, o r Quality Assurance Officer.

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

A measured volume of sample is serially extracted with methylene chloride using a separatory funnel. Depending on the analytes to be detected, it may be necessary to adjust the pH of the aqueous sample prior to extraction (Table 1).

Any water is removed from the sample extract by filtering through a powder funnel containing approximately 60g of baked anhydrous sodium sulfate. The extract is then concentrated and, as needed, exchanged into a solvent compatible with the cleanup or determinative step being employed. The various cleanup methods used summarized in Table 1.

2.1 Method Modifications from Reference

None.

3. Reporting Limits

Reporting Limit information can be found in the analytical method SOPs.

4. Interferences

4.1 The most common cause of contamination is from improperly cleaned glassware and lab supplies. All glassware and re-useable extraction equipment must be scrupulously cleaned, following the Organic Extraction Glassware Cleaning and Handling SOP/1953 and Work instruction 10995, Solvent rinsing/filtering guide.

4.2 Impurities in solvents and reagents may also yield artifacts and/or interferences that may compromise the results of sample analysis. All of these materials must be demonstrated to be free from interferences under the conditions of extract preparation and analysis by preparing method blanks with each extraction batch. The same solvents and reagents are used for the method blank and the associated samples.

4.3 Phthalate esters contaminate many types of products used in the laboratory. Plastic materials must not contact the samples or extracts, as phthalates could be easily leached from the plastic. The exception is in the use of various pre-packed reagent cartridges (Florisil, Silica gel) used in the extract cleanup steps. Each new lot of cartridges is checked for contamination, and is monitored on an on-going basis through the analysis of method blanks.

4.4 Additional specific interference or contamination concerns are addressed in the various analytical SOPs.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

5.1 Lab coats, safety glasses, and gloves must be worn when handling samples, extracts, standards or solvents and when washing glassware.

5.2 All extract concentration steps must be performed in the extraction hoods. All solvent and extract transfers must also be handled in the hood.

5.3 All expired stock standards, working standards, and spent sample extracts must be placed into the waste bucket in the lab, for future disposal by the Hazardous Waste Manager. The container must be properly labeled with hazard warning labels indicating the container contents.

5.4 Bottles containing flammable solvents must be stored in the flammables cabinet or in the vented cabinets found under the hoods.

5.5 All waste solvents must be transferred to the satellite waste storage containers located in the extraction lab. Separate containers are provided for chlorinated and non-chlorinated solvents and must be used accordingly. Under no circumstances are solvents to be poured down the sink drains.

5.6 Inspect all glassware prior to use. Do not use any glassware that is chipped, cracked or etched if it could present a safety hazard. Damaged glassware is put aside for repair, otherwise discard the piece.

5.7 All Field Samples must be opened and handled in a hood.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Sample collection and preservation requirements are described in the various analytical method SOPs.

6.2 Sample Preservation

None.

6.3 Sample Shipping

See applicable Sample Custody SOP.

6.4 Sample Handling

All aqueous samples are stored, refrigerated, in the Organic Extraction Custody Refrigerators. Samples are removed from the refrigerator by the Chemist immediately prior to sample extraction. The Chemist must take custody of the samples by signing them out utilizing the LIMS, see Work Instruction 2517 ELN Procedure and Work Instruction 2421 Labeling and Generating Work Groups and Batches.

Visually inspect the samples prior to starting the extraction process, as described in Section 10.1. Typically the entire content of the 1L amber jar is used for extraction. After the sample or sample aliquot is measured, the samples or empty sample containers are scanned to “empty” or returned to the Refrigerator.

7. Equipment and Supplies

7.1 Separatory Funnel: 2 or 3-Liter, glass or Teflon, with polytetrafluoroethylene (PTFE) stopcock and cap. 3L separatory funnels are to be used for extractions requiring 2L of sample.

7.2 Erlenmeyer Flasks: 250 and 500 mL.

7.3 Centrifuge Tubes.

7.4 Syringes: 1mL, 250µL, 100µL

7.5 Sodium Sulfate Stainless Steel filtering funnels. Add a plug of glass wool to the base of the 104mm glass funnel. Add approximately 60grams of baked sodium sulfate.

7.6 Glass wool.

7.7 Kuderna-Danish (KD) Apparatus: Assemble by attaching the Concentrator Tube to the Evaporation Flask using the Plastic clip. Add the Macro column to the Evaporation Flask. The Micro Snyder Column is attached directly to the Concentrator Tube using the Plastic Clip.

7.7.1 Evaporation Flask: 500mL KD flask.

7.7.2 Concentrator Tube: 25mL, graduated.

7.7.3 3-Ball Macro Snyder Column.

7.7.4 Micro Snyder Column.

7.7.5 Plastic Kek clips.

7.8 Boiling Chips: Solvent extracted, approximately 10/40 mesh (silicon carbide, or equivalent).

7.9 Graduated Cylinders: 25, 50, 100, 250, 500, and 1000 mL, class "A".

7.10 S-EVAP Water Bath with Solvent Collection Capability: Heated. Capable of temperature control (0.1C). Baths are located in a hood. Baths are equipped with chilled water condensers for solvent collection.

7.11 Buchi Concentration System: Base Unit, Chiller, Pump, Block, Controller and 180mL Glass Vessels.

7.12 N-EVAP: Organomation; utilized for micro blow down.

7.13 pH Paper: Multibanded, wide range.

7.14 Filter Paper: Whatman #4 185mm

7.15 Screw-top vials: 22mL volume.

7.16 Automatic Separatory Funnel Shaker

7.17 KI Paper Strips: 0.05mg/L residual chlorine sensitivity.

7.18 Multi-Position Stirring Plates.

7.19 Magnetic Stirring Bars.

7.20 Brady Labelling System: Thermal label generator.

8. Reagents and Standards

Pesticide or reagent grade chemicals are used in all tests. All reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.1 Reagent Water: All references to water in this method refer to reagent water from Alpha's DI water treatment system.

8.2 Sodium hydroxide solution (10N), NaOH: Dissolve 400g NaOH in reagent water and dilute to 1000mL. For basification of samples. Reagent expires one year after preparation.

8.3 Sulfuric acid solution (1:1 v/v), H_2SO_4: Prepare by slowly adding 500mL of concentrated H_2SO_4 to 500mL of reagent water, in a 1-Liter beaker placed in an ice water bath. For acidification of all non-EPH samples. Expires 1 year from date of preparation.

8.4 Hydrochloric Acid, 6N, 2:1: Place a 2000mL Beaker or equivalent in an ice water bath. Add 50mL of DI water to the beaker. Slowly add 100mL of Concentrated HCL. Mix with a stirring rod and allow to cool in a hood. Used for the acidification of EPH samples. Expires 1 year from date of preparation.

8.5 Sodium Sulfate (Na_2SO_4): Granular anhydrous; purified by baking at 400ºC for 4 hours in a shallow tray. Store in closed glass containers. All references to sodium sulfate in this method refer to this prepared reagent.

8.6 Methylene Chloride: Pesticide quality or equivalent. No expiration date listed.

8.7 Hexane: Pesticide quality or equivalent. No expiration date listed.

8.8 Acetone: Pesticide quality or equivalent. No expiration date listed.

8.9 1:1 Acetone/Methylene Chloride: Using a Graduated Cylinder measure 2 Liters of Acetone and transfer into a 4-Liter glass bottle. Using a graduated cylinder, add 2 Liters of Methylene Chloride into the same 4-Liter bottle. Mix.

8.10 Spiking Solutions: The various surrogate and LCS/MS spiking solutions used in the extraction steps are listed in the Separatory Funnel Extraction Guide WI# 19781. The preparation and expiration dates of these solutions are described in the analytical SOPs.

8.11 Sodium Thiosulfate Crystals ($Na_2S_2O_3$): J.T. Baker; 5-Hydrate crystal.

8.12 Silica Gel: VWR, Cat# TX4694MAAA. 60 - 200 mesh, chromatography grade. Activated by baking at 140 °C for a minimum of 14 hours in a shallow tray. The silica gel is stored in the oven or desiccator until ready for use. All references to silica gel in this method refer to this prepared reagent.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

Each extraction batch contains various QC samples used to ensure the validity of the sample results. The particular QC elements performed for a given extraction batch are determined by the requirements of the determinative method. The purpose and definition of the QC samples performed are listed below. The specific QC requirements of the analytical methods are listed in WI/19781 Separatory Funnel Extraction Guide.

9.1 Blank

Blanks, or method blanks, are measured aliquots of reagent water (for aqueous extractions) that are treated identically to the associated samples. Surrogates are added, and the blanks are carried through all stages of the sample extraction, concentration, and cleanup procedures. Blanks serve to ensure that no systematic contamination exists. A blank is extracted with each batch of 20 or less samples.

For 608 and 625 a blank is extracted with each batch of 10 or less samples.

For 608.3 and 625.1 a blank is extracted with each batch of 20 or less samples.

9.2 Laboratory Control Sample (LCS/LCSD)

LCS samples are measured aliquots of reagent water (for aqueous extractions) that are spiked with a solution containing known amounts of target compounds, in addition to the surrogate solution. The LCS is carried through all stages of the sample extraction, concentration, and cleanup procedures. LCS samples serve as batch specific quantitative checks of the extraction. An LCS is extracted with each batch of 20 or less samples.

For samples to be analyzed by EPA 608 and 625, a LCS is extracted with each batch of 10 or less samples.

For samples to be analyzed by EPA 608.3 and 625.1, a LCS is extracted with each batch of 20 or less samples.

An LCSD is performed in addition to an LCS for most methods, as well as in lieu of the MS/MSD or Duplicate when there is insufficient sample volume available. The required solutions and volumes are listed in the Separatory Funnel Extraction Guide WI# 19781.

9.3 Initial Calibration Verification (ICV)

Not Applicable.

9.4 Continuing Calibration Verification (CCV)

Not Applicable.

9.5 Matrix Spike

MS and MSDs are field samples spiked with a known quantity of the target analyte(s). They are prepared by taking additional sample aliquots, and adding the appropriate amounts of surrogate and spiking solutions. The MS/MSD are carried through all stages of the sample extraction, concentration, and cleanup procedures. MS samples serve as a measure of extraction accuracy, by allowing the comparison of the found amount(s) of target analyte(s) with the spiked amount(s). An MS/MSD set also allows for the calculation of the extraction precision, by comparing the results of the two samples.

For samples to be analyzed by EPA 608 and 625, a MS is extracted with batch of 10 samples or less.

For samples to be analyzed by EPA 608.3 and 625.1, a MS is extracted with batch of 20 samples or less.

For samples with a state of origin of New Jersey, a MS and MSD/DUP must be extracted for every twenty samples within a 24hr period.

9.6 Laboratory Duplicate

Duplicates are laboratory selected replicate samples, prepared by taking an additional sample aliquot of a sample. The duplicate is carried through all stages of the sample extraction, concentration, and cleanup procedures. Duplicates serve as a measure of the extraction precision, by comparing the results of the sample and duplicate.

For samples to be analyzed by EPA 608 and 625, a DUP is extracted with batch of 10 samples or less.

For samples to be analyzed by EPA 608.3 and 625.1, a DUP is extracted with batch of 20 samples or less.

For samples with a state of origin of New Jersey, a MS and MSD/DUP must be extracted for every twenty samples within a 24hr period.

9.7 Method-specific Quality Control Samples

9.7.1 Surrogates

Surrogates are compounds specified by the analytical method that are added to all samples and QC samples prior to beginning the extraction process. Surrogate recoveries are calculated and serve as a sample specific quantitative check of the extraction. The various spiking solutions are prepared according to the directions found in the analytical SOPs. The required solutions and volumes used are listed in WI/19781 Separatory Funnel Extraction Guide.

9.8 Method Sequence

See Section 10.

10. Procedure

All glassware and Separatory Funnels must be cleaned following the procedure described in the glassware washing SOP/1953. In addition, the glassware must be rinsed with acetone and methylene chloride, or baked until reaching a temperature of 400C just prior to use. The Separatory Funnels are rinsed with a 1:1 mixture of Acetone/Methylene Chloride.

All water extractions follow the LEAN “one-piece flow”. All extraction information is recorded by the chemist performing the work in the ELN (Electronic Lab Notebook) see WI/2517. In addition to recording the extraction, concentration, clean-up and vialing information, the analyst must note any anomalies during the extraction procedure in the comments section of the ELN. Generating Work Groups, Batches and Labeling is described in Work Instruction WI/2421.

10.1 Sample Extraction

10.1.1 Carefully examine the sample prior to beginning the extraction process. The sample should be a single phase, with minimal or no sediment or solid material present. If this is not the case, contact the Extraction Lab Supervisor or Manager to determine how to handle the sample. The supervisor may need to contact login or the project manager to determine how the client would like the sample handled.

10.1.2 Common volumes for extraction include 140mL, 275mL, 500mL, 1000mL, 1200mL or 2000mL of sample to meet reporting limits. If 1200mL is required, this will be specified by the client. The containers that samples are received in will determine the extraction volume and procedure (I.e. standard procedure versus low volume initiative).

10.1.3 If the sample volume is less than the top of the sample collection bottle, do not use another container to bring it to 1L. Mark the current volume level on the sample bottle using a permanent marker for later volume determination.

10.1.4 Field and QC samples being analyzed for either EPA 608, EPA 625, EPA 625.1 or EPA 608.3 must be checked for residual chlorine prior to any pH adjustments or extraction. Invert the sample several times to ensure that sample is well mixed, then dip one KI test strip into sample for 10 seconds with a gentle constant back and forth motion. Wait 30 seconds and then refer to the chart on the KI strip container, if chlorine is detected at a level less than 0.1mg/L proceed with the extraction. Otherwise, record in the ELN that the TRC is positive (+). Then add sodium thiosulfate to the sample, mix and re-check, repeat until the chlorine level is less than 0.1mg/L. Record in the ELN.

10.1.5 For One-piece flow sample processing and labeling, see Labeling and Generating Work Groups and Batches Work Instruction WI/2421 and Separatory Funnel Extraction Guide WI/19781.

10.1.6 Using an appropriate size Class "A" Graduated Cylinder, measure the correct amount of reagent water for blanks and LCSs. For QC requirements, see WI/19781 Separatory Funnel Extraction Guide. For samples, use the entire sample bottle (See10.1.2). Unless high analyte concentrations are anticipated, a smaller sample volume may be taken and diluted to the appropriate volume with reagent water. On occasion, the client may provide limted sample volumes, note this in the ELN. If the sample volume is higher than 3/4 of the container, then mark the water level on the amber and use the entire contents. If the sample volume is less than 3/4 of the container mark the water level on the amber and add reagent water to reach a final volume appropriate for the container (e.g. 1000mL, 500mL, 275mL, 140mL, etc.). Notate in the ELN the sample volume extracted and addition of DI water.

10.1.6.1 See WI# 19781 Separatory Funnel Extraction Guide for specific extraction volumes and spiking requirements. Extraction volume may affect the extract final volume (see WI#18528 S-Evap/N-Evap Concentration Standard Process or WI# 19781 Separatory Funnel Extraction Guide).

10.1.6.2 The for TCLP samples, TCLP fluid is used instead of DI water for the QC. For SPLP samples, QC will require the use of SPLP fluid.

10.1.7 Transfer the sample from the sample container or graduated cylinder into a labeled 2L separatory funnel for all extraction methods requiring less than 2L of sample volume. For extraction methods that require 2L of sample volume use a 3L glass separatory funnel.

10.1.7.1 A colored dot should be placed on the separatory funnel to match the ring stand above the collection glassware for each sample. This is used as a tool to prevent samples being drained into the incorrect collection vessel.

10.1.8 Adjust the pH, if necessary, to the pH indicated in Table 1 or WI# 19781 Separatory Funnel Extraction Guide (including QC samples), using the base and acid solutions listed in Sections 8.2-8.4. (**NOTE:** Do not alter the pH for neutral extractions.) The amount of acid or base required to achieve the desired pH is highly sample dependant, but is typically 1-10 mL.

10.1.9 Using the appropriately sized syringe, add the appropriate volume(s) of the surrogate/spiking solutions to the sample/QC as required; see WI/19781 Separatory Funnel Extraction Guide.

10.1.10 If there is no significant sediment or non-aqueous material present in the sample container, use methylene chloride to rinse the sample cylinder (or bottle) and transfer this rinseate into the separatory funnel**.**If the sample container does contain sediment or solid material that is not considered part of the sample, add methylene chloride directly into the separatory funnel. A comment must be placed in the ELN stating that the bottle was not rinsed due to the presence of sediment in the container. **NOTE:** Depending on the volume of the container that the sample is received in, it will determine the solvent volume used to rinse the container/extract the sample. See WI# 19781 Separatory Funnel Extraction Guide.

10.1.11 If the sample was transferred directly from the sample bottle, refill the bottle with water to the mark made in Section 10.1.3 and measure the volume using a graduated cylinder. Record the volume of sample that was in the bottle into the ELN.

10.1.12 Tightly cap the separatory funnel and vent the separatory funnel into a hood. Place the funnel into the holder on the Automatic Shaker, cap upward. Secure the locks. Repeat

steps 10.1.6-10.1.10 for the remaining samples to be extracted. **NOTE:** If extracting using the 3L glass separatory funnel, the chemist must shake the vessel by hand.

10.1.13 Turn the unit on. Press 'Select' three times to open the timing menu. Press the 'up arrow' to set the number of minutes to be equal to '2'.

NOTE: For EPH extractions set the number of minutes to be equal to '3'.

10.1.14 Press "Start". Press the 'up arrow' to set the rpm to be equal to '150'.

10.1.15 When the Automatic Shaker stops, allow the organic layer to separate from the water phase for a minimum of 10 minutes. Remove the first separatory funnel and align in the hood over the appropriate collection vessel (see WI/2421). If the emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample and may include stirring, filtration of the emulsion through glass wool, centrifugation, addition of sodium chloride, or other physical methods.

10.1.16 **Before decanting the solvent layer**, check the pH of the sample with wide-range pH paper and record the value in the ELN. If the pH of the sample has not been satisfied as required by the determinative method, re-adjust the pH as indicated in Table1 or WI# 19781 Separatory Funnel Extraction Guide, using the base and acid solution. Repeat sections 10.1.10-10.1.13 until the appropriate pH is reached. Do not drain the solvent layer until the appropriate pH is reached.

10.1.17 Filter the extract through a funnel packed with glass wool and one 1/4cup scoop of sodium sulfate, collecting the filtrate in a the appropriate concentration glassware with the appropriate sample label. See WI 10995, Solvent rinsing/filtering guide. Add the same amount (60 or 30mL) of solvent to the separatory funnel (unless it is a 625 or 608. See 10.1.17.3) and place the sample back on the shaker table. Repeat steps 10.1.14 through 10.1.16 for each sample on the shaker table.

10.1.17.1 Note: The chemist should drain the solvent until approximately 1mL of solvent remains in the separatory funnel in order to prevent water from reaching the sodium sulfate funnel.

10.1.17.2 Note: Due to contamination issues, pesticide samples should be filtered through a funnel containing a paper filter with one 1/4cup scoop of sodium sulfate, rather than glasswool.

10.1.17.3 For 625 and 608 samples, DCM must be added to the sample container before it is transferred to the separatory funnel before every shake, not just the first shake.

10.1.18 For all analyses repeat the extractions two more times for two minutes each, using fresh portions of solvent (Section 10.1.10-10.1.16). Collect all three solvent extract portions in the same labeled collection vessel (KD apparatus, Erlenmeyer flask. or Buchi vessel).

10.1.18.1 For EPH and EPH-TPH-10 analyses, repeat the extractions two more times for three minutes each, using fresh portions of solvent (Section10.1.9-10.1.15).

10.1.19 If additional pH adjustment during extraction is required (ex. ABN), adjust the pH of the aqueous phase to the desired pH indicated in Table 1 or WI# 19781 Separatory Funnel Extraction Guide. Repeat the extraction process Section 10.1.9-10.1.16.

10.1.19.1 For ETPH and EPH-TPH-10 analyses, the sample extract is filtered through a stainless steel funnel containing glass wool and approximately 60g of sodium sulfate into a 250mL Erlenmeyer flask. Add 3 grams of activated Silica Gel and a stir bar to the extract. Place the sample on a stirring plate and stir for 5 minutes at 650 RPM. Filter the extract through a stainless steel funnel

containing filter paper, collecting the filtrate in a Buchi vessel for concentration (See Section 10.2.3).

10.1.19.2 Proceed to sample concentration. Note all DRO, ETPH, and EPH-TPH-10 products are concentrated using the Bucchi Concentration System.

10.2 Sample Concentration Techniques

10.2.1 KD Technique: For samples requiring hexane exchange (See Table 1).

10.2.1.1 S-Evap baths are heated to 75°C and 95°C. Attach a three-ball Snyder column to the top of the flask. Place the KD apparatus on the hot water bath heated to approximately 75°C so that the concentrator tube is partially immersed in the hot water, and so that the entire lower rounded surface of the flask is bathed in hot water vapor. Attach the chilled water condenser to the top of the Synder Column. Adjust the position of the apparatus as required to complete the concentration. At the proper rate of distillation, the balls in the column will actively chatter, but the chambers will not flood with solvent.

10.2.1.2 When the sample volume reaches 10-15mL, remove the condenser from the Synder Column and add 25mL of hexane using a graduated cylinder. Add the hexane to the top of the Snyder Column. Allow the sample to continue to concentrate until the volume reaches 10-15mL or boiling reduces in intensity. Add an additional 25mL of hexane and allow the sample to become fully exchanged with hexane (boiling reduces in intensity and the Snyder columns stop chattering). Remove the KD concentration setup and move to the 95C bath. Re-attach the condenser and continue with the concentration until the extract volume is reduced to 10-15mL.

10.2.1.3 Remove the KD apparatus from the water bath. Wipe the flask and its lower ground glass joint with a dry paper towel to remove any moisture from the outside of the glassware. Allow to cool before disassembling the KD apparatus. Move the sample label from the KD Flask to the concentrator tube (See WI/2421).

10.2.1.4 Place the Concentrator tube on the N-EVAP. The N-EVAP is set at 65 °C for samples extracted in Hexane with the nitrogen flow at 5 - 7. Samples remain on N-EVAP until they are reduced to the appropriate final volume (see S-Evap/N-Evap Concentration Standard Process, WI#18528).

10.2.1.5 The extract is now ready for sample cleanup or vialing (See Table 1). Refer to the relevant Clean-up SOP or proceed with extract vialing (See WI/3827, Extract Vialing Procedure, WI/2426, GC Extract Vialing Procedure and WI/2423, GC/MS Extract Vialing Procedure).

10.2.2 Alternative Concentration Technique: Buchi

The Buchi is a self-contained sample concentration and solvent recovery system that utilizes vacuum, heat and oscillation to concentrate samples. The Buchi will recover >95% of solvent emissions.

10.2.2.1 Setup: All DRO, ETPH, EPH-TPH-10, and 8270/SIM products require Buchi concentration. The samples are filtered into the Buchi concentration vessels. The samples are equalized with the extraction solvent. Soil samples will be adjusted to 80mL, while liquid samples will have a 180mL volume. This will allow for even concentration.

10.2.2.2 Empty or drain the solvent collection flask into the appropriate hazardous waste container.

10.2.2.3 Turn “on” the Base Unit, Chiller (set at 5˚C), Pump, Block and Controller. Push “start” button on the chiller to begin circulation and chilling of fluid. Push the “start” button on the block once and turn “off” the oscillation. This will begin cooling the condenser. Pre-heat the block to 65C as noted on the panel readout.

10.2.2.4 On the Controller, press program #1(Gradient and DCM program should be selected). This program will show the following, DCM 50min., 600MBAR for 1 minute, 540MBAR for 17min., and 460MBAR for 32 min. Total run time will be 50 minutes.

10.2.2.5 Add 5mL of water into each block cell position. Place the labeled samples (glass vessel) into the block. Note, all positions on the block must have a vessel in order to form a vacuum. Use empty vessels if necessary to fill the 12-position block.

10.2.2.6 Place the block lid on top of the sample extracts and aligning the set screws to the holes in the lid. Tighten the two knobs on to the block, hand tight. Tighten the knobs evenly or the lid will not form a vacuum.

10.2.2.7 Push “Start” on the controller. The controller should immediately drop and read 600MBAR. If the vacuum does not drop, there is a leak with the vessels. Reposition and retry.

10.2.2.8 Check the sample solvent level after 50min., by shutting off the controller and removing the lid. Remove samples that have a final volume 1.0mL. Place an empty vessel in its place and change the program to “Touchup”. If necessary, restart the program and check the solvent volume every 5minutes. Note, by looking at the solvent condensing rate on the condenser, you can gauge the solvent remaining in the vessel.

10.2.2.9 Once all samples have been concentrated to 1.0mL, the extract is ready to be vialed. Refer to the relevant Clean-up SOP or proceed with extract vialing (See WI 3827 Extract Vialing Procedure, WI 2426 GC Extract Vialing Procedure and WI 2423 GC/MS Extract Vialing Procedure).

10.2.2.10 Samples requiring a final volume of less than 1mL should be transferred to a concentrator tube and placed on the N-Evap until the required volume is reached.

10.2.2.11 Samples that will not concentrate to the required final volume should be transferred to a concentrator tube and placed on the N-Evap in order to try to reduce the volume further. Samples that will not continue to concentrate should have the final volume documented in the ELN and proceed to the relevant clean-up or extract vialing procedure.

10.3 Preventive Maintenance

10.3.1 Buchi Concentrators

10.3.1.1 **Refrigeration Re-circulator:** The Refrigeration Re-circulator should be checked once every shift (when in use) to insure it is at the specified temperature, running correctly and that the level of coolant is constant with manufactures recommendation.

10.3.1.2 **Buchi System**: Contact factory representative with inquiries and maintenance issues.

10.3.1.2.1 Buchi Vacuum Cover: The Buchi unit vacuum cover requires cleaning in a fume hood using acetone.

10.3.2 Water Bath

10.3.2.1 The water bath should be kept full at all times. Add reagent water as necessary.

10.3.2.2 Keep unit clean. Avoid solvent spills on or around unit. Clean periodically with a damp cloth.

11. Data Evaluation, Calculations and Reporting

Not Applicable.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedence, improper preservation and observed sample headspace are noted on the nonconformance report form.

When analysis of samples indicates possible extraction problems, such as poor surrogate recoveries, poor LCS/MS/MSD recoveries, or suspected contamination in blanks or samples, re-extractions are required. Depending on the particular failure, the re-extraction may be of a specific sample or the entire extraction batch.

The analyst that determines the need for re-extraction must fill out a sample re-extract request form. This form notes the reason for the re-extraction request along with any special requirements, and the date and time that the re-extract is needed. Re-extraction request forms are maintained on file to help track the cause for re-extractions, and to be used as a tool in improving systems to minimize the need for re-extractions.

Depending on the results of the re-extraction, the first, second, or both sets of results may be reported to the client, along with a narrative report detailing the problems encountered and the resolution.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

SOP/2121 Chemical Hygiene Plan

SOP/1732 DL/LOD/LOQ Generation

SOP/1739 DOC Generation

SOP/1728 Waste Management and Disposal SOP

SOP/1953 Organic Extraction Glassware Cleaning and Handling

WI/2421 Labeling and Generating Work Groups and Batches

WI/2517 LIMS Electronic Laboratory Notebook Procedure

WI/3827 Extract Vialing Procedure

WI/10995 Solvent Rinsing/Filtering

WI/19781 Separatory Funnel Extraction Guide

16. Attachments

Table 1 – Specific Extraction Conditions for Various Determinative Methods

Table 1

Specific Extraction Conditions for Various Determinative Methods

Determinative Method	Initial Extraction pH	Secondary Extraction pH	Extraction Solvent	Concentration method	Final Volume	Appropriate Cleanup Technique
Pest/8081	5 – 9	None	DCM	S-Evap/N-Evap	1, 5,10 mL	Florisil *b*c
PCB/8082	5 – 9	None	DCM	S-Evap/N-Evap	1, 5 mL	Sulfuric acid/Copper
608	5 – 9	None	DCM	S-Evap/N-Evap	1, 5, 10mL	Florisil *b
608.3	5 – 9	None	DCM	S-Evap/N-Evap	1, 5, 10mL	Florisil *b
8270	< 2	> 11	DCM	Buchi	1 mL	n/a
625	< 2	> 11	DCM	Buchi	1 mL	n/a
625.1	< 2	> 11	DCM	Buchi	1 mL	n/a
TPH-DRO	As received	None	DCM	Buchi	1 mL	n/a
NJ TPH	As received	None	DCM	S-Evap/N-Evap	1 mL	n/a
MA-EPH	< 2	None	DCM	S-Evap/N-Evap	1 mL	Silica Gel Fractionation
NJ-EPH	<2	None	DCM	S-Evap/N-Evap	1mL	Silica Gel Fractionation
EPH-TPH-10	<2	None	DCM	Buchi	1mL	Silica Gel
CT-ETPH	As received	None	DCM	Buchi	1 mL	Silica Gel
Herbicide	See relevant SOP					

- 8270, 625, and 625.1 methods are for extraction of both acidic and base/neutral compounds.
- *b - Sample extracts may require additional cleanup by Method 3660A to remove elemental sulfur, as determined by the sample extract appearance once florisil cleanup has been performed.
- *c – Liquid matrix samples do not require florisil cleanup unless requested by the client or required for analysis (exception: Pest-608).
- For the full list of products in each category, see Separatory Extraction Guide WI#19781.

For additional products, QC's, solvent, reagents, surrogate/spike solutions, and extraction volumes see Qualtrax ID 19781 "Separatory Extraction Guide."

TCLP Extraction

Metals and Semi-Volatile Organics

Reference Method: **EPA 1311**

Reference: **SW-846**, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Update I, July 1992

State of Connecticut, DEP, TCLP by SW-846 Method 1311, Version 2.0, December 2006

1. Scope and Application

The TCLP is designed to determine the mobility of both organic and inorganic analytes present in liquid, solid, and multiphasic wastes.

Definitions: See Alpha Laboratories Quality Manual Appendix A.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Extractions Manager, Laboratory Services Manager, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

For liquid wastes, (i.e., those containing less than 0.5% dry solid material), the waste, after filtration through a 0.6 to 0.8 μm glass fiber filter, is defined as the TCLP extract.

For wastes containing greater than or equal to 0.5% solids, the liquid, if any, is separated from the solid phase and stored for later analysis; the particle size of the solid phase is reduced, if necessary. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. The extraction fluid employed is a function of the alkalinity of the solid phase of the waste. Following extraction, the liquid extract is separated from the solid phase by filtration through a 0.6 to 0.8 μm glass fiber filter.

If compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract, and these are analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

For additional detailed instruction, see TCLP and SPLP Work Instruction (ID# 17618).

2.1 Method Modifications from Reference

None.

3. Reporting Limits

The Reported Detection Limit is determined by the amount of sample used for preparation. Therefore a review of Client requirements for Reporting Limits is necessary prior to sample preparation. Refer to analytical method SOPs.

4. Interferences

4.1 The most common cause of contamination is from improperly cleaned glassware and lab supplies. All glassware and re-useable extraction equipment must be scrupulously cleaned, following the Organic Extraction Glassware Cleaning and Handling SOP/1953 and Work Instruction 10995, Solvent rinsing guide.

4.2 Impurities in solvents and reagents may also yield artifacts and/or interferences that may compromise the results of sample analysis. All of these materials must be demonstrated to be free from interferences under the conditions of extract preparation and analysis by preparing method blanks with each extraction batch. The same solvents and reagents are used for the method blank and the associated samples.

4.3 Phthalate esters contaminate many types of products used in the laboratory. Plastic materials must not contact the samples or extracts, as phthalates could be easily leached from the plastic. The exception is in the use of various pre-packed reagent cartridges (Florisil, Silica gel) used in the extract cleanup steps. Each new lot of cartridges is checked for contamination, and is monitored on an on-going basis through the analysis of method blanks.

4.4 Additional specific interference or contamination concerns are addressed in the various analytical SOPs.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents. This includes wearing personal protective equipment such as a labcoat, safety glasses, gloves and respirator (as necessary).

5.1 Lab coats, safety glasses, and gloves must be worn when handling samples, extracts, standards or solvents and when washing glassware.

5.2 All extract concentration steps must be performed in the extraction hoods. All solvent and extract transfers must also be handled in the hood.

5.3 All expired stock standards, working standards, and spent sample extracts must be placed into the waste bucket in the lab, for future disposal by the Hazardous Waste Manager. The container must be properly labeled with hazard warning labels indicating the container contents.

5.4 Bottles containing flammable solvents must be stored in the flammables cabinet or in the vented cabinets found under the hoods.

5.5 All waste solvents must be transferred to the satellite waste storage containers located in the extraction lab. Separate containers are provided for chlorinated and non-chlorinated solvents and must be used accordingly. Under no circumstances are solvents to be poured down the sink drains.

5.6 Inspect all glassware prior to use. Do not use any glassware that is chipped, cracked or etched if it could present a safety hazard. Damaged glassware is put aside for repair, otherwise discard the piece.

5.7 All Field Samples must be opened and handled in a hood.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples are collected in glass containers. Soils are collected in 8oz amber glass jars, and aqueous samples are collected in 500mL amber glass jars.

6.2 Sample Preservation

Preservatives are not added to samples before extraction.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

Samples may be refrigerated at 4 ± 2°C, unless refrigeration results in irreversible physical change to the waste. If precipitation occurs, the entire sample (including precipitate) should be extracted.

Samples must undergo TCLP extraction within the time periods listed in Table 1.

7. Equipment and Supplies

7.1 Agitation Apparatus: End-over-end 30 ± 2 rpm. Calibrated Quarterly.

7.2 Vacuum Filtration Unit

7.3 0.6 to 0.8 µm Glass Fiber Filter Paper: 70mm, 90mm, and 120mm, and acid prewashed 47mm for all metals analysis.

7.4 2L Glass/Plastic-coated Bottles

7.5 pH Meter: +/- 0.01 units resolution

7.6 pH test paper: 0-14 pH range

7.7 Laboratory Balance: +/- 0.1 g tolerance

7.8 250mL Beaker or equivalent

7.9 Watch Glass: Used with Beaker (Section 7.8)

7.10 Stirbars

7.11 Hot Plate: 1x1ft (120V) Barnestead-Thermolyne – Type 2200

7.12 2L Graduated Cylinder Class A

7.13 250mL Graduated Cylinder Class A

7.14 Teflon coated Sieve: 9.5mm (0.375-inch)

7.15 Oven: Capable of maintaining 100 ± 2°C

7.16 Automatic Shaker Table

7.17 Vessel to store fluid #1

7.18 Vessel to store fluid #2

7.19 Calibrated thermometer: Temperature calibration point 50°C. Range: 0-100°C.

7.20 Bottle Top Dispenser: 0-5ml, 0-100mL

7.21 1L Amber Bottle: Thermo Scientific, Item # 341-0950, Jar Tall Amber WM.

7.22 120mL Plastic Bottle: Greenwood, Product # 07-120WMF24503, Natural HDPE WM Packer assembled w/38-400 F-217 Lined Cap.

7.23 250mL Plastic Bottle: Greenwood, Product # 07-250OB45F22603, Natural HDPE WM Oblong Bottle Assembled w/45-400 F-217 Lined Cap.

8. Reagents and Standards

8.1 Hydrochloric Acid: 1 N: Dilute 83 ml conc. HCl to 1 liter with reagent water. Store at room temperature; expires one year from date of prep.

8.2 Sodium Hydroxide: 1N: Dissolve 40 grams NaOH in 1 liter of reagent water. If larger volumes are required, prepare using similar ratios. Store at room temperature; expires one year from date of prep.

8.3 Glacial acetic acid: CH_3COOH. Store at room temperature; expires according to manufacturer's specifications.

8.4 Reagent Water, (ASTM Type II): All references to water within this SOP refer to reagent water unless otherwise specified. Reagent water is interference-free.

8.5 Extraction Fluid #1: Add 5.7 mL glacial acetic acid to 500 mL of reagent water, add 64.3 mL of 1 N NaOH, and dilute to a volume of 1 liter. Check the pH of the solution after preparation and record the value in the Extraction Fluid Logbook. The pH is within 4.93 $\pm$ 0.05 when correctly prepared. In addition, the pH of the fluid is verified prior to each use and the pH recorded in the extraction logbook. Store at room temperature; no expiration date, however the pH must be maintained.

NOTE: Larger volumes or extraction fluid may be prepared, provided the ratios remain the same. For example, a typical preparation of 50L of Extraction Fluid #1:

46500mL of reagent water + 285 mL Glacial Acetic Acid + 3215mL 1N NaOH

8.6 Extraction Fluid #2: Dilute 5.7 mL glacial acetic with reagent water to a volume of 1 liter. Check pH of solution which should be 2.88 ± 0.05 when correctly prepared. Record the pH in the Extraction Fluid Logbook.

The pH is checked prior to use to ensure that this fluid is prepared accurately. If the pH is not within the above specifications, the fluid is discarded and fresh extraction fluid prepared. Store at room temperature; no expiration date, however the pH must be maintained.

NOTE: Larger volumes or extraction fluid may be prepared, provided the ratios remain the same. For example, a typical preparation of 20L or 50L Extraction Fluid #2:

19,886mL of reagent water + 114mL Glacial Acetic Acid = 20L of fluid.

49,715mL of reagent water + 285mL Glacial Acetic Acid = 20L of fluid

8.7 Trace Nitric Acid (1:1 HNO3): 500mL tHNO3 diluted to 1 liter with DI water. Store at room temperature in hood. Expires one year from date of prep.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

A minimum of **one blank** (using the same extraction fluid as used for the samples) must be prepared and analyzed for every batch or 20 extracts, whichever comes first. The Blank vessel is a randomly selected container from the stock and is not one designated for blank use only.

9.2 Laboratory Control Sample (LCS)

Not applicable.

9.3 Initial Calibration Verification (ICV)

Not applicable.

9.4 Continuing Calibration Verification (CCV)

Not applicable.

9.5 Matrix Spike

Not applicable.

9.6 Laboratory Duplicate

Not applicable.

9.7 Method-specific Quality Control Samples

None.

9.8 Method Sequence

- Determine analysis to be performed (Metals, ABN, Pest, PCB, Herbicides)
- Check sample; ensure there is enough sample for the extraction
- Determine the %solids of the waste sample

- Determine particle size reduction requirement
- Determine the extraction fluid to be used
- Extract the sample for TCLP
- Filter the extract.
- Aliquot extract to proper preparation departments
- Prepare extracts according to analytical protocols

10. Procedure

10.1 Equipment Set-up

10.1.1 Preliminary Evaluation

Perform preliminary TCLP evaluations on a minimum of 100g aliquot of waste. If there is limited sample volume, the chemist must contact the project manager. This aliquot may not actually undergo TCLP extraction. These preliminary evaluations include:

(1) determination of the percent solids (Section 10.1.1.1);

(2) determination of whether the waste contains insignificant solids and is, therefore, its own extract after filtration (Section 10.1.1.1.2);

(3) determination of whether the solid portion of the waste requires particle size reduction (Section 10.1.1.2); and

(4) determination of which of the two extraction fluids are to be used for the nonvolatile TCLP extraction of the waste (Section 10.1.1.3)

10.1.1.1 Determine % Solids

10.1.1.1.1 If waste contains no free liquid, proceed to Section 10.1.1.2 where the waste is the solid portion. If the waste contains less than 0.5% solids, proceed to Section 10.1.1.1.2.

10.1.1.1.2 Mulitphasic waste - separate liquid and solid portion

10.1.1.1.2.1 Pre-weigh a filter. Pre-weigh a beaker or graduated cylinder that will hold the initial filtrate. Record both weights in the extraction logbook.

10.1.1.1.2.2 Assemble the vacuum filtration unit.

10.1.1.1.2.3 Weigh a subsample of the waste (100 gram minimum), or pour 100mL of the sample into a cylinder, and record the weight or volume in the extraction logbook.

10.1.1.1.2.4 Transfer the waste sample to the filter holder, spreading evenly over the filter.

10.1.1.1.2.5 Allow enough time for the liquid to pass through the filter (at least 15 minutes).

10.1.1.1.2.6 The material in the filter holder is defined as the SOLID PHASE of waste and the filtrate is defined as the LIQUID PHASE.

Note: The SOLID PHASE may appear liquid in some samples. Do Not replace original filter. Use only one filter.

10.1.1.1.2.7 Weigh the filtrate filled container and calculate the weight of the LIQUID PHASE as follows:

$$W_{LP} = W_F - W_C$$

Where:

W_{LP} = Weight of the LIQUID PHASE
W_F = Weight of the filtrate filled container
W_C = Weight of container

10.1.1.1.2.8 Calculate the weight of the SOLID PHASE using the following formula:

$$W_{SP} = W_W - W_{LP}$$

Where:

W_{SP} = Weight of the SOLID PHASE
W_W = Weight of the waste sample (Section 10.1.1.1.2.3)
W_{LP} = Weight of the LIQUID PHASE (Section 10.1.1.1.2.7)

10.1.1.1.2.9 Calculate the percent solids using the following formula:

$$\%\ \text{Solids} = \frac{W_{sp}}{W_w \times 100}$$

Where:

W_{SP} = Weight of the SOLID PHASE (Section 10.1.1.1.2.8)
W_W = Weight of the waste sample (Section 10.1.1.1.2.3)

10.1.1.1.2.10 If the % Solids determined above, section, is less than 0.5%, then the filtrate is the TCLP extract. Filter sample until enough filtrate is obtained for analysis and proceed to Section 10.3.1.3.6.

10.1.1.1.2.11 If the % Solids determined above in section is greater than 0.5%, all liquids entrained in the filter and waste must be removed to determine the % dry solids. Remove the solid phase and filter from the filtration apparatus, dry at 100 $\pm$ 2° C, cool and weigh. Record weight in the extraction logbook.

10.1.1.1.2.12 Calculate the percent dry solids using the following formula:

$$\%\ \text{dry solids} = \frac{W_{DW} - W_F}{W_W \times 100}$$

Where:

$\mathbf{W_{DW}}$ = Weight of dried waste + filter (Section 10.1.1.1.2.11)
$\mathbf{W_F}$ = Weight of the filter (Section 10.1.1.1.2.1)
$\mathbf{W_W}$ = Weight of waste sample (Section 10.1.1.1.2.3)

10.1.1.1.2.13 If the % dry solids determined above, is less than 0.5%, then the filtrate is the TCLP extract. Filter sample until enough filtrate is obtained for analysis and proceed to Section 10.3.1.3.6.

10.1.1.1.2.14 If the % dry solids determined above is greater than 0.5%, the sample is considered to be multi-phasic. Determine the weight of the dry waste by subtracting the weight of the filter from the weight of the dry waste and filter as determined follows and record in extraction logbook:

Weight of dry solids= $\mathbf{W_{DW} - W_F}$

Where:

$\mathbf{W_{DW}}$ = Weight of dried waste + filter (Section 10.1.1.1.2.11)
$\mathbf{W_F}$ = Weight of the filter (Section 10.1.1.1.2.1)

Proceed to Section 10.3.1.3. (**NOTE:** Do not dispose of dried waste + filter.)

10.1.1.2 Determine whether waste requires particle size reduction

Evaluate the solid phase of the waste sample (Section 10.1.1.1.2.6), to determine if particle size reduction is necessary. Particle size reduction is required unless the solid has a surface area greater than 3.1 cm^2 per gram, or is smaller than 1 cm in its narrowest dimension (i.e.: will not pass through a 9.5 mm (0.375-inch) standard sieve).

If the surface area is smaller or the particle size is larger than described above, prepare the solid portion of the waste for extraction by crushing, cutting or grinding the waste to a surface area or particle size as described above.

NOTE: Surface area criteria is meant for filamentous (e.g. paper, cloth, etc.) waste materials. Actual measurement of surface area is not required, nor is it recommended. For materials that do not obviously meet the criteria, those samples should be reduced to meet the 1 cm dimension criteria.

10.1.1.3 Extraction Fluid Determination

10.1.1.3.1 Weigh out a 5g subsample of the solid phase waste, if the sample is all solid. If the sample is multiphase, weigh out a 5g subsample of the dried waste from Section 10.1.1.1.2.14.

10.1.1.3.2 Transfer the 5g aliquot to a 250mL beaker and add 96.5 mL of reagent water. (NOTE: Less weight and volume may be used, provided the ratio remains the same.)

10.1.1.3.3 Place beaker in the automatic shaker (Section 7.16) for 5 minutes.

10.1.1.3.4 Measure using pH strip and record the pH in the extraction logbook.

10.1.1.3.4.1 If pH is less than 5.0, use extraction fluid #1, proceed to Section 10.3.

10.1.1.3.4.2 If pH is greater than 5.0, add 3.5 mL 1N HCl, slurry briefly, cover with a watch glass, heat to 50°C+/- 2°C on a hotplate and hold for 10 minutes.

Note: heating block load tests have shown heating times of up to 25 minutes prior to the sample reaching the specified temperature. The 10 minute hold begins only after the temperature is reached and monitored using a calibrated thermometer placed in a sample or one representative of the batch.

Cool solution to room temperature and record pH. If the pH is less than 5.0, use extraction fluid #1. Otherwise, use extraction fluid #2.

10.2 Initial Calibration

Not applicable.

10.3 Equipment Operation and Sample Processing

10.3.1 Extraction of SOLID PHASE

10.3.1.1 A 100gram minimum aliquot of SOLID PHASE is extracted to produce the TCLP extract, as outlined below. In some cases, a larger sample size may be required in order to produce enough liquid for the required analysis. If the amount of extract generated by a single TCLP extraction will not be sufficient to perform all of the analysis, more than one extraction may be performed and the extracts combined for analysis. If there is limited sample volume, the chemist must contact the project manager.

10.3.1.2 100% Solid Waste

10.3.1.2.1 If the waste is 100% solid, weigh out an aliquot (100 gram minimum), follow Sections 10.4.1.2 (particle size reduction) and then proceed to 10.3.1.3.2 (extraction fluid amount).

10.3.1.3 Liquid or Multiphasic Waste

10.3.1.3.1 Quantitatively transfer the solid material, along with the filter (from Section 10.1.1.1.2.14), into a glass extractor bottle (plastic may be used if only metals are being analyzed).

Repeat sections 10.1.1.1.2 through .6 if additional final volume is needed for the intended analysis.

10.3.1.3.2 Determine the amount of extraction fluid to add to the extractor vessel as follows:

$$V_{EF} = \frac{20 \text{ X } WD_W}{100}$$

Where:

$\mathbf{V_{EF}}$ = Volume of extraction fluid required.
(Density = 0.998g/L, therefore assume volume is equivalent to weight.)

$\mathbf{WD_W}$ = Weight of waste filtered (dry weight if multiphasic)

10.3.1.3.3 Add the volume of the appropriate extraction fluid (determined in Section 10.1.1.3) to the extraction vessel. Glass amber 2.5 L for organics and metals, 2.5L plastic if for metals only analysis.

10.3.1.3.4 Close the extractor bottle tightly. Secure in rotary agitation device, and rotate at 30 ± 2 rpm for 18 ± 2 hours while maintaining room temperature at 23 ± 2° C. Record the agitation device (Tumbler) ID in the extraction logbook.

10.3.1.3.4.1 The room temperature (°C) and the extraction time are recorded in the extraction logbook at both the beginning and the end of extraction.

10.3.1.3.4.2 To verify that the proper room temperature is maintained during extraction, a Maximum/Minimum thermometer is reset at the beginning of the extraction period. This thermometer is reviewed at the end of the extraction period and the Maximum and Minimum temperatures are noted in the extraction logbook.

10.3.1.3.4.3 If proper room temperature is not maintained during the extraction period, it is considered a variation from the method and must be written into a laboratory narrative and submitted with the reported data. The Department Supervisor is immediately notified to determine the proper corrective action to be taken.

10.3.1.3.5 Following extraction, vacuum filter the extract through glass fiber filters (acid precleaned for metals analysis). For this final filtration, the glass fiber filter may be changed to facilitate quicker filtration but should be kept to a minimum when practical. Use of pre-filters is prohibited for TCLP.

10.3.1.3.6 Prepare the TCLP extract as follows:

10.3.1.3.6.1 If the waste contained no initial liquid phase, the filtered extract obtained is defined as the TCLP extract. Proceed to 10.3.1.3.7.

10.3.1.3.6.2 If the waste contained a liquid phase, check its compatibility with the extraction fluid determined in Section 10.1.1.3. If the liquids are compatible (miscible), then the initial liquid phase (Section 10.1.1.1.2.6) and the filtered extract are combined and analyzed together.

10.3.1.3.6.3 If the initial liquid phase of the waste obtained in Section 10.1.1.1.2.6, is not compatible with the filtered extract, do not combine the liquids. Analyze these liquids, collectively defined as the TCLP extract, separately and mathematically combine the results, as described below. Determine the volume of the individual phases, conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

$$(mg/L),\quad \text{Final Analyte Concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

Where:

V_1 = Volume of the first phase (L)
C_1 = Analyte concentration first phase (mg/L)
V_2 = Volume of the second phase (L)
C_2 = Analyte concentration second phase(mg/L)

10.3.1.3.7 Record the pH of the TCLP extract in the extraction logbook prior to preservation of samples.

10.3.1.3.8 Metals analysis- transfer filtrate (or combined filtrates) to a 120 mL plastic bottle and add 2.5 mL of 1:1 HNO_3 preservative to all samples.

10.3.1.3.8.1 Check the pH of the preserved samples to ensure that the sample is properly preserved at a pH of less than 2.

10.3.1.3.9 Non-volatile Organics- transfer filtrate (or combined filtrates) to two 1L glass amber bottles, adhere pre-made sample ID labels and transfer to extraction department refrigerators.

10.3.2 Extraction of Lamps

Lamps (for both TCLP extraction and 120x extraction) are broken down into small particles (See Section 10.1.1.2) and extracted using Extraction Fluid #1. The extraction procedure from Section 10.3.1.3.2 through 10.3.1.3.8 is followed with the exception that the entire lamp weight including endcaps is used. 7L plastic extraction vessels are utilized for larger lamps that exceed 100g total weight.

10.4 Continuing Calibration

Not Applicable.

10.5 Preventive Maintenance

None.

11. Data Evaluation, Calculations and Reporting

None.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

12.1 All Holding time exceedences, improper preservation and Extraction Anomalies are to be reported to a Supervisor or Manager. Non Conformance Reports may need to be issues through the Qualtrax System.

12.2 Refer to determinative method SOPs for additional Corrective Action information.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP 1732 These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

Not Applicable.

13.2.2 Continuing (DOC)

Not Applicable.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP 1732 MDL/LOD/LOQ Generation

SOP 1739 IDC/DOC Generation

SOP 1728 Waste Management and Disposal SOP

WI 17618 TCLP and SPLP Work Instruction

16. Attachments

Table 1: Maximum Holding Times

Table 1

MAXIMUM HOLDING TIMES

Sample Maximum Holding Times (Days)

	From: Field collection **To:** TCLP extraction	**From:** TCLP extraction **To:** Preparative extraction	**From:** Preparative extraction **To:** Determinative analysis	**Total Elapsed Time**
Semi-volatiles	14	7	40	61
Mercury	28	N/A	28	56
Metals, except mercury	180	N/A	180	360

TCLP/SPLP Extraction - Volatile Organics EPA 1311/1312

Reference: EPA 1311, EPA 1312 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Update I, July 1992

1. Scope and Application

Matrices: The TCLP/SPLP extraction process is designed to determine the mobility of both organic and inorganic analytes present in liquid, solid, and multiphasic wastes.

Definitions: Refer to Alpha Analytical Quality Manual.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the INSTRUMENT and in the interpretation of INSTRUMENT data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

For liquid wastes, (i.e., those containing less than 0.5% dry solid material), the waste, after filtration through a 0.6 to 0.8 μm glass fiber filter, is defined as the TCLP/SPLP extract.

For wastes containing greater than or equal to 0.5% solids, the liquid, if any, is separated from the solid phase and stored for later analysis. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. A special extractor vessel is used when testing for volatile analytes. Following extraction, the liquid extract is separated from the solid phase by filtration through a 0.6 to 0.8 μm glass fiber filter.

If compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract, and these are analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

2.1 Method Modifications from Reference

None.

3. Reporting Limits

The analytical method detection limits determined by the laboratory are on file for review. See the 8260 SOP 2108.

4. Interferences

Potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents. This includes wearing personal protective equipment such as a lab coat, safety glasses, gloves and respirator (as necessary).

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

When the waste is to be evaluated for volatile analytes, care shall be taken to minimize the loss of volatiles. Samples shall be collected and stored in a manner intended to prevent the loss of volatile analytes (e.g., samples are collected in Teflon-lined septum capped vials and stored at 4 ± 2° C). Samples are opened only immediately prior to extraction.

SPLP samples may be collected in 25g En Core sampling device.

6.2 Sample Preservation

Preservatives shall not be added to samples before extraction. For SPLP samples arriving in En Cores they must be extruded and tumbled within 48 hours of collection. Extrude the entire sample or up to 25g if the sample weighs more than 25g since the vessel can only hold 500 mLs of fluid

6.3 Sample Shipping

No specific requirements.

6.4 Sample Handling

Samples may be refrigerated unless refrigeration results in irreversible physical change to the waste. If precipitation occurs, the entire sample (including precipitate) must be extracted.

TCLP/SPLP extracts are prepared for analysis and analyzed as soon as possible following extraction. Extracts or portions of extracts for organic analyte determinations shall not be allowed to come into contact with the atmosphere (i.e., no headspace) to prevent losses.

Samples must undergo TCLP/SPLP extraction within the time periods listed in Table 1.

7. Equipment and Supplies

7.1 Agitation Apparatus: End-over-end 30 ± 2 rpm.

7.2 Zero Headspace Extraction Vessel

7.3 Glass Fiber Filters: 0.6 to 0.8µm, 90mm

7.4 50mL Gastight Syringe

7.5 Graduated Cylinder: 1L

7.6 Laboratory Balance: 0.01g tolerance

7.7 Pressure Gauge: 0-7 bar

7.8 Pressure Filtration Device

7.9 Drying Oven: 100 ± 2 °F

7.10 Fluid Metering Pump

7.11 pH Meter: The meter should be accurate to + 0.05 units at 25°C

8. Reagents and Standards

8.1 Analytical Standards: The standards shall be prepared according to the appropriate volatile organic analysis method 8260 SOP 2108.

8.2 Hydrochloric Acid (HCl), 1N: Dilute 83 mL conc. HCl to 1L with DI water.

8.3 Nitric Acid (HNO_3), 1N: Dilute 64 mL conc. HNO_3 to 1L with DI water.

8.4 Sodium Hydroxide (NaOH), 1N: Dissolve 40 gs NaOH in 1L of water.

8.5 Glacial acetic acid: CH_3COOH.

8.6 Ottawa sand: SiO_2

8.7 Extraction Fluid #1: Add 5.7 mL glacial acetic acid to 500 mL of DI water, add 64.3 mL of 1N NaOH, and dilute to a volume of 1L. Check pH of solution which must be 4.93 $\pm$ 0.05 when correctly prepared. The laboratory reagent number is referenced in the TCLP sample logbook.

NOTE: This extraction fluid is monitored frequently for impurities. The pH is checked prior to use to ensure that this fluid is prepared accurately. If impurities are found or the pH is not within the above specifications, the fluid shall be discarded and fresh extraction fluid prepared.

8.8 Extraction Fluid #2: Organic-free reagent water.

8.9 Methanol: Purge and Trap Grade or equivalent. Store in flammables cabinet.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank

A minimum of **one blank** must be analyzed for every batch or 20 extracts, whichever comes first. The blank is prepared by weighing 20g of Ottawa sand (SiO_2) and utilizing the same extraction fluid as used for the samples. The blank is then analyzed following the same procedures as analytical samples.

9.2 Laboratory Control Sample (LCS)

A laboratory control sample is not extracted. Refer to the 8260 procedure for the LCS information.

9.3 Initial Calibration Verification (ICV)

Not Applicable.

9.4 Continuing Calibration Verification (CCV)

Not Applicable.

9.5 Matrix Spike

Upon a client's request, a spike solution composed of all regulated compounds can be added to the TCLP/SPLP extract before analysis. See Method 8260 for more information.

9.6 Laboratory Duplicate

Not Applicable.

9.7 Method-specific Quality Control Samples

Not Applicable.

9.8 Method Sequence

The extraction sequence is:

- Disassemble ZHE; clean all parts with warm H_2O and soap; dry.
- Check sample; ensure there is enough sample for analysis.
- Determine %solids of the waste.
- Extract the TCLP/SPLP.
    - Record the temperature of the extraction room.
    - Following extraction, perform a leak test on the ZHE canisters.
- Draw sample with syringe from ZHE after extraction.
- Prepare sample for 8260 analysis

10. Procedure

10.1 Equipment Set-up

10.1.1 Preliminary Evaluation

10.1.1.1 Determine % Solids :

10.1.1.1.1 If waste contains no free liquid, proceed to Section 10.1.1.2 where the waste is the solid portion.

10.1.1.1.2 If the waste contains less than 0.5% solids, proceed to Section 10.3.2.

10.1.1.1.3 Multiphasic waste - separate liquid and solid portion

The following is performed by the Metals Prep Department.

10.1.1.1.3.1 Using a pressure filtration device pre-weigh the filter and the container to receive the initial filtrate. Record the weight in the laboratory notebook.

10.1.1.1.3.2 Assemble the pressure filter according to the manufacturer's directions.

10.1.1.1.3.3 Weigh a subsample of the waste (100 g minimum) and record the weight.

10.1.1.1.3.4 Transfer the waste sample to the filter holder spreading evenly over the filter.

10.1.1.1.3.5 Gradually apply a gentle pressure of 1-10psi until the pressurizing gas moves through the filter, proceed to Section 10.1.1.1.3.8.

10.1.1.1.3.6 If no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10psi increments.

10.1.1.1.3.7 Stop the filtration when pressurizing gas passes through the filter, or 50psi is reached and no additional liquid passes through the filter in any 2 minute interval.

10.1.1.1.3.8 The material in the filter holder is defined as the SOLID PHASE of waste and the filtrate is defined as the LIQUID PHASE.

Note: the SOLID PHASE may appear liquid in some samples. Do Not replace original filter. Use only one filter.

10.1.1.1.3.9 Weigh the filtrate filled container and record the weight in the laboratory notebook. Calculate the weight of the LIQUID PHASE as follows:

$$W_{LP} = W_F - W_C$$

Where:

W_{LP} = Weight of the LIQUID PHASE
W_F = Weight of the filtrate filled container
W_C = Weight of container

10.1.1.1.3.10 Calculate the weight of the SOLID PHASE using the following formula:

$$W_{SP} = W_W - W_{LP}$$

Where:

W_{SP} = Weight of the SOLID PHASE
W_W = Weight of the waste sample
W_{LP} = Weight of the LIQUID PHASE

10.1.1.1.3.11 Calculate the percent solids using the following formula:

$$\%Solids = \frac{W_{sp}}{W_w x 100}$$

Where:

W_{SP} = Weight of the SOLID PHASE
W_W = Weight of the waste sample

10.1.1.1.3.12 If the % Solids determined in Section 10.1.1.1.3.11, is less than 0.5%, then proceed to Section 10.3.2, where the filtrate is the TCLP/SPLP extract.

10.1.1.1.3.13 Remove the solid phase and filter from the filtration apparatus, dry at 100 ± 2° F, cool and weigh. Record the weight in the laboratory notebook.

10.1.1.1.3.14 Calculate the percent dry solids using the following formula:

$$\%\ \text{dry solids} = \frac{W_{DW} - W_F}{W_W \times 100}$$

Where:

W_{DW} = Weight of dried waste + filter
W_F = Weight of the filter
W_W = Weight of waste sample

10.1.1.1.3.15 If the % dry solids determine above, 10.1.1.3.14, is less than 0.5%, then proceed to Section 10.3.2, where the filtrate is the TCLP extract.

10.1.1.2 Determine whether waste requires particle size reduction

The following is performed by the Metals Prep Department.

Using fresh portion of the waste, obtain another solid phase of the waste sample by pressure filtration as determined in Sections 10.1.1.1.1. Use enough wet sample to obtain a dry portion for tumbling: at least 100 g. Evaluate the solid phase for particle size. Particle size reduction is required unless the solid has a surface area greater to 3.1 cm^2 per g, or is smaller than 1 cm in its narrowest dimension.

10.1.1.3 Determine Compatibility of Liquids

The following is performed by the Metals Prep Department.

If a LIQUID PHASE was filtered, check its compatibility with the extraction fluid. If the liquids are compatible (miscible), then only one TCLP/SPLP extract needs to

be analyzed. If the liquids are not compatible, then the two liquids will have to be analyzed separately. Record in the laboratory notebook.

10.2 Initial Calibration

Not Applicable.

10.3 Equipment Operation and Sample Processing

10.3.1 After the preliminary examination has been completed, the sample used in the following step comes from supplied VOA sample vials. The extraction for Volatile Organics is accomplished in a zero headspace extractor, ZHE.

10.3.2 Waste containing less than 5% dry solids

10.3.2.1 For waste containing less than 5% dry solids, weigh out up to a 500 g subsample and record its weight. Filter enough of the sample so that two 40 mL VOA vials may be filled with the filtered liquid. The liquid portion of the waste, after filtration, is defined as the TCLP/SPLP extract for volatile organic analysis. Proceed to Section 10.3.4.20.

10.3.3 100% Solid Waste

10.3.3.1 If particle size reduction is required (Section 10.1.1.2.), prepare the waste for extraction by crushing, cutting, or grinding the waste to a surface area or particle size as described in Section 10.1.1.2. Waste and appropriate reduction equipment should be refrigerated to 4 ± 2° C prior to particle size reduction.

10.3.3.2 Weigh 20 gs of prepared waste and record the weight. If a 25 g Encore is supplied extrude and record the weight.

10.3.3.3 Quantitatively transfer the sample to the ZHE.

10.3.3.4 Assemble the ZHE according to manufacturer's instructions. Add the appropriate type and amount of fluid using the fluid metering pump. The fluid added is 20 times the amount of sample. For example, for a 20 g sample, add 400 mL of extraction fluid for TCLPs or 400 mL of organic free DI water for SPLPs. For a 25 g sample, 500 mLs of the appropriate fluid is added.

10.3.3.5 Position the ZHE in the vertical position with the liquid inlet/outlet valve on top and open.

10.3.3.6 Gradually apply pressure to the ZHE and bleed out any headspace.

10.3.3.7 Proceed to Section 10.3.4.16.

10.3.4 Waste containing greater than 5% dry solids

10.3.4.1 Determine the amount of waste to charge into the ZHE as follows:

$$\text{Weight of waste to charge the ZHE} = \frac{20 \times 100}{\%\ \text{solids}}$$

Weigh out a subsample of the waste of the appropriate size and record the weight

10.3.4.2 If particle size reduction of the solid portion of the waste was required in Section 10.1.1.2, prepare the waste for extraction by crushing, cutting or grinding the solid portion of the waste to a surface area or particle size as described in Section 10.1.1.2. Waste and appropriate reduction equipment should be refrigerated to 4 ± 2° C prior to particle size reduction.

10.3.4.3 Quantitatively transfer the entire sample quickly to the ZHE. Assemble the ZHE in accordance with the manufacturer's instructions.

10.3.4.4 Place the device in the vertical position with the inlet/outlet valve on top and open.

10.3.4.5 Gradually apply pressure to the ZHE and bleed out any headspace quickly.

10.3.4.6 At the first appearance of liquid from the liquid inlet/outlet valve quickly close the valve.

10.3.4.7 Attach a 50mL gastight syringe to the liquid inlet/outlet valve and open the valve.

10.3.4.8 Begin applying gentle pressure to force the liquid phase of the sample into the filtrate collection container. When no additional liquid has passed through the filter in any 2-minute interval, proceed to Section 10.3.4.9.

10.3.4.9 Stop filtration by closing the liquid inlet/outlet valve.

10.3.4.10 Disconnect and weigh the 50mL gastight syringe. Record the weight in the laboratory notebook.

10.3.4.11 The material in the ZHE is defined as the solid phase of the waste and the filtrate is defined as the liquid phase.

10.3.4.12 If the liquid phase is compatible with the extract (Section 10.1.1.3), store the filtrate at 4 ± 2°C until analysis. Then combine and analyze the filtrate as one extract.

10.3.4.13 If the liquid phase is not compatible with the solid extract, then analyze the filtrates separately and mathematically combine the results (Section 10.3.4.22).

10.3.4.14 Determine the weight of extraction fluid #1 for TCLP or organic free DI water for SPLP to add to the ZHE as follows:

Weight of extraction fluid = $20(\mathbf{W_S + W_T - W_{TF}})$

Where:

$\mathbf{W_S}$ = Weight Sample
$\mathbf{W_T}$ = Weight of pre-weighed 50mL syringe
$\mathbf{W_{TF}}$ = Weight of filtrate in 50mL syringe

For 100% solid samples, the above formula reduces to:

Weight of extraction fluid = 20 x weight of sample

10.3.4.15 Add extraction fluid by backing piston off the filter and sucking fluid into the ZHE. Do not open the ZHE.

10.3.4.16 Re-pressurize the ZHE to 15 psi (1.03 bar) and place in the rotary agitation apparatus. Rotate at 30 ± 2 rpm for 18 ± 2 hours.

10.3.4.17 Post-extraction measure the final pressure of the ZHE canister. If pressure has decreased below 15 PSI the sample must be reset.

10.3.4.18 Following the agitation period separate the material in the extractor vessel into its liquid and solid phases.

10.3.4.18.1 100% solid waste samples: Filter enough liquid phase to fill two 40 mL vials. The filtrate is defined as the TCLP/SPLP extract. Proceed to Section 10.3.4.20.

10.3.4.18.2 Compatible liquid phases: Filter the entire extract into a 50mL gastight syringe. Transfer the contents of the syringe into 40mL VOA vials. Combine the extracts (Section 10.3.4.22) and analyze together.

10.3.4.18.3 Non-compatible liquid phases. Filter a sufficient quantity of the solid extract to fill two 40 mL VOA vials and analyze. The two liquids are collectively defined as the TCLP/SPLP extract and results are mathematically combined after individual analysis.

10.3.4.19 Following collection of the TCLP/SPLP extract immediately prepare the extract for analysis and store with minimal headspace at 4 ± 2°C until analyzed.

10.3.4.20 Follow the volatile organic analyses method 8260 SOP 2108 to analyze the TCLP/SPLP extract.

10.3.4.21 If the individual liquid phases (i.e. non-compatible liquids) are analyzed separately, determine the volume of the individual phases, conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

$$\text{Final Analyte Concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

Where:

V_1 = Volume of the first phase, (L)
C_1 = Analyte concentration first phase, (mg/L)
V_2 = Volume of the second phase, (L)
C_2 = Analyte concentration second phase, (mg/L)

10.4 Continuing Calibration

Not Applicable.

10.5 Preventive Maintenance

Tumbler rotation speed is verified annually and documented in the TCLP Annual Calibration Log, Form No.: 13855.

Balances are calibrated semi-annually by an instrument service company. Certificates are kept on file.

ZHEs are cleaned after extraction in soapy water. Filter screens and o-rings may be changed if they show degradation or the extraction vessel can't maintain pressure. Extremely contaminated vessels maybe further solvent rinsed with methanol

11. Data Evaluation, Calculations and Reporting

Calculations are included in Section 10.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedance, improper preservation and observed sample headspace are noted on the nonconformance report form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

Review of standards, blanks and standard response for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form.

13. Method Performance

13.1 Detection Limit Study (DL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the DL, LOD, and/or LOQ as outlined in Alpha SOP ID 1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP ID 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

Not Applicable.

13.2.2 Continuing (DOC)

Not Applicable.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Hazardous Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

2121 Chemical Hygiene Plan

1732 Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ) SOP

1739 Demonstration of Capability (DOC) Generation SOP

1728 Hazardous Waste Management and Disposal SOP

2108 8260 SOP

13855 TCLP Annual Calibration Form

16. Attachments

Table1: Maximum Holding Times
Table 2: TCLP Analyte List

Table 1

MAXIMUM HOLDING TIMES

Sample Maximum Holding Times (Days)

	From: Field collection **To:** TCLP/SPLP extraction	**From:** TCLP/SPLP extraction **To:** Preparative extraction	**From:** Preparative extraction **To:** Determinative analysis	**Total Elapsed Time**
Volatiles	14/48 (hrs.)	N/A	14	28
Semi-volatiles	14	7	40	61
Mercury	28	N/A	28	56
Metals, except mercury	180	N/A	180	360

Table 2
TCLP Analyte List

Analyte	CAS No.
1,1 - Dichloroethene	75-35-4
1,2 - Dichloroethane	0107-06-02
1,4 - Dichlorobenzene	106-46-7
2-Butanone	78-93-3
Benzene	71-43-2
Carbon tetrachloride	56-23-5
Chlorobenzene	108-90-7
Chloroform	67-66-3
Tetrachloroethene	127-18-4
Trichloroethene	79-01-6
Vinyl chloride	75-01-4

Total Dissolved Solids Dried at 180ºC

Total Volatile Dissolved Solids at 550°C

Method Reference: **SM 2540C (TDS) and 2540E (TVDS)**, Standard Methods for the Examination of Water and Wastewater. APHA-AWWA-WEF. Standard Methods Online.

1. Scope and Application

Matrices: This method is suitable for the determination of Total Dissolved Solids (TDS) and Total Volatile Dissolved Solids (TVDS) in potable, surface, and saline waters, as well as domestic and industrial wastewaters.

Definitions: Refer to Alpha Analytical Quality Manual.

"Solids" refer to matter suspended or dissolved in water or wastewater. Solids may affect water or effluent quality adversely in a number of ways. Waters with high dissolved solids generally are of inferior palatability and may induce an unfavorable physiological reaction in the transient consumer. For these reasons, a limit of 500mg dissolved solids/L is desirable for drinking waters. Highly mineralized waters also are unsuitable for many industrial applications. Waters high in suspended solids may be esthetically unsatisfactory for such purposes as bathing. Solids analyses are important in the control of biological and physical wastewater treatment processes and for assessing compliance with regulatory agency wastewater effluent limitations.

"Total solids" is the term applied to the material residue left in the vessel after evaporation of a sample and its subsequent drying in an oven at a defined temperature. Total solids include "total suspended solids," the portion of total solids retained by a filter, and "total dissolved solids," the portion that passes through the filter.

"Fixed solids" is the term applied to the residue of total, suspended, or dissolved solids after heating to dryness for a specified time at a specified temperature. The weight loss on ignition is called "volatile solids." Determinations of fixed and volatile solids do not distinguish precisely between inorganic and organic matter because the loss on ignition is not confined to organic matter. It includes losses due to decomposition or volatilization of some mineral salts

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

A well-mixed sample is filtered through a standard glass fiber filter, and the filtrate is evaporated to dryness in a weighed dish and dried to constant weight at 180ºC. The increase in dish weight represents the total dissolved solids. The filtrate from the total suspended solids determination (refer to SOP ID 2220) may be used for determination of total dissolved solids. Samples

submitted for Total Volatile Dissolved Solids analysis are also dried at a higher temperature to a constant weight.

2.1 Method Modifications from Reference

None

3. Reporting Limits

The RDL is 10mg/L using a 100mL sample and analytical balance sensitive to 0.1mg, with an upper limit of 2000mg/L. A lower RDL may be achieved by using a larger sample (repeated evaporation of 100mL aliquots) or the use of a more sensitive balance.

4. Interferences

4.1 Highly mineralized water with a significant concentration of calcium, magnesium, chloride, and/or sulfate may be hygroscopic and require prolonged drying, proper desiccation and rapid weighing.

4.2 Samples high in bicarbonate require careful and possibly prolonged drying at 180ºC to ensure complete conversion of bicarbonate to carbonate.

4.3 Exclude large, floating particles or submerged agglomerates of non-homogeneous materials from the sample if it is determined that their inclusion is not desired in the final result.

4.4 Disperse visible floating oil and grease with a blender before withdrawing a sample portion for analysis.

4.5 Because excessive residue in the dish may form a water-trapping crust, limit sample to no more than 200mg residue.

4.5.1 For samples high in dissolved solids, thoroughly wash the filter to ensure removal of dissolved material. Mix small samples with a magnetic stirrer. If suspended solids are present, pipet with wide-bore pipets. If part of a sample adheres to the sample container, consider this in evaluating and reporting results. Some samples dry with the formation of a crust that prevents water evaporating; special handling is required. Avoid using a magnetic stirrer with samples containing magnetic particles.

4.5.2 Prolonged filtration times resulting from filter clogging may produce high results owing to increased colloidal materials captured on the clogged filter.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Use resistant-glass or plastic bottles, provided that the material in suspension does not adhere to container walls. Sampling, subsampling, and pipetting two-phase or three-phase samples may introduce serious errors. Make and keep such samples homogeneous during transfer. Use special handling to ensure sample integrity when subsampling.

6.2 Sample Preservation

None.

6.3 Sample Shipping

No specific requirements.

6.4 Sample Handling

Begin analysis as soon as possible because of the impracticality of preserving the sample. Refrigerate sample at 4 ±2 ºC up to the time of analysis to minimize microbiological decomposition of solids. In no case hold sample more than 7 days. Bring samples to room temperature before analysis.

7. Equipment and Supplies

7.1 Glass-Fiber Filter Disks: 47mm diameter, without organic binder, Type A/B, size 1.0um.

7.2 Filtration Apparatus: With a membrane filter funnel.

7.3 Side-arm Flask: Of sufficient capacity for sample size selected.

7.4 Evaporation Dishes: 100mL, porcelain, 90mm diameter.

7.5 Dessicator: With a dessicant containing a color indicator of moisture concentration or an instrumental indicator.

7.6 Drying Oven: 75 - 80ºC.

7.7 Drying Oven: 180ºC ± 2ºC.

7.8 Analytical Balance: Capable of weighing to 0.1mg.

7.9 Magnetic Stirrer: With TFE stirring bar.

7.10 Wide-bore Pipets.

7.11 Graduated Cylinders: 100mL, plastic or glass.

7.12 Wax Crayon or Sharpie pen

7.13 Muffle Furnace: Capable of 550°C.

8. Reagents and Standards

8.1 Reagent Water: Deionized (DI) water.

8.2 LCS Solution: To a 1L volumetric flask, add 0.5g of K_2SO_4. Bring to volume with DI water. Transfer the solution to a 1L plastic bottle that is labeled with the contents, preparation date, expiration date, and the initials of the preparer. Solution is stored at room temperature. The LCS Solution is valid for 1 year.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank

Filter one blank of DI water per batch of 20 samples or less. Results must be less than the Reporting Limit (RL). If Blank results are less than 10 times the RL and the sample results are greater than 10 times the RL, the data is reported. If Blank results are greater than 10 times the RL, and there is sufficient sample volume remaining, the entire batch is reset and reanalyzed. A narrative is included with the report.

9.2 Laboratory Control Sample (LCS)

Filter one LCS per batch of 20 samples or less. Results must be within ± 20% of the true value. If acceptance criteria are not met then the entire sample batch must be reset. If there is insufficient sample volume remaining, the data is submitted and a narrative is included with the final report.

9.3 Initial Calibration Verification (ICV)

Not Applicable.

9.4 Continuing Calibration Verification (CCV)

Not Applicable.

9.5 Matrix Spike

Not Applicable.

9.6 Laboratory Duplicate

Filter one duplicate sample per batch of 10 samples or less. Duplicate determinations should agree within 5% of their average weight (or 10% RPD). If acceptance criteria are not met, and there is sufficient sample volume remaining and sample is within holding time, the sample is reset. Otherwise, the data is submitted and a narrative is included with the final report.

9.7 Method-specific Quality Control Samples

None.

9.8 Method Sequence

- Prepare the evaporation dishes.
- Weigh the clean evaporation dishes.

- Filter the appropriate volumes of sample, and QC samples.
- Rinse the filter with DI water.
- Transfer the filtrate into a clean, pre-weighed evaporation dish, and rinse the filter flask.
- Evaporate the filtrate in a 75 - 80°C oven.
- Once dry, transfer the dishes into the 180°C oven.
- Dry for a minimum of 2 hours in a 180°C oven.
- Cool in a dessicator until temperature is constant.
- Weigh the dishes until the weight change is less than 4% of the previous weight or 0.5mg, whichever is less.
- Total Volatile Suspended Solids are also dried for a minimum of 2 hours in a 550 °C muffle furnace.
- Cool in a dessicator until temperature is constant.
- Weigh the dishes until the weight change is less than 4% of the previous weight or 0.5mg, whichever is less.
- Calculate results.

10. Procedure

10.1 Equipment Set-up

10.1.1 Preparation of Evaporation Dishes

10.1.1.1 Label evaporating dishes with a unique ID using a wax crayon or Sharpie pen.

10.1.1.2 Bake clean evaporating dishes to 180 ± 2ºC for at least two hours in an oven, but preferably overnight for the greatest stability.

10.1.1.3 Store the evaporation dishes in a dessicator until needed.

10.2 Initial Calibration

Not Applicable.

10.3 Equipment Operation and Sample Processing

10.3.1 Filtration

10.3.1.1 Remove the evaporating dishes from the dessicator and weigh them immediately before use. Record the weight on the computer worksheet. Also record the date/time/analyst's initials for the analysis on the computer worksheet in the required areas.

10.3.1.2 Insert a glass-fiber filter disk (Section 7.1) with wrinkled side up in the filtration apparatus. Attach a side-arm flask.

Note: Record the manufacturer and lot number of the filter used in the computer worksheet.

10.3.1.3 Apply vacuum, and wash the disk with three successive 20mL portions of DI water.

10.3.1.4 Continue suction to remove all traces of water, and discard washings.

10.3.1.5 Selection of Sample Size: Choose a sample volume to yield 200mg dried residue. Generally a 100mL sample volume is used. When very low dissolved solids are encountered (less than 100mg/L), less dried residue may be collected. Compensate for this by using a high-sensitivity balance (0.001mg) or by increasing the amount of sample volume.

10.3.1.6 Samples

10.3.1.6.1 Apply vacuum.

10.3.1.6.2 Shake sample thoroughly to ensure it is well homogenized prior to use. Pour 100mL of sample (or selected sample volume, Section 10.3.1.5) into a clean 100mL graduated cylinder. Pour this volume into the filtering apparatus.

10.3.1.6.3 If the sample contains sediment, oil, or any other substance, stir the sample using a magnetic stirrer, and while stirring, pipet a measured volume onto the seated filter.

10.3.1.7 Quality Control Samples

10.3.1.7.1 LCS

Using a 100mL graduated cylinder, shake well, and measure 50mL of the LCS Solution (Section 8.2) and pour into the filtering apparatus.

10.3.1.7.2 Blank

Using a 100mL graduated cylinder, measure 100mL of DI water and pour into the filtering apparatus.

10.3.1.8 Rinse the filter with three successive 10mL volumes of DI water, allowing complete drainage between washings, and continue suction for about 3 minutes after filtration is complete.

10.3.1.9 Transfer the total filtrate (with washings) to a weighed evaporating dish and evaporate to dryness in an oven at 75 - 80ºC. Record the date, time in, and the oven temperature in the computer worksheet.

10.3.1.10 Then, transfer the samples to dry for at least two hours in an oven at 180 ±2ºC.

- Following removal from the drying oven (Section 10.3.1.9), record the date, time out, and the oven temperature in the computer worksheet.
- Following the placement of the samples and QC samples in the 180 ±2ºC oven (Section 10.3.1.10), record the date, time in, and the oven temperature in the computer worksheet.

10.3.1.11 Remove the dishes from the oven and cool them in a dessicator to constant temperature. Record the date, time out, and the oven temperature in the computer worksheet.

10.3.1.12 Record the weight of the dishes on the computer worksheet.

10.3.1.13 Repeat drying cycle of drying, cooling, desiccating, and weighing until a constant weight is obtained or until the weight change is less than 4% of the previous weight or 0.5mg, whichever is less.

10.3.1.14 Total Volatile Dissolved Solids

10.3.1.14.1 If TVDS analysis is requested, transfer the crucible to a muffle furnace at 550°C. Record the date, time in, and the oven temperature in the computer worksheet.

10.3.1.14.2 Dry the crucible for a minimum of 2 hours at 550°C and cool in a dessicator to a constant room temperature.

- Following placement of the samples and QC samples in the furnace, record the date, time in, and the furnace temperature in the computer worksheet.
- Following the placement of the samples and QC samples in the dessicator, record the date, time out, and the furnace temperature in the computer worksheet.

10.3.1.14.3 Using an analytical balance, weigh the crucible and record the weight on the computer worksheet.

10.3.1.14.4 Repeat the cycle of drying, cooling, dessicating and weighing until a constant weight is obtained or until the weight change is less than 4% of the previous weight, or 0.5mg, whichever is less. Record each weight on the computer worksheet.

10.4 Continuing Calibration

Not Applicable.

10.5 Preventive Maintenance

10.5.1 Prior to each use, the filtering apparatus is rinsed thoroughly with DI water.

10.5.2 As needed, the filtering apparatus is washed in the industrial dishwasher.

10.5.3 As necessary, wipe the inside of the filtering apparatus with a paper towel and then rinse thoroughly with DI water

10.5.4 Ensure that there is an adequate supply of oil in the vacuum pump

10.5.5 The temperature of the laboratory ovens is recorded constantly on a data logger system, and monitored once daily by laboratory analysts.

10.5.6 The data logger system and the laboratory ovens are calibrated on an annual basis by an instrument service company. Certificates are kept on file in the QA Department.

11. Calculations

$$\text{mg TDS/L} = \frac{(\mathbf{A} - \mathbf{B}) \times 1000}{\text{sample volume, (mL)}}$$

where:

A = Final weight (weight of dish + dried residue, mg)
B = Initial weight (weight of clean dish, mg)

$$\text{mg TVDS / L} = \frac{(\mathbf{G} - \mathbf{N}) \times 1000}{\text{sample volume, (mL)}}$$

where:

G = Final Weight at 105 °C (weight of dish + dried residue, mg)
N = Weight at 550 °C (weight of dish + dried residue, mg)

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedence and improper preservation are noted on the nonconformance report form.

Perform routine preventative maintenance as outlined in Section 10.5.

Review of blanks and LCS for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form.

If the LCS recovery falls outside the designated acceptance range, the laboratory performance is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result is suspect and is only reported for regulatory compliance purposes with the appropriate nonconformance action form. Immediate corrective action includes reanalyzing all affected samples by using any retained sample before the expiration of the holding time.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1734 and 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/1732 MDL/LOD/LOQ Generation

SOP/1734, 1739 IDC/DOC Generation

SOP/1728 Waste Management and Disposal SOP

16. Attachments

None.

Total Sulfide, Methylene Blue Method

References: **SM 4500S2-D:** Standard Methods for the Examination of Water and Wastewater. APHA-AWWA-WEF. Standard Methods Online.

EPA 9030B: SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update III, 1997.

1. Scope and Application

Matrices: This method is applicable to water, wastewater and seawater samples.

Definitions: Refer to Alpha Analytical Quality Manual.

Sulfide often is present in groundwater, especially in hot springs. Its common presence in wastewaters comes partly from the decomposition of organic matter, sometimes from industrial wastes, but mostly from the bacterial reduction of sulfate. Hydrogen sulfide escaping into the air from sulfide-containing wastewater causes odor nuisances. The threshold odor concentration of H_2S in clean water is between 0.025 and 0.25µg/L. H_2S is very toxic and has claimed the lives of numerous workers in sewers. It attacks metals directly and has indirectly caused serious corrosion of concrete sewers because it is oxidized biologically to H_2SO_4 on the pipe wall.

From an analytical standpoint, three categories of sulfide in water and wastewater are distinguished:

- Total sulfide includes dissolved H_2S and HS-, as well as acid-soluble metallic sulfides present in suspended matter. The S_{-2} is negligible, amounting to less than 0.5% of the dissolved sulfide at pH 12, less than 0.05% at pH 11, etc. Copper and silver sulfides are so insoluble that they do not respond in ordinary sulfide determinations; they can be ignored for practical purposes.
- Dissolved sulfide is that remaining after suspended solids have been removed by flocculation and settling.

Un-ionized hydrogen sulfide may be calculated from the concentration of dissolved sulfide, the sample pH, and the practical ionization constant of H_2S.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

Sulfide is separated from the sample matrix by suspending the sample in a hydrochloric acid solution and vigorous agitation. Hydrogen sulfide gas is released from the sample and collected in a gas scrubbing bottle containing sodium hydroxide. The methylene blue method is based on the reaction of sulfide, mild oxidizing agent potassium dichromate, and dimethyl-p-phenylenediamine to produce methylene blue.

2.1 Method Modifications from Reference

Soil samples don't require preservation and have hold time of 7 days

For water samples: whole sample is filtered and filter with sediment is being analyzed.

Lab is purchasing commercially available coloring reagents from HACH.

Lab is using DI instead of dagassed reagent water.

3. Reporting Limits

The reported detection limit is 0.1 mg/L for liquid samples and 0.5 mg/kg for soils samples

4. Interferences

4.1 Strong reducing agents interfere in the methylene blue test by preventing formation of the blue color. Thiosulfate at concentrations about 10mg/L may retard color formation or completely prevent it. Sulfide itself prevents the reaction if its concentration is very high, in the range of several hundred milligrams per liter. To avoid the possibility of false negative results, use the antimony method to obtain a qualitative result in industrial wastes likely to contain sulfide but showing no color by the methylene blue method. Iodide, which is likely to be present in oil-field wastewaters, may diminish color formation if its concentration exceeds 2mg/L. Ferrocyanide produces a blue color; distillation will minimize interference.

4.2 Eliminate interferences due to sulfite, thiosulfate, iodide, and many other soluble substances (but not ferrocyanide), by precipitating ZnS.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Take samples with minimum aeration in either a glass or plastic bottle. Fill the bottle completely and cover. Aqueous samples are collected in a 250mL plastic bottle, soil/solid samples are collected in an amber glass container.

6.2 Sample Preservation

All samples must be preserved with 2N zinc acetate (Section 8.5) and 6N sodium hydroxide (Section 8.6).

Use four drops of 2N zinc acetate solution per 100mL aqueous sample. Adjust the pH to greater than 9.0 with 6N NaOH.

Soils require no preservative.

6.3 Sample Shipping

No specific requirements.

6.4 Sample Handling

Samples are stored refrigerated at 4 ± 2° C. Aqueous and soil samples are analyzed within a 7-day holding time.

7. Equipment and Supplies

7.1 Spectrophotometer, Genesys 10vis (or equivalent): For use at a wavelength of 664nm with cells providing light paths of 1cm.

7.2 Test Tubes (centrifuge tubes): 25mL capacity

7.3 Pipets: Glass, volumetric, various volumes or Auto-pippets.

7.4 Volumetric Flasks: 100mL and 1L volumes, 250 ml and 50 ml

7.5 Erlenmeyer Flasks: 125mL volume

7.6 Three-neck Flask: 500mL volume, 24/40 standard taper joints.

7.7 Dropping funnel: 250mL volume, 24/40 joint.

7.8 Purge gas inlet tube: 24/40 joint with coarse frit.

7.9 Purge gas outlet: 24/40 joint reduced to ¼ in. tube.

7.10 Gas scrubbing bottles: 125mL volume, with ¼ inch o.d. inlet and outlet tubes.

7.11 Tubing: ¼ inch o.d. polytetrafluoroethylene (PTFE) or polypropylene.

7.12 Magnetic Stirrer

7.13 Nitrogen gas: 5.0 Ultra pure.

7.14 Nitrogen regulator

7.15 Flowmeter

7.16 Teflon-coated stirring bar

7.17 Analytical Balance: Capable of weighing 0.1g.

7.18 Glass fiber filter disks: 90mm

7.19 Filtration Apparatus: With membrane filter funnel.

7.20 Suction flask

7.21 Vacuum pump

7.22 Graduated cylinders

8. Reagents and Standard

8.1 2 different sources. Stock Sulfide Solution, approximately 0.05M: Weigh 1.1045 grams of $Na_2S \cdot 9H_2O$ (Section 8.9) and add 140 ml of DI. Store at room temperature. Final concentration is 1097 mg/L. Stable for 2 weeks.. Standard has to be re-made, if degradation of LCS and ICV is noticeable.

8.1.1 LCS/ICV/CCV/ Spike Working Solution (65.82 mg/l): In 50 ml volumetric flask add 10 ml of 1N NAOH (8.6) solution, then adds 3 ml of Stock Sulfide Solution (8.1), bring volume up to 50 ml with DI. Gently mix; can not be mixed vigorously. Different stocks are used to prep ICV and LCS solution and CCV and Spike solution. Solution is stable for one week.

8.1.2 Intermediate calibration standard 10.97 mg/l: Pipet 1 ml of Stock Sulfide Calibration Solution (8.1) into 100ml volumetric flask. Bring volume up to 100ml with DI. Make daily.

8.2 Sulfide 1 Reagent: Available commercially from HACH Company. Store at 4 ± 2°. Reagent expires upon manufacturer's specified date.

8.3 Sulfide 2 Reagent: Available commercially from HACH Company. Store at 4 ± 2°. Reagent expires upon manufacturer's specified date.

8.4 Sulfide Calibration Solutions: These solutions are used to generate the calibration curve in Section 10.1. Prepare a series of sulfide solutions by diluting the Intermediate Calibration Sulfide Standard second source (Section 8.1.2). These solutions are prepared fresh prior to generating a calibration curve.

Into seven 100mL volumetric flasks, pipet 0mL, 0.5 ml 1mL, 2.0mL, 4mL, and 8mL,12 ml and 16 ml of Intermediate Calibration Sulfide Solution (Section 8.1.2). Bring each to volume with DI.

8.5 Zinc Acetate Solution: Dissolve 220g $Zn(C_2H_3O_2)_2 \cdot 2H_2O$ in 500mL water. This solution is used as the preservative during sampling. Store at room temperature. Solution is stable for one year.

8.6 Sodium Hydroxide Solution, NaOH, 1N, 6N and 0.2N: For 1N NaOH, dissolve 40g NaOH in 1L of DI water. For 6N, dissolve 240g NaOH in 1L of DI water. This solution is used as a preservative during sampling. Store at room temperature. Solution is stable for 6 month. For 0.2N NaOH, add 200 ml of 1.0 N NaOH in 1L of DI water. Stable for 1 month.

8.7 HCL Solution, 2N: Add 175mL concentrated HCL (Section 8.10) to 500mL of DI water. Dilute to 1L with DI water. Store at room temperature. Make daily.

8.8 HCL (50%): Carefully add 250mL of concentrated HCL (Section 8.10) to 250mL DI water. Store at room temperature. Solution is stable for 6 month.

8.9 $Na_2S \cdot 9H_2O$: Store at 4 +/- 2 degrees C, out of direct light.

8.10 Concentrated HCL: Store at room temperature.

8.11 Ottawa sand or equivalent.

8.12

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blanks

9.1.1 Method Blank: Distill and analyze one Method Blank of DI water (for liquid samples) or 10g of Ottawa sand (for soil samples) per batch of 20 samples or less. The Method Blank results must be less than the Reporting Limit. Refer to Section 12 for any corrective actions.

9.1.2 ICB/CCB: The ICB/CCB is not distilled. It consists 25 ml of DI and is analyzed following each ICV/CCV. Results must be less than the Reporting Limit. Refer to Section 12 for any corrective actions.

9.2 Laboratory Control Sample (LCS)

Distill and analyze one LCS (Section 8.1.1) per batch of 20 samples or less.

For waters: in 250 ml volumetric flask add approximately 50 ml of DI, 6 drops on 2N Zn Acetate, swirl and add 1 ml of LCS working Solution (8.1.1), adjust pH with 6N NaOH solution (@ 6 drops) to pH greater then 9. Bring volume up to 250 ml with DI. Let solution to sit for several minutes prior to preparation (filtration).

For soils: weigh 10g of Ottawa sand and add 1ml of LCS working solution (8.1.1) directly to reaction flask.

Follow distillation and colorimetric process.

% Recovery must be within 75 – 125%. Refer to Section 12 for any corrective actions.

9.3 Initial Calibration Verification (ICV)

The ICV is not distilled. In 100 ml volumetric flask add 20 ml of 1N NAOH solution, 1 ml of LCS working solution; bring volume up to 100 ml with DI. Done fresh prior to analysis

ICV is analyzed at the beginning of analytical batch.

% Recovery must be within 75 – 125%. Refer to Section 12 for any corrective actions.

9.4 Continuing Calibration Verification (CCV)

The CCV is not distilled. In 100 ml volumetric flask add 20 ml of 1N NAOH solution, 1 ml of CCV working solution; bring volume up to 100 ml with DI. Done fresh prior to analysis. It consists of the CCV Standard (Section 8.1.1) and is analyzed after every 10 samples in the batch.

% Recovery must be within 75 – 125%. Refer to Section 12 for any corrective actions.

9.5 Matrix Spike

Prepare and analyze one matrix spike per batch of 20 samples or less. The matrix spike consists of the 2ml of Working spike solution (8.1.1) added directly to reaction flask prior to distillation. % Recovery must be within in-house control limits. Refer to Section 12 for any corrective actions.

9.6 Laboratory Duplicate

Prepare and analyze one sample in duplicate per batch of 20 samples or less. %RPD must be ≤ 25%. Refer to Section 12 for any corrective actions.

9.7 Method-specific Quality Control Samples

Upon client specification

9.8 Control Limits

The laboratory maintains performance records to document the quality of data that is generated. Method accuracy for samples is assessed and records maintained. After the analysis of 20 spiked samples, and 20 laboratory control samples, calculate the average percent recovery (R) and the standard deviation of the percent recovery (S).

Control limits for the method parameters are generated. The control limits are calculated based on in-house performance data. The limits are compared to the control limits found in the reference method.

9.9 Method Sequence

- Generate a calibration curve.(done once a year)
- Filter water samples or weigh out soil samples
- Add turned filters (for water samples) or aliquot of soil to reaction flask
- Assemble distillation system and distill for at least an hour.
- Disassemble distillation unit and bring distillate up to final volume.
- Start colorimetric procedure.
- Add 1mL Sulfide 1 Reagent to 25mL of sample and swirl gently to mix.
- Add 1mL of Sulfide 2 Reagent.
- Wait 5 – 15 minutes.
- Read the absorbance of samples and QC samples.
- Calculate results.

10. Procedure

10.1 Initial Calibration

Prepare a calibration curve using the selected standards from Section 8.4

Analyze the standards as in Section 10.2.2 and 10.2.3 below, and plot concentration vs. absorbance. Determine the slope and the y-intercept. The correlation coefficient must be $\geq$ 0.995. A calibration curve is generated once a year of when significant changes are noted.

10.2 Equipment Operation and Sample Processing

10.2.1 Sample Preparation

10.2.1.1 **Soil samples:** Weigh 10 grams of the sample directly in reaction flask with a clean stir bar. Record the weight on excel form/ prep book.

10.2.1.2 **Aqueous samples:** Filter entire sample volume through a 90mm glass fiber filter and rinse with DI water. Place filter into the reaction flask with a clean Teflon stir bar. Record the volume of sample filtered on excel form/ prep book.

10.2.1.3 Add 200mL of 2N HCL Solution (Section 8.7) into the thistle tube, making sure that the stopcock is closed.

10.2.1.4 Add 20mL of 1N NaOH (Section 8.6) and 50mL of DI to the 250mL collection vessel.

10.2.1.5 Assemble the distillation train.

10.2.1.6 Connect the thistle flask to the center position of the reaction flask

10.2.1.7 Connect the collection vessel to the left side of the reaction flask, and the impinger tube to the collection vessel. The impinger tube must be below the surface of the NaOH solution. Make sure that all ground glass joints are sealed.

10.2.1.8 Connect the Nitrogen gas to the right side of the collection vessel. The gas inlet must be below the surface of the reaction flask solution.

10.2.1.9 Making sure that the train is a closed system, purge the train with Nitrogen for 5 minutes. Open the valve on the Nitrogen tank (Section 7.13) and open up the regulator so that 45 psi of Nitrogen is flowing (this will effectively give a flow rate of about 150mL/min).

10.2.1.10 Once the system has been purged for 5 minutes, open the stopcock fully on the thistle tube and release the 2N HCL solution into the reaction vessel. At this point the sample should be stirring. Close the stopcock as soon as all of the 2N HCL solution has been delivered to the reaction flask. Let the reaction progress for 1 hour.

10.2.1.11 After one hour has passed, dissemble the train and bring the total volume of the collection vessel to 100mL with DI**.**

10.2.2 **Color Development:** Transfer 25mL sample to a test tube. Add 1.0mL Sulfide 1 reagent (Section 8.2) and swirl to mix. Add 1mL of Sulfide 2 reagent (Section 8.3). Let the reaction continue for 5 minutes. Excessive mixing causes low results by loss of H_2S as a gas before it has had time to react. The presence of sulfide will be indicated by the appearance of blue color. Color development usually is complete in about 1 minute, but a longer time is often required for fading out of the initial pink color. Wait 5 to 15 minutes and make color comparisons. If zinc acetate was used, wait at least 10 minutes before making a visual color comparison.

10.2.3 **Absorbance Determination:** Set the spectrophotometer to a wavelength of 664nm. Zero the instrument with a portion of the reagent blank sample using a 1 cm cell. Read the absorbance of the samples and QC samples and record this information on the excel format.

If the absorbance is greater than the highest standard in the calibration curve, the sample must be diluted with 0.2N NaOH and reanalyzed. Record on excel format any dilutions that are prepared.

10.3 Continuing Calibration

See Sections 9.4

10.4 Preventive Maintenance

An instrument service company calibrates the spectrophotometers on a semi-annual basis. Certificates are kept on file.

11. Calculations

Calculate sulfide concentration using the calibration curve generated in Section 10.1:

$$\text{Sulfide (mg/L)} = \frac{\text{Absorbance} - \text{y intercept}}{\text{Slope}} \times \text{Dilution factor}$$

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedance and improper preservation are noted on the nonconformance report form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

Review of standards, blanks and standard response for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form.

If the ICV, CCV or LCS recovery of any parameter falls outside the designated acceptance range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked samples is suspect and is only reported for regulatory compliance purposes with the appropriate nonconformance action form. Immediate corrective action includes reanalyzing all affected samples by using any retained sample before the expiration of the holding time.

If the Method Blank result is greater than the reporting limit, and all associated sample results are non-detect, or greater than 10x the concentration of the Method Blank, the data may be reported and a narrative submitted for inclusion on the final report. If associated sample results are positive, but at a concentration less than 10x the concentration of the Method Blank, those samples are re-distilled and reanalyzed.

If the LCS fails acceptance criteria, the entire batch must be redigested and reanalyzed. If there is insufficient sample volume to redigest, then the Client Services Department is notified and the sample results are reported as estimated, with a narrative to be included on the final report.

If the Matrix Spike fails acceptance criteria, the non-conformance is narrated and included on the final report.

If %RPD for the Duplicate fails acceptance criteria, the non-conformance is narrated and included on the final report.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732 These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/1732 MDL/LOD/LOQ Generation
SOP/1739 IDC/DOC Generation
SOP/1728 Waste Management and Disposal SOP

16. Attachments

None.

Total Suspended Solids Dried at 103-105ºC

Total Volatile Suspended Solids Dried at 500°C

References: **SM 2540 D (for TSS) and SM 2540 E (for TVSS)**, Standard Methods for the Examination of Water and Wastewater. APHA-AWWA-WEF. Standard Methods Online.

1. Scope and Application

Matrices: This method is suitable for the determination of Total Suspended Solids (TSS) and Total Volatile Suspended Solids (TVSS) in potable, surface, and saline waters, as well as domestic and industrial wastewaters.

Definitions: Refer to Alpha Analytical Quality Manual.

"Solids" refer to matter suspended or dissolved in water or wastewater. Solids may affect water or effluent quality adversely in a number of ways. Waters with high dissolved solids generally are of inferior palatability and may induce an unfavorable physiological reaction in the transient consumer. For these reasons, a limit of 500mg dissolved solids/L is desirable for drinking waters. Highly mineralized waters also are unsuitable for many industrial applications. Waters high in suspended solids may be esthetically unsatisfactory for such purposes as bathing. Solids analyses are important in the control of biological and physical wastewater treatment processes and for assessing compliance with regulatory agency wastewater effluent limitations.

"Total solids" is the term applied to the material residue left in the vessel after evaporation of a sample and its subsequent drying in an oven at a defined temperature. Total solids include "total suspended solids," the portion of total solids retained by a filter, and "total dissolved solids," the portion that passes through the filter.

"Fixed solids" is the term applied to the residue of total, suspended, or dissolved solids after heating to dryness for a specified time at a specified temperature. The weight loss on ignition is called "volatile solids." Determinations of fixed and volatile solids do not distinguish precisely between inorganic and organic matter because the loss on ignition is not confined to organic matter. It includes losses due to decomposition or volatilization of some mineral salts.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

A well-mixed sample is filtered through a weighed standard glass-fiber filter, and the residue retained on the filter is dried to a constant weight. The increase in the weight of the filter represents the total suspended solids. If the suspended material clogs the filter and prolongs filtration, the difference between the total solids and the total dissolved solids may provide an estimate of the total suspended solids. Samples submitted for Total Volatile Suspended Solids analysis are also dried at a higher temperature to a constant weight.

2.1 Method Modifications from Reference

Filters are prepared and analyzed at 500°C.

Method specifies that duplicate determination should agree within 5% of their average weight; Alpha is using in-house control limits.

3. Reporting Limits

The RDL for both TSS and TVSS analysis is 5mg/L using 1000mL of sample and an analytical balance sensitive to 0.1mg. A lower RDL may be achieved by using a larger sample (more volume) or the use of a more sensitive balance.

Note: TSS-low method should be used for samples where client requested reporting limit lower than 5 mg/L. Reporting limit for low TSS-LOW method is 1.0 mg/L. Analyst must use 5 digit balances in order to achieve low reporting limit.

4. Interferences

4.1 Highly mineralized water with a significant concentration of calcium, magnesium, chloride, and/or sulfate may be hygroscopic and require prolonged drying, proper desiccation and rapid weighing.

4.2 Mix small samples with a magnetic stirrer. If suspended solids are present, pipet with widebore pipets. If part of a sample adheres to the sample container, consider this in evaluating and reporting results. Some samples dry with the formation of a crust that prevents water evaporation; special handling is required to deal with this. Avoid using a magnetic stirrer with samples containing magnetic particles.

4.3 Exclude large, floating particles or submerged agglomerates of non-homogeneous materials from the sample if it is determined that their inclusion is not desired in the final result.

4.4 Disperse visible floating oil and grease with a blender before withdrawing a sample portion for analysis.

4.5 Because excessive residue in the dish may form a water-trapping crust, limit sample to no more than 200mg residue.

4.6 For samples high in dissolved solids, thoroughly wash the filter to ensure removal of dissolved material.

4.7 Prolonged filtration times resulting from filter clogging may produce high results due to increased colloidal materials captured on the clogged filter.

4.8 If the sample has high sediment/sand content, it is necessary to use a smaller volume for analysis. This will allow for a smooth transfer of the filter to the drying tin.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples are collected in glass or plastic bottles, provided that the material in suspension does not adhere to container walls.

Sampling, subsampling, and pipetting two-phase or three-phase samples may introduce serious errors. Make and keep such samples homogeneous during transfer. Use special handling to insure sample integrity when subsampling.

6.2 Sample Preservation

None.

6.3 Sample Shipping

No specific requirements.

6.4 Sample Handling

Begin analysis as soon as possible because of the impracticality of preserving the sample. Refrigerate sample at 4 ± 2ºC up to the time of analysis to minimize microbiological decomposition of solids. In no case hold sample more than 7 days. Bring samples to room temperature before analysis.

7. Equipment and Supplies

7.1 Drying Tins: Aluminum.

7.1.1 50mm diameter: For use with 47mm filter disks (Section 7.2)

7.1.2 140mL capacity: For use with 90mm filter disks (Section 7.2)

7.2 Glass-Fiber Filter Disks: 90mm and 47mm diameter, without organic binder, Type A/B, size 1.0um; use with drying tins from Section 7.1.

Whatman Grade 934-AH ready-to-use (RTU) filters must be used : part# 9907-90 for 90 mm and 9907-047 for 47 mm filters

Note: Ready to use filters (RTU) can be used only for regular TSS method ;original filters must be used for method TSS-LOW, (must be weighed using 5 digit balances)

7.3 Filtration Apparatus: With a membrane filter funnel.

7.4 Side-arm Flask: Of sufficient capacity for sample size selected.

7.5 Dessicator: With a dessicant containing a color indicator of moisture concentration.

7.6 Drying Oven: For operation at 103 - 105ºC.

7.7 Analytical Balance: Capable of weighing to 0.1mg. % digit balances (capable of weighting 0.01 mg) must be used for TSS-LOW method.

7.8 Magnetic Stirrer: With TFE stirring bar.

7.9 Wide-bore Pipets: Various volumes.

7.10 Graduated Cylinders: Glass or plastic at 100mL, 500mL, 1000mL volumes.

7.11 Muffle Furnace: Capable of 500°C.

7.12 Porcelain Crucibles

7.13 Tweezers: Flat tip, non-piercing.

7.14 Wax crayon or Sharpie pen

7.15 Vacuum Pump for use at filtration station

8. Reagents and Standards

8.1 Reagent Water: Deionized (DI) water.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank

Filter one blank of 1000mL of DI water per batch of 20 samples or less. Results must be less than the Reporting Limit (RL). If Blank results are less than 10 times the RL and the sample results are greater than 10 times the RL, the data is reported. If Blank results are greater than 10 times the RL, and there is sufficient sample volume remaining, the entire batch is reset and reanalyzed. A narrative is included with the report.

9.2 Laboratory Control Sample (LCS)

Not Applicable.

9.3 Initial Calibration Verification (ICV)

Not Applicable.

9.4 Continuing Calibration Verification (CCV)

Not Applicable.

9.5 Matrix Spike

Not Applicable.

9.6 Laboratory Duplicate

Filter one sample in duplicate per batch of 10 samples or less. Duplicate determinations should be within in-house control limits. Current control limits are ≤ 11% and may be re-evaluated. If acceptance criteria are not met, and there is sufficient sample volume remaining and sample is within holding time, the sample is reset. Otherwise, the data is submitted a narrative is included with the final report.

Additionally, the average between sample and duplicate tare-corrected weights in g must be calculated. Duplicate determinations should agree within 5% of their average weight. This calculation has to be performed on excel format.

9.7 Method-specific Quality Control Samples

None.

9.8 Method Sequence

- Prepare the filters.
- Weigh the clean filters, and/or drying tins.
- Filter the appropriate volumes of sample, and QC samples.
- Rinse the filter with DI water.
- Transfer the filter into to its corresponding drying tin.
- Total Suspended Solids are dried for a minimum of 2 hours in a 103 – 105 °C oven.
- Cool in a dessicator until temperature is constant.
- Weigh the filters until the weight change is less than 4% of the previous weight or 0.5mg, whichever is less.
- Total Volatile Suspended Solids are also dried for a minimum of 2 hours in a 500 °C muffle furnace.
- Cool in a dessicator until temperature is constant.
- Weigh the filters until the weight change is less than 4% of the previous weight or 0.5mg, whichever is less.
- Calculate results.

Note: For regular TSS method (not TSS-LOW) use prepped filters (RTU filters). Record filter weight provided by the vendor.

10. Procedure

10.1 Equipment Set-up

10.1.1 Preparation of Glass-Fiber Filter Disk

10.1.1.1 Attach a side-arm flask to the filtration apparatus.

10.1.1.2 Insert a glass-fiber filter disk (Section 7.2) with the wrinkled side up into the filtration apparatus.

Note: Record the manufacturer and lot number of the filter in the batch computer worksheet, when generated.

10.1.1.3 Apply the vacuum, and wash the disk with three successive 20mL portions of DI water.

10.1.1.4 Continue the suction to thoroughly remove all traces of water, and discard washings.

10.1.1.5 Using tweezers, remove the filter from filtration apparatus and transfer to a drying tin (Section 7.1).

10.1.1.6 Dry the filter and tin in an oven at 103-105ºC for at least two hours.

Note: For TVSS filters must be pre-washed and dried at 500ºC for at least 2 hours.

10.1.1.7 Cool in a dessicator to a constant temperature, and weigh. Record the weights on the computer worksheet. Print a copy of the worksheet for later use in Section 10.3.

10.1.1.8 The dried filters may be used immediately, or stored in a dessicator until needed. Proceed to Section 10.3.

10.2 Initial Calibration

Not Applicable.

10.3 Equipment Operation and Sample Processing

10.3.1 Label drying tins with sample IDs. At the beginning of analysis, record the date/time/analyst's initials in the computer worksheet. Batch samples in groups of 10 or less, for each product, and enter the associated WG# into each computer worksheet. The worksheet is then ready for use when sample filtration begins (from Section 10.3.4).

10.3.2 Assemble filtering apparatus with a prepared filter from Section 10.1.1, attach a side-arm flask and apply vacuum. Wet filter with a small volume of DI water to seat it

10.3.3 Selection of Sample Sizes

Choose a sample volume to yield between 10 and 200mg dried residue. If more than 10 minutes are required to complete filtration, it is necessary to use another prepped filter (from Section 10.1.1) with a decreased sample volume.

If Total Dissolved Solids are to be determined, collect the filtrate into a clean side-arm flask. Refer to SOP/2219.

10.3.3.1 **Clean samples:** Shake sample container to ensure a well-mixed solution prior to pouring. Use a graduated cylinder to measure sample volume. Begin with a sample volume of 100mL. Continue filtering successive volumes of 100mL until the desired volume of 1000mL is reached. However, if the filtration begins to slow in efficiency during this time, do not add any more additional sample.

10.3.3.2 **Samples containing sediment or other material:** Begin with a smaller sample volume. (An initially large volume of these types of samples will likely clog the filter and require reanalysis.)

10.3.3.2.1 These types of samples require initial stirring on a magnetic stirrer. While stirring, withdraw a volume of sample using a wide-bore pipet (Section 7.9). Pipet the sample onto the filter, and then while holding the pipet over the filter, rinse the pipet with DI water and filter this rinseate as well. This will ensure that all of the solid material is transferred to the filter.

10.3.3.3 **Sludge samples:** These samples are always pipetted to the filter as stated above in Section 10.3.3.2.1. A small sample size is desirable to prevent crust formation and filter curling during the drying step of the procedure.

10.3.4 Write the volume of sample filtered on the computer worksheet that was printed in Section 10.1.1.7. next to the corresponding sample number, along with any necessary comments regarding matrix interference, limited volume, etc.

10.3.5 Wash with three successive 10mL volumes of DI water, allowing complete drainage between washings, and continue suction for about 3 minutes after filtration is complete.

10.3.6 Samples with high dissolved solids may require additional washings.

10.3.7 Using tweezers, carefully remove the filter from filtration apparatus and transfer to the corresponding, labeled drying tin.

10.3.8 Place the filter in the tin into a 103-105ºC oven to dry for a minimum of 2 hours. Record the date, time in and the oven temperature in the computer worksheet.

10.3.9 After drying, remove tin from the oven to cool in a dessicator to a constant temperature. Record the date, time out and the oven temperature in the computer worksheet.

10.3.10 Weigh the tin on an analytical balance. Record the weights on the computer worksheet, and the sample volumes filtered as written down from Section 10.3.3. Enter the sample volumes from the logbook onto the computer worksheet discussed in Section 10.1.1.7.

10.3.11 Repeat the cycle of drying, cooling, desiccating, and weighing until a constant weight is obtained or until the weight change is less than 4% of the previous weight or 0.5mg, whichever is less. Record each weight on the computer worksheet.

10.3.12 Total Volatile Suspended Solids

10.3.12.1 If TVSS analysis is requested, using tweezers, transfer the filter from the drying tin to a labeled crucible.

10.3.12.2 Place the crucible into a 500ºC muffle furnace for a minimum of 2 hours, and record the date, time in, and the oven temperature in the computer worksheet. Following removal from muffle furnace, place in a dessicator to cool to a constant temperature. Record the date, time out and the oven temperature in the computer worksheet.

10.3.12.3 Using an analytical balance, tare a clean drying tin. Remove the filter from the crucible with tweezers and transfer the filter to the drying tin.

10.3.12.4 Record the weights on the computer worksheet.

10.3.12.5 Repeat the cycle of drying, cooling, desiccating, and weighing until a constant weight is obtained or until the weight change is less than 4% of the previous weight or 0.5mg, whichever is less. Record each weight on the computer worksheet.

10.4 Continuing Calibration

Not Applicable.

10.5 Preventive Maintenance

10.5.1 Prior to each use, the filtering apparatus is rinsed thoroughly with DI water.

10.5.2 As needed, the filtering apparatus is washed in the industrial dishwasher.

10.5.3 As necessary, wipe the inside of the filtering apparatus with a paper towel and then rinse thoroughly with DI water

10.5.4 Ensure that there is an adequate supply of oil in the vacuum pump.

10.5.5 Monitor automated data logger system once daily for temperature of oven.

11. Calculations

$$\text{mg TSS / L} = \frac{(\mathbf{A} - \mathbf{B}) \times 1000}{\text{sample volume, (mL)}}$$

where:

A = Final weight (weight of filter + dried residue, g)
B = Initial weight (weight of clean filter, g)

$$\text{mg TVSS / L} = \frac{(\mathbf{G} - \mathbf{N}) \times 1000}{\text{sample volume, (mL)}}$$

where:

G = Final Weight at 105 °C (weight of dish + dried residue, g)
N = Weight at 500 °C (weight of dish + dried residue, g)

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedence and improper preservation are noted on the nonconformance report form.

Perform routine preventative maintenance as outlined in Section 10.5.

Review of blanks and duplicate samples for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

The MDL for TSS and TVSS is a calculated value; therefore, the MDL studies are not performed.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1734 and 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

Analysis of a weight-bearing, real-world sample is chosen to run four times in duplicate between two analysts (one of which must be a trained analyst), and the results must be within 10%.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

Analysis of a weight-bearing, real-world sample is chosen to run four times in duplicate between two analysts (one of which must be a trained analyst), and the results must be within 10%.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/1732 MDL/LOD/LOQ Generation

SOP/1734, 1739 IDC/DOC Generation

SOP/1728 Waste Management and Disposal SOP

16. Attachments

None.

Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)

References: **Method 8260C**, SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, 2006.

Method 5035A, SW-846, Closed System Purge & Trap and Extraction for Volatile Organics in Soil and Waste Samples. Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, Draft Revision I, July 2002.

Method 5030B, Purge & Trap for Aqueous Samples. SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update III, December, 1996.

Method 5030C, Purge & Trap for Aqueous Samples. SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update IV, May, 2003.

1. Scope and Application

Matrices: Method 8260 is used to determine volatile organic compounds in a variety of solid waste matrices. This method is applicable to nearly all types of samples, regardless of water content, including various air sampling trapping media, ground and surface water, aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments.

Definitions: Refer to Alpha Analytical Quality Manual.

The compounds listed in Table 5 may be determined by this method.

There are various techniques by which these components may be introduced into the GC/MS system. Purge-and-trap, by Methods 5030C (aqueous samples) and 5035A (solid and waste oil samples), is the most commonly used technique for volatile organic analytes. However, other techniques are also appropriate and necessary for some analytes. One technique is direct injection of an aqueous sample (concentration permitting).

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the gas chromatograph/mass spectrometers and in the interpretation of mass spectra and their use as a quantitative tool. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

The volatile compounds are introduced into the gas chromatograph by the purge-and-trap method or by direct injection. The analytes are introduced to a narrow-bore capillary column for analysis. The Gas Chromatograph (GC) is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS) interfaced to the GC.

Analytes eluted from the capillary column are introduced into the mass spectrometer via a direct connection. Identification of target analytes is accomplished by comparing their mass spectra with

the electron impact (or electron impact-like) spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard, comparing sample response to the calibration standards.

2.1 Method Modifications from Reference

None.

3. Reporting Limits

Table 1 lists our typical reporting limits.

4. Interferences

4.1 Impurities in the purge gas, organic compounds out-gassing from the plumbing ahead of the trap, and solvent vapors in the laboratory account for the majority of contamination problems. The analytical system must be free from contamination under the conditions of the analysis. Running laboratory reagent blanks as described in Section 9.1 and 10.3 demonstrates the system is free of contamination. The use of non-Teflon plastic tubing, non-Teflon thread sealants, or flow controllers with rubber components in the purge and trap system must be avoided.

4.2 Sample contamination occurs by diffusion of volatile organics (particularly fluorocarbons and methylene chloride) through the septum seal into the sample during shipment and storage. A trip blank or a field reagent blank prepared from reagent water and carried through the sampling and handling protocol serves as a check on such contamination.

4.2.1 Storage blanks shall be analyzed if contamination is suspect. If contamination is confirmed by positive detections in the sample storage blanks, all data from samples contained in the relative refrigerator or freezer shall be evaluated for possible contamination. If the samples contain suspected contamination, the Client Services department shall be notified in order to contact the necessary clients regarding the contamination. Samples shall be reanalyzed if so desired by the client. If suspected contamination is not confirmed by storage blanks, no further action shall be pursued concerning said blanks. It is recommended that further action be taken to determine the possible cause of suspected contamination.

4.3 Contamination by carry-over can occur whenever high level and low level samples are sequentially analyzed. Whenever a highly concentrated sample is being encountered, it should be followed by an analysis of reagent water (instrument blank) to check for potential contamination. If carry-over is suspected, then numerous instrument blanks may be required; additionally all affected samples are rerun for confirmation. In case of severe contamination, preventive maintenance of the entire system may be required.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

The following method analytes have been tentatively classified as known or suspected human or mammalian carcinogens: benzene, carbon tetrachloride, 1,4-dichlorobenzene, 1,2-dichloroethane, hexachlorobutadiene, 1,1,2,2-tetrachloroethane, 1,1,2-trichloroethane, chloroform, 1,2-dibromoethane, tetrachloroethene, trichloroethene, and vinyl chloride. Pure standard materials and stock standard solutions of these compounds should be handled in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

5.1 Lab coats, safety glasses, and gloves must be worn when handling samples, standards, or solvents.

5.2 All stock solution standard preparation must be performed in the volatiles hood. Initial calibration, continuing calibration, laboratory control sample and client sample dilutions do not need to be performed in the hood.

5.3 All expired standards must be placed into the waste bucket in the lab for future disposal. The container must be labeled properly with hazard warning labels indicating the container contents.

5.4 Bottles containing Methanol must be stored in the flammables cabinet.

6. Sample Collection, Preservation, Storage, Shipping and Handling

6.1 Sample Collection and Preservation

6.1.1 Aqueous Samples

Grab samples are collected in standard 40mL amber glass screw-cap vials with Teflon lined silicon septa (VOA vial). Two or more VOA vials should be filled per sample location. EPA Method 8260 requires that samples be acidified to eliminate the possibility of biological degradation. Unless otherwise directed for project-specific reasons, all VOA vials are delivered to the client with approximately 2 – 4 drops of 1:1 HCl added to the vial, which is sufficient to adjust the pH of the sample to < 2. Prepared trip blanks are provided to the client to accompany field samples for QC purposes.

Fill the sample vial to the point of overflowing so that no headspace is contained within. Samples must be introduced into the vials gently to reduce agitation, which might drive off volatile compounds or cause loss of the HCl preservative.

Seal the bottle so that no air bubbles are in the VOA vial. If preservative has been added, shake vigorously for one minute. Invert the bottle and tap to check for air bubbles. Recollect the samples if any air bubbles are present.

Maintain the hermetic seal on the VOA vial until time of analysis.

6.1.2 Soil Samples

The recommended sampling method for soil samples is EPA 5035A. Method 5035A provides for two distinct sampling procedures, depending on the required reporting limits and suspected or known concentration levels of target analytes. These methods are referred to as the High Level and Low Level methods. Both are listed below, but depending on the samples only one of the methods may be required. If concentration levels are unknown, it is recommended that samples be collected using both procedures. The Lab will analyze the high level sample first, followed by the low level sample if the

results from the high level analysis show that the sample is clean or contains analytes at low levels. The typical reporting levels of the two methods are listed in Table 1.

6.1.2.1 High Level Soil Samples

Collect sample in a standard 40mL amber glass screw-cap vial with Teflon lined silicon septa (VOA vial). The vial is provided containing 15mL of Purge and Trap Grade methanol, and is labeled and weighed prior to addition of sample. Record the weight of the vial with methanol on the vial label. Prepared trip blanks are provided to the client to accompany field samples for QC purposes.

Approximately 15g of soil is added to the vial in the field, making sure that the sample is completely covered by the methanol.

Maintain the hermetic seal on the VOA vial until the time of analysis.

An additional sample of the soil must also be obtained (without methanol) to be used for the determination of soil moisture content to allow for the calculation of the dry weight results, and to calculate the methanol dilution effect. (See Sections 11.1.2.2.2 and 11.1.2.2.3)

6.1.2.2 Low Level Soil Samples

Collect sample in a standard 40mL amber glass screw-cap vials with Teflon lined silicon septa (VOA vial). Two samples should be taken per sample location. Vials are provided containing a magnetic stirring bar and 5 mL of either 200g/L sodium bisulfate solution or water, prepared by a certified vendor. These vials are labeled and weighed prior to addition of sample. Record the weight of the vial with the stirring bar and preservative on the vial label.

Approximately 5g of soil is added to the vial in the field, making sure that the sample is completely covered by the sodium bisulfate solution or water.

Maintain the hermetic seal on the VOA until the time of analysis.

6.2 Sample Handling and Storage

Document client specific sample handling, preservation and collection criteria in the project file. The laboratory Log-in staff documents sample temperature at the time of receipt.

Record deviations from this SOP or client specific criteria on the chain of custody form.

Record holding time exceedence, improper preservation and observed sample headspace on the nonconformance report form.

6.2.1 Aqueous Samples

Ice or refrigerate all samples from the time of collection until analysis, maintaining the sample temperature between 1 and 4 °C. Sample receiving personnel note on the sample delivery group form when samples received at the laboratory are not within the temperature criteria. If more than one vial is received for a sample the vials are stored in separate refrigerators. Storing the vials apart provides a useful check if laboratory contamination of a sample is suspected. Samples must be analyzed within 14 days of collection. Unpreserved samples requiring aromatic analysis must be analyzed within 7 days of collection.

6.2.2 High Level Soil Samples

Ice or refrigerate all samples from the time of collection until analysis, maintaining the sample temperature between 2 and 6 °C. Sample receiving personnel note on the

nonconformance report form when samples received at the laboratory are not within the temperature criteria.

6.2.3 Low Level Soil Samples

Ice or refrigerate samples preserved with water or sodium bisulfate from the time of collection until analysis, maintaining the sample temperature between 2 and 6 °C. Samples preserved with water are to be immediately frozen after sampling. Sample receiving personnel note on the nonconformance report form when samples received at the laboratory are not within the temperature criteria.

6.3 Sample Shipping

Samples requiring shipment to the laboratory are shipped in ice-packed coolers via an overnight delivery service in accordance with applicable Department of Transportation regulations.

7. Equipment and Supplies

7.1 Purge and Trap System (For Aqueous samples and High Level Soils): The purge-and-trap system consists of two separate pieces of equipment: a purging device (autosampler) (Varian Archon/8100, Tekmar Solatek, EST Centurion) coupled to the desorber (concentrator) (Tekmar Velocity or EST Encon).

7.1.1 Purge gas = Helium, analytical grade (99.999%).

7.1.2 The purging device is configured with 25 mL sample purge tubes, and the helium purge gas is introduced at the bottom of the water column as finely divided bubbles

7.1.3 The trap used in the desorber is typically a Supelco “K” trap. Different traps may be used if equivalent performance is demonstrated.

7.1.4 The desorber is capable of rapidly heating the trap to 260^{o}C. The trap is not heated above manufacturer’s specifications

7.2. Purge and Trap System (For Low Level Soil Samples): The purge and trap system consists of two separate pieces of equipment: a purging device (autosampler) coupled to the desorber (concentrator) (Varian Archon/8100, Tekmar Solatek, EST Centurion with EST Encon, Tekmar Velocity, or equivalents).

7.2.1. Purge gas = Helium, analytical grade (99.999%).

7.2.2. The autosampler purging device is a closed system, designed to accept the 40mL VOA vials. The VOA vial, containing the soil sample, water (or sodium bisulfate), and stirring bar is placed into the autosampler tray. The instrument automatically adds reagent water, internal standards, and surrogates to the unopened VOA vial. The vial is heated to 40 °C, and the helium purge gas is introduced into the aqueous portion to purge the volatile components onto the trap.

7.2.3. The trap used in the desorber is typically a Supelco “K” trap. Different traps may be used if equivalent performance is demonstrated.

7.2.4. The desorber is capable of rapidly heating the trap to 260 °C. The trap is not heated above manufacturer specifications.

7.3 Gas Chromatography/Mass Spectrometer/Data System:

7.3.1 Gas Chromatograph, Agilent 6890/7890 or equivalent: An analytical system complete with a temperature-programmable gas chromatograph with appropriate interface for sample introduction device. The system includes all required accessories, including syringes, analytical columns, and gases. The capillary column is directly coupled to the source of the GC/MS system.

7.3.2 Typical Gas Chromatographic Columns:

7.3.2.1 Column 1: Restek 502.2, 40 meter, 0.18mm ID, or equivalent.

7.3.2.2 Column 2: Restek RTX-VMS, 30 meter, 0.25mm ID, or equivalent

7.3.3 Mass Spectrometer, Agilent 5973/5975/5978 or equivalent: Scanning from 35 to 300 amu every 2 seconds or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for 4-Bromofluorobenzene (BFB) which meets all of the criteria in Table 3, when 50ng of the GC/MS tuning standard (BFB) are injected through the GC. For all SIM analysis, the mass spectrometer must also be able to acquire data in a dual acquisition mode (SIM and full scan).

7.3.4 Data System: Hewlett-Packard EnviroQuant software is used for data acquisition, and allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program.

Thruput Target 4.12 software or Enviroquant E.02.02 (or equivalent) is used for data processing, and allows searching of any GC/MS data file for ions of a specified mass, and plotting such ion abundances versus time or scan-number.

The most recent version of the EPA/NIST Mass Spectral Library is loaded onto the Target / Enviroquant data system.

7.4 Wiretrol or Micro syringes: 10µL - 1,000µL.

7.5 Syringes: 5mL, 10mL, or 25mL, glass with Luerlock tip.

7.6 Balances: Top-loading, capable of weighing 0.01g.

7.7 Vials: 2mL, 4mL.

7.8 Disposable Pipets.

7.9 Volumetric Flasks: Class A, appropriate sizes, with ground-glass stoppers.

7.10 Eppendorf Pipets.

8. Reagents and Standards

Reagent grade organic chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all organic reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

Great care must be taken to maintain the integrity of all standard solutions. Standards in methanol are stored at –10ºC or less, in amber vials with PTFE-lined screw-caps.

8.1 Organic-free Reagent Water:

All references to water in this method refer to organic-free reagent water, which is tap water passed through activated carbon and air bubbled through.

8.2 Methanol:

Purge and Trap Grade or equivalent. Store in flammables cabinet.

8.3 Stock Solutions:

All stock standard solutions are purchased from commercial vendors as ampulated certified solutions. When an ampulated stock solution is opened, it is transferred to a labeled amber screw-cap vial with minimal headspace. The expiration date of the stock solution is either the vendor specified expiration date or 6 months from the date the ampule was opened, whichever is sooner. Typical stock standard concentrations are listed in Table 4.

8.4 Intermediate Standards: Intermediate standards are prepared volumetrically by diluting the appropriate stock standard(s) with methanol. Initial Calibration solutions expire 2 months from the date of preparation, or sooner if daily continuing calibration checks do not achieve the method acceptance criteria. If the Intermediate Standards are used as a second source to verify a valid Initial Calibration solution, there is no expiration date.

8.4.1 Internal Standard Solutions:

The internal standards are Fluorobenzene, Chlorobenzene-d_5, and 1,4-Dichlorobenzene-d_4. The intermediate IS solution is prepared by diluting the stock solution(s) with methanol to a concentration of 100 µg/mL. The appropriate amount of IS solution is added to the water or soil sample or QC sample to achieve a final concentration of 100 ng/sample or standard. Internal standard is added at the same concentration to all standards, samples, and QC samples.

8.4.2 Surrogate Standard Solutions:

The surrogate standards are Dibromofluoromethane, 1,2-Dichloroethane-d_4, Toluene-d_8, and 4-Bromofluorobenzene. The intermediate surrogate solution is prepared by diluting the stock solution(s) with methanol to a concentration of 100 µg/mL. The appropriate amount of surrogate solution is added to the water or soil sample or QC sample to achieve a final concentration of 100 ng/sample.

8.4.3 Target Compound Solutions:

The target analytes routinely reported by this method are listed in the beginning of this SOP. The intermediate target compound solutions are prepared by diluting the stock solution(s) with methanol. This set of solutions, at concentrations of 200 µg/mL, is used for preparation of the calibration standards.

8.4.4 4-Bromofluorobenzene (BFB) Tune solution:

A solution containing BFB at a concentration of 50 µg/mL is prepared by volumetrically diluting the BFB stock solution. 1 µL of this solution is direct-injected or purged into the GC/MS system to verify system performance prior to any standard or sample analysis.

8.5 Calibration Standards:

There are two types of calibration standards used for this method – initial calibration standards and calibration verification standards.

8.5.1 Initial Calibration Standards:

Initial calibration standards can be prepared at the levels listed in Table 4 (other/different levels are allowed). The Initial Calibration needs to have a minimum of 5 standards, 6 if a quadratic curve fit is used. Prepare these solutions in organic-free reagent water. The standards correspond to the range of concentrations found in typical samples and do not exceed the working range of the GC/MS system. Initial calibration should be mixed from fresh stock standards and dilution standards when generating an initial calibration curve.

8.5.2 Initial Calibration Verification Standard (ICV):

The initial calibration verification standard is at the same concentration as the level 3 initial calibration standard. This standard is made from a second source than the Initial Calibration Standards.

8.5.3 Continuing Calibration Verification Standard:

The continuing calibration verification standard, or calibration check standard, should be analyzed near the action level of the project. Since most projects are focused on achieving low reporting limits, the continuing calibration verification standard is at the same concentrations as the level 3 initial calibration standard. This standard is run at the beginning of each analytical sequence, following the BFB tune standard, to verify system performance.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

Blank samples must be matrix specific, i.e. methanol samples need to have methanol in the blank; sodium bisulfate samples need to have a sodium bisulfate blank analyzed; TCLP samples need a TCLP blank.

Analyze a matrix-specific blank each day prior to sample analysis to demonstrate that interferences from the analytical system are under control. The blank must contain the internal standards and surrogates.

Analyze the reagent water blank from the same source of water used for preparing the standards, QC samples and making sample dilutions. The method blank must not contain any target analytes at or above the compound reporting limits.

9.2 Laboratory Control Sample (LCS)/ Laboratory Control Sample Duplicate (LCSD)

A LCS/LCSD pair is analyzed at the beginning of each analytical sequence. Since the LCS contains the same compounds at the same concentrations as the continuing calibration check standard, the same analysis is used to satisfy both QC elements. The LCS/LCSD acceptance criteria are based on in-house control limits, unless specified by project/regulation.

9.3 Initial Calibration Verification (ICV)

Refer to Section 10.2.5.

9.4 Continuing Calibration Verification (CCV)

Refer to Section 10.4.4.

9.5 Matrix Spike/ Matrix Spike Duplicate

Upon Client Request, a matrix spike/matrix spike duplicate pair may be analyzed with each batch of 20 or less samples. The MS/MSD are sample aliquots spiked with the target compounds at the same concentration as the continuing calibration standard. The MS/MSD acceptance criteria are based on in-house control limits. If the MS/MSD does not meet the criteria, but the LCSD does, the failure may be attributed to sample matrix. Report the MS/MSD, including a narrative sheet for inclusion with the client report.

9.6 Laboratory Duplicate

Not applicable.

9.7 Method-specific Quality Control Samples

9.7.1 Internal Standards

Area counts of the internal standard peaks in all samples and QC samples must be between 50-200% of the areas of the internal standards in the QC check standard.

If any individual percent recovery falls outside the range, that parameter has failed the acceptance criteria. For calibration standards, CCVs, LCS/LCSD or blanks the internal standard must be within the range for data to be reported to the clients. For samples, matrix spikes and duplicates: if the data is not within the range, the sample is rerun to confirm that the failure is due to sample matrix. A nonconformance report form is completed to ensure client notification and reporting if matrix effect is confirmed.

9.7.2 Surrogates

Surrogates are added to each field sample and QC sample. The laboratory must evaluate surrogate recovery data from individual samples versus the surrogate control limits developed by the laboratory. The surrogate acceptance criteria are listed in Table 2. Since the SIM analysis is acquired in dual mode, the surrogates from the full scan are used to evaluate the entire sample (SIM and full scan).

9.8 Method Sequence

In a 12-hour period, the typical analytical sequence is as follows:

- BFB
- QC Check Standard/Laboratory Control Sample/LCSD
- Method Blank
- Samples
- MS/MSD (upon Client request, may be run any time after the Method Blank)

10. Procedure

10.1 Equipment Set-up

Typical instrument operating conditions are listed below. Alternate conditions are allowed, as long as method performance criteria can be met.

10.1.1 GC Conditions:

Temperature 1:	35°C	Carrier gas:	Helium, 99.999%
Hold Time 1:	4 minutes	Carrier mode:	Constant flow
Ramp 1:	6°C/minute	Carrier flow:	1 mL/minute
Temperature 2:	150°C		
Hold Time 2:	0 minutes		
Ramp 2:	8°C/minute		
Temperature 3:	220°C		
Final Time:	1 minute		

10.1.2 MS Conditions:

Mass scan range:	35 – 260 amu
Scan time:	0.5 minutes/scan
Source temperature:	230°C

10.1.3 Velocity Concentrator Purge and Trap Conditions:

Purge time:	11 minutes
Dry purge:	2 minutes
Desorb preheat:	250°C
Desorb temp:	255°C
Desorb time:	2 minutes
Bake temp:	290°C
Bake time:	10 minutes

10.1.4 Encon Concentrator Purge and Trap Conditions:

Purge time: 11 minutes
Dry purge: 1 minute

Desorb preheat: 245°C
Desorb temp: 255°C
Desorb time: 1 minute

Bake temp: 270°C
Bake time: 10 minutes

10.2 Initial Calibration

10.2.1 The initial calibration is performed at a minimum of five (5) concentration levels listed in Table 4, the low level of the either at or below the reporting limit. The calibration is performed using instrument conditions listed in Section 10.1.

BFB must be analyzed prior to analysis of the initial calibration standards, and must pass the criteria listed in Table 3. The mass spectrum of BFB should be acquired in the following manner:

(1) Three scans (the peak apex scan, the scan immediately preceding the apex and the scan immediately following the apex) are acquired and averaged.
(2) Background subtraction is performed using a single scan of no more than 20 scans prior to the elution of BFB.

This is done automatically with the ThruPut Target / Enviroquant software.

10.2.1.1 Low Level/High Level Soil Curve on Archon or Centurion: To prepare a calibration standard, add the appropriate volume of standard solution(s) to a 50mL volumetric flask using a micro syringe. Remove the needle quickly and mix by inverting the flask 3 times. Pour several mLs of the aqueous standard into the waste vessel, then gently fill a 5mL syringe with standard and transfer to a 40mL VOA vial containing a magnetic stir bar. Load the vial onto autosampler.

10.2.1.2 Aqueous/High Level Soil Curve on Solatek or Centurion: To prepare a calibration standard, add the appropriate volume of standard solution(s) to a 100mL volumetric flask using a micro syringe. Remove the needle quickly and mix by inverting the flask 3 times. Pour several mLs of the aqueous standard into the waste vessel, then gently fill a 40mL VOA vial to the top. Load the vial onto the autosampler.

10.2.2 Establish the GC operating conditions by loading the appropriate GC method. Typical instrument conditions are listed in Section 10.1. The same operating conditions are used for calibration and sample analyses. Create the analytical sequence using the HP Enviroquant data acquisition software.

Relative Response Factors: The internal standard calibration technique is used. In each calibration standard, calculate the relative response factor for each analyte and the relative standard deviation (RSD) of the response factors using the Target / Enviroquant data processing software. The response factors are calculated using the areas of the characteristic (quantitation) ion for each target analyte and internal standard. The calculations are performed automatically using the Target / Enviroquant software, using the formulae listed in Alpha's Quality Manual.

10.2.3 Initial Calibration Criteria: The following sections outline the method acceptance criteria for an initial calibration curve. All criteria must be met for the calibration to be deemed acceptable, and for sample analysis to proceed.

10.2.3.1 Relative Standard Deviation Criteria: If the RSD for each target analyte is less than or equal to 20%, then the response for this compound is considered linear over the calibration range and the mean calibration factor can be used to quantitate sample results. If the 20% RSD criterion is not met for an analyte linear regression may be used if **r** $\geq$ 0.990, weighted linear with a weighting factor of 1/SD2 and r > 0.990, or quadratic fit if $\mathbf{r^2} \geq 0.995$. A minimum of six points is required and the low point of the calibration must be re-quantitated and recover within 70-130% to be deemed acceptable. The calibration must be repeated for any compounds that fail. If more than 10% of the compounds exceed the 20% RSD limit and do not achieve the minimum correlation coefficient for alternative curve fits, sample analysis cannot proceed.

10.2.3.2 Minimum Response Factors: Table 1 lists the suggested minimum response factors for the most common analytes. Each calibration level must be evaluated against the specified criteria. Analytes that fall below the criteria, but are greater than or equal to 0.05, are narrated for inclusion on the final report. There are certain very poor purgers (1,4-Dioxane, Acrolein, ketones, alcohols and other water soluble compounds) that should meet a 0.001 response factor. If an analyte falls below 0.05 (or 0.001 for 1,4-Dioxane, Acrolein, ketones, alcohols and other water soluble compounds), then corrective action must be taken to resolve the problem before analysis can proceed.

10.2.4 Evaluation of Retention Times: The relative retention times used for identification of target analytes are +/- 0.06 RRT (Relative Retention Time) units, based on the most recent standard run. It has been determined that these limits work well, being wide enough to eliminate false-negative results while being tight enough to eliminate false positive results. Due to the selectivity of the mass spectrometer, compound identification is more definitive than when using a less selective detector.

10.2.5 Initial Calibration Verification: After each calibration and before the analysis of samples, an ICV must be analyzed at or near the midpoint of the curve. The ICV must be prepared using a different source than the Initial Calibration and must contain all target analytes. The percent recoveries must be between 70% and 130% for target analytes except for “difficult” analytes (Table 7), which must exhibit percent recoveries between 40% and 160%. Corrective action is required if greater than 10% of all analytes are outside the prescribed criteria.

10.3 Equipment Operation and Sample Processing

The same GC, MS, and Purge and Trap conditions used for the initial calibration must be employed for sample analysis. After verification of system performance by analysis of BFB, the continuing calibration standard and method blank, samples are analyzed and processed as described below.

10.3.1 Analysis of Samples

10.3.1.1 All samples are initially screened using Tekmar 7000 Headspace Autosamplers.

10.3.1.2 Retrieve sample VOA vials from the sample refrigerator just prior to loading onto the purge and trap system. High level soil samples must be shaken for 1 – 2 minutes to extract the volatile components into the methanol. Let sample settle

prior to taking methanol aliquot. Low level soil sample should be shaken briefly to ensure that the stir bar is loose, and will spin on the Archon or Centurion unit.

10.3.1.3 Low level soil samples: (Archon or Centurion)
Take the low level VOA vial and place directly into the rack of the Archon sampling unit. Surrogate and internal standards are added automatically by the Archon prior to sample purging.

10.3.1.4 Aqueous samples: (Solatek or Centurion)
Load the VOA vial directly on the sampling rack. Dilutions may be prepared volumetrically and poured into VOA vials ensuring there is no headspace left in the vial. The auto-sampler will then sample 10mL from the VOA vial.

10.3.1.5 High level soil samples: (Archon/Solatek/Centurion)

Shake for 2 minutes, ensuring the methanol has completely penetrated the soil in the vial.

10.3.1.5.1 Through liquid path
Load a maximum of 430µL or appropriate dilution of the methanol into a half-full VOA vial. Fill the VOA vial up to the top with water and cap with no headspace. Allow the auto-sampler to sample 10mL out of the VOA vial which would be equivalent to injecting 100µL of the methanol extract. Prepare dilutions accordingly.

10.3.1.5.2 Through soil path
Into a VOA vial with a stir bar added, load 4.9mL of water plus a maximum of 100 µL of methanol or appropriate dilution of methanol extract from a 5mL luerlock syringe. Cap the vial and load onto the auto-sampler.

10.3.2 Qualitative Analysis:

10.3.2.1 The qualitative identification of each compound is based on retention time and on comparison of the sample mass spectrum with the reference mass spectrum. The reference mass spectrum must be generated by the laboratory on the same GC/MS system. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds are identified as present when the following criteria are met:

10.3.2.1.1 The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. The Target / Enviroquant data system is configured to make this check.

10.3.2.1.2 The relative retention time (RRT) of the sample component is within ±0.06 RRT units of the RRT of the standard component.

10.3.2.1.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%.)

10.3.2.1.4 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs (i.e., m and p-xylene).

10.3.2.1.5 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important.

10.3.2.1.6 Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification of compounds. When analytes coelute (i.e., only one chromatographic peak is apparent), the identification criteria may be met, but each analyte spectrum will contain extraneous ions contributed by the coeluting compound.

10.3.2.2 For samples containing non-target analytes, a library search will be performed at client request. Compound identification will be classified as “tentative”, and the concentration will be reported as an estimate as no quantitative standards are run for these compounds.

1) Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.
2) The relative intensities of the major ions should agree within ±20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%.)
3) Molecular ions present in the reference spectrum should be present in the sample spectrum.
4) Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.
5) Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks.

10.3.3 Quantitative Analysis:

10.3.3.1 Quantitation of a target compound detected in a sample is performed automatically by the Enviroquant data processing software, using the formulae found in Alpha’s Quality Manual. Either the average response factor or calibration curve will be used for sample quantitation, depending on how the particular analyte was processed in the initial calibration curve.

10.3.3.2 If non-target compounds are to be reported, the quantitation is performed automatically by the Enviroquant software using the total area of the compound and the nearest internal standard, and assuming a relative response factor of 1.0.

10.4 Continuing Calibration

Calibration verification consists of three steps that are performed at the beginning of each 12-hour analytical shift.

10.4.1 Prior to the analysis of samples or calibration standards, inject or purge 1 µL (50 ng) of the 4-Bromofluorobenzene standard (Section 8.4.4) into the GC/MS system. The resultant mass spectra for the BFB must meet the criteria given in Table 3 before sample analysis begins.

10.4.2 The initial calibration curve for each compound of interest must be verified once every 12 hours prior to sample analysis. This is accomplished by analyzing the continuing calibration check standard (Section 8.5.3).

10.4.3 A method blank must be analyzed prior to any samples, typically immediately following the continuing calibration check standard, to ensure that the analytical system is free of contaminants. The method blank must not contain any target analytes at or above the required compound reporting limits.

10.4.4 The percent difference or drift for each target analyte must be less than or equal to 20% (30% for all SIM compounds). If greater than 20% of target analytes exceed the %D criteria corrective action must be taken prior to the analysis of samples. If less than or equal to 20% of compounds exceed the criteria, corrective action is not required.

10.4.5 The continuing calibration standard must also be evaluated for the suggested minimum response factor criteria, as specified in section 10.2.3.2

10.4.6 Internal Standard Retention Time:

The retention times of the internal standards in the calibration verification standard are evaluated after data acquisition. If the retention time for any internal standard changes by more than 30 seconds from that in the mid-point standard level of the most recent initial calibration sequence, then the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

10.4.7 Internal Standard Response:

If the area for any of the internal standards in the calibration verification standard changes by a factor of two (-50% to +100%) from that in the mid-point standard level of the most recent initial calibration sequence, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, re-analysis of samples analyzed while the system was malfunctioning is required.

10.5 Preventive Maintenance

Routine preventive maintenance should be performed on the analytical system. This includes replacement of GC septa and periodic rinsing or replacement of purge and trap tubes and sparge needles. The trap should be replaced every six months, or sooner if performance criteria cannot be met. Periodic cleaning (typically twice per year) of the mass spectrometer ion source is required. More frequent source cleaning may be needed, especially if dirty samples are analyzed.

If system performance deteriorates, additional maintenance may be required. This includes replacement of injector ports and seals, clipping several inches off of the front end of the GC column, or in extreme cases the replacement of the GC column. Flushing or replacement of purge and trap lines may be necessary if they become contaminated or develop active sites.

Perform routine preventative maintenance as described throughout this SOP. Record all maintenance in the instrument logbook.

11. Data Evaluation, Calculations and Reporting

11.1.1 LIMS Data Corrections

Please note that the Laboratory Information Management System (LIMS) automatically adjusts soil sample results to account for the % Total Solids of the sample (as determined per Alpha SOP/07-38) and the methanol preservation dilution effect.

11.1.2 Data Calculations

11.1.2.1 Results of Aqueous Sample Analysis:

$$\text{Concentration (ug/L)} = \frac{(Conc.)\,(Vp)\,(DF)}{(Vs)}$$

where:

Conc. = On-column concentration obtained from the quantitation report.
Vp = Volume purged, 10 mL is standard
Vs = Volume of sample purged
DF = Dilution factor, for manually prepared dilutions, not instrumental "dilutions".

11.1.2.2 Results of Sediment/Soil, Sludge, and Waste Analysis:

All solids including soils, sediments, and sludges must be reported on a dry-weight basis.

11.1.2.2.1 Low-Level Samples:

$$\text{Concentration (ug/Kg)} = \frac{(Conc.)\,(Vp)\,(DF)}{(W)\,(\%S)}$$

11.1.2.2.2 High-Level Samples:

$$\text{Concentration (ug/Kg)} = \frac{\text{(Conc.) (Vp) (5000) (DF)}}{\text{(W) (Ve) (\%S)}}$$

where:

Conc. = On-column concentration obtained from the quantitation report.
DF = Dilution factor, for manually prepared dilutions, not instrumental "dilutions".
Ve = Extract volume, mL
Vp = Volume purged, 5 mL is standard
W = Aliquot of sample (wet), g
%S = Sample % solid
5000 = Constant representing the final volume of the methanol extraction.

11.1.2.2.3 High-Level Samples Corrected for Total Water/Solvent Mixture (V_t):

Samples that are extracted prior to analysis in a water miscible solvent such as methanol are diluted by the total volume of the water/solvent mixture. The total mixture volume can only be calculated based on the sample moisture present as determined by the % moisture calculation.

$$\text{\% moisture} = \frac{\text{g of sample – g of dry sample}}{\text{g of sample}} \times 100$$

$$V_t = \frac{\text{[mL of solvent + (\%moisture x g of sample)]}}{100} \times 1000\text{mL/mL}$$

The calculated V_t value is now added to the volume of methanol in the sample (typically 5000µL), and the corrected concentration is calculated using the equation below:

$$\text{Corrected concentration (mg/Kg)} = \frac{\text{(Conc.) } (V_t + \text{methanol vol.}) \text{ (Vp) (DF)}}{\text{(W) (Ve) (\%S)}}$$

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

All batch and sample specific QC criteria outlined in section 10 are evaluated by the analyst prior to approval of the data. When any QC criteria fail, the cause for the failure must be identified and corrected. This may include instrument recalibration followed by sample reanalysis, sample cleanup, or sample re-extraction. If it is determined that the failure is due to sample matrix effects, a project narrative report is written by the analyst for inclusion in the data report. If there is insufficient sample volume to perform the re-analysis for confirmation, this is also noted in the narrative and included in the client report.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/08-05. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/08-12 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/08-05 MDL/LOD/LOQ Generation

SOP/08-12 IDC/DOC Generation

SOP/14-01 Waste Management and Disposal SOP

16. Attachments

TABLE 1: 8260 REPORTING LIMITS

TABLE 2: 8260 QC ACCEPTANCE CRITERIA

TABLE 3: BFB TUNING CRITERIA

TABLE 4: STANDARD SOLUTIONS and ICAL Levels

TABLE 5: 8260C Volatile Internal Standards with Corresponding Target Compounds and Surrogates Assigned for Quantitation

TABLE 6: 8260C Quantitation Ions

TABLE 7: Difficult Analytes

Table 1
Standard Reported Detection Limits
US EPA METHOD 8260C and 5035A/8260C

Analyte	Recommended Minimum Response Factor	RDL (µg/L)	RDL(µg/KG)(1)	RDL (µg/KG) (2)
Acetone (3,4,5)	0.100	5.0	10	250
Acrolein (5)		5.0	25	1250
Acrylonitrile (3,4)		5.0	5	200
Allyl Chloride (7)		N/A	5	250
Benzene (3,4,5)	0.500	0.5	1	50
Bromobenzene (3,4)		2.5	5	250
Bromochloromethane (3,4,5)		2.5	5	250
Bromodichloromethane (3,4,5)	0.200	0.5	1	50
Bromoform (3,4,5)	0.100	2.0	4	200
Bromomethane (3,4,5)	0.100	1.0	2	100
2-Butanone (3,4,5)	0.100	5.0	10	500
Butyl acetate (7)		N/A	5	50
n-Butyl benzene (3,4)		0.5	1	50
sec-Butyl benzene (3,4)		0.5	1	50
tert-Butyl benzene (3,4)		2.5	5	250
Carbon disulfide (3,4,5)	0.100	5.0	10	500
Carbon tetrachloride (3,4,5)	0.100	0.5	1	50
Chlorobenzene (3,4,5)		0.5	1	50
Chlorodifluoromethane (7)		N/A	5	250
Chloroethane (3,4,5)	0.100	1.0	2	100
2-Chloroethylvinyl ether (3)		10.0	20	1000
Chloroform (3,4,5)	0.200	0.75	1.5	75
Chloromethane (3,4,5)	0.100	2.5	5	250
o-Chlorotoluene (3,4)		2.5	5	250
Cyclohexane (5)	0.100	10	20	1000
Cyclohexanone		10	20	1000
p-Chlorotoluene (3,4)		2.5	5	250
cis-Decahydronaphthalene (7)		N/A	5	250
trans-Decahydronaphthalene (7)		N/A	5	250
n-Decane (7)		N/A	5	250
Dibromochloromethane (3,4,5)	0.100	0.5	1	50
1,2-Dibromo-3-chloropropane (3,4,5)	0.050	2.5	5	250
1,2-Dibromoethane (3,4,5)	0.100	2.0	5	250
Dibromomethane (3,4)		5.0	10	500
1,2-Dichlorobenzene (3,4,5)	0.400	2.5	5	250
1,3-Dichlorobenzene (3,4,5)	0.600	2.5	5	250
1,4-Dichlorobenzene (3,4,5)	0.500	2.5	5	250
1,4-Dichlorobutane (3,4)		5.0	10	500
trans-1,4-Dichloro-2-butene (3,4)		2.5	5	250

Dichlorodifluoromethane (3,4,5)		5.0	10	500
1,1-Dichloroethane (3,4,5)	0.200	0.75	1.5	75
1,2-Dichloroethane (3,4,5)	0.100	0.5	1	50
1,1-Dichloroethene (3,4,5)	0.100	0. 5	1	50
cis-1,2-Dichloroethene (3,4,5)	0.100	0.5	1	50
trans-1,2-Dichloroethene (3,4,5)	0.100	0.75	1.5	75

Table 1 (continued)

Standard Reported Detection Limits

US EPA METHOD 8260C and 5035A/8260C

Analyte	Recommended Minimum Response Factor	RDL (µg/L)	RDL(µg/KG)(1)	RDL (µg/KG) (2)
1,2-Dichloropropane (3,4,5)	0.100	1.75	3.5	175
1,3-Dichloropropane (3,4)		2.5	5	250
2,2-Dichloropropane (3,4)		2.5	5	250
1,1-Dichloropropene (3,4)		2.5	2.5	250
cis-1,3-Dichloropropene (3,4,5)	0.200	0.5	1	50
p-Diethylbenzene (4)		2.0	4	200
Diisopropyl Ether (6)		2.0	4	200
1,4-Dioxane (5) (non-SIM)		250	100	5000
trans-1,3-Dichloropropene (3,4,5)	0.200	0.5	1	50
Ethanol (7)		N/A	1000	50000
Ethyl acetate		10.0	20	1000
Ethylbenzene (3,4,5)	0.100	0.5	1	50
Ethyl ether (3,4)		2.5	5	250
4-Ethyltoluene (4)		2.0	4	200
Ethyl methacrylate (3,4)		5.0	10	500
Ethyl-Tert-Butyl-Ether (6)		2.0	4	200
Freon-113 (5)		10.0	20	1000
n-Heptane (7)		N/A	5	250
Hexachlorobutadiene (3,4)		0.5	5	250
Hexachloroethane (7)		N/A	5	250
Hexane		1.0	1.0	50
2-Hexanone (3,4,5)	0.100	5.0	10	500
Iodomethane		5.0	5.0	250
Isopropyl Alcohol (IPA)		25		
Isopropylbenzene (3,4,5)	0.100	0.5	1	50
p-Isopropyltoluene (3,4)		0.5	1	50
Limonene (7)		N/A	5	250
Methyl Acetate (5)	0.100	20	20	1000
Methylene chloride (3,4,5)	0.100	3.0	10	500
Methyl Cyclohexane (5)	0.100	20	4	200
Methyl isothiocyanate (7)		N/A	5	250
Methyl Methacrylate		1.0	5	250

4-Methyl-2-pentanone (3,4,5)	0.100	5.0	10	500
Methyl-tert-butyl-ether (3,4,5)	0.100	1.0	2	100
Naphthalene (3,4)		2.5	5	250
Nitrobenzene (7)		N/A	5	250
2-Nitropropane (7)		N/A	5	250
n-Nonane (7)		N/A	5	250
n-Octane (7)		N/A	5	250
n-Butanol (5)		100	200	10000
n-Propylbenzene (3,4)		0.5	1	50
n-Propyl bromide		5.0		
Pentachloroethane		2.0	N/A	N/A
Styrene (3,4,5)	0.300	1.0	2	100
Tert-Butyl Alcohol (5)		30	100	5000
Tertiary-Amyl Methyl Ether (6)		2.0	4	200
Analyte	**Recommended Minimum Response Factor**	**RDL (µg/L)**	**RDL(µg/K**	**RDL(µg/K**
1,1,1,2-Tetrachloroethane (3,4)		0.5	1	50
1,2,4,5-Tetramethylbenzene (4)		2.0	4	200
1,1,2,2-Tetrachloroethane (3,4,5)	0.300	0.5	1	50
Tetrachloroethene (3,4,5)	0.200	0.5	1	50
Tetrahydrofuran (3)		10.0	20	1000
Toluene (3,4,5)	0.400	0.75	1	75
1,2,3-Trichlorobenzene (3,4,5)		2.5	5	250
1,2,4-Trichlorobenzene (3,4,5)	0.200	2.5	5	250
1,3,5-Trichlorobenzene (6)		2.0	5	250
1,1,1-Trichloroethane (3,4,5)	0.100	0.5	1	50
1,1,2-Trichloroethane (3,4,5)	0.100	0.75	1.5	75
Trichloroethene (3,4,5)	0.200	0.5	1	50
Trichlorofluoromethane (3,4,5)	0.100	2.5	5	250
1,2,3-Trichloropropane (3,4)		5.0	10	500
1,2,4-Trimethylbenzene (3,4)		2.5	5	250
1,3,5-Trimethylbenzene (3,4)		2.5	5	250
n-Undecane (7)		N/A	5	250
Vinyl acetate (3,4)		5.0	10	500
Vinyl chloride (3,4,5)	0.100	1.0	2	100
m/p-Xylenes (3,4,5)	0.100	1.0	2	100
o-Xylene (3,4,5)	0.300	1.0	2	100
1,4-Dioxane (5) SIM		3.0		
1,1,2,2-Tetrachloroethane SIM		0.1		

(1) Detection Limits are for Low-level Aqueous preserved samples.

(2) Detection Limits are for High-level Methanol preserved samples.
(3) Analyte reported by standard 8260 reporting list.
(4) Analyte reported by New York TCL reporting list.
(5) Analyte reported by New Jersey TCL reporting list.
(6) Analyte reported for New Hampshire in addition to standard 8260 reporting list.
(7) Analyte only reported for New York TCL report upon client request.

Note: Reporting Limits are based on standard 8260 reporting list, RL's may vary for New York and New Jersey reporting lists.

Table 2

QUALITY CONTROL ACCEPTANCE CRITERIA

Surrogate Spike Percent Recovery	Aqueous Limits		Soil Limits	
	Lower Control Limit	Upper Control Limit	Lower Control Limit	Upper Control Limit
1,2-Dichloroethane-d_4	70%	130%	70%	130%
4-Bromofluorobenzene	70%	130%	70%	130%
Toluene-d_8	70%	130%	70%	130%
Dibromofluoromethane	70%	130%	70%	130%

Table 3

BFB (4-BROMOFLUOROBENZENE) MASS INTENSITY CRITERIA

m/z	Required Intensity (relative abundance)
50	15 to 40% of m/z 95
75	30 to 60% of m/z 95
95	Base peak, 100% relative abundance
96	5 to 9% of m/z 95
173	Less than 2% of m/z 174
174	Greater than 50% of m/z 95
175	5 to 9% of m/z 174
176	Greater than 95% but less than 101% of m/z 174
177	5 to 9% of m/z 176

Table 4

Stock Standard Concentrations and Suggested Calibration Concentration Levels

Soil	Stock (µg/mL)	Level 0 (ug/kg)	Level 1 (ug/kg)	Level 1.5 (ug/kg)	Level 2 (ug/kg)	Level 3 (ug/kg)	Level 4 (ug/kg)	Level 5 (ug/kg)	Level 6 (ug/kg)	Level 7 (ug/kg)	Level 8 (ug/kg)
Fluorobenzene	2500	20	20	20	20	20	20	20	20	20	20
Dichlorodifluoromethane	2000	0.5	1	2	4	20	40	60	100	200	300
Chlorodifluoromethane	2000	0.5	1	2	4	20	40	60	100	200	300
Chloromethane	2000	0.5	1	2	4	20	40	60	100	200	300
Vinyl chloride	2000	0.5	1	2	4	20	40	60	100	200	300
Bromomethane	2000	0.5	1	2	4	20	40	60	100	200	300
Chloroethane	2000	0.5	1	2	4	20	40	60	100	200	300
Trichlorofluoromethane	2000	0.5	1	2	4	20	40	60	100	200	300
Ethyl ether	2000	0.5	1	2	4	20	40	60	100	200	300
Ethanol	2000	N/A	20	N/A	80	200	400	600	1000	3000	4000
1,1-Dichloroethene	2000	0.5	1	2	4	20	40	60	100	200	300
Carbon disulfide	2000	0.5	1	2	4	20	40	60	100	200	300
Freon-113	2000	0.5	1	2	4	20	40	60	100	200	300
Iodomethane	2000	0.5	1	2	4	20	40	60	100	200	300
Acrolein	2000	0.5	1	2	4	20	40	60	100	200	300
Allyl chloride	2000	0.5	1	2	4	20	40	60	100	200	300
Methylene chloride	2000	0.5	1	2	4	20	40	60	100	200	300
Isopropyl alcohol	2000	N/A	20	N/A	80	200	400	600	1000	3000	4000
Acetone	2000	0.5	1	2	4	20	40	60	100	200	300
trans-1,2-Dichloroethene	2000	0.5	1	2	4	20	40	60	100	200	300
Methyl acetate	2000	0.5	1	2	4	20	40	60	100	200	300
Hexane	2000	0.5	1	2	4	20	40	60	100	200	300
Methyl tert-butyl ether	2000	0.5	1	2	4	20	40	60	100	200	300
tert-Butyl alcohol	2000	2.5	5	10	20	100	200	300	500	1000	1500
Diisopropyl ether	2000	0.5	1	2	4	20	40	60	100	200	300
1,1-Dichloroethane	2000	0.5	1	2	4	20	40	60	100	200	300
Halothane	2000	0.5	1	2	4	20	40	60	100	200	300
Acrylonitrile	2000	0.5	1	2	4	20	40	60	100	200	300
Ethyl tert-butyl ether	2000	0.5	1	2	4	20	40	60	100	200	300
Vinyl acetate	2000	0.5	1	2	4	20	40	60	100	200	300
cis-1,2-Dichloroethene	2000	0.5	1	2	4	20	40	60	100	200	300
2,2-Dichloropropane	2000	0.5	1	2	4	20	40	60	100	200	300
Bromochloromethane	2000	0.5	1	2	4	20	40	60	100	200	300
Cyclohexane	2000	0.5	1	2	4	20	40	60	100	200	300

Soil	Stock (µg/mL)	Level 0 (ug/kg)	Level 1 (ug/kg)	Level 1.5 (ug/kg)	Level 2 (ug/kg)	Level 3 (ug/kg)	Level 4 (ug/kg)	Level 5 (ug/kg)	Level 6 (ug/kg)	Level 7 (ug/kg)	Level 8 (ug/kg)
Chloroform	2000	0.5	1	2	4	20	40	60	100	200	300
Ethyl acetate	2000	0.5	1	2	4	20	40	60	100	200	300
Carbon tetrachloride	2000	0.5	1	2	4	20	40	60	100	200	300
Tetrahydrofuran	2000	0.5	1	2	4	20	40	60	100	200	300
Dibromofluoromethane	2500	20	20	20	20	20	20	20	20	20	20
1,1,1-Trichloroethane	2000	0.5	1	2	4	20	40	60	100	200	300
2-Butanol	2000	N/A	20	N/A	80	200	400	600	1000	3000	4000
2-Butanone	2000	0.5	1	2	4	20	40	60	100	200	300
1,1-Dichloropropene	2000	0.5	1	2	4	20	40	60	100	200	300
Heptane	2000	0.5	1	2	4	20	40	60	100	200	300
Benzene	2000	0.5	1	2	4	20	40	60	100	200	300
tert-Amyl methyl ether	2000	0.5	1	2	4	20	40	60	100	200	300
1,2-Dichloroethane-d4	2500	20	20	20	20	20	20	20	20	20	20
1,2-Dichloroethane	2000	0.5	1	2	4	20	40	60	100	200	300
Isobutyl alcohol	2000	N/A	20	N/A	80	200	400	600	1000	3000	4000
2-Methyl-2-butanol	2000	2.5	5	10	20	100	200	300	500	1000	1500
Methyl cyclohexane	2000	0.5	1	2	4	20	40	60	100	200	300
Trichloroethene	2000	0.5	1	2	4	20	40	60	100	200	300
n-Butanol	2000	N/A	20	N/A	80	200	400	600	1000	3000	4000
Dibromomethane	2000	0.5	1	2	4	20	40	60	100	200	300
1,2-Dichloropropane	2000	0.5	1	2	4	20	40	60	100	200	300
4-penten-2-ol	2000	2.5	5	10	20	100	200	300	500	1000	1500
2-Chloroethyl vinyl ether	2000	0.5	1	2	4	20	40	60	100	200	300
Bromodichloromethane	2000	0.5	1	2	4	20	40	60	100	200	300
Ethyl acrylate	2000	0.5	1	2	4	20	40	60	100	200	300
Methyl methacrylate	2000	0.5	1	2	4	20	40	60	100	200	300
1,4-Dioxane	2000	N/A	40	80	200	1000	2000	3000	5000	10000	15000
cis-1,3-Dichloropropene	2000	0.5	1	2	4	20	40	60	100	200	300
Chlorobenzene-d5	2500	20	20	20	20	20	20	20	20	20	20
Octane	2000	0.5	1	2	4	20	40	60	100	200	300
Toluene-d8	2500	20	20	20	20	20	20	20	20	20	20
Toluene	2000	0.5	1	2	4	20	40	60	100	200	300
4-Methyl-2-pentanone	2000	0.5	1	2	4	20	40	60	100	200	300
Tetrachloroethene	2000	0.5	1	2	4	20	40	60	100	200	300
2-Nitropropane	2000	0.5	1	2	4	20	40	60	100	200	300

Soil	Stock (µg/mL)	Level 0 (ug/kg)	Level 1 (ug/kg)	Level 1.5 (ug/kg)	Level 2 (ug/kg)	Level 3 (ug/kg)	Level 4 (ug/kg)	Level 5 (ug/kg)	Level 6 (ug/kg)	Level 7 (ug/kg)	Level 8 (ug/kg)
Chloropicrin	2000	N/A	50	N/A	100	200	300	400	500	600	700
trans-1,3-Dichloropropene	2000	0.5	1	2	4	20	40	60	100	200	300
Methyl isothiocyanate	2000	0.5	1	2	4	20	40	60	100	200	300
4-Methyl-2-pentanol	2000	N/A	20	N/A	80	200	400	600	1000	3000	4000
Ethyl methacrylate	2000	0.5	1	2	4	20	40	60	100	200	300
1,1,2-Trichloroethane	2000	0.5	1	2	4	20	40	60	100	200	300
Chlorodibromomethane	2000	0.5	1	2	4	20	40	60	100	200	300
1,3-Dichloropropane	2000	0.5	1	2	4	20	40	60	100	200	300
1,2-Dibromoethane	2000	0.5	1	2	4	20	40	60	100	200	300
n-Butyl Acetate	2000	0.5	1	2	4	20	40	60	100	200	300
2-Hexanone	2000	0.5	1	2	4	20	40	60	100	200	300
Nonane	2000	0.5	1	2	4	20	40	60	100	200	300
Chlorobenzene	2000	0.5	1	2	4	20	40	60	100	200	300
Ethylbenzene	2000	0.5	1	2	4	20	40	60	100	200	300
1,1,1,2-Tetrachloroethane	2000	0.5	1	2	4	20	40	60	100	200	300
p/m Xylene	2000	1	2	4	8	40	80	120	200	400	600
o Xylene	2000	1	2	4	8	40	80	120	200	400	600
Styrene	2000	1	2	4	8	40	80	120	200	400	600
1,4-Dichlorobenzene-d4	2500	20	20	20	20	20	20	20	20	20	20
Bromoform	2000	0.5	1	2	4	20	40	60	100	200	300
Butyl acrylate	2000	0.5	1	2	4	20	40	60	100	200	300
Isopropylbenzene	2000	0.5	1	2	4	20	40	60	100	200	300
4-Bromofluorobenzene	2500	20	20	20	20	20	20	20	20	20	20
Bromobenzene	2000	0.5	1	2	4	20	40	60	100	200	300
Decane	2000	0.5	1	2	4	20	40	60	100	200	300
n-Propylbenzene	2000	0.5	1	2	4	20	40	60	100	200	300
1,4-Dichlorobutane	2000	0.5	1	2	4	20	40	60	100	200	300
1,1,2,2-Tetrachloroethane	2000	0.5	1	2	4	20	40	60	100	200	300
4-Ethyltoluene	2000	0.5	1	2	4	20	40	60	100	200	300
2-Chlorotoluene	2000	0.5	1	2	4	20	40	60	100	200	300
1,3,5-Trimethylbenzene	2000	0.5	1	2	4	20	40	60	100	200	300
1,2,3-Trichloropropane	2000	0.5	1	2	4	20	40	60	100	200	300
trans-1,4-Dichloro-2-butene	2000	0.5	1	2	4	20	40	60	100	200	300
4-Chlorotoluene	2000	0.5	1	2	4	20	40	60	100	200	300

Soil	Stock (µg/mL)	Level 0 (ug/kg)	Level 1 (ug/kg)	Level 1.5 (ug/kg)	Level 2 (ug/kg)	Level 3 (ug/kg)	Level 4 (ug/kg)	Level 5 (ug/kg)	Level 6 (ug/kg)	Level 7 (ug/kg)	Level 8 (ug/kg)
tert-Butylbenzene	2000	0.5	1	2	4	20	40	60	100	200	300
Pentachloroethane	2000	0.5	1	2	4	20	40	60	100	200	300
n-Butyl methacrylate	2000	0.5	1	2	4	20	40	60	100	200	300
1,2,4-Trimethylbenzene	2000	0.5	1	2	4	20	40	60	100	200	300
Limonene	2000	0.5	1	2	4	20	40	60	100	200	300
sec-Butylbenzene	2000	0.5	1	2	4	20	40	60	100	200	300
p-Isopropyltoluene	2000	0.5	1	2	4	20	40	60	100	200	300
1,3-Dichlorobenzene	2000	0.5	1	2	4	20	40	60	100	200	300
1,4-Dichlorobenzene	2000	0.5	1	2	4	20	40	60	100	200	300
trans-Decahydronaphthalene	2000	0.5	1	2	4	20	40	60	100	200	300
Undecane	2000	0.5	1	2	4	20	40	60	100	200	300
p-Diethylbenzene	2000	0.5	1	2	4	20	40	60	100	200	300
n-Butylbenzene	2000	0.5	1	2	4	20	40	60	100	200	300
Hexachloroethane	2000	0.5	1	2	4	20	40	60	100	200	300
1,2-Dichlorobenzene	2000	0.5	1	2	4	20	40	60	100	200	300
cis-Decahydronaphthalene	2000	0.5	1	2	4	20	40	60	100	200	300
1,2,4,5-Tetramethylbenzene	2000	0.5	1	2	4	20	40	60	100	200	300
1,2-Dibromo-3-chloropropane	2000	0.5	1	2	4	20	40	60	100	200	300
1,3,5-Trichlorobenzene	2000	0.5	1	2	4	20	40	60	100	200	300
Nitrobenzene	2000	0.5	1	2	4	20	40	60	100	200	300
Hexachlorobutadiene	2000	0.5	1	2	4	20	40	60	100	200	300
1,2,4-Trichlorobenzene	2000	0.5	1	2	4	20	40	60	100	200	300
Naphthalene	2000	0.5	1	2	4	20	40	60	100	200	300
1,2,3-Trichlorobenzene	2000	0.5	1	2	4	20	40	60	100	200	300
1,3-Dioxolane	2000	N/A	25	N/A	50	100	250	500	1000	1500	2000

Stock Standard Concentrations and Suggested Calibration Concentration Levels

Water	Stock (µg/mL)	Level 11 (ug/L)	Level 1 (ug/L)	Level 2 (ug/L)	Level 3 (ug/L)	Level 4 (ug/L)	Level 5 (ug/L)	Level 6 (ug/L)	Level 7 (ug/L)	Level 8 (ug/L)	Level 9 (ug/L)	Level 10 (ug/L)
							Optional		Optional		Optional	
Fluorobenzene	2500	10	10	10	10	10	10	10	10	10	10	10
Dichlorodifluoromethane	2000		0.5	2	10	30	50	80	100	120	160	200
Chloromethane	2000		0.5	2	10	30	50	80	100	120	160	200
Vinyl chloride	2000	0.2	0.5	2	10	30	50	80	100	120	160	200
Bromomethane	2000		0.5	2	10	30	50	80	100	120	160	200
Chloroethane	2000		0.5	2	10	30	50	80	100	120	160	200
Trichlorofluoromethane	2000		0.5	2	10	30	50	80	100	120	160	200
Ethyl ether	2000		0.5	2	10	30	50	80	100	120	160	200
Ethanol	2000		0.5	2	10	30	50	80	100	120	160	200
1,1-Dichloroethene	2000		0.5	2	10	30	50	80	100	120	160	200
Carbon disulfide	2000		0.5	2	10	30	50	80	100	120	160	200
Freon-113	2000		0.5	2	10	30	50	80	100	120	160	200
Iodomethane	2000		0.5	2	10	30	50	80	100	120	160	200
Acrolein	2000		0.5	2	10	30	50	80	100	120	160	200
Methylene chloride	2000		0.5	2	10	30	50	80	100	120	160	200
Isopropyl alcohol	2000		2.5	10	50	150	250	400	500	600	800	1000
Acetone	2000		0.5	2	10	30	50	80	100	120	160	200
trans-1,2-Dichloroethene	2000		0.5	2	10	30	50	80	100	120	160	200
Methyl acetate	2000		0.5	2	10	30	50	80	100	120	160	200
Methyl tert-butyl ether	2000		0.5	2	10	30	50	80	100	120	160	200
tert-Butyl alcohol	2000		2.5	10	50	150	250	400	500	600	800	1000
Diisopropyl ether	2000		0.5	2	10	30	50	80	100	120	160	200
1,1-Dichloroethane	2000		0.5	2	10	30	50	80	100	120	160	200
Halothane	2000		0.5	2	10	30	50	80	100	120	160	200
Acrylonitrile	2000		0.5	2	10	30	50	80	100	120	160	200
Ethyl tert-butyl ether	2000		0.5	2	10	30	50	80	100	120	160	200
Vinyl acetate	2000		0.5	2	10	30	50	80	100	120	160	200
cis-1,2-Dichloroethene	2000		0.5	2	10	30	50	80	100	120	160	200
2,2-Dichloropropane	2000		0.5	2	10	30	50	80	100	120	160	200
Bromochloromethane	2000		0.5	2	10	30	50	80	100	120	160	200
Cyclohexane	2000		0.5	2	10	30	50	80	100	120	160	200
Chloroform	2000		0.5	2	10	30	50	80	100	120	160	200
Ethyl acetate	2000		0.5	2	10	30	50	80	100	120	160	200

Water	Stock (µg/mL)	Level 11 (ug/L)	Level 1 (ug/L)	Level 2 (ug/L)	Level 3 (ug/L)	Level 4 (ug/L)	Level 5 (ug/L)	Level 6 (ug/L)	Level 7 (ug/L)	Level 8 (ug/L)	Level 9 (ug/L)	Level 10 (ug/L)
							Optional		Optional		Optional	
Carbon tetrachloride	2000	0.2	0.5	2	10	30	50	80	100	120	160	200
Tetrahydrofuran	2000		0.5	2	10	30	50	80	100	120	160	200
Dibromofluoromethane	2500	10	10	10	10	10	10	10	10	10	10	10
1,1,1-Trichloroethane	2000		0.5	2	10	30	50	80	100	120	160	200
2-Butanol	2000		2.5	10	50	150	250	400	500	600	800	1000
2-Butanone	2000		0.5	2	10	30	50	80	100	120	160	200
1,1-Dichloropropene	2000		0.5	2	10	30	50	80	100	120	160	200
Benzene	2000	0.2	0.5	2	10	30	50	80	100	120	160	200
tert-Amyl methyl ether	2000		0.5	2	10	30	50	80	100	120	160	200
1,2-Dichloroethane-d4	2500	10	10	10	10	10	10	10	10	10	10	10
1,2-Dichloroethane	2000		0.5	2	10	30	50	80	100	120	160	200
Isobutyl alcohol	2000		2.5	10	50	150	250	400	500	600	800	1000
2-Methyl-2-butanol	2000		2.5	10	50	150	250	400	500	600	800	1000
Methyl cyclohexane	2000		0.5	2	10	30	50	80	100	120	160	200
Trichloroethene	2000	0.2	0.5	2	10	30	50	80	100	120	160	200
n-Butanol	2000		2.5	10	50	150	250	400	500	600	800	1000
Dibromomethane	2000		0.5	2	10	30	50	80	100	120	160	200
1,2-Dichloropropane	2000		0.5	2	10	30	50	80	100	120	160	200
4-penten-2-ol	2000		2.5	10	50	150	250	400	500	600	800	1000
2-Chloroethyl vinyl ether	2000		0.5	2	10	30	50	80	100	120	160	200
Bromodichloromethane	2000		0.5	2	10	30	50	80	100	120	160	200
Ethyl acrylate	2000		0.25	1	5	15	25	40	50	60	80	100
Methyl methacrylate	2000		0.25	1	5	15	25	40	50	60	80	100
1,4-Dioxane	2000		100	400	500	600	1000	800	1000	1200	1600	2000
cis-1,3-Dichloropropene	2000		0.5	2	10	30	50	80	100	120	160	200
Chlorobenzene-d5	2500	10	10	10	10	10	10	10	10	10	10	10
Toluene-d8	2500	10	10	10	10	10	10	10	10	10	10	10
Toluene	2000		0.5	2	10	30	50	80	100	120	160	200
4-Methyl-2-pentanone	2000		0.5	2	10	30	50	80	100	120	160	200
Tetrachloroethene	2000		0.5	2	10	30	50	80	100	120	160	200
Chloropicrin	2000		30	50	80	120	200	160	400	200	320	400
trans-1,3-Dichloropropene	2000		0.5	2	10	30	50	80	100	120	160	200
4-Methyl-2-pentanol	2000		2.5	10	50	150	250	400	500	600	800	1000

Water	Stock (µg/mL)	Level 11 (ug/L)	Level 1 (ug/L)	Level 2 (ug/L)	Level 3 (ug/L)	Level 4 (ug/L)	Level 5 (ug/L)	Level 6 (ug/L)	Level 7 (ug/L)	Level 8 (ug/L)	Level 9 (ug/L)	Level 10 (ug/L)
							Optional		Optional		Optional	
Ethyl methacrylate	2000		0.5	2	10	30	50	80	100	120	160	200
1,1,2-Trichloroethane	2000		0.5	2	10	30	50	80	100	120	160	200
Chlorodibromomethane	2000		0.5	2	10	30	50	80	100	120	160	200
1,3-Dichloropropane	2000		0.5	2	10	30	50	80	100	120	160	200
1,2-Dibromoethane	2000		0.5	2	10	30	50	80	100	120	160	200
2-Hexanone	2000		0.5	2	10	30	50	80	100	120	160	200
Chlorobenzene	2000		0.5	2	10	30	50	80	100	120	160	200
Ethylbenzene	2000		0.5	2	10	30	50	80	100	120	160	200
1,1,1,2-Tetrachloroethane	2000		0.5	2	10	30	50	80	100	120	160	200
p/m Xylene	2000		1	4	20	60	100	160	200	240	320	400
o Xylene	2000		1	4	20	60	100	160	200	240	320	400
Styrene	2000		1	4	20	60	100	160	200	240	320	400
1,4-Dichlorobenzene-d4	2500	10	10	10	10	10	10	10	10	10	10	10
Bromoform	2000		0.5	2	10	30	50	80	100	120	160	200
Butyl acrylate	2000		0.25	1	5	15	25	40	50	60	80	100
Isopropylbenzene	2000		0.5	2	10	30	50	80	100	120	160	200
4-Bromofluorobenzene	2500	10	10	10	10	10	10	10	10	10	10	10
Bromobenzene	2000		0.5	2	10	30	50	80	100	120	160	200
n-Propylbenzene	2000		0.5	2	10	30	50	80	100	120	160	200
1,4-Dichlorobutane	2000		0.5	2	10	30	50	80	100	120	160	200
1,1,2,2-Tetrachloroethane	2000		0.5	2	10	30	50	80	100	120	160	200
4-Ethyltoluene	2000		0.5	2	10	30	50	80	100	120	160	200
2-Chlorotoluene	2000		0.5	2	10	30	50	80	100	120	160	200
1,3,5-Trimethylbenzene	2000		0.5	2	10	30	50	80	100	120	160	200
1,2,3-Trichloropropane	2000		0.5	2	10	30	50	80	100	120	160	200
trans-1,4-Dichloro-2-butene	2000		0.5	2	10	30	50	80	100	120	160	200
4-Chlorotoluene	2000		0.5	2	10	30	50	80	100	120	160	200
tert-Butylbenzene	2000		0.5	2	10	30	50	80	100	120	160	200
Pentachloroethane	2000		0.5	2	10	30	50	80	100	120	160	200
n-Butyl methacrylate	2000		0.25	1	5	15	25	40	50	60	80	100
1,2,4-Trimethylbenzene	2000		0.5	2	10	30	50	80	100	120	160	200
sec-Butylbenzene	2000		0.5	2	10	30	50	80	100	120	160	200

Water	Stock (µg/mL)	Level 11 (ug/L)	Level 1 (ug/L)	Level 2 (ug/L)	Level 3 (ug/L)	Level 4 (ug/L)	Level 5 (ug/L)	Level 6 (ug/L)	Level 7 (ug/L)	Level 8 (ug/L)	Level 9 (ug/L)	Level 10 (ug/L)
							Optional		Optional		Optional	
p-Isopropyltoluene	2000		0.5	2	10	30	50	80	100	120	160	200
1,3-Dichlorobenzene	2000		0.5	2	10	30	50	80	100	120	160	200
1,4-Dichlorobenzene	2000		0.5	2	10	30	50	80	100	120	160	200
p-Diethylbenzene	2000		0.5	2	10	30	50	80	100	120	160	200
n-Butylbenzene	2000		0.5	2	10	30	50	80	100	120	160	200
1,2-Dichlorobenzene	2000		0.5	2	10	30	50	80	100	120	160	200
1,2,4,5-Tetramethylbenzene	2000		0.5	2	10	30	50	80	100	120	160	200
1,2-Dibromo-3-chloropropane	2000		0.5	2	10	30	50	80	100	120	160	200
1,3,5-Trichlorobenzene	2000		0.5	2	10	30	50	80	100	120	160	200
Hexachlorobutadiene	2000		0.5	2	10	30	50	80	100	120	160	200
1,2,4-Trichlorobenzene	2000		0.5	2	10	30	50	80	100	120	160	200
Naphthalene	2000		0.5	2	10	30	50	80	100	120	160	200
1,2,3-Trichlorobenzene	2000		0.5	2	10	30	50	80	100	120	160	200
1,3-Dioxolane	2000		10	40	100	250	N/A	500	N/A	750	N/A	1000
Pentachloroethane	2000		0.5	2	10	30	50	80	100	120	160	200
1,4-Dioxane (SIM)	100		0.5	2	10	20	30	50	100	200	N/A	N/A
1,1,2,2-Tetrachloroethane (SIM)	100		0.05	0.1	0.2	0.5	1	2	5	10	N/A	N/A

- For Low Level Soil analysis, the calibration levels are the same in µg/Kg units.
- For High Level Soil analysis, the calibration levels are at 50x the levels listed due to sample preparation requirements.

TABLE 5
8260C Volatile Internal Standards with Corresponding Target Compounds and Surrogates Assigned for Quantitation

Fluorobenzene	Chlorobenzene-d5	1,4-Dichlorobenzene-d4
Dichlorodifluoromethane	Toluene-d8 (surr)	Isopropylbenzene
Chloromethane	Toluene	Bromoform
Vinyl Chloride	Ethyl Methacrylate	1,4-dichloro-2-butane
Bromomethane	Trans-1,3-dichloropropene	1,1,2,2,-tetrachloroethane
Chloroethane	1,1,2-trichloroethane	4-bromofluorobenzene (surr)
Trichlorofluoromethane	2-hexanone	1,2,3-trichloropropane
Ethyl Ether	1,3-dichloropropane	trans-1,4-dichloro-2-butene
Freon 113	Tetrachloroethene	n-propylbenzene
Acrolein	Chlorodibromomethane	Bromobenzene
Acetone	1,2-dibromoethane	4-ethyltoluene
Ethanol	Chlorobenzene	1,3,5-trimethybenzene
1,1,-dichloroethene	1,1,1,2-tetrachloroethane	2-chlorotoluene
Tert-Butyl Alcohol	Ethylbenzene	4-chorotoluene
Methyl Acetate	p/m xylene	tert-butylbenzene
Carbon Disulfide	o xylene	1,2,4-trimethylbenzene
Methylene Chloride	Styrene	sec-butylbenzene
Acrylonitrile	Octane	p-isopropyltoluene
Methyl Tert Butyl Ether	2-Nitropropane	1,3-dichlorobenzene
Halothane	Methyl isothiocyanate	1,4-dichlorobenzene
Trans-1,2-dichloroethene	n-Butyl acetate	n-butylbenzene
Diisopropyl Ether	Nonane	p-diethylbenzene
Vinyl Acetate		1,2-dichlorobenzene
1,1-dichloroethane		1,2,4,5-tetramethylbenzene
Ethyl-Tert-Butyl-Ether		1,2-dibromo-3-chloropropane
2-butanone		1,3,5-trichlorobenzene
2,2-dichloropropane		1,2,4-trichlorobenzene
Cis-1,2-dichloroethene		Hexachlorobutadiene
Chloroform		Naphthalene
Bromochloromethane		1,2,3-trichlorobenzene
Tetrahydrofuran		Cyclohexanone
Dibromofluoromethane (surr)		Nitrobenzene

1,1,1-trichloroethane		Pentachloroethane
Cyclohexane		Decane
1,1-dichloropropene		Limonene
Carbon Tetrachloride		Trans-Decahydronaphthalene
Tertiary-Amyl Methyl Ether		Undecane
1,2-dichloroethane-d4 (surr)		Hexachloroethane
1,2-dichloroethane		Cis-Decahydronaphthalene
Benzene		
Trichloroethene		
Methyl Cyclohexane		
1,2-dichloropropane		
Bromodichloromethane		
1,4-Dioxane		
Dibromomethane		
2-Chloroethylvinyl Ether		
4-methyl-2-pentanone		
Cis-1,3-dichloropropene		
Iodomethane		
Methyl methacrylate		
n-Butanol		
Ethyl acetate		
Isopropyl Alcohol (IPA)		
Hexane		
n-Propyl bromide		
Chlorodifluoromethane		
Allyl chloride		
Heptane		

TABLE 6
8260C Quantitation Ions

Analyte	Quantitation Ion	Analyte	Quantitation Ion
Dichlorodifluoromethane	85	Ethyl Methacrylate	69
Chloromethane	50	Trans-1,3-dichloropropene	75
Vinyl Chloride	62	1,1,2-trichloroethane	83
Bromomethane	94	2-hexanone	43
Chloroethane	64	1,3-dichloropropane	76
Trichlorofluoromethane	101	Tetrachloroethene	166
Ethyl Ether	74	Chlorodibromomethane	129
Freon 113	101	1,2-dibromoethane	107
Acrolein	56	Chlorobenzene	112
Acetone	43	1,1,1,2-tetrachloroethane	131
1,1,-dichloroethene	96	Ethylbenzene	91
Tert-Butyl Alcohol	59	p/m xylene	106
Methyl Acetate	43	o xylene	106
Carbon Disulfide	84	Styrene	104
Methylene Chloride	76	Isopropylbenzene	105
Acrylonitrile	53	Bromoform	173
Methyl Tert Butyl Ether	73	1,4-dichloro-2-butane	55
Halothane	117	1,1,2,2,-tetrachloroethane	83
Trans-1,2-dichloroethene	96	1,2,3-trichloropropane	75
Diisopropyl Ether	45	Trans-1,4-dichloro-2-butene	53
Vinyl Acetate	43	n-propylbenzene	91
1,1-dichloroethane	63	Bromobenzene	156
Ethyl-Tert-Butyl-Ether	59	4-ethyltoluene	105
2-butanone	43	1,3,5-trimethybenzene	105
2,2-dichloropropane	77	2-chlorotoluene	91
Cis-1,2-dichloroethene	96	4-chorotoluene	91
Chloroform	83	tert-butylbenzene	119
Bromochloromethane	128	1,2,4-trimethylbenzene	105
Tetrahydrofuran	42	sec-butylbenzene	105
1,1,1-trichloroethane	97	p-isopropyltoluene	119
Cyclohexane	56	1,3-dichlorobenzene	146
1,1-dichloropropene	75	1,4-dichlorobenzene	146
Carbon Tetrachloride	117	n-butylbenzene	91
Tertiary-Amyl Methyl Ether	73	p-diethylbenzene	119
1,2-dichloroethane	62	1,2-dichlorobenzene	146
Benzene	78	1,2,4,5-tetramethylbenzene	119
Trichloroethene	95	1,2-dibromo-3-chloropropane	75
Methyl Cyclohexane	83	1,3,5-trichlorobenzene	180
1,2-dichloropropane	63	1,2,4-trichlorobenzene	180
Bromodichloromethane	83	Hexachlorobutadiene	225
1,4-dioxane	88	Naphthalene	128
Dibromomethane	93	1,2,3-trichlorobenzene	180
2-Chloroethylvinyl Ether	63	Ethanol	45
4-methyl-2-pentanone	58	Cyclohexanone	55
Cis-1,3-dichloropropene	75	Ethyl acetate	43

TABLE 6
8260C Quantitation Ions (continued)

Analyte	Quantitation Ion	Analyte	Quantitation Ion
Toluene	92	Iodomethane	142
Methyl methacrylate	69	n-Butanol	56
Pentachloroethane	167	Isopropyl Alcohol (IPA)	45
Hexane	57	n-Propyl bromide	43
Chlorodifluoromethane	51	Iodomethane	142
Allyl chloride	76	Heptane	71
Octane	85	2-Nitropropane	41
Methyl isothiocyanate	73	n-Butyl Acetate	43
Nonane	57	Decane	57
Limonene	68	Undecane	57
trans-Decahydronaphthalene	138	cis-Decahydronaphthalene	138
Hexachloroethane	117	Nitrobenzene	77

Table 7

List of 8260 Difficult Analytes:

1,1,2,2-Tetrachloroethane
1,2-Dibromo-3-chloropropane (DBCP)
1,4-Dioxane
2-Butanone
2-chloroethylvinyl ether
2-Hexanone
2,2-dichloropropane
4-Methyl-2-pentanone
Acetone
Bromoform
Bromomethane
Carbon disulfide
Chloroethane
Chloromethane
cis-1,3-Dichloropropene
Dichlorodifluoromethane (Freon 12)
Ethanol
Iodomethane
Isobutyl Alcohol
Naphthalene
Nitrobenzene
n-butanol
Styrene
Tert-Butyl Alcohol
Trichlorofluoromethane (Freon 11)
Isopropyl Alcohol (IPA)

VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

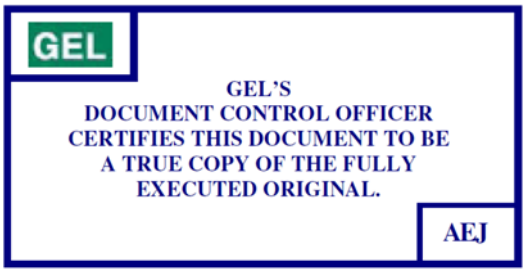
STANDARD OPERATING PROCEDURE FOR TOTAL DISSOLVED SOLIDS

(GL-GC-E-001 REVISION 16)

APPLICABLE TO METHODS:
EPA 160.1
Standard Methods 22nd Edition 2540 C-2011

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1.0 STANDARD OPERATING PROCEDURE FOR TOTAL DISSOLVED SOLIDS

2.0 METHOD CODE

2.1 EPA 160.1

2.2 Standard Methods 22nd Edition 2540 C-2011

3.0 METHOD OBJECTIVE / PURPOSE

This procedure is used to measure total dissolved solids (TDS) in waters and wastewaters. TDS are those solids capable of passing through a standard glass fiber filter and dried to a constant weight at 180° C.

4.0 METHOD SUMMARY

4.1 Summary: In this gravimetric procedure, a water sample is filtered through a glass fiber filter into a preweighed porcelain crucible. The filtrate is evaporated to dryness to a constant weight at 180° C. The increase in the dish weight represents the total dissolved solids.

4.2 Synonym: Residue, Total Filterable (Dried at 180° C)

5.0 APPLICABLE MATRICES

5.1 Groundwater

5.2 Drinking water

5.3 Domestic and industrial wastewater

NOTE: Clients may request that this analysis be performed on miscellaneous liquid samples. In these cases, the procedure is modified as necessary.

6.0 HOLDING TIME

Seven days filtration from the time and date of collection unless otherwise specified by contract.

7.0 SAMPLE CONTAINER, PRESERVATION, COLLECTION, AND STORAGE REQUIREMENTS

7.1 Samples may be stored in glass or plastic containers.

7.2 Preservation:

7.2.1 No preservatives are required. Preserved samples should never be analyzed.

7.2.2 Temperature and pH are documented at login. Refer to GL-SR-E-001 for Sample Receipt, Login, and Storage.

7.3 Refrigerate sample at 0° ≤ 6° C until analysis to minimize microbiological decomposition of solids.

8.0 INTERFERENCES

8.1 Highly mineralized waters containing significant concentrations of calcium, magnesium, chloride, and/or sulfate may be hygroscopic and will require prolonged drying, desiccation, and rapid weighing.

8.2 Samples containing high concentrations of bicarbonate will require careful and possibly prolonged drying at 180° C to ensure that all the bicarbonate is converted to carbonate.

8.3 Too much residue in the evaporating dish will crust over and entrap water that will not be driven off during drying. Total residue should be limited to about 200 mg.

9.0 PERFORMANCE CHARACTERISTICS

9.1 Method concentration range: 10 mg/L to 20,000 mg/L

9.2 Method detection limit (MDL): Refer to current MDL study.

9.3 Method precision: 0-5% RPD.

9.4 Method accuracy: 95-105% recovery.

10.0 DEFINITIONS

10.1 Hygroscopic: Attracting, absorbing, and retaining atmospheric moisture.

10.2 Gravimetric: Pertaining to measurement by weight.

10.3 Desiccant: Material used to absorb moisture present.

10.4 AlphaLIMS: The Laboratory Information Management System used at GEL.

10.5 Method Blank (MB): An aliquot of reagent water or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The MB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

10.6 Laboratory Control Standard (LCS): An aliquot of reagent water or other blank matrix to which known quantities of the method analytes are added in the laboratory. The LCS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

10.7 Laboratory Duplicate (DUP, LCSD, MSD or PSD): Aliquots of a sample taken from the same container and processed in the same manner under identical laboratory conditions. The aliquot is analyzed independently from the parent sample and the results are compared to measure precision and accuracy.

10.8 Method Detection Limit (MDL): The minimum concentration of an analyte that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero.

10.9 Statistical Process Control (SPC) Limits: Statistically derived limits that establish Acceptable Ranges for recoveries of analytes of interest, including LCS, MS, MSD, PS, PSD and internal standards.

10.10 CRDL: Contract Required Detection Limit

11.0 ANALYST VERIFICATION

Technicians and analysts do not analyze client samples without supervision until trained by qualified personnel and upon the successful analysis of a proficiency sample. Training records are maintained as quality records.

12.0 DOCUMENTATION OF DATA

As data are obtained, they are recorded in AlphaLIMS.

13.0 SAFETY PRECAUTIONS AND HAZARD WARNINGS

13.1 Wear eye protection with side shields while performing procedures in the lab.

13.2 Treat all chemicals and samples as potential health hazards and limit exposure to these chemicals to the lowest level possible. GEL maintains a current awareness file of OSHA regulations regarding the safe handling of the chemicals in the laboratory as well as a reference file of Material Safety Data Sheets (MSDS). These documents and client sample MSDS forms are maintained in the laboratory.

14.0 SAMPLE RECEIPT FOR ANALYSIS

14.1 The analyst/technician gives the list of samples needed to the sample custodian. The sample custodian removes the appropriate samples from the cooler and either delivers them to the analyst/technician or places them on the "pick-up" shelf in the main cooler.

14.2 Analysts and technicians are responsible for retrieving their own samples when the sample custodian is not available.

15.0 INSTRUMENTATION / EQUIPMENT / GLASSWARE

15.1 Sartorius Basic BA210S or comparable analytical balance, capable of weighing to 0.0005g.

NOTE: The balance must be calibrated in accordance with the procedure outlined in the GL-LB-E-002 for Balances.

15.2 VWR 13703M or comparable drying oven for operation at 180° ± 2° C

15.3 VWR 13703M or comparable drying oven for operation at approximately 98° C

15.4 Desiccator with a color-indicating desiccant

15.5 Porcelain crucibles, 100 mL capacity or larger

15.6 Glass fiber filter paper, 4.7 cm

15.7 Filtration flask (Minimum size is 250 mL)

15.8 Magnetic filter funnel

15.9 Tongs

15.10 Vacuum source

15.11 Graduated cylinders (Typical capacity required is 100 mL)

15.12 Thermometers are verified in accordance with GL-QS-E-007 for Thermometer Verification, and capable of recording temperatures up to 182° C.

16.0 REAGENTS

16.1 Sodium chloride, NaCl

16.2 ASTM Type I Deionized (DI) water (Refer to GL-LB-E-016)

17.0 PREPARATION OF SAMPLES

Not Applicable

18.0 PREPARATION OF STANDARDS

18.1 Documentation of standards and their preparation is maintained in AlphaLIMS in accordance with to GL-LB-E-007 for Laboratory Standards Documentation.

18.2 Laboratory Control Sample (LCS), 300 mg/L sodium chloride, NaCl: Weigh 0.300 g of NaCl and dilute to 1000 mL with deionized water. The LCS should be discarded after six months.

19.0 INSTRUMENT / EQUIPMENT START-UP PROCEDURES

19.1 Preparation of desiccators:

19.1.1 Ensure that the desiccant is activated by observing the blue color indicator.

NOTE: If < 50% of the indicator desiccant is blue, change the desiccant.

19.1.2 Desiccator must be sealed. Any moisture absorbed by the crucibles can cause erroneous results.

19.1.3 Label desiccators to be used with the date and what they will contain.

19.2 Preparation of crucibles:

19.2.1 Crucibles are cleaned by the glassware technicians, according to GL-LB-E-003 for Glassware Preparation, and stored in a drying oven.

19.2.2 Crucibles are marked by the analyst with a grease pencil and identified with letters and/or numbers.

19.2.3 The analyst is responsible for the preparation of the crucibles as follows:

19.2.3.1 Place cleaned crucibles in the drying oven at 180 ± 2° C.

19.2.3.2 Set the temperature at 180° C.

19.2.3.3 Crucibles should remain at the specified temperature for a minimum of one hour.

NOTE: The oven/furnace temperature should not be allowed to drop below 100° C before the crucibles are removed and placed in desiccators.

19.2.3.4 Using tongs, remove the crucibles from the oven or furnace and place in a desiccator immediately.

19.2.3.5 Allow crucibles to cool in desiccators to room temperature before use. If it is possible, allow crucibles to cool for at least 12 hours.

19.3 Filter Papers (if prewashed, certified filters are being used, no filter preparation is needed):

19.3.1 Place glass fiber filter in filter apparatus.

19.3.2 Rinse with 100 mL of deionized water.

19.3.3 Apply vacuum until water is removed from the filter.

19.3.4 Place the filter in an oven to dry at approximately 100° C and store in a desiccator until needed.

NOTE: If necessary, the filter can be washed just prior to use and can be used without drying in an oven.

20.0 QUALITY CONTROL (QC) REQUIREMENTS

20.1 Frequency of QC:

20.1.1 A matrix duplicate should be analyzed for every batch of ≤ 10 samples and for each set of ten samples in batches with ≥ 10 samples.

20.1.2 A method blank (MB) and laboratory control sample (LCS) should be analyzed once for every batch containing ≤ 20 samples.

NOTE: LCS duplicates are analyzed if required by contract or requested by client or if matrix QC is not available

20.2 Acceptance limits:

20.2.1 Matrix Relative Percent Difference (RPD): 0-5% RPD.
20.2.2 Method Blank: Less than the CRDL.
20.2.3 LCS: 95%-105% recovery.

20.3 Handling out-of-control situations:

20.3.1 Notify the Group Leader or Team Leader immediately.

20.3.2 If the LCS, LCS RPD, matrix RPD, or blanks fall outside of current acceptance limits, the samples to which the unacceptable QC pertains must be reanalyzed.

20.3.3 The analyst should document, in case narrative for the batch, the specific QC that is out of control and cross-reference data from any subsequent reanalysis.

21.0 SUGGESTED RUN SEQUENCE

21.1 Method blank

21.2 LCS

21.3 Samples 1 through x where x ≤ 10

21.4 Sample x duplicate

21.5 Repeat steps 21.3 through 21.4 for every group of ten samples in the batch.

22.0 PROCEDURE

22.1 Calibration of equipment/instrumentation:

22.1.1 Balance

Ensure that the analytical balance to be used has been calibrated and is within control limits before use. Refer to the balance logbook and GL-LB-E-002 for balances, for limits.

22.1.2 Oven

22.1.2.1 Make sure that the oven to be used is at a temperature that is within acceptance limits.

22.1.2.2 Control limits for the drying oven are documented on the temperature log.

22.1.2.3 The oven temperature log format provides the required documentation to ensure that the oven temperature was in control during the drying process.

22.1.2.4 Refer to GL-LB-E-004 for Temperature Monitoring.

22.2 Analysis:

22.2.1 Remove the prepared crucibles from the desiccator.

NOTE: Crucible should be removed from the desiccator just immediately prior to weighing.

NOTE: NEVER touch the crucibles with your hands. Always use tongs or Kimwipes.

22.2.2 Record the crucible ID, sample number, and the weight of each crucible to be used in AlphaLIMS in the appropriate columns.

22.2.3 Place a prepared glass fiber filter in the filter apparatus.

22.2.4 Thoroughly mix the sample to be analyzed.

22.2.5 Filter the sample through the glass fiber filter into a clean filter flask. Volume of filtrate needed is 70 mL.

22.2.6 Wash filter with three successive 10 mL volumes of DI water, allowing complete drainage between washings, and continue suction for about 3 minutes after filtration is complete.

22.2.7 Transfer total filtrate (with washings) to the designated preweighed crucible.

NOTE: Volume of filtrate used may vary depending upon the matrix of the sample. If more than 10 minutes are required to complete filtration, decrease sample volume.

22.2.8 Place the crucible and filtrate in an oven at approximately 98 ° C until the filtrate evaporates to dryness.

22.2.9 Once sample has evaporated dryness, dry for a minimum of 1 hour in an oven at 180 ± 2° C.

CAUTION: The temperature of the oven must be recorded or results may be considered invalid.

22.2.10 Rinse the used funnel thoroughly before beginning to filter the next sample.

22.2.11 Repeat steps 22.2.3 through 22.2.10 for all samples, blanks, duplicates, and LCS to be analyzed.

22.2.12 Using tongs to remove the crucibles from the oven and cool to room temperature in a desiccator.

22.2.13 Weigh and record the weight of each crucible in AlphaLIMS in the appropriate column.

22.2.14 Put the crucibles back into an oven at 180 ± 2° C for a minimum of one hour.

22.2.15 Repeat 22.2.12 through 22.2.14 until a constant weight is obtained. Constant weight is defined by EPA 160.1 as weight change of ≤ 0.0005 g.

22.3 Calculation/reporting of results:

22.3.1 Calculate TDS as follows:

$$\text{TDS in mg/L} = \frac{(A-B) \times 1{,}000}{C}$$

A = final weight of dried residue + crucible (g)
B = initial weight of crucible (g)
C = volume of filtrate used (L)

22.3.2 Results are reported in mg/L.

22.3.3 LCS recoveries are calculated as follows:

$$\frac{\text{Actual [TDS] mg/L obtained}}{\text{Known [TDS] mg/L}} \times 100$$

22.3.4 RPDs are calculated as follows:

$$\frac{\text{[TDS mg/L] sample - [TDS mg/L] duplicate}}{\text{Average [TDS mg/L] of the sample + duplicate}}$$

23.0 INSTRUMENT AND EQUIPMENT SHUT-DOWN PROCEDURES

Not Applicable

24.0 DATA REVIEW, VALIDATION, AND APPROVAL PROCEDURES

Refer to GL-GC-E-092 for General Chemistry Data Review and Packaging.

25.0 DATA TRANSMITTAL

When a batch is given "DONE" status, the data are automatically available to reporting personnel.

26.0 RECORDS MANAGEMENT

All logbooks and data generated as a result of this procedure are maintained as quality records in accordance with GL-QS-E-008 for Quality Records Management and Disposition.

27.0 ROUTINE INSTRUMENT / EQUIPMENT MAINTENANCE

27.1 Ovens and Thermometers

Refer to GL-LB-E-004 for Temperature Monitoring and Documentation Requirements for Refrigerators, Freezers, Ovens, Incubators, and other Similar Devices.

27.2 Balances

Refer to GL-LB-E-002 for Balances.

27.3 Procedure for Maintenance of Desiccant

27.3.1 Analysts are responsible for ensuring that the desiccators are maintained by replacing the desiccant whenever 50% of the blue indicator starts to turn pink.

27.3.2 Dry desiccant for reuse as follows:

27.3.2.1 Spread desiccant out in an aluminum pan.

27.3.2.2 Place in drying oven capable of maintaining 105° C for one hour or more.

27.3.2.3 Remove from oven.

NOTE: All desiccants should be blue. If not, discard the dessicant.

27.3.2.4 Place in a desiccator immediately to cool.

28.0 LABORATORY WASTE HANDLING AND DISPOSAL

For the proper handling and disposal of all types of wastes from this procedure, refer to GL-LB-G-001, the Laboratory Waste Management Plan.

29.0 METHOD VARIATIONS

29.1 Step 19.3.2 differs from the method. The method mentions three 20 mL rinses of the filter paper with deionized water. As written, this SOP asks for the filter papers to be rinsed only once with 100 mL of deionized water.

29.2 Methods SM 2540 C-2011 and EPA 160.1 require that the filtrate be evaporated in a steam bath before being dried at 180° C. This SOP replaces the steam bath with an oven at approximately 98° C.

30.0 METHOD VERIFICATION

MDLs are calculated in accordance with GL-LB-E-001 for Determination of Method Detection Limits.

31.0 REFERENCES

31.1 Federal Register 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants under the Clean Water Act," April 4, 1995.

31.2 Manual of Methods For Chemical Analysis of Water and Wastes. US EPA Technology Transfer, EPA-625/6-74-003a, 1976.

31.3 Methods for Chemical Analysis of Water and Wastes. EPA 600/4-79-020. March 1979. Method 160.1, "Total Dissolved Solids."

31.4 Standard Methods for the Examination of Water and Wastewater, 22nd Edition Method 2540 C-2011.

31.5 Compilation of ASTM Standard Definitions, sponsored by ASTM Committee on Terminology. 7th Edition, American Society for Testing and Materials 1990.

32.0 HISTORY

Revision 16: Updated acceptance criteria.
Revision 15: TDS calculation revised.
Revision 14: Updated to include model of drying oven and analytical balance being used to comply with SCDHEC audit finding.
Revision 13: Updated Method reference for Standard Methods 2540 C-2011.
Revision 12: Replace Type II with Type I Deionized water.
Revision 11: Changed drying temperature to approximately 98 ° C and added filter washings to procedure.

VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE
FOR
pH

(GL-GC-E-008 REVISION 24)

APPLICABLE TO METHODS:
EPA 150.1, SW-846 Method 9040B/9040C,
SW-846 Method 9041A, SW-846 Method 9045C/9045D
Exhibit D Semivolatiles, OLMO4.2, 10.1.4.1 and
Standard Methods 22nd Edition, SM 4500-H^+-00

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1.0 STANDARD OPERATING PROCEDURE FOR pH

2.0 METHOD CODE

2.1 Matrix - Water or multiphase waste where at least 20% of the sample is aqueous

2.1.1 EPA 150.1

2.1.2 Standard Methods 22nd Edition, 4500-H^+-00

2.1.3 SW-846 Method 9040B/9040C

2.2 Matrix – Solids and Sludges

2.2.1 SW-846 Method 9045C/9045D

2.3 Matrix - Liquid samples, where measurement by the methods listed in 2.1 is not possible, and oils

2.3.1 SW-846 Method 9041A (pH paper method) - This procedure is described in Section 22.1 of this SOP.

3.0 METHOD OBJECTIVE/PURPOSE

The purpose of this standard operating procedure (SOP) is to describe the procedures used to determine pH.

4.0 METHOD SUMMARY

4.1 For Methods EPA 150.1, Standard Methods 4500-H^+, SW-846 9040B/9040C, and SW-846 9045C/9045D: The pH of a sample is determined electrometrically using either a glass electrode in combination with a reference potential or a combination electrode.

4.2 For SW-846 Method 9041A: The pH is determined with pH paper. Skip directly to Section 22.1 for this procedure.

5.0 APPLICABLE MATRICES

5.1 Groundwater

5.2 Drinking water

5.3 Domestic and industrial wastewater

5.4 Soil

5.5 Sludge

5.6 Oil

NOTE: Clients may request that this analysis be performed on miscellaneous liquid or solid samples. In these cases the procedure is modified as necessary.

6.0 HOLDING TIME

The Environmental Protection Agency (EPA) ruled on April 4, 1995, that the holding time for pH analysis is 15 minutes or less. Since transport of the samples to the laboratory for analysis exceeds this holding time, analysis occurs upon arrival.

7.0 SAMPLE CONTAINER/PRESERVATION/COLLECTION/STORAGE REQUIREMENTS

7.1 Samples can be collected in plastic or glass containers with no headspace.

7.2 Samples are unpreserved.

7.3 Samples are refrigerated at 0° ≤ 6° C until the start of analysis in accordance with GL-SR-E-001 for Sample Receipt, Login, and Storage.

8.0 INTERFERENCES/LIMITATIONS

8.1 Sodium error at pH levels greater than 10 can be reduced or eliminated by using a low sodium error electrode.

8.2 Oily material or particulate matter can coat the electrode and impair response. This is removed by gentle wiping and distilled water rinsing or by soaking in 10% hydrochloric acid if needed.

8.3 Temperature affects the pH of different samples in different ways. For this reason both pH and temperature should be recorded at time of analysis.

8.4 The Environmental Protection Agency (EPA) ruled on April 4, 1995, that the holding time for pH analysis is 15 minutes or less. Since transport of the samples to the laboratory for analysis exceeds this holding time, analysis occurs upon arrival. All pH samples will be flagged as being analyzed out of holding.

8.5 The fill hole on the pH probe MUST be in the open position during calibration and analysis for proper performance. It may be closed to prevent evaporation/concentration when not in use.

9.0 PERFORMANCE CHARACTERISTICS

9.1 Method range: 0 to 14 pH units (s.u.)

9.2 Calibration range: 4 to 10 pH units (s.u.)

NOTE: Add a pH 2 buffer for acidic or a pH 12 buffer for caustic wastes when a sample is to be analyzed for corrosivity characterization.

9.3 Method precision: Refer to current SPC limits

9.4 Method accuracy: Refer to current SPC limits

10.0 DEFINITIONS

10.1 AlphaLIMS: The Laboratory Information Management System used at GEL Laboratories, LLC.

10.2 Buffer Solution: Solutions with known pH values that are used to perform daily calibrations of the pH meter.

10.3 Calibration Standard (CAL): A solution prepared from the primary dilution standard solution or stock standard solutions and the internal standards and surrogate analytes. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

10.4 Continuing Calibration Verification (CCV) Standard: An aliquot of reagent water or other blank to which known quantities of the method analytes are added in the laboratory. The CCV is analyzed exactly like a sample, periodically throughout the run sequence. Its purpose is to determine whether the analytical sequence is in control during sample analysis. It may be prepared from the same source as the calibration standards, and is usually of varied concentration.

10.5 Laboratory Duplicate (DUP, LCSD, MSD, PSD): Aliquots of a sample taken from the same container and processed in the same manner under identical laboratory conditions. The aliquot is analyzed independently from the parent sample and the results are compared to measure precision and accuracy.

10.6 Laboratory Control Standard (LCS): An aliquot of reagent water or other blank matrix to which known quantities of the method analytes are added in the laboratory. The LCS is analyzed exactly like a sample, and whether the laboratory is capable of making accurate and precise measurements.

10.7 Statistical Process Control (SPC) Limits: Statistically derived limits that establish acceptable ranges for recoveries of analytes of interest, including LCS, MS, MSD, PS, PSD, and internal standards.

10.8 Stock Standard Solution: A concentrated solution containing one or more method analytes prepared in the laboratory using certified reference materials or purchased from a reputable commercial source.

10.9 Refer to GL-QS-B-001 the Quality Assurance Plan for additional lab-wide used definitions.

11.0 ANALYST VERIFICATION

Technicians and analysts do not analyze samples without supervision until trained by qualified personnel and upon successful analysis of a proficiency sample. Training records are maintained as quality records.

12.0 DOCUMENTATION OF DATA

12.1 Sample preparation data are recorded in AlphaLIMS.

12.2 As analytical data are obtained they are recorded in AlphaLIMS.

13.0 SAFETY PRECAUTIONS AND HAZARD WARNINGS

13.1 Wear eye protection with side shields while performing procedures in the laboratory.

13.2 Treat all chemicals and samples as potential health hazards, and limit exposure to these chemicals to the lowest level possible. GEL maintains a current awareness file of OSHA regulations regarding the safe handling of the chemicals in the laboratory as well as a reference file of Material Safety Data Sheets (MSDS). These documents and client sample MSDSs are maintained in the laboratory.

13.3 Personal protective equipment

13.3.1 Approved gloves are required when handling chemicals or samples.

13.3.2 To prevent clothes and skin from being exposed to corrosive materials, wear a lab coat.

13.4 Prior to handling radioactive samples analysts must have had radiation safety training and understand their full responsibilities in radioactive sample handling. Some general guidelines follow:

13.4.1 Wear a dosimeter at all times while working in the lab to monitor radioactive exposure.

13.4.2 Wear a plastic apron over lab coat when working with radioactive samples.

13.4.3 Protect counter tops with counter paper or work from radioactive sample handling trays.

13.4.4 Prohibit admittance to immediate work area.

13.4.5 Post signs indicating radioactive samples are in the area.

13.4.6 Take swipes of the counter tops upon completion of work. Deliver those swipes to the designated swipe count box.

13.4.7 Segregate radioactive wastes. Radioactive waste containers are obtained from Waste Management.

13.5 All samples, chemicals, extracts, and extraction residues must be transferred, delivered, and disposed of safely according to all related SOPs.

13.5.1 Segregate solid wastes from liquid wastes in the satellite area containers.

13.5.2 Segregate oil wastes from water-soluble wastes in the satellite area containers.

13.6 Never leave gas cylinders unchained or untied, including when they are on the moving carts.

13.7 In the event of an accident or medical emergency, call for help immediately. When time and safety permit, an accident report form should be completed and turned in to the safety committee.

13.8 Fire escape routes are posted in the lab and all personnel should be familiar with them. In addition, fire safety equipment such as fire extinguishers is located in the lab. Training is available on the proper operation of this equipment.

14.0 SAMPLE RECEIPT FOR ANALYSIS

14.1 Login personnel are responsible for delivering samples for pH analysis to the pH work area in accordance with GL-SR-E-001 for Sample Receipt, Login, and Storage.

14.2 Analysts and technicians are responsible for retrieving their own samples when the sample custodian is not available.

15.0 INSTRUMENTATION/EQUIPMENT/GLASSWARE

15.1 Orion Star A111 pH meter or equivalent.

15.2 Orion 8302BNUMD or any other comparable combination pH electrode

15.3 Temperature compensation probe (ATC) Orion 927005 or equivalent.

15.4 Beakers (minimum capacity needed, 25 mL)

15.5 Magnetic stir plate

15.6 Stirrer magnets

15.7 If Method 9045C/9045D is being used:

15.7.1 Centrifuge

15.7.2 Centrifuge tubes (minimum capacity, 50 mL)

15.7.3 Top-loader balance

15.7.4 Spatula

15.7.5 Separatory funnel (optional)

15.8 If Method 9041A is being used:

15.8.1 Wide range pH strips

15.8.2 Narrow range pH strips

16.0 REAGENTS

16.1 pH 4 Calibrating Buffer

16.2 pH 7 Calibrating Buffer (Two sources are required. A second source is needed for a LCS).

NOTE: If the client specifies, a pH 2 buffer will be run as the LCS for a corrosivity batch containing acidic samples. For a corrosivity batch containing caustic samples, a pH 12 buffer will be run.

16.3 pH 10 Calibrating Buffer

16.4 Laboratory deionized reagent water (DI)

16.5 10% Hydrochloric acid (HCL)

16.6 Orion Ag/AgCl Reference Electrode Filling Solution or equivalent.

17.0 PREPARATION OF SAMPLES

Sample preparation for soils, sludges, oils, miscellaneous solids, and liquids where less than 20% of the sample is aqueous:

NOTE: No sample preparation is required when more than 20% of the sample is aqueous. To determine if the sample is > 20% aqueous, look at the sample in the container. If the sample clearly has an aqueous portion that appears to be at least ¼ of the sample volume, proceed with section 22.0. If there is a question concerning the % volume of liquid material in a sample container, request assistance from an experienced analyst or technician.

17.1 Place a beaker or centrifuge tube on the top-loader balance. Tare the balance.

NOTE: The balance must be verified in accordance with GL-LB-E-002 for Balances.

17.2 Transfer 20 g soil/sediment to the container. Add 20 mL of DI water and stir or shake for 5 minutes.

17.3 The mixture is allowed to stand for about one hour to allow most of the suspended material to settle out. Alternatively, the sample may be filtered or centrifuged for at least 5 minutes.

17.4 The aqueous layer is decanted for pH measurement using the calibration pH electrode as described in Section 22.0.

NOTE: If the sample is hygroscopic or another problematic matrix, begin the procedure again using 20 g of sample and 40 mL of DI water.

NOTE: If the sample is classified as a waste material, the mixed suspension is either allowed to stand for 15 minutes or filtered or centrifuged for at least 5 minutes.

NOTE: If the supernatant is multiphasic, decant the oily phase or use a separatory funnel to obtain the aqueous phase to use for the pH determination.

NOTE: If limited sample volume is received, use smaller 1:1 ratio of grams of sample to mL of DI water for the pH determination.

18.0 PREPARATION OF STANDARDS

18.1 Documentation of standards is maintained in accordance with GL-LB-E-007 for Laboratory Standards Documentation.

18.2 Laboratory Control Sample(s): Commercially available pH buffer of 7.00 ± 0.01 @ 25°C. This must be from a different source than the calibrating pH 7 buffer.

18.3 Calibration standards:

18.3.1 pH buffers: 4, 7, and 10 pH units.

NOTE: If a sample is being analyzed for corrosivity, the pH meter check must also include buffers of pH 2 and pH 12. These additional buffers are not for calibration, but for checks only.

18.3.2 The buffers for calibration are stored in the Immediates area in the cabinets near the pH meter.

18.3.3 Buffers are discarded when they exceed the manufacturer's expiration date.

19.0 INSTRUMENT/EQUIPMENT START-UP PROCEDURES

The combination pH electrode must be plugged into the pH BNC jack on the rear part of the meter. The temperature compensating electrode is plugged into the ATC jack. A diagram for this is shown on page 7 of the Model 370 Instruction Manual. Make sure the LogR mode is off.

20.0 QUALITY CONTROL (QC) REQUIREMENTS

20.1 The pH calibration must be checked by measuring the pH of all buffers used to calibrate the meter. The pH must be within 0.05 pH units of the pH 4, pH 7, and pH 10 buffer solution values and within 0.1 pH units of the pH 2 and pH 12 buffer solution values. The slope value must read 92%-102%. If not, the calibration must be repeated until these criteria are met.

20.2 Frequency of QC:

20.2.1 A matrix duplicate is run for every group of ≤ 10 samples and for each set of ten samples in batches with > 10 samples.

20.2.2 A laboratory control sample is run once per batch unless specified otherwise by the client.

20.2.3 A continuing calibration verification (CCV) is run after every 5 samples and after the last sample in the run.

20.3 Acceptance limits:

20.3.1 Matrix relative percent difference (RPD): Refer to current SPC limits.

20.3.2 LCS: must be ± 0.05 s.u.

20.3.3 CCV must be ± 0.05 s.u.

20.4 Handling out-of-control situations:

All QC data are calculated as soon as the results for the QC samples are known.

20.4.1 If the matrix RPD falls outside of the SPC limits, reanalyze that sample.

20.4.2 If the LCS RPD, or LCS recovery falls outside of the SPC limits, the pH meter is to be recalibrated and all samples bracketed by the LCS and LCS DUP must be reanalyzed.

21.0 RUN SEQUENCE

21.1 Calibration standards: pH 4, 7 and 10 buffer solutions

21.2 pH 7 LCS (2nd Source Standard)

21.3 CCV (Continuing Calibration Verification)

21.4 Samples 1 through x where x ≤ 10

21.5 Sample x duplicate

21.6 Repeat steps 21.3 through 21.5 for every group of 10 samples in the batch. Run an additional CCV after the last analytical sample in the run.

22.0 PROCEDURE

22.1 For sample analysis by SW-846 Method 9041A, "pH Paper Method," for solvents and other samples that could possibly damage the electrodes, paper pH strips may be used to determine pH. Wide range pH strips are listed as 0-14 pH units. Narrow range pH strips come in five different pH unit ranges: 0-2.5, 2.5-4.5, 4.0-7.0, 6.5-10.0, and 11.0-13.0 pH units.

NOTE: This method is not considered to be as accurate for pH measurement as pH meters; for this reason, pH measurements taken by Method 9041A cannot be used to define a waste as alkaline and non-corrosive.

NOTE: If a sample masks color changes on the pH paper, this method is not reliable.

22.1.1 Place a wide range pH strip into a small aliquot of sample.

22.1.2 The color change of the wide range pH strip is compared to the color patterns listed on the pH strip box. Use this value to determine which narrow range pH strips to use. Select the appropriate narrow range strip.

22.1.3 Place the narrow range pH strip into a small aliquot of sample. Compare the color change to the color pattern on the narrow range strip box.

22.1.4 Record the pH in AlphaLIMS.

NOTE: Each batch of pH paper must be calibrated (checked) against certified pH buffers. The paper must read within 0.5 pH units of the certified pH of the buffer. This calibration is recorded in the pH calibration log.

22.1.5 Analyze each sample in duplicate by repeating steps 22.1.1 through 22.1.3.

22.1.6 Using a thermometer verified in accordance with GL-QS-E-007 for Thermometer Verification, measure the temperature of one of the sample

aliquots used during narrow range pH analysis. This is the temperature of the sample at the time the pH was measured.

22.1.7 Record the temperature of the sample in AlphaLIMS.

22.2 Calibration for methods using a pH meter:

22.2.1 Rinse the electrode with DI water and place into pH 4 buffer. Press 'CAL'. Wait for 'READY' to appear and press CAL again, making sure the screen now says '4'.

22.2.2 Repeat step 1 using the pH 7 buffer, making sure the screen now says '4, 7' after pressing CAL.

22.2.3 Repeat step 1 using the pH 10 buffer. When "READY' appears, press 'mode(enter) to save and end calibration. Slope will then be displayed, record this in LIMS.

22.3 Sample analysis by methods using a pH meter:

22.3.1 After the meter is calibrated using section 22.2, the unit will proceed to measure mode. The sample pH and temperature is automatically displayed.

22.3.2 Shake each sample prior to analysis. Approximately 40 mL of sample is poured into a beaker.

NOTE: If the sample was prepared as described in 17.1, decanted layer is the sample aliquot.

22.3.3 Place the beaker containing the sample on a magnetic stirring plate and add a clean magnetic stirring bar. Adjust the stir control to maintain a slow, steady rate of stirring.

22.3.4 Lower the pH electrode and the temperature compensating probe into the sample.

22.3.5 When the "Ready" status is displayed, record the pH, temperature, and time for the sample into the appropriate columns of the pH data entry screen in AlphaLIMS.

22.3.6 Repeat steps 22.3.2 through 22.3.5 with successive sample aliquots until the pH values obtained differ by < 0.1 pH units.

22.3.7 Run the samples in the sequence described in section 21.0.

NOTE: If only a few samples are ready to be run at a time, a duplicate sample is run for each set, regardless of whether or not few samples have been run since the last set of QC. The reason for this is to prove electrode repeatability throughout the day.

22.3.8 After all the pH samples present have been analyzed pH electrode is rinsed with DI water and placed back in the pH 7 buffer for storage.

22.4 Calculation/reporting of results:

22.4.1 Each sample result has a corresponding temperature and run time. These are recorded in AlphaLIMS. The final pH reading obtained for a sample after the criteria in 22.3.6 have been met is the one that is reported.

22.4.2 When submitting data for data entry, record the analyst's name, date of analysis, time, temperature, and pH values in AlphaLIMS.

22.4.3 After every duplicate sample value is recorded, the RPD (relative percent difference) of the sample and the duplicate are calculated. This is done by subtracting the lower value from the higher value, dividing by the average of the two and multiplying by 100 as shown in this formula:

$$\frac{\text{High value - Low value}}{\text{Average of the two values}} \times 100 = \text{RPD}$$

22.4.4 If the RPD is within the current control limits for pH, the duplicate is acceptable.

22.4.5 If the RPD is greater than the control limit, the sample and the duplicate are rerun. If the RPD is outside of the acceptance limits after being rerun, the pH meter is recalibrated and all samples analyzed prior to this duplicate and after the last acceptable duplicate are reanalyzed.

22.4.6 LCS RPDs are calculated using the formula described in 22.4.3.

22.4.7 LCS recoveries are calculated using this formula:

$$\frac{\text{actual value obtained}}{\text{theoretical value}} \times 100 = \%\ \text{LCS recovery}$$

23.0 INSTRUMENT/EQUIPMENT SHUT-DOWN PROCEDURES

23.1 After all the samples have been run, both the pH electrode and temperature compensating probe are rinsed thoroughly with DI water.

23.2 The electrode and probe are placed in a beaker filled with pH 7 buffer.

24.0 DATA REVIEW, VALIDATION, AND APPROVAL PROCEDURE

Refer to GL-LB-E-005 and GL-GC-E-092 for data review and validation procedures.

25.0 DATA TRANSMITTAL

When a batch is issued "DONE" status, the data become available to reporting personnel.

26.0 RECORDS MANAGEMENT

All data associated with the performance of this procedure, including relevant logbooks, are maintained as quality records in accordance with GL-QS-E-008 for Quality Records Management and Disposition.

27.0 ROUTINE INSTRUMENT/EQUIPMENT MAINTENANCE

27.1 The combination pH electrode is drained and refilled with the appropriate filling solution as needed.

27.2 For problems that occur during operation, refer to the troubleshooting checklists located in the Orion Ross pH Electrode Instruction Manual or the Orion Model EA 940 Expandable Ion Analyzer Instruction Manual.

28.0 LABORATORY WASTE HANDLING AND DISPOSAL

For the proper disposal of sample and reagent wastes from this procedure, refer to the Laboratory Waste Management Plan, GL-LB-G-001.

29.0 METHOD VARIATION

The pH test paper method SW-846 9041A is used only for miscellaneous samples. For safety reasons, the interference screening procedure described in section 3.1 of SW-846 9041A has not been included in this SOP.

30.0 REFERENCES

30.1 Method for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, Method 150.1, “pH, Electrometric”.

30.2 Orion Model 370 PerpHect Ion Selective pH Meter Instruction Manual

30.3 Orion 9272 pH Electrode Instruction Manual

30.4 Test Methods for Evaluating Solid Waste: Laboratory Manual Physical/ Chemical Methods, Volume 1C, SW-846, 3rd Edition, June 1997. USEPA Office of Solid Waste and Emergency Response, Washington, DC. 20460:

30.4.1 Method 9045D, “Soil and Waste pH,” Revision 4, August 2002.

30.4.2 Method 9045C, “Soil and Waste pH,” Revision 3, January 1995.

30.4.3 Method 9040C. “pH Electrometric Measurement,” Revision 3, August 2002.

30.4.4 Method 9040B, “pH Electrometric Measurement,” Revision 2, January 1995.

30.4.5 Method 9041A, “pH Paper Method,” Revision 1, July 1992.

30.5 Standard Methods, 22nd Edition, Method 4500-H^+-00, "pH value."

30.6 Exhibit D, Semivolatiles, Section 10.1.4.1, OLMO4.2

31.0 HISTORY

Revision 20: Change LCS requirement to ± 0.05 pH units.

Revision 21: Updated Standard Methods reference

Revision 22: Updated pH meter and flagging requirements.

Revision 23: Added notes requiring fill hole on probe to be in open position during calibration and analysis.

Revision 24: Updated for current process for the pH meter.

VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE FOR TOTAL SUSPENDED SOLIDS

(GL-GC-E-012 REVISION 16)

APPLICABLE TO METHODS:

Standard Methods 22nd Edition 2540 D-2011

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1.0 STANDARD OPERATING PROCEDURE FOR TOTAL SUSPENDED SOLIDS

2.0 METHOD CODE

2.1 Standard Methods 22nd Edition 2540 D-2011

3.0 METHOD OBJECTIVE/PURPOSE

This procedure is used to measure total suspended solids (TSS) in waters and wastewaters. TSS are those solids that are retained by a standard glass fiber filter and dried to a constant weight at 103 to 105 °C.

4.0 METHOD SUMMARY

4.1 Summary: In this gravimetric procedure, a water sample is filtered through a preweighed glass fiber filter. The filter is then dried to a constant weight at 103 to 105 °C. The weight of the residue on the filter represents the total suspended solids.

4.2 Synonym: Residue, total non-filterable

5.0 APPLICABLE MATRICES

5.1 Groundwater

5.2 Drinking water

5.3 Domestic and industrial wastewater

NOTE: Clients may request that this analysis be performed on miscellaneous liquid samples. In these cases the procedure is modified as necessary.

6.0 HOLDING TIME

Holding time is seven days from the time and date of collection until the start of analysis unless otherwise specified by contract.

7.0 SAMPLE CONTAINER/PRESERVATION/COLLECTION/STORAGE REQUIREMENTS

7.1 Samples may be stored in glass or plastic containers.

7.2 No preservatives are required. Preserved samples should not be analyzed.

7.3 Non-homogeneous particles such as leaves, sticks, fish, and lumps of fecal material should be excluded from the sample.

7.4 Refrigerate samples at $0 \leq 6$ °C until the start of analysis to minimize microbiological decomposition of solids.

8.0 INTERFERENCES/LIMITATIONS

8.1 Too much residue on the filter will entrap water and may require prolonged drying.

8.2 For samples that are high in dissolved solids, thoroughly wash the filter to ensure the removal of the dissolved material.

8.3 Prolonged filtration times resulting from filter clogging may produce high results due to the excessive capture of solids on the clogged filter.

9.0 PERFORMANCE CHARACTERISTICS

9.1 Method concentration range: 4 mg/L to 20,000 mg/L

9.2 Method detection limit (MDL): Refer to current MDL study.

9.3 Method precision: 0-5% RPD.

9.4 Method accuracy: 95-105% recovery.

10.0 DEFINITIONS

10.1 Desiccant: Material used to absorb moisture.

10.2 Gravimetric: Pertaining to measurement by weight.

10.3 Hygroscopic: Attracting, absorbing, and retaining atmospheric moisture.

10.4 Laboratory Control Standard (LCS): An aliquot of reagent water or other blank matrix to which known quantities of the method analytes are added in the laboratory. The LCS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

10.5 Method Blank (MB): An aliquot of reagent water or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The MB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus

10.6 Statistical Process Control (SPC) Limits: Statistically derived limits that establish acceptable ranges for recoveries of analytes of interest, including LCS, MS, MSD, PS, PSD and internal standards.

10.7 Laboratory Duplicate (DUP): Aliquots of a sample taken from the same container and processed in the same manner under identical laboratory conditions. The aliquot is analyzed independently from the parent sample and the results are compared to measure precision and accuracy.

10.8 Method Detection Limit (MDL): The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero.

10.9 CRDL: Contract Required Detection Limit

11.0 ANALYST VERIFICATION

Technicians and analysts do not analyze client samples without supervision until trained by qualified personnel and upon successful analysis of a proficiency sample. Training records are maintained as quality records.

12.0 DOCUMENTATION OF DATA

As data are obtained, they are recorded in AlphaLIMS.

13.0 SAFETY PRECAUTIONS AND HAZARD WARNING

13.1 Wear eye protection with side shields while performing procedures in the lab.

13.2 Treat all chemicals and samples as potential health hazards and limit exposure to

these chemicals to the lowest level possible. GEL maintains a current awareness file of OSHA regulations regarding the safe handling of the chemicals in the laboratory as well as a reference file of Material Safety Data Sheets (MSDS). These documents and client sample MSDS forms are maintained in the laboratory.

13.3 All personnel performing this procedure are trained in and follow the procedures in the Safety, Health and Chemical Hygiene Plan, GL-LB-N-001.

14.0 SAMPLE RECEIPT FOR ANALYSIS

14.1 The analyst/technician gives the list of samples needed to the sample custodian. The sample custodian removes the appropriate samples from cooler and either delivers them to the analyst/technician or places them on the "pick-up" shelf in the main cooler.

14.2 Analysts and technicians are responsible for retrieving their own samples when the sample custodian is not available.

15.0 INSTRUMENTATION/EQUIPMENT/GLASSWARE

15.1 Sartorius Basic BA210S or comparable analytical balance capable of weighing to 0.0001 g

NOTE: The balance must be calibrated in accordance with the procedure outlined in GL-LB-E-002 for Balances.

15.2 VWR 1370FM or comparable drying oven for operation at 103 to 105 °C

NOTE: The oven's temperature is monitored in accordance with GL-LB-E-004.

15.3 Desiccator with a color indicating desiccant

15.4 Glass fiber filter paper, 4.7 cm

NOTE: If filters are not certified to be pre-washed they must be prepared according to step 19.2.

15.5 Filtration flask (minimum size: 1 L)

15.6 Magnetic filter funnel

15.7 Tweezers

15.8 Vacuum source

15.9 Disposable pipets

15.10 Thermometer verified according to GL-QS-E-007 for Thermometer Verification and capable of measuring temperatures of 103 to 105 °C.

15.11 Aluminum weigh boats

15.12 Volumetric flasks and/or beakers, various sizes

15.13 Stirring apparatus and stir bars

15.14 Graduated cylinders of various sizes

16.0 REAGENTS

16.1 ASTM Type I deionized (DI) water. (Refer to GL-LB-E-016)

16.2 Desiccant

16.3 Celite

17.0 PREPARATION OF SAMPLES

NOTE: It is recommended that if less than 250 mL is required, sample is stirred with a magnetic stirrer at a speed to shear larger particles to a more uniform particle size.

18.0 PREPARATION OF STANDARDS

18.1 500 mg/L Celite standard.

NOTE: This standard serves as the LCS.

18.2 Add 0.0500 g of Celite to a 100 mL volumetric flask that is partially filled with DI water.

18.3 Bring to volume with deionized water. Mix well.

18.4 Prepare a separate standard for each LCS and LCS duplicate.

18.5 Document preparation of standards in AlphaLIMS according to GL-LB-E-007 for Laboratory Standards Documentation and GL-GC-E-004 for General Chemistry Standards, Definitions, and Preparation.

19.0 INSTRUMENT/EQUIPMENT START-UP PROCEDURE

19.1 Preparation of desiccators:

19.1.1 Ensure that the desiccant is activated by observing the blue color indicator.

NOTE: If < 50% of the indicator desiccant is blue, change the desiccant.

19.1.2 Desiccator must be sealed. Any moisture absorbed by the filters can cause erroneous results.

19.2 Filter papers

NOTE: If pre-washed filter papers are being used, proceed to step 22.

19.2.1 Place a glass fiber filter in the filter apparatus.

19.2.2 Rinse with three successive portions of approximately 20 mL of DI water.

19.2.3 Apply vacuum until the water is removed from the filter.

19.2.4 Place the filter on aluminum foil in an oven to dry at 103 to 105 °C for at least one hour.

19.2.5 Remove the filter from the oven and cool it in a desiccator for approximately 30 minutes.

19.2.6 Weigh each filter and record its weight in the appropriately labeled column of AlphaLIMS.

19.2.7 Repeat the cycle of rinsing, drying, desiccating, and weighing until a constant weight is obtained for each filter.

19.2.8 Store the washed and dried filter in a desiccator.

19.2.9 Alternatively, pre-washed filters may be used as purchased.

20.0 QUALITY CONTROL (QC) REQUIREMENTS

20.1 Frequency of QC:

20.1.1 A matrix duplicate is analyzed for every batch of ≤ 10 samples and for each set of ten samples in batches with > 10 samples.

20.1.2 A MB and LCS are analyzed for every batch containing ≤ 20 samples.

NOTE: An LCS duplicate is analyzed per client request.

20.2 Acceptance limits:

20.2.1 Matrix Relative Percent Difference (RPD): 0-5% RPD.

NOTE: If the difference between the sample concentration and duplicate concentration is less than or equal to the PQL and within a PQL value of each other, the results are acceptable even though the RPD may exceed current SPC limits.

20.2.2 Method blank: CRDL ≤ MB ≤ CRDL or ≤ ½ CRDL for any state of North Carolina samples.

20.2.3 LCS: 95-105% recovery.

20.2.4 LCS RPD: 0-5% RPD.

20.3 Handling out-of-control situations:

20.3.1 Notify the Group Leader immediately.

20.3.2 If the matrix or LCS RPD, MB, and/or LCS recovery fall outside of current acceptance limits, the samples to which the unacceptable quality control (QC) pertains must be reanalyzed.

20.3.3 Document in the case narrative the specific QC that is out of control and cross-reference data from any subsequent reanalysis.

20.3.4 If filtering times are greater than 10 minutes, use less sample aliquot.

21.0 RUN SEQUENCE

21.1 MB

21.2 LCS

21.3 Samples 1 through x where $x \leq 10$

21.4 Sample Duplicate

21.5 Repeat 21.3 through 21.4 for every 10 samples in the batch.

22.0 PROCEDURE

22.1 Calibration of equipment/instrumentation:

22.1.1 Balance

Ensure that the analytical balance to be used has been calibrated and it is within control limits before use. Refer to the balance logbook and GL-LB-E-002 for Balances.

22.1.2 Oven

22.1.2.1 Make sure that the oven to be used is at a temperature that is within acceptance limits.

22.1.2.2 Control limits for the drying oven are documented on the temperature log.

22.1.2.3 The oven temperature log format provides the required documentation to ensure that the oven temperature was in control during the drying process.

22.1.2.4 Refer to GL-LB-E-004 for Temperature Monitoring and Documentation Requirements for Refrigerators, Freezers, Ovens, Incubators and Other Similar Devices.

22.2 Analysis:

22.2.1 Label an appropriate number of aluminum weigh boats (one per sample and for each QC sample).

22.2.2 Remove, immediately prior to weighing, the pre-washed glass fiber filter, prepared as described in section 19.0, from the desiccator.

NOTE: NEVER touch the filter with your hands. Always use tweezers or tongs.

22.2.3 Record the weigh boat ID, sample number, and the weight of each filter to be used in the appropriate columns in the data entry screen in AlphaLIMS.

22.2.4 Dry the filters for at least one hour at 103 to 105 °C.

22.2.5 Remove the filters from the oven and cool in a desiccator until they reach room temperature. This takes approximately 30 minutes.

22.2.6 Weigh each filter and record its weight a second time in the appropriately labeled column of AlphaLIMS.

22.2.7 Repeat the cycle of drying, cooling, desiccating, and weighing until a constant weight is obtained for each filter. Constant weight is defined as a weight loss or gain of ≤ 0.5 mg or 0.0005 g.

22.2.8 Place the filter on the filter apparatus and wet with a small amount of reagent grade water, to seat the filter.

22.2.8.1 If aliquots greater than 250 mL are to be used, shake container and rapidly transfer the sample to the filter by means of a graduated cylinder

NOTE: It is recommended that for volumes less than 250 mL, thoroughly shake the sample to be analyzed. Add a magnetic stir bar and stir at a speed to shear larger particles, to obtain a more uniform particle size.

22.2.8.2 While stirring, pipet a measured volume onto the seated filter, pipeting from the approximated midpoint of the container, but not in the vortex.

22.2.9 Record the volume of sample used in AlphaLIMS.

NOTE: The volume of sample to be used can be increased or decreased depending upon the matrix of the sample. If there is minimal residue (< 1.0 mg) on the filter after filtering 100 mL, additional 100 mL aliquots of the sample must be filtered. In some cases the filter may clog, preventing additional sample from being filtered. The analyst may use less than 1000 mL of sample if the filtration time takes ≥ 10 minutes.

NOTE: 500 mL of DI water must be filtered for the MB.

22.2.10 Apply the vacuum and filter the sample.

22.2.11 Rinse the graduated cylinder three times with DI water and transfer the rinsate to the funnel to ensure that all residue is transferred.

22.2.12 Remove any lingering residue from the funnel by rinsing thoroughly with three successive volumes of DI water (approximately 10 mL each).

22.2.13 Continue to apply the suction after the filtration is complete in order to remove as much water as possible.

22.2.14 Carefully remove the filter from the filter apparatus.

22.2.15 Place the filter back in its aluminum weigh boat.

22.2.16 Repeat steps 22.2.8 through 22.2.15 for each sample or QC sample in the batch.

22.2.17 Dry the filters for at least one hour at 103 to 105 °C.

NOTE: The amount of residue on the filter should fall in the range of 10 to 200 mg.

22.2.18 Remove the weigh boats and filters from drying oven and cool to room temperature in a dessicator.

22.2.19 Weight and record the weight of each filter in AlphaLIMS in the appropriate column and place back in its weigh boat.

22.2.20 Put the weigh boats and filters back in the oven at 103°C to 105°C degree oven for at least an hour.

22.2.21 Repeat steps 22.2.18 through 22.2.20 until constant weight is obtained.

22.3 Calculation/reporting of results:

22.3.1 Calculate TSS as follows:

$$\text{TSS (mg/L)} = \frac{A - B \times 1{,}000}{C}$$

Where:

A = Final weight of the filter plus residue in g

B = Weight of the filter paper in g (This is the last weight of the filter obtained prior to filtration.)

C = Volume of the sample in (L)

22.3.2 Results are reported in mg/L.

22.3.3 RPDs are calculated as follows:

$$\frac{([\text{TSS mg/L}]\ \text{sample} - [\text{TSS mg/L}]\ \text{duplicate})}{\text{Average}\ [\text{TSS mg/L}]\ \text{of sample and duplicate}} \text{X}\ 100$$

23.0 INSTRUMENT/EQUIPMENT SHUT-DOWN PROCEDURE

Not Applicable

24.0 DATA REVIEW, VALIDATION, AND APPROVAL PROCEDURE

Refer to GL-GC-E-092 for General Chemistry Data Review and Packaging.

25.0 DATA TRANSMITTAL

When a batch is issued a status of DONE, the data are automatically available to reporting personnel.

26.0 RECORDS MANAGEMENT

All logbooks and data generated as a result of this procedure are maintained as quality records in accordance with GL-QS-E-008 for Quality Records Management and Disposition.

27.0 ROUTINE INSTRUMENT/EQUIPMENT MAINTENANCE

27.1 Ovens and Thermometers

Refer to GL-LB-E-004 for Temperature Monitoring and Documentation Requirements for Refrigerators, Freezers, Ovens, Incubators, and other Similar Devices.

27.2 Balances

Refer to GL-LB-E-002 for Balances.

27.3 Procedure for the maintenance of desiccant

27.3.1 Analysts are responsible for ensuring that the desiccators are maintained by replacing the desiccant whenever 50% of the blue indicator starts to turn pink.

27.3.2 Dry desiccant for reuse as follows

27.3.2.1 Spread desiccant out in an aluminum pan.

27.3.2.2 Place in drying oven capable of maintaining 105 °C for one hour or more.

27.3.2.3 Remove from the oven.

NOTE: All desiccants should be blue.

27.3.2.4 Place in a desiccator immediately to cool.

28.0 LABORATORY WASTE HANDLING AND DISPOSAL

For the proper disposal of sample and reagent wastes from this procedure, refer to the Laboratory Waste Management Plan, GL-LB-G-001.

29.0 METHOD VARIATION AND VERIFICATION

29.1 Step 19.2.2 differs from SM 2540 D-2011. Both methods mention three 20 mL

rinses of the filter paper with deionized water during filter preparation. As written, this SOP asks for the filter papers to be rinsed only once with 100 mL of DI water.

29.2 Constant weight is defined in SM 2540 D-2011 as a weight loss or gain of < 4% of the previous weight or 0.0005 g. Constant weight is defined in this SOP as a difference equal to or less than 0.0005 g.

29.3 MDLs are calculated in accordance with GL-LB-E-001 for Determination of Method Detection Limits.

29.4 Step 22.2.8 differs from SM 2540 D-2011, which calls for the pipetting of the sample aliquot to the filter instead of transferring by a graduated cylinder.

30.0 REFERENCES

30.1 Federal Register 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants under the Clean Water Act," April 4, 1995.

30.2 Standard Methods for the Examination of Water and Wastewater, 22nd Edition, Method 2540 D-2011.

30.3 Compilation of ASTM Standard Definitions. Sponsored by ASTM Committee on Terminology, 7th Edition, Philadelphia, American Society for Testing and Materials, 1990.

30.4 Manual of Methods for Chemical Analysis of Water and Wastes. EPA Technology Transfer, EPA-625/6-74-003a, 1976.

31.0 HISTORY

Revision 16: Added process improvements for drying. Updated Acceptance Limits.

Revision 15: TSS calculation revised.

Revision 14: Revised to include model of analytical balance and drying oven being used to comply with SCDHEC audit finding.

Revision 13: Revised quality control requirements for method blank for state of North Carolina samples.

Revision 12: Updated Standard Methods reference for MURII compliance.

VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE

FOR

ALKALINITY: TOTAL, BICARBONATE, CARBONATE, HYDROXIDE, AND PHENOLPHTHALEIN

(GL-GC-E-033 REVISION 13)

APPLICABLE TO METHODS:
EPA Method 310.1,
Standard Methods 22nd Edition, SM 2320 B-97

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1.0 STANDARD OPERATING PROCEDURE FOR ALKALINITY: TOTAL, BICARBONATE, CARBONATE, HYDROXIDE, AND PHENOLPHTHALEIN

2.0 METHOD CODE

2.1 EPA Method 310.1

2.2 Standard Methods 22nd Edition, SM 2320 B-97

3.0 METHOD OBJECTIVE AND PURPOSE

This standard operating procedure (SOP) describes the method used to determine the concentration of alkalinity (total, bicarbonate, carbonate, and hydroxide alkalinity) present in water samples.

4.0 METHOD SUMMARY

4.1 Summary: A sample aliquot is titrated with standardized sulfuric acid (H_2SO_4) solution to the phenolphthalein alkalinity (pH of 8.3) endpoint and then to the total alkalinity (pH of 4.5) endpoint. The total alkalinity concentration corresponds directly to the amount of titrant used to reach the pH 4.5 endpoint. Bicarbonate, carbonate, and hydroxide alkalinity can be determined for a sample using an alkalinity relationship table.

4.2 Synonym: Alkalinity as calcium carbonate, $CaCO_3$

5.0 APPLICABLE MATRICES

5.1 Groundwater

5.2 Drinking water

5.3 Domestic and industrial wastewater

NOTE: Clients may request that this analysis be performed on miscellaneous liquid or solid samples. In these cases, the procedure is modified as necessary.

6.0 HOLDING TIME

The holding time is 14 days from the date and time of collection until the start of analysis unless otherwise specified by contract.

7.0 SAMPLE CONTAINER, PRESERVATION, COLLECTION, AND STORAGE REQUIREMENTS

7.1 Samples are collected in glass or plastic containers.

7.2 Samples are unpreserved. Since the alkalinity concentration determination is based on the pH of a sample, preserved samples are not analyzed.

7.3 During collection, samples should not be agitated and/or exposed to the air for a prolonged period. Both conditions could affect the pH of the sample.

7.4 Samples are stored at $0 \leq 6^{\circ}$ C in accordance with GL-SR-E-001 for Sample Receipt, Login, and Storage.

8.0 INTERFERENCES/LIMITATIONS

8.1 Interferences:

8.1.1 Oil and grease can coat the pH electrode and cause a sluggish response.

8.1.2 Salts present in large amounts can cause interference in the pH measurements.

8.1.3 Samples containing high concentrations of mineral acids should be titrated to an electromagnetic endpoint of 3.9. Refer to ASTM Standards, part 3, "WATER," D-1067, Method D (1976).

8.2 Limitations

8.2.1 Avoid titration volumes greater than 50 mL by using one of the following:

8.2.1.1 Use a more concentrated sulfuric acid titrant.

8.2.1.2 Use a smaller sample volume.

8.2.2 The sample cannot be filtered, diluted, or concentrated prior to analysis without compromising the integrity of the results.

9.0 PERFORMANCE CHARACTERISTICS

9.1 Method detection limit (MDL): Refer to current MDL limits.

9.2 Method precision: Refer to current SPC limits.

9.3 Method accuracy: Refer to current SPC limits.

10.0 DEFINITIONS

10.1 AlphaLIMS: The Laboratory Information Management System used at GEL Laboratories, LLC.

10.2 Laboratory Control Standard (LCS): An aliquot of reagent water or other blank matrix to which known quantities of the method analytes are added in the laboratory. The LCS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements.

10.3 Laboratory Duplicate (DUP, LCSD, MSD or PSD): Aliquots of a sample taken from the same container and processed in the same manner under identical laboratory conditions. The aliquot is analyzed independently from the parent sample, and the results are compared to measure precision and accuracy.

10.4 Method Detection Limit (MDL): The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero.

10.5 Spike (Matrix Spike or Post Spike): An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS or PS is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS or PS corrected for background concentrations.

10.6 Statistical Process Control (SPC) Limits: Statistically derived limits that establish acceptable ranges for recoveries of analytes of interest, including LCS, MS, MSD, PS, PSD, and internal standards.

11.0 ANALYST VERIFICATION

Before a technician/analyst is allowed to analyze samples without supervision, he/she will be trained by qualified personnel. Training records are maintained as quality records.

12.0 DOCUMENTATION OF DATA

As data are obtained, they are recorded in AlphaLIMS.

13.0 SAFETY PRECAUTIONS AND HAZARD WARNINGS

13.1 Wear eye protection with side shields while performing procedures in the lab.

13.2 Treat all chemicals and samples as potential health hazards, and limit exposure to these chemicals to the lowest level possible. GEL maintains a current awareness file of OSHA regulations regarding the safe handling of the chemicals in the laboratory as well as a reference file of Material Safety Data Sheets (MSDS.) These documents and client sample MSDS forms are maintained in the laboratory.

13.3 Use care when handling concentrated forms of sulfuric acid.

14.0 SAMPLE RECEIPT FOR ANALYSIS

14.1 The analyst/technician gives the list of samples needed to the sample custodian. The sample custodian removes the appropriate samples from cooler and either delivers them to the analyst/technician or places them on the "pick-up" shelf in the main cooler.

14.2 Analysts and technicians are responsible for retrieving their own samples when the sample custodian is not available.

15.0 INSTRUMENTATION/EQUIPMENT/GLASSWARE

15.1 Beakers: Capacity of 150 mL required

15.2 Graduated cylinders: Minimum capacity of 100 mL is needed

15.3 Magnetic stir plate

15.4 Stir bar

15.5 Magnetic stir bar retriever

15.6 Squeeze bottle for deionized water

15.7 Hot plate

15.8 Burette with 0.05 mL increments: Recommended capacity, 10 mL to 20 mL

15.9 Watch glass

15.10 pH meter and probe

15.11 Oven capable of maintaining 250 ± 50˚ C

NOTE: The temperature of the oven is monitored in accordance with GL-LB-E-004.

15.12 Volumetric flask, 1 L

15.13 Aluminum weigh boat

15.14 Desiccator and desiccant

15.15 Analytical balance: Capable of weighing to 0.0001 g

NOTE: Balances are calibrated in accordance with GL-LB-E-002 for Balances.

16.0 REAGENTS

16.1 Reagents

16.1.1 Sodium carbonate (Na_2CO_3)

16.1.2 Sulfuric acid (H_2SO_4)

16.1.3 ASTM type I deionized (DI) water: See GL-LB-E-016

16.1.4 pH buffers

16.1.4.1 pH = 4

16.1.4.2 pH = 7

16.1.4.3 pH =10

16.1.5 Sodium borate ($Na_2B_4O_7 \bullet 10H_2O$)

16.2 Solutions

16.2.1 0.05 N sodium carbonate solution (Na_2CO_3)

16.2.1.1 Dry 3 to 5 g of sodium carbonate in an oven at 250 ± 50° C for 4 hours.

16.2.1.2 Remove the sodium carbonate from the oven, and cool in a desiccator.

16.2.1.3 Weigh 2.5 ± 0.2 g of the dried sodium carbonate and transfer to a 1 L volumetric flask.

NOTE: Record the weight of the sodium carbonate to the nearest mg in the standardization logbook. The weight is needed in the standardization of the sulfuric acid titrant.

16.2.1.4 Bring to volume with deionized water and mix well.

16.2.1.5 Discard after one week.

16.2.2 0.1 N sulfuric acid titrant

16.2.2.1 Transfer 3.0 mL of concentrated sulfuric acid to a 1 L volumetric flask.

16.2.2.2 Bring to volume with deionized water.

16.2.3 Standardization of the 0.1 N sulfuric acid titrant

NOTE: This must be performed monthly

16.2.3.1 Transfer 40.0 mL of 0.05 N sodium carbonate to a 250 mL beaker.

16.2.3.2 Add 60 mL of deionized water.

16.2.3.3 Put a stir bar in the beaker and place on a stir plate.

16.2.3.4 Measure the pH of the solution.

NOTE: Refer to GL-GC-E-008 for pH.

16.2.3.5 Fill a buret with the 0.1 N sulfuric acid solution.

16.2.3.6 Record the initial volume of titrant in the standardization log.

16.2.3.7 Using the sulfuric acid solution as the titrant, titrate the sodium carbonate solution to a pH of approximately 5. Record the volume of titrant used.

16.2.3.8 Remove the beaker from the stir plate.

16.2.3.9 Place a watch glass over the beaker and put the beaker on a hot plate.

16.2.3.10 Boil the solution gently for 3 to 5 minutes.

16.2.3.11 Cool to room temperature.

16.2.3.12 Rinse any moisture or residue on the watch glass into the same beaker of sodium carbonate.

16.2.3.13 Titrate the sodium carbonate solution again with the sulfuric acid titrant until a pH of 4.5 is reached.

16.2.3.14 Record the volume of titrant used in the standardization logbook. Repeat Steps 16.2.3.1 to 16.2.3.13 two more times.

16.2.3.15 Calculate the exact normality of the sulfuric acid solution using this formula:

$$\text{Normality of the } H_2SO_4 \text{ solution} = \frac{A \times B}{53.00 \times C}$$

Where:

A = g of Na_2CO_3 used in preparing Na_2CO_3 solution (refer to 16.2.1.3)

B = volume in mL of the Na_2CO_3 solution transferred to the beaker (refer to 16.2.3.1)

C = total volume of the 0.1 N H_2SO_4 acid titrant used to reach a pH of 4.5.

16.2.4 0.02 N sulfuric acid: Dilute 200 mL of the 0.1 N sulfuric acid to 1L with DI water.

16.2.5 Standardization of the 0.02 N sulfuric acid

16.2.5.1 Standardize the sulfuric acid solution monthly.

16.2.5.2 Transfer 15.0 mL of 0.05 N sodium carbonate solution to a 250 mL beaker.

16.2.5.3 Add 85 mL of deionized water.

16.2.5.4 Put a stir bar in the beaker and place on a stir plate.

16.2.5.5 Measure the pH of the solution.

NOTE: Refer to GL-GC-E-008 for pH.

16.2.5.6 Fill a buret with the 0.02 N sulfuric acid solution.

16.2.5.7 Record the initial volume of titrant in the standardization log.

16.2.5.8 Using the sulfuric acid solution as the titrant, titrate the sodium carbonate solution to a pH of approximately 5. Record the volume of titrant used.

16.2.5.9 Remove the beaker from the stir plate.

16.2.5.10 Place a watch glass over the beaker and put the beaker on a hot plate.

16.2.5.11 Boil the solution gently for 3 to 5 minutes.

16.2.5.12 Cool to room temperature.

16.2.5.13 Rinse any moisture or residue on the watch glass into the same beaker of sodium carbonate.

16.2.5.14 Titrate the sodium carbonate solution again with the sulfuric acid titrant until a pH of 4.5 is reached.

16.2.5.15 Record the volume of titrant used in the standardization logbook.

16.2.5.16 Calculate the exact normality of the sulfuric acid solution using this formula:

$$\text{Normality of the } H_2SO_4 \text{ solution} = \frac{A \times B}{53.00 \times C}$$

Where:

A = grams of Na_2CO_3 used in preparing Na_2CO_3 solution (refer to 16.2.1.3)

B = volume in mL of the Na_2CO_3 solution transferred to the beaker (refer to 16.2.5.2)

C = total volume of the 0.02 N H_2SO_4 acid titrant used to reach a pH of 4.5.

16.2.6 Sodium borate LCS: Weigh 3.814 g of sodium borate decahydrate ($Na_2B_4O_7 \cdot 10H_2O$) into a 1000 mL volumetric flask. Dilute to volume with DI water. Discard after six months.

17.0 PREPARATION OF SAMPLES

Not applicable

18.0 PREPARATION OF STANDARDS

Refer to step 16.2

19.0 INSTRUMENT/EQUIPMENT START-UP PROCEDURE

Refer to GL-GC-E-008 for pH.

20.0 QUALITY CONTROL (QC) REQUIREMENTS

20.1 Frequency of QC:

20.1.1 Matrix duplicate is analyzed for every batch of ≤ 10 samples and for each set of ten samples in batches with > 10 samples.

20.1.2 LCS is analyzed with each alkalinity batch of ≤ 20 samples. Recovery must be 100% ± 10%.

20.1.3 Prepare LCS by diluting 5 mL of sodium borate solution (16.2.6) to 100 mL with DI water. Analyze as if it were a sample.

20.1.4 A matrix spike (MS) is prepared with each alkalinity batch of ≤ 10 samples. Add 5 mL of LCS (sodium borate) solution to 100 mL of sample.

20.2 Acceptance limits:

20.2.1 Matrix relative percent difference (RPD): Refer to current SPC limits.

20.2.2 LCS: 90 – 110%.

20.2.3 MS: Refer to current SPC limits.

21.0 RUN SEQUENCE

21.1 LCS

21.2 Samples 1 through x where x ≤ 10

21.3 Sample x duplicate

21.4 Sample x matrix spike

21.5 Repeat steps 21.3 and 21.5 for the next 10 samples.

22.0 PROCEDURE

22.1 Analysis:

22.1.1 Calibrate the pH meter as described in GL-GC-E-008 for pH.

22.1.2 Record the analyst initials, date, and time of analysis in AlphaLIMS.

22.1.3 Using a graduated cylinder, transfer 10 ml to 100 mL of sample beaker or disposable container.

22.1.4 Record the volume of sample used in AlphaLIMS.

22.1.5 Measure the pH of the sample. (Refer to the GL-GC-E-008 for pH.)

22.1.6 Record this initial pH in AlphaLIMS.

22.1.7 Fill the buret with one of the standardized sulfuric acid titrants, and record the normality of the chosen titrant in AlphaLIMS.

22.1.7.1 Use 0.02 N sulfuric acid if the alkalinity is expected to be < 1000 mg/L of calcium carbonate.

22.1.7.2 Use 0.1 N sulfuric acid if the alkalinity is expected to > 1000 mg/L calcium carbonate.

NOTE: If more than 50 mL of 0.02 N titrant is required, reanalyze the sample using 0.1 N titrant.

22.1.8 Titrate the sample with the sulfuric acid titrant until the appropriate endpoint is obtained.

22.1.8.1 Phenolphthalein alkalinity: Endpoint = pH of 8.3

22.1.8.2 Total alkalinity: Endpoint = pH of 4.5

22.1.8.3 Total alkalinity less than 20 mg/L: Endpoint = pH of 4.5, then titrate to pH 4.2.

22.1.9 Record the total volume of titrant used in AlphaLIMS.

22.1.10 Repeat steps 22.1.3 through 22.1.9 for each sample and quality control sample in the batch.

22.2 Calculation/reporting of results:

22.2.1 Calculate the total alkalinity using this formula:

alkalinity in mg/L = $\frac{Vt \times N \times 50000}{Vs}$ where

Vt = volume in mL of H_2SO_4 used to reach the pH 4.5 endpoint

N = normality of the H_2SO_4 titrant

Vs = volume in mL of sample used

22.2.2 Calculate the total alkalinity for concentrations less than 20 mg/L using this formula:

Alkalinity in mg/L =

$$\frac{((2 \times V_t) - V_{t2}) \times N \times 50000)}{V_s}$$

where

Vt = volume in mL of H_2SO_4 used to reach the pH 4.5 endpoint

V_{t2} = total volume in mL of H_2SO_4 used to reach the pH 4.2 endpoint

N = normality of the H_2SO_4 titrant

Vs = volume in mL of sample used

22.2.3 Calculation of the phenolphthalein alkalinity (P)

Calculate P using this formula

P in mg/L = $\frac{Vt \times N \times 50000}{Vs}$ where

Vt = volume in mL of H_2SO_4 used to reach the pH 8.3 endpoint

N = normality of the H_2SO_4 titrant

Vs = volume in mL of sample used

22.2.4 Calculation of carbonate, bicarbonate, and hydroxide alkalinity.

These forms of alkalinity are calculated using the calculations found in Standard Methods, 18th, 19th, 20th, and 21st Editions, Method 2320B, Table 2320: II.

23.0 INSTRUMENT/EQUIPMENT SHUT-DOWN PROCEDURE

Refer to the GL-GC-E-008 for pH for shutdown procedures.

24.0 DATA REVIEW, VALIDATION, AND APPROVAL PROCEDURE

Refer to GL-GC-E-092 for General Chemistry Data Review and Packaging.

25.0 DATA TRANSMITTAL

When a batch is issued "DONE" status, it is automatically transferred via AlphaLIMS to reporting personnel.

26.0 RECORDS MANAGEMENT

All logbooks and data generated as a result of this procedure are maintained as quality records in accordance with GL-QS-E-008 for Quality Records Management and Disposition.

27.0 ROUTINE INSTRUMENT/EQUIPMENT MAINTENANCE

Refer to GL-GC-E-008 for pH for instrument maintenance.

28.0 LABORATORY WASTE HANDLING AND DISPOSAL

For the proper disposal of sample and reagent wastes from this procedure, refer to the Laboratory Waste Management Plan, GL-LB-G-001.

29.0 METHOD VERIFICATION

29.1 Method detection limit studies are performed in accordance with GL-LB-E-001 for Method Detection Limits.

29.2 EPA method 310.1 states that analysis should begin as soon as it is practical. This SOP allows 14 days from the date of collection until the start of analysis in accordance with the holding time listed in the Federal Register Volume 49 Number 209 Part 136.3 Table II.

30.0 REFERENCES

30.1 Methods of Chemical Analysis of Water and Wastes. Alkalinity. EPA 600/4 -79-020. Method 310.1 (1979).

30.2 Standard Methods for the Examination of Water and Wastewater 22nd Edition. SM 2320 B-97. "Alkalinity, Titration Method".

30.3 Federal Register 40 CFR Part 136 "Guidelines Establishing Test Procedures for the Analysis of Pollutants under the Clean Water Act," April 4, 1995.

31.0 HISTORY

Revision 9: Updated endpoint and calculations for Total Alkalinity Determinations

Revision 10: Updated Standard Methods reference

Revision 11: Updated run sequence.

Revision 12: Added Titration Steps.

Revision 13: Removed Method Blank requirements.

VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE FOR SULFIDE (METHYLENE BLUE METHOD)

(GL-GC-E-052 REVISION 11)

APPLICABLE TO METHOD:
EPA Method 376.2, Modified
Hach Method 8131
Standard Methods 22nd Edition, 4500S^{2-} D-2000

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1.0 STANDARD OPERATING PROCEDURE FOR SULFIDE (METHYLENE BLUE METHOD)

2.0 METHOD CODE

2.1 Hach Method 8131

2.2 Standard Methods 22nd Edition, 4500-S^{2-} D-2000

2.3 EPA Method 376.2, Modified

3.0 METHOD OBJECTIVE / PURPOSE

This method detects the presence of sulfide in aqueous samples. GEL uses the HACH instrumentation and chemical reagents described in HACH Method 8131, equivalent to SM 4500-S^2-D.

4.0 METHOD PURPOSE

The concentration of sulfide in water samples is measured by treating the samples with a methylene blue indicator and comparing spectrophotometric measurements of absorbance's from samples to that of a series of known standards prepared at the time of analysis.

5.0 APPLICABLE MATRICES

5.1 Groundwater

5.2 Drinking water

5.3 Domestic and industrial wastewater

NOTE: Clients may request that this analysis be performed on miscellaneous liquid or solid samples. In these cases the procedure is modified as necessary.

6.0 HOLDING TIME

Chemically preserved and chilled samples may be held up to 7 days prior to analysis.

7.0 SAMPLE CONTAINER/PRESERVATION/COLLECTION/STORAGE REQUIREMENTS

7.1 Samples are collected in glass or plastic containers.

7.2 Samples are preserved with NaOH and Zinc Acetate.

7.3 Samples are stored at 0° ≤ 6° C until the start of analysis in accordance with GL-SR-E-001 for Sample Receipt, Login, and Storage.

8.0 INTERFERENCES

8.1 For turbid samples, prepare a sulfide-free background blank as follows:

Measure 25 mL of sample into a small beaker or disposable medicine cup. Bromine water dropwise with swirling until a permanent yellow color just appears. Add phenol solution dropwise until the yellow color disappears. Use this solution as a background blank in the sample concentration calculation.

8.2 Avoid excessive agitation of samples to minimize sulfide loss

8.3 Strong reducing agents interfere in the methylene blue test by preventing formation of the blue color.

8.4 Thiosulfate at concentrations above 10 mg/L may retard color formation or completely prevent it.

8.5 Sulfide itself prevents the reaction if its concentration is very high, in the range of several hundred mg/L. To avoid the possibility of false negative results if suspected, the sample may be spiked. A low recovery could indicate high sulfide concentrations. These may require dilutions. Some Sulfide loss may occur when the sample is diluted.

8.6 Iodide, which is likely to be present in oil-field wastewaters, may diminish color formation if its concentration exceeds 2 mg/L.

8.7 Ferro cyanide produces a blue color.

8.8 Oxygen can convert sulfide to an unmeasurable form.

9.0 PERFORMANCE CHARACTERISTICS

9.1 Calibration range - 0.1 to 0.6 mg/L as S

9.2 Method detection limit (MDL) - Refer to current method detection limit study.

9.3 Method precision - Refer to current SPC limits.

9.4 Method accuracy - Refer to current SPC limits.

10.0 DEFINITIONS

10.1 Calibration Standard (CAL): A solution prepared from the primary dilution standard solution or stock standard solutions and the internal standards and surrogate analytes. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

10.2 Continuing Calibration Blank (CCB): An aliquot of reagent water or other blank matrix that is analyzed after each CCV. The CCB is used to determine whether the analytical sequence is in control during sample analysis.

10.3 Continuing Calibration Verification (CCV) Standard: An aliquot of reagent water or other blank matrix to which known quantities of the method analytes are added in the laboratory. The CCV is analyzed exactly like a sample, periodically throughout the run sequence. Its purpose is to determine whether the analytical sequence is in control during sample analysis. It may be prepared from the same source as the calibration standards, and is usually of varied concentration.

10.4 Independent Calibration Blank (ICB): An aliquot of reagent water or other blank matrix that is analyzed after each ICV. The ICB is used to determine whether there is carryover contamination after injection of the mid-level ICV.

10.5 Independent Calibration Verification (ICV): A solution of method analytes of known concentrations that is used to fortify an aliquot of Blank or sample matrix. The ICV is obtained from a source external to the laboratory and different from the

source of calibration standards. It is used to check laboratory performance with externally prepared test materials.

10.6 Laboratory Duplicate (DUP, LCSD, MSD or PSD): Aliquots of a sample taken from the same container and processed in the same manner under identical laboratory conditions. The aliquot is analyzed independently from the parent sample and the results are compared to measure precision and accuracy.

10.7 Linear Calibration Range (LCR): The concentration range over which the instrument response is linear.

10.8 Method Detection Limit (MDL): The minimum concentration of an analyte that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero.

10.9 Spike (Matrix Spike or Post Spike): An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS or PS is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS or PS corrected for background concentrations.

10.10 Statistical Process Control (SPC) Limits: Statistically derived limits which establish Acceptable Ranges for recoveries of analytes of interest, including LCS, MS, MSD, PS, PSD and internal standards.

10.11 Refer to SOP GL-QS-B-001 the Quality Assurance Plan for additional lab-wide used definitions.

11.0 ANALYST VERIFICATION

Before a technician/analyst is allowed to analyze samples without supervision, he/she will be trained by qualified personnel and will be required to successfully analyze a proficiency sample as discussed in GL-HR-E-002. Training records are maintained as quality records.

12.0 DOCUMENTATION OF DATA

As data is obtained, it is recorded in AlphaLIMS.

13.0 SAFETY PRECAUTIONS AND HAZARD WARNINGS

13.1 Wear safety glasses while in the laboratory.

13.2 Treat all chemicals and samples as a potential health hazard and limit exposure to these chemicals to the lowest level possible. GEL maintains a current awareness file of OSHA regulations regarding the safe handling of the chemicals in the laboratory as well as a reference file of Material Safety Data Sheets (MSDS.) These documents and client sample MSDS forms are maintained in the laboratory.

13.3 Sulfide 2 Reagent contains potassium dichromate. The final solutions will contain hexavalent chromium at a concentration that is regulated as a hazardous waste by Federal RCRA. Refer to the MSDS for safe handling and disposal instructions.

14.0 SAMPLE RECEIPT FOR ANALYSIS

14.1 The analyst/technician gives the list of samples needed to the sample custodian. The list is typically in the form of a computer-generated batch sheet and is placed on the clipboard outside of the main cooler. The sample custodian removes the appropriate samples from the cooler and either delivers them to the analyst/technician or places them on the "pick-up" shelf in the main cooler.

14.2 Analysts and technicians are responsible for retrieving their own samples when login personnel are not available.

15.0 INSTRUMENTATION/EQUIPMENT/GLASSWARE

15.1 Spectrophotometer for use at 664 nm with a light path of 1 cm or 2 cm

15.2 Cell, 1 cm or 2 cm

15.3 Glass volumetric flasks (100 mL)

15.4 Graduated cylinder 25 mL

15.5 Micropipet with tips; range required 0.1-1 mL

NOTE: The pipet used must be calibrated in accordance with GL-LB-E-010 for Maintenance and Use of Air Displacement Pipets.

15.6 Beakers (25 mL).

15.7 Medicine cups.

15.8 Analytical balance capable of measuring 0.0001 g.

NOTE: Balances are calibrated in accordance with GL-LB-E-002 for Balances.

16.0 REAGENTS

16.1 Deionized water (DI), Type I Reagent Water (Refer to GL-LB-E-016)

16.2 Sodium sulfide crystals, $Na_2S•9H_2O$

16.3 Sodium hydroxide pellets (NaOH)

16.4 Sulfide I reagent (Hach 1816-32)

16.5 Sulfide II reagent (Hach 1817-32)

16.6 Bromine water, 30 g/L (Hach 2211-20)

16.7 Phenol solution, 30 g/L (Hach 2112-20)

17.0 PREPARATION OF SAMPLES

Determine soluble sulfides by centrifuging the sample in a completely filled, capped tube and analyzing the supernatant. Insoluble sulfides are then estimated by subtracting the soluble sulfides concentration from the total sulfide result.

18.0 PREPARATION OF STANDARDS

18.1 Documentation of standards and their preparation is maintained in AlphaLIMS in accordance with GL-LB-E-007 for Laboratory Standards Documentation.

18.2 Preparation of stock sulfide solution

18.2.1 Partially fill a 1000 mL volumetric flask with deionized water.

18.2.2 Weigh approximately 4 g of NaOH pellets and transfer to the flask.

18.2.3 Weigh 0.7500 g Na_2S*9H_2O crystals into a small weigh boat and transfer quantitatively to the flask with NaOH.

18.2.4 Bring to volume with deionized water and invert several times to mix. This standard is 100 mg/L as S. It is stable for one week if refrigerated. The sodium sulfide crystals should also be stored in the refrigerator when not in use.

18.3 Preparation of calibration standards. These standards are unstable and must be prepared fresh daily.

18.3.1 0.1 mg/L calibration standard

Pipet 0.1 mL of the 100 mg/L Na_2S stock standard into a 100 mL volumetric flask and bring to volume with deionized (DI) water.

18.3.2 0.2 mg/L calibration standard

Pipet 0.2 mL of the 100 mg/L Na_2S stock standard into a 100 mL volumetric flask and bring to volume with deionized (DI) water.

18.3.3 0.3 mg/L calibration standard

Pipet 0.3 mL of the 100 mg/L Na_2S stock standard into a 100 mL volumetric flask and bring to volume with deionized (DI) water.

18.3.4 0.4 mg/L calibration standard

Pipet 0.4 mL of the 100 mg/L Na_2S stock standard into a 100 mL volumetric flask and bring to volume with deionized (DI) water.

18.3.5 0.6 mg/l calibration standard

Pipet 0.6 mL of the 100 mg/l Na_2S stock standard into a 100 ml volumetric flask and bring to volume with deionized (DI) water.

18.3.6 Refer to GL-LB-E-007 for Laboratory Standards Documentation.

Laboratory control sample (LCS) and initial calibration verification (ICV): Prepare a 0.4 mg/L Na_2S standard as described in 18.3.4 using a second source 100 mg/L stock standard.

18.3.7 Continuing Calibration Verification (CCV): Prepare 0.4 mg/L standard as described in 18.3.4.

18.3.8 Matrix Spike: Refer to 22.2.9.

19.0 INSTRUMENT/EQUIPMENT START-UP PROCEDURES

19.1 Turn **MODE SWITCH** of the spectrophotometer to **ABS**.

19.2 Adjust the wavelength to 664 nm.

19.3 Allow the instrument to warm up for at least 15 minutes.

19.4 Insert the stray light filter (filter range: 420 - 890 nm) into position if necessary.

19.4.1 The stray light filter is wiped prior to use with a Kimwipe.

19.4.2 Changing the stray light filter:

19.4.2.1 Removing a filter:

19.4.2.1.1 Push back handle of filter slightly to disengage the lock.

19.4.2.1.2 Lift the filter straight up.

19.4.2.2 Inserting a filter:

19.4.2.2.1 Carefully insert the proper filter into position.

19.4.2.2.2 The filter will automatically lock into place.

19.4.2.2.3 Do not touch the surface of the filter with your fingers. If you do, clean the filter prior to insertion.

19.5 Insert the appropriate cuvette holder.

19.6 Fill the appropriate cuvette with the prepared calibration blank, clean the outside surface with a Kimwipe, and place in the cuvette holder. Make sure the lid of the cuvette holder is closed.

20.0 QUALITY CONTROL (QC) REQUIREMENTS

20.1 Correlation coefficient: 0.995 or greater for the calibration curve(s).

20.2 Frequency of QC:

20.2.1 A matrix spike and matrix spike duplicate (and duplicate upon client request) are analyzed for every batch of $\leq$ 10 samples and for each set of ten samples in batches with > 10 samples.

20.2.2 A Method Blank (MB) and LCS are analyzed once for each set of 20 samples in a batch or once for batches that contain $\leq$ 20 samples.

NOTE: LCS duplicates are analyzed if required by client contract.

20.3 Acceptance limit:

20.3.1 Matrix Spike and Duplicate relative percent difference (RPD): ± 15%

20.3.2 Matrix spike recovery: 75-125%.

20.3.3 Method blank, ICB and CCB: < 0.1 mg/L or < 0.05 mg/L (½ RL) for North Carolina batches.

20.3.4 LCS: 85%-115% of known value

20.3.5 LCS RPD: ± 15%

20.3.6 CCV and ICV: 90-110% unless otherwise specified by certification or contract.

20.3.7 Calculated curve readbacks are: 50%-150% for the first point (low-end) and 90%-110% for all the other points. These recoveries are displayed on the logbook.

20.4 Handling out-of-control situations:

20.4.1 If the correlation coefficient is < 0.995:

20.4.1.1 Check the spectrophotometer and make sure that the proper filter and wavelength are being used.

20.4.1.2 Reanalyze the method blank and calibration standards.

20.4.1.3 If after performing the above two steps and the correlation coefficient is still unacceptable, prepare the standards again and check the filter. Begin the calibration procedure again.

20.4.2 If the method blank analyzed during the run has a [sulfide] ≥ 0.1 mg/L or ≥ 0.05 mg/L for North Carolina samples, prepare another blank for analysis.

20.4.3 If the RPD falls outside of current acceptance limits, sample must be rerun. Contact the Project Manager if sample volume is limited.

20.4.4 If the spike recovery falls outside of current acceptance limits, sample must be rerun. Contact the Project Manager if sample volume is limited.

20.4.5 If the reading of the sample is greater than the highest standard, the sample must be rerun at a higher dilution.

20.4.6 If check standard falls out of acceptable recovery limits, prepare another check standard and analyze. If check standard recovery still is unacceptable, rerun calibration curve and all associated samples.

21.0 RUN SEQUENCE

21.1 Sample Run Sequence

21.1.1 Calibration blank

21.1.2 Calibration standards: 0.1, 0.2, 0.3, 0.4 and 0.6 mg/L

21.1.3 ICV, ICB and LCS (0.4 mg/L sulfide standard from a different source than calibration standards)

21.1.4 Method blank

21.1.5 Sample 1

21.1.6 Sample 1 spike

21.1.7 Sample 1 spike duplicate (and sample duplicate, if client-requested)

21.1.8 Samples 2 through x where x $\leq$ 10

21.1.9 CCV and CCB

NOTE: Repeat steps 21.1.5 - 21.1.9 for each additional set of 10 (or less in batches with less than 20 samples) samples in the batch.

22.0 PROCEDURE

22.1 Calibration

22.1.1 Measure 25 mL of deionized water into a small beaker or disposable medicine cup. This is analyzed as a calibration blank.

22.1.2 Measure 25 mL of each standard to be analyzed into a small beaker or disposable medicine cup.

22.1.3 Using a micropipet, add 1 mL of Hach Sulfide I reagent to each calibration blank and standards to be analyzed. Gently swirl to mix.

22.1.4 Using a fresh tip, transfer 1 mL of Hach Sulfide II reagent to each sample to be analyzed. Immediately swirl to mix. Allow 5 minutes for color development.

22.1.5 With the calibration blank-filled cuvette still in the cuvette holder, set the **MODE SWITCH** to **ABS**.

22.1.6 Adjust the **100%T/0A COARSE** knob to approximately **0.000**.

22.1.7 Adjust the **100%T/0A FINE** knob to exactly **0.000**.

22.1.8 Remove the cuvette from the holder.

22.1.9 Replace the calibration blank in the cuvette with the first calibration standard that was prepared as described in section 22.1.1, 22.1.3, and 22.1.4.

NOTE: When replacing one solution in the cuvette with another, the cuvette is rinsed three times with the second solution prior to filling the cuvette with this solution. This aids in decreasing the chance of cross-contamination.

22.1.10 Carefully, wipe the outside of the cuvette with a Kimwipe to remove any fingerprints or water from the outside surface of the cuvette. This should be done prior to each insertion of the cuvette into the cuvette holder.

22.1.11 Insert the cuvette into the cuvette holder and close the holder's lid. The instrument will read the sample's absorbance.

NOTE: It is extremely important that the cuvette be placed into the holder in the same manner and direction each time during a set of analysis. This will decrease interferences that may be due to flaws in the cuvette's surface. For the same

reason, the same cuvette should be used throughout the entire analysis of a batch of samples.

22.1.12 Read and record the absorbance of each calibration standard by repeating steps 22.1.8 - 22.1.11.

NOTE: To minimize cross-contamination, analyze the standards in this order: 0.1mg/L, 0.2 mg/L, 0.3 mg/L, 0.4 mg/L and 0.6 mg/L.

22.1.13 Enter absorbance's for all standards in AlphaLIMS. AlphaLIMS will calculate the correlation coefficient, slope and y-intercept.

22.1.14 If the correlation coefficient is 0.995 or greater, then begin the analysis of samples.

22.1.15 If the correlation coefficient is less than 0.995, refer to section 20.4.

22.2 Analysis

22.2.1 Measure 25 mL of each sample to be analyzed into a small beaker or disposable medicine cup.

22.2.2 Using a micropipet add 1 mL of Hach Sulfide I reagent to each sample to be analyzed.

22.2.3 Using a fresh tip, transfer 1 mL of Hach Sulfide II reagent to each sample to be analyzed.

22.2.4 Allow 5 to 10 minutes for color development.

22.2.5 After rinsing the cuvette several times with treated sample, fill the cuvette with the treated sample.

22.2.6 Place the sample-filled cuvette in the holder and measure the absorbance.

NOTE: If the absorbance of the sample is greater than that of the 0.6 mg/L standard, then the sample is diluted until its absorbance reads between the low and high standards, ideally around the mid-range standard.

22.2.7 Record the absorbance and dilution factor (if applicable) in AlphaLIMS.

22.2.8 Method blanks, matrix duplicates, matrix spikes, LCS, and check standards are analyzed as described in the run sequence in section 21.

22.2.9 Sample Spike:

22.2.9.1 Add 0.1 mL of the 100 mg/L stock standard to 25 mL of sample.

22.2.9.2 Treat the spiked sample in the same manner as the other samples.

22.2.9.3 Nominal concentration of the spike is 0.4 mg/L.

22.3 Calculating and reporting results

22.3.1 Results are reported in mg/L.

22.3.2 Sample Spike Recovery:

22.3.2.1 After the concentration of the spiked sample has been obtained, the sample spike recovery (SR) can be calculated using this formula:

$$\frac{[H_2S \text{ spiked sample }] - [H_2S \text{ sample }]}{\text{Nominal concentration}} (100) = \%SR$$

22.3.2.2 If the percent spike recovery falls within the current sulfide matrix spike control limits, the spike is acceptable.

NOTE: Occasionally a sample will exhibit a low spike recovery due to sample matrix interference. If this is suspected, dilute the sample until the interference exhibited by the matrix is no longer substantial and rerun the sample and the spike at this dilution.

22.3.3 LCS:

22.3.3.1 LCS RPDs are calculated using the formula in 22.3.2.

22.3.3.2 LCS recoveries are calculated using this formula:

$$\frac{\text{actual value obtained}}{\text{theoretical value}}(100) = \%\text{LCS recovery}$$

22.3.3.3 If either the LCS RPDs or LCS % recovery fall outside of current control limits, the batch of samples are reanalyzed.

23.0 INSTRUMENT/EQUIPMENT SHUT-DOWN PROCEDURES

23.1 Remove the cuvette.

23.2 Turn the spectrophotometer's **MODE SWITCH** to **OFF**.

24.0 DATA REVIEW, VALIDATION AND APPROVAL PROCEDURES

Refer to GL-GC-E-092 for General Chemistry Data Packaging and Validation, and GL-LB-E-005 for Data Review/Validation.

25.0 DATA TRANSMITTAL

When a batch is given General Chemistry departmental "DONE" status, the data is automatically available to reporting personnel.

26.0 RECORDS MANAGEMENT

All data associated with the performance of this procedure, including relevant logbooks, are maintained as quality records in accordance with GL-QS-E-008 for the Management and Disposition of Quality Records.

27.0 ROUTINE INSTRUMENT/EQUIPMENT MAINTENANCE

Refer to the Operator's Reference Manual Model 340 Spectrophotometer for the routine maintenance of the spectrophotometers.

28.0 LABORATORY WASTE HANDLING AND DISPOSAL

For the proper disposal of sample and reagent wastes from this procedure, refer to the Laboratory Waste Management Plan, GL-LB-G-001.

29.0 METHOD VERIFICATION

29.1 Method detection limit studies are conducted in accordance with GL-LB-E-001 for the Determination of Method Detection Limits.

29.2 Modifications to the method:

29.2.1 The reference method requires immediate sample analysis; the modified method allows up to 14 days prior to analysis provided samples are preserved with zinc acetate and NaOH, and stored at 0° ≤ 6° C.

29.2.2 The modified method specifies commercially prepared Hach reagents to condition sample and produce color development whereas the reference method gives instructions for preparation of these reagents in the laboratory. The two Hach reagents are designed to replace the amine-sulfuric acid reagent, ferric chloride solution, 1:1 sulfuric acid, diammonium hydrogen phosphate, and methylene blue solutions cited in the reference method.

29.2.3 EPA Method 376.2 suggests using a wavelength of 625nm. Standard Methods 4500-S^{2-} D suggests using a wavelength of 664nm. GEL uses 664nm as the wavelength for Sulfide determination, due to better analytical results.

30.0 REFERENCES

30.1 Standard Methods for the Examination of Water and Wastewater. 22nd Edition. Method 4500-S^{2-} D-2000.

30.2 Hach Method 8131

30.3 Operator's Reference Manual Model 340 Spectrophotometer. Sequoia-Turner Corporation. May 1982.

30.4 EPA Methods for Chemical Analysis for Water and Waste, Method 376.2, “Sulfide (Colorimetric, Methylene Blue)”

31.0 HISTORY

Revision 6: Updated method reference to Standard Methods 22nd Edition, added safety note pertaining to Sulfide 2 Reagent.
Revision 7: Updated the reference to a standard used.
Revision 8: Updated the Holding Time for this procedure as required by 40 CFR, Part 136, Table II.
Revision 9: updated SPC limits for batch QC.
Revision 10: Added SM 4500 and North Carolina requirements for blanks.
Revision 11: Revised to clarify spike duplicate requirements

VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

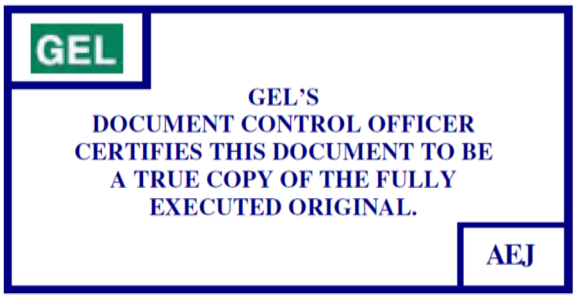
STANDARD OPERATING PROCEDURE
FOR
FLASHPOINT BY SETAFLASH

(GL-GC-E-066 REVISION 13)

APPLICABLE TO METHODS:
SW-846 Method 1020A
SW-846 Method 1020B
ASTM D3278-96

PROPRIETARY INFORMATION

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1.0 STANDARD OPERATING PROCEDURE FOR FLASHPOINT BY SETAFLASH

2.0 METHOD CODE

2.1 SW-846 Method 1020A

2.2 SW-846 Method 1020B

2.3 ASTM D3278-96

3.0 METHOD OBJECTIVE/PURPOSE

This method determines the flashpoint of a liquid within a specified temperature range.

4.0 METHOD SUMMARY

The sample is introduced into the Setaflash test cup and slowly heated. Sample vapors are exposed to a flame at 9˚ F increments until a flash is observed or until the test limit temperature is reached.

5.0 APPLICABLE MATRICES

5.1 Groundwater

5.2 Drinking water

5.3 Domestic and industrial wastewater

5.4 Oil

NOTE: Clients may request that this analysis be performed on miscellaneous liquid or solid samples. In these cases, the procedure is modified as necessary.

6.0 HOLDING TIME

SW-846 Method 1020A does not address holding times. Samples, however, should be analyzed as soon as possible, due to volatiles that may be present.

7.0 SAMPLE CONTAINER/PRESERVATION/COLLECTION/STORAGE REQUIREMENTS

7.1 Container: Glass or metal containers with at least a 25 mL capacity.

NOTE: Samples collected and stored in plastic bottles may result in higher flashpoint due to loss of volatiles.

7.2 Preservation: Samples are unpreserved.

7.3 Collection: Obtain at least a 25 mL sample and store in a clean, tightly closed glass or metal container with minimum headspace.

7.4 Storage: Samples are stored at 0° $\leq$ 6° C until the start of analysis in accordance with GL-SR-E-001 for Sample Receipt, Login, and Storage.

8.0 INTERFERENCES/LIMITATIONS

The Setaflash method is appropriate for liquids with expected flashpoints from 32° F to 230° F. The normal reporting range for Setaflash is between 75° F and 200° F.

9.0 PERFORMANCE CHARACTERISTICS

9.1 Calibration range: 32° F to 200° F

9.2 Method precision: Refer to current SPC limits.

9.3 Method accuracy: Refer to current SPC limits.

10.0 DEFINITIONS

10.1 Flashpoint: The lowest temperature at which application of the test flame ignites the vapor above the sample.

10.2 Flashpoint-140-S: Samples tested for flashpoint up to 140° F.

10.3 Flashpoint-200-S: Samples tested for flashpoint up to 200° F.

10.4 AlphaLIMS: The Laboratory Information Management System used at GEL.

10.5 Laboratory Control Standard (LCS): An aliquot of reagent water or other blank matrix to which known quantities of the method analytes are added in the laboratory. The LCS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

10.6 Laboratory Duplicate (DUP, LCSD, MSD or PSD): Aliquots of a sample taken from the same container and processed in the same manner under identical laboratory conditions. The aliquot is analyzed independently from the parent sample and the results are compared to measure precision and accuracy.

10.7 Continuing Calibration Verification (CCV): An aliquot of reagent water or other blank matrix to which known quantities of the method analytes are added in the laboratory. Its purpose is to determine whether the analytical sequence is in control during sample analysis. It may be prepared from the same source as the calibration standards and is usually of varied concentrations.

10.8 Statistical Process Control (SPC) Limits: Statistically derived limits that establish acceptable ranges for recoveries of analytes of interest, including LCS, MS, MSD, PS, PSD, and internal standards.

10.9 Matrix Spike (MS): An aliquot of a known sample to which known quantities of the method analytes are added in the laboratory. The MS is analyzed like a sample and its purpose is to determine whether the sample matrix contributes to the analytical results.

11.0 ANALYST VERIFICATION

Technicians and analysts do not analyze client samples without supervision until trained by qualified personnel and upon successful analysis of a proficiency sample. Training records are maintained as quality records.

12.0 DOCUMENTATION OF DATA

As data are obtained, they are recorded in AlphaLIMS.

13.0 SAFETY PRECAUTIONS AND HAZARD WARNINGS

13.1 Wear eye protection with side shields while performing procedures in the lab.

13.2 Treat all chemicals and samples as potential health hazards, and limit exposure to these chemicals to the lowest level possible. GEL maintains a current awareness file of OSHA regulations regarding the safe handling of the chemicals in the laboratory as well as a reference file of Material Safety Data Sheets (MSDS). These documents and client sample MSDS forms are maintained in the laboratory.

13.3 Gloves are required when handling the chemicals in this procedure.

13.4 Prior to handling radioactive samples, analysts must have had radiation safety training and understand their full responsibilities in radioactive sample handling. Some general guidelines follow:

13.4.1 Wear a plastic apron over lab coat when working with radioactive samples.

13.4.2 Protect counter tops with counter paper or work from radioactive sample handling trays.

13.4.3 Prohibit admittance to immediate work area.

13.4.4 Post signs indicating radioactive samples are in the area.

13.4.5 Take swipes of the counter tops upon completion of work. Deliver those swipes to the designated swipe count box.

13.4.6 Segregate radioactive wastes. Radioactive waste containers are obtained from Waste Management.

13.5 All samples, chemicals, extracts, and extraction residues must be transferred, delivered, and disposed of safely according to all related SOPs.

13.5.1 Segregate solid wastes from liquid wastes in the satellite area containers.

13.5.2 Segregate oil wastes from water-soluble wastes in satellite area containers.

13.6 Never leave gas cylinders unchained or untied, even when on the moving carts.

14.0 SAMPLE RECEIPT FOR ANALYSIS

14.1 The analyst/technician gives the list of samples needed to the sample custodian. The sample custodian removes the appropriate samples from cooler and either delivers them to the analyst/technician or places them on the "pick-up" shelf in the main cooler.

14.2 Analysts and technicians are responsible for retrieving their own samples when the sample custodian is not available.

15.0 INSTRUMENTATION/EQUIPMENT/GLASSWARE

15.1 Setaflash tester

15.2 Series 8 or equivalent

15.3 Disposable syringes with a minimum capacity of 2 mL

15.4 Shallow-lipped planchet that fits in heating manifold.

16.0 REAGENTS

p-Xylene

17.0 LABORATORY CONTROL STANDARD

The solvent, p-xylene, is used as the laboratory control standard (LCS) and CCV source. The standard has a flashpoint of 81° F.

18.0 INSTRUMENT/EQUIPMENT START-UP PROCEDURE

18.1 Plug in the Setaflash tester.

18.2 Turn on the instrument using the on/off switch on the rear.

19.0 QUALITY CONTROL (QC) REQUIREMENTS

19.1 Frequency of QC:

19.1.1 A matrix duplicate is run for every batch of ≤ 10 samples and for each set of ten samples in batches with > 10 samples.

19.1.2 A LCS is run once for each set of 20 samples in a batch or once for batches that contain ≤ 20 samples.

19.1.3 A CCV is run after every 10 samples and at the end of the analysis sequence.

19.2 Acceptance limits:

19.2.1 Matrix relative percent difference (RPD): Refer to current SPC control limits.

19.2.2 LCS, LCSD and CCV: 81° ± 2°

19.2.3 LCS RPD: Refer to current SPC control limits.

20.0 RUN SEQUENCE

20.1 LCS

20.2 Sample 1

20.3 Sample 1 duplicate

20.4 Sample 2 through x where x ≤ 10

20.5 CCV (after every 10 samples)

20.6 Repeat steps 21.2 through 21.4 for each set of 10 samples in the batch.

20.7 CCV at end of analysis

21.0 PROCEDURE

21.1 Setaflash 8 Series verification

The setaflash instrument is calibrated yearly for barometric pressure and temperature and verified in accordance with GL-QS-E-007.

21.2 Analysis:

21.2.1 Close the lid over test cup, securing it with a locking lever. If using high radioactive sample matrix, place shallow-lipped planchet in test cup. If using a modified method to analyze solids, place approximately 2 g of material into the test cup or planchet.

21.2.2 Using a disposable syringe, introduce a 2 mL aliquot of sample into the test cup through the sample port.

NOTE: The batch sheet indicates the test that the samples are logged for and informs the analyst of the temperature limit required for the test. Samples are accepted for Setaflash 140° F and Setaflash 200° F

21.2.3 Select a test method, enter user initials, sample ID and comments as needed on instrument.

21.2.4 Select test temperature and press ✓.

21.2.5 Wait for the cup temperature to stabilize.

21.2.6 Press ► button to start the test

NOTE: The test ends when one of the following occurs:

1. The instrument detects a flash
2. The instrument does not detect a flash by the end of the test temperature range.
3. The instrument will beep twice if a flash is detected or once if no flash is detected at the end of the test.

NOTE: Some samples may cause the test flame to grow larger or produce a halo. These effects are not flashes. A flash is the sudden appearance of a large flame, which propagates itself over the surface of the sample.

21.2.7 Once the test has ended press the home button to return to main menu.

21.2.7.1 If a flash is detected during the non-mandatory ramp search, use a fresh sample to run method 3278 ramp beginning at 5 F° lower than detected flash temperature.

21.2.7.2 If a flash is observed, repeat the determination use a fresh sample to run method 3278 finite beginning at 2 °F lower than the detected flash temperature. Continue tests at 1 °F intervals until a flash is detected.

21.2.8 Recording Data

21.2.8.1 Record the actual and adjusted temperature in AlphaLIMS. The mean of the duplicate runs is reported to the nearest 1° F provided the difference between the two values does not exceed 2° F.

21.2.8.2 If no flash is observed, record a (999) temperature in AlphaLIMS.

21.2.9 Preparing for next sample

21.2.9.1 Close the gas control valve on the Setaflash tester.

21.2.9.2 Soak up any previous sample in the Setaflash tester with paper tissues.

21.2.9.3 Clean the lid and shutter assembly.

21.2.9.4 Clean the sample port with a pipe cleaner.

21.2.10 Repeat steps 22.2.1 through 22.2.10 for each sample in the batch.

21.3 Calculation/reporting of results:

21.3.1 If a flash was not observed before the designated temperature was reached, the result is reported as greater than that temperature.

21.3.1.1 Flashpoint-140: If no flash was observed at temperatures ≤ 140° F, report (999). LIMS will report > 140° F.

21.3.1.2 Flashpoint-200: If no flash was observed at temperatures

≤ 200° F, report (999). LIMS will report > 200° F.

22.0 INSTRUMENT/EQUIPMENT SHUT-DOWN PROCEDURE

22.1 Close inlet gas valve on Setaflash unit.

22.2 Close main gas valve.

22.3 Unplug Setaflash unit.

23.0 DATA REVIEW, VALIDATION, AND APPROVAL PROCEDURE

Refer to GL-GC-E-092 for General Chemistry Data Review and Packaging.

24.0 DATA TRANSMITTAL

When a batch is given General Chemistry departmental "DONE" status, it automatically becomes available to reporting personnel.

25.0 RECORDS MANAGEMENT

All logbooks and data generated as a result of this procedure are maintained as quality records in accordance with GL-QS-E-008 for Quality Records Management and Disposition.

26.0 ROUTINE INSTRUMENT/EQUIPMENT MAINTENANCE

Refer to Setaflash Models 01SF & 03SF Technical Manual for any equipment maintenance.

27.0 LABORATORY WASTE HANDLING AND DISPOSAL

For the proper disposal of sample and reagent wastes from this procedure, refer to the Laboratory Waste Management Plan, GL-LB-G-001.

28.0 METHOD VERIFICATION

Method is verified by ensuring the accuracy of the Setaflash tester with p-xylene (LCS). Repeatability is verified by testing p-xylene (in duplicate) and/or sample duplicates.

29.0 METHOD VARIATIONS

ASTM 3278-96 recommends the LCS limit to be 81 $\pm$ 1.5° F. Due to thermometer limitations, GEL uses 81 $\pm$ 2.0° F.

30.0 REFERENCES

30.1 Test Methods for Evaluation Solid Waste Physical/Chemical Methods, Volume 1C, Method 1020A.

30.2 Standard Test Methods for Flashpoint of Liquids by Small Scale Closed-Cup Apparatus. ASTM D3278-96.

30.3 Setaflash Series 8 manual.

30.4 Test Methods for Evaluation Solid Waste Physical/Chemical Methods, Volume 1C, Method 1020B, Revision 2, November 2004.

31.0 HISTORY

Revision 13: Added new process for instrument.

Revision 12: Updated methods and criteria.

Revision 11: Removed small circle reference in procedure.

Revision 10: Added solids testing step.

VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE

FOR

REACTIVE CYANIDE AND SULFIDE

(GL-GC-E-069 REVISION 12)

APPLICABLE TO METHODS:
EPA SW-846, Chapter Seven, 7.3.3, Interim Guidance for Reactive Cyanide
EPA SW-846, Chapter Seven, 7.3.4, Interim Guidance for Reactive Sulfide

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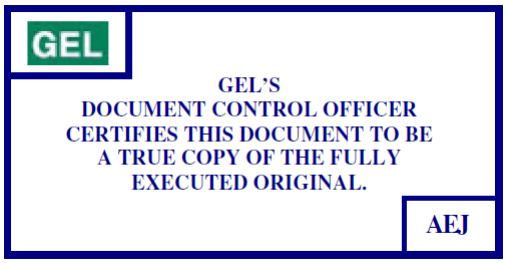


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1.0 STANDARD OPERATING PROCEDURE FOR REACTIVE CYANIDE AND SULFIDE

2.0 METHOD CODE

2.1 EPA SW-846 Chapter Seven, 7.3.3, Interim Guidance for Reactive Cyanide

2.2 EPA SW-846 Chapter Seven, 7.3.4, Interim Guidance for Reactive Sulfide

3.0 METHOD OBJECTIVE/PURPOSE

The objective of this method is to describe the procedure for measuring the concentrations of reactive cyanide and reactive sulfide given off under specific conditions in samples of various matrices.

4.0 METHOD SUMMARY

In this method, an aliquot of dilute sulfuric acid is added to a portion of waste inside a closed system. Any gas given off is swept by a flow of nitrogen gas into a sodium hydroxide scrubber solution. The concentrations of cyanide and sulfide are then determined by the conventional analysis methods for these analytes.

5.0 APPLICABLE MATRICES

5.1 Soil

5.2 Sludge

5.3 Oil

5.4 Liquid

NOTE: Clients may request that this analysis be performed on miscellaneous liquid or solid samples. In these cases, the procedure is modified as necessary.

6.0 HOLDING TIME

The holding time for liquid samples is 7 days, and solid samples have 28 days.

7.0 SAMPLE CONTAINER, PRESERVATION, COLLECTION, AND STORAGE REQUIREMENTS

7.1 Container type

Samples should be collected in a closed, light-resistant container such as an amber glass bottle.

7.2 Preservation

7.2.1 Solid and liquid samples are not preserved.

7.3 Collection

7.3.1 Samples should be collected with as little aeration as possible to avoid possible loss of sulfides.

7.3.2 Sample should be collected with zero headspace.

7.4 Storage

Samples should be stored in a cool, dark place from time of collection to time of sample preparation.

8.0 INTERFERENCES/LIMITATIONS

8.1 The interferences to this method are undetermined.

8.2 The purpose of this method is to determine whether a waste contains a concentration of cyanide and sulfide above or below the EPA guidance level (250 mg HCN/kg and 500 mg H_2S/kg). Because of the deficiencies in the method, this value should not be used as an exact concentration measurement.

9.0 PERFORMANCE CHARACTERISTICS

9.1 Method concentration range: The current EPA guidance level is:

9.1.1 Total releasable cyanide: 250mg HCN/kg waste

9.1.2 Total releasable sulfide: 500mg H_2S/kg waste

9.2 Calibration range: Refer to GL-GC-E-095 for Cyanide Analysis by Lachat QuikChem 8000 FIA and GL-GC-E-082 for Acid-Soluble Sulfides.

9.3 Method detection limit (MDL): Due to the deficiencies of the method, no MDL is determined. The reporting limit for reactive cyanide is listed in AlphaLIMS as 250 ppm. The reporting limit for reactive sulfide is listed in AlphaLIMS as 500 ppm.

9.4 Method precision: Refer to current Statistical Process Control (SPC) limits.

10.0 DEFINITIONS

10.1 AlphaLIMS: GEL's Laboratory Information Management System.

10.2 Laboratory Control Sample (LCS): A standard, usually of the same matrix as the sample batch being organized, is taken through the same prep process as the samples, then analyzed with the batch.

10.3 Method Blank (MB): An aliquot of reagent water or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards and surrogates that are used with other samples. The MB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

10.4 Laboratory Duplicate (DUP, LCSD, MSD or PSD): Aliquots of a sample taken from the same container and processed in the same manner under identical laboratory conditions. The aliquot is analyzed independently from the parent sample and the results are compared to measure precision and accuracy.

10.5 Statistical Process Control (SPC) Limits: Statistically derived limits that establish acceptable ranges for recoveries of analytes of interest, including LCS, MS, MSD, PS, PSD and internal standards.

10.6 Stock Standard Solution: A concentrated solution containing one or more method analytes prepared in the laboratory using certified reference materials or purchased from a reputable commercial source.

11.0 ANALYST VERIFICATION

Technicians and analysts do not analyze client samples without supervision until they have been fully trained and have demonstrated the ability to generate acceptable data. Training records are maintained as quality records.

12.0 DOCUMENTATION OF DATA

12.1 Sample preparation data are recorded in the AlphaLIMS.

12.2 As data are acquired, computer printouts of the cyanide data are generated. These dated hard copies of the data are stored/kept in the Cyanide hard copy binder. Data from the printouts are uploaded to AlphaLIMS in accordance with the General Chemistry SOP for data entry/data review.

12.3 The reactive sulfide data are recorded in AlphaLIMS.

13.0 SAFETY PRECAUTIONS AND HAZARD WARNINGS

13.1 Wear eye protection with side shields while in the laboratory.

13.2 Treat all chemicals and samples as potential health hazards, and limit exposure to these chemicals to the lowest level possible. GEL maintains a current awareness file of OSHA regulations regarding the safe handling of the chemicals in the laboratory as well as a reference file of Material Safety Data Sheets (MSDS). Individual sample MSDS forms provided by the clients are also maintained.

14.0 SAMPLE RECEIPT FOR ANALYSIS

14.1 The analyst/technician gives the list of samples needed to the sample custodian. The sample custodian removes the appropriate samples from the cooler and scans them to AlphaLIMS using a bar code scanner. The analyst then takes custody of the samples and scans the batch to the code for the area where the samples are to be prepped or analyzed.

14.2 Analysts and technicians are responsible for retrieving their own samples when the sample custodian is not available.

15.0 INSTRUMENTATION, EQUIPMENT, AND GLASSWARE

15.1 Instrumentation: Refer toGL-GC-E-095 for Cyanide Analysis by Lachat QuikChem 8000 FIA and GL-GC-E-082 for Acid-Soluble Sulfides.

15.2 Equipment

15.2.1 6-place Stirring apparatus

15.2.2 Stand to hold stirring apparatus and glassware in place

15.2.3 Nitrogen gas supply

15.2.4 Analytical balance capable of weighing out to at least 3 decimal places

15.2.5 Air manifold

15.3 Glassware

15.3.1 (6) 500 mL two-neck flat-bottom flask with ground glass joints

15.3.2 6 scrubber tubes

15.3.3 6 glass frits

15.3.4 6 Addition funnels with pressure equalizing tubes

15.3.5 Magnetic stir bars

16.0 REAGENTS

NOTE: All chemicals should be of ACS grade or equivalent unless otherwise specified.

16.1 Sodium hydroxide, NaOH

16.2 Sodium sulfide nonahydrate ($Na_2S•9H_2O$)

16.3 Sulfuric acid, H_2SO_4

16.4 Potassium cyanide, KCN

16.5 Deionized (DI) water, H_2O

16.6 Reagent preparation

16.6.1 0.25 N Sodium hydroxide solution

16.6.1.1 Add 10 g sodium hydroxide pellets to a 1 L volumetric flask.

16.6.1.2 Bring up to volume with DI water and put on a stir plate until completely dissolved.

16.6.1.3 Store in a suitable container. This solution may be made in bulk and stored in a carboy.

16.6.2 0.01 N Sulfuric acid solution.

16.6.2.1 Add 0.28 mL sulfuric acid to a 1 L volumetric flask already approximately half full of DI water.

16.6.2.2 Bring to volume with DI water and mix well.

16.6.2.3 Store this 0.01 N solution in a suitable closed container.

17.0 PREPARATION OF SAMPLES

17.1 Sample screening

17.1.1 Working under a hood, place a small portion of each sample in a 50 mL beaker or other suitable container.

17.1.2 Carefully place a few drops of dilute sulfuric acid solution on the sample.

17.2.3 If any violent reaction takes place, the sample is not applicable for the Reactive Cyanide/Sulfide procedure.

17.2 Setup

17.2.1 Pour 50 mL 0.25 N sodium hydroxide solution into each scrubber tube.

17.2.2 Fill each addition flask with 200 mL 0.01 N sulfuric acid solution.

17.2.3 Close the airflow path of the system and turn on the nitrogen by rotating the handle on the regulator until it is parallel to the countertop.

17.2.4 Add 10 g or 10 mL (depending on matrix) of sample to each flask.

17.3 Acidification

17.3.1 Open the stopcocks on the addition flasks so that each sample flask slowly fills up to the halfway point. At this time start the timer for the 30-minute test period.

17.3.2 Begin stirring the flasks at approximately 30 rpm (such that no vortex is created) at the same time the acid is added.

17.3.3 At the end of the thirty-minute test period, turn off the nitrogen flow and disconnect each scrubber tube.

17.3.4 Pour the solution in each scrubber tube into a suitably labeled container.

17.3.5 After the contents of each scrubber tube are poured into a suitable container, 25 mL of sample should be set aside for each analysis.

18.0 PREPARATION OF STANDARDS

18.1 Documentation of standards and their preparation is kept in the RFA Standards logbook and the Sulfide Standards logbook in accordance with GL-LB-E-007 for Laboratory Standards Documentation.

18.2 Laboratory control samples

18.2.1 1000 mg/L Cyanide reference solution

18.2.1.1 Dissolve 2.51 g KCN and 1.79 g NaOH in a 1 L volumetric flask half filled with DI water.

18.2.1.2 Bring up to volume with DI water and store in a 1 L Nalgene container. Potassium cyanide (KCN) solution, (1 mL =1000 µg CN^-).

18.2.1.3 Standardize with 0.0192 N silver nitrate, $AgNO_3$. Dilute to appropriate concentration to achieve 1 mL = 1000 µg of CN^-

18.2.1.4 Alternatively a certified standard may be purchased.

NOTE: This solution may be used for up to 6 months after preparation.

18.2.2 536 mg/L Hydrogen sulfide reference solution

18.2.2.1 Dissolve 4.02 g $Na_2S•9H_2O$ and 4 g NaOH pellets in a 1 L volumetric flask filled approximately half full of DI water.

18.2.2.2 Dilute to volume and store in a 1 L Nalgene container.

NOTE: This solution may be used for up to 1 month after preparation.

18.2.3 10 mL of stock is added to reaction flask to make the LCS Standard.

19.0 INSTRUMENT/EQUIPMENT START-UP PROCEDURE

19.1 Refer to GL-GC-E-095 for Cyanide Analysis by Lachat QuikChem 8000 FIA.

19.2 Refer to GL-GC-E-082 for Acid-Soluble Sulfides.

20.0 QUALITY CONTROL (QC) REQUIREMENTS

20.1 Batch QC

20.1.1 A sample duplicate is prepped for every batch of < 10 samples and for each set of ten samples in batches with > 10 samples.

20.1.2 An LCS and method blank are prepped at least once for every batch of 20 samples or less.

20.2 Acceptance limits

20.2.1 Method blank: Must be less than the PQL (Practical Quantitation Limit).

20.2.2 Sample duplicate: Refer to current SPC limits

20.2.3 LCS: Refer to current SPC limits

21.0 RUN SEQUENCE

21.1 Refer to GL-GC-E-095 for Cyanide Analysis by Lachat QuikChem 8000 FIA.

21.2 Refer to GL-GC-E-082 for Acid-Soluble Sulfides.

NOTE: Samples for reactive cyanide and sulfide may be run in the same run as samples for total cyanide and sulfide.

22.0 PROCEDURE

22.1 Analysis.

22.1.1 Refer to GL-GC-E-095 for Cyanide Analysis by Lachat QuikChem 8000 FIA. Samples for reactive cyanide are analyzed using the same procedure.

22.1.2 Refer to GL-GC-E-082 for Acid-Soluble Sulfides.

22.2 Calculation/reporting of results

22.2.1 The reporting limit in AlphaLIMS is 250 ppm for reactive cyanide, 500 ppm for reactive sulfide (These are the guidance levels determined by the EPA).

22.2.2 The value given as a result is only useful for characterizing whether a sample falls above or below the EPA guidance levels.

23.0 INSTRUMENT/EQUIPMENT SHUT-DOWN PROCEDURE

23.1 After all samples have been prepared, make sure the nitrogen stopcock has been turned to the “off” position.

23.2 Remove the sample flasks and deposit sample waste into the carboy for cyanide wastes.

23.3 Take all glassware to Glassware Prep

24.0 DATA REVIEW, VALIDATION, AND APPROVAL PROCEDURE

Refer to GL-GC-E-092 for data review and validation procedures.

25.0 DATA TRANSMITTAL

When a batch is issued "DONE" status, the data become available to reporting personnel.

26.0 RECORDS MANAGEMENT

All data associated with the performance of this procedure, including relevant logbooks, are maintained as quality records in accordance with GL-QS-E-008 for the Management and Disposition of Quality Records.

27.0 ROUTINE INSTRUMENT/EQUIPMENT MAINTENANCE

None required for this SOP.

28.0 LABORATORY WASTE HANDLING AND DISPOSAL

For the proper disposal of sample and reagent wastes from this procedure, refer to the Laboratory Waste Management Plan, GL-LB-G-001.

29.0 METHOD VERIFICATION AND VARIATION

29.1 This method is intended only to determine whether or not a sample will release concentrations of cyanide and/or sulfide above or below the EPA guidance levels (250 mg/L for cyanide, 500 mg/L for sulfide) under the specified conditions. It does not provide data about the concentrations of cyanide or sulfide present in the sample.

29.2 According to numerous sources at the EPA, recoveries of the reference solutions should be low. Recoveries of 10 to 30% are likely.

29.3 Sodium hydroxide is used instead of potassium hydroxide as the preserving agent for the cyanide reference solution.

30.0 REFERENCES

30.1 Test Methods for Evaluating Solid Waste, Volume 1C, Chapter 7, 7.3, Test Method to Determine Hydrogen Cyanide Released from Wastes, Revision 1, July 1992.

30.2 Test Methods for Evaluating Solid Waste, Volume 1C, Chapter 7, 7.3, Test Method to Determine Hydrogen Sulfide Released from Wastes, Revision 1, July 1992.

30.3 GL-GC-E-095 for Cyanide Analysis by Lachat QuikChem 8000 FIA.

30.4 GL-GC-E-082 for Acid-Soluble Sulfides.

31.0 HISTORY

Revision 10: Added definitions and references.

Revision 11: Procedure updated to comply with current process being used.

Revision 12: Updated concentration of reference solution used.

VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE FOR AMMONIA-NITROGEN SAMPLE PREPARATION

(GL-GC-E-072 REVISION 18)

APPLICABLE TO METHODS:
EPA Method 350.1
Standard Methods 22nd Edition, 4500-NH_3 B-2011

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1.0 STANDARD OPERATING PROCEDURE FOR AMMONIA-NITROGEN SAMPLE PREPARATION

2.0 METHOD CODE

2.1 EPA Method 350.1 (Colorimetric, Automated Phenate)

2.2 Standard Methods 22nd Edition, 4500-NH_3 B-2011

3.0 METHOD OBJECTIVE/PURPOSE

The purpose of this method is to describe the preparation procedure performed on samples of various matrices to be tested for ammonia-nitrogen by automated colorimetric analysis.

4.0 METHOD SUMMARY

In this method, a borate buffer solution is added to the sample to stabilize the pH at 9.5. The sample is then distilled on a steam distillation or a simple ammonia distillation apparatus. The distillate is collected in a solution of 0.04 N sulfuric acid. This distillate is then refrigerated until time of analysis by the automated colorimetric method.

5.0 APPLICABLE MATRICES

5.1 Domestic and industrial wastewater

5.2 Soil (method modified accordingly)

5.3 Sludge (method modified accordingly)

NOTE: Clients may request that this analysis be performed on miscellaneous liquid or solid samples. In these cases, the procedure is modified as necessary.

6.0 HOLDING TIME

Holding time for non-South Carolina samples is 28 days from the time and date of collection until the start of analysis unless otherwise specified by contract. Samples reported for compliance in South Carolina must be analyzed within 24 hours of sample preparation.

7.0 SAMPLE CONTAINER/PRESERVATION/COLLECTION/STORAGE REQUIREMENTS

All liquid samples are preserved at the time of collection with sulfuric acid to a pH $\leq$ 2 and stored at 0° $\leq$ 6° C.

8.0 INTERFERENCES/LIMITATIONS

8.1 Alkalinity over 500 mg/L as calcium carbonate ($CaCO_3$) and acidity over 100 mg/L as calcium carbonate are interferences removed by distillation.

8.2 Sample color and turbidity may also interfere with the colorimetric analysis. This may be removed from most samples by filtration or distillation.

8.3 All samples are checked with potassium iodide (KI) starch paper for the presence of residual chlorine. Any sample testing positive must have the chlorine removed by adding sodium thiosulfate solution to the sample before distillation.

9.0 PERFORMANCE CHARACTERISTICS

9.1 Method concentration range: 0.05 to 2.00 mg/L ammonia-nitrogen

9.2 Calibration range: Refer to GL-GC-E-106.

9.3 Method Detection Limit (MDL): MDL studies are performed bi-annually.

10.0 DEFINITIONS

10.1 AlphaLIMS: The Laboratory Information Management System used at GEL Laboratories, LLC.

10.2 Calibration Standard (CAL): A solution prepared from the primary dilution standard solution or stock standard solutions and the internal standards and surrogate analytes. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

10.3 Kjeldahl Nitrogen: Refers to all organic plus all ammonia-nitrogen in a sample.

10.4 Laboratory Control Standard (LCS): An aliquot of reagent water or other blank matrix to which known quantities of the method analytes are added in the laboratory. The LCS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

10.5 Laboratory Duplicate (DUP, LCSD, MSD or PSD): Aliquots of a sample taken from the same container and processed in the same manner under identical laboratory conditions. The aliquot is analyzed independently from the parent sample and the results are compared to measure precision and accuracy.

10.6 Method Blank (MB): An aliquot of reagent water or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The MB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

10.7 Method Detection Limit (MDL): The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero.

10.8 Spike (Matrix Spike or Post Spike): An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS or PS is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS or PS corrected for background concentrations.

10.9 Stock Standard Solution: A concentrated solution containing one or more method analytes prepared in the laboratory using certified reference materials or purchased from a reputable commercial source.

10.10 Refer to GL-QS-B-001 the Quality Assurance Plan for additional lab-wide used definitions.

11.0 ANALYST VERIFICATION

Before a technician/analyst is allowed to analyze samples without supervision, he/she is trained by qualified personnel and required to successfully analyze a proficiency sample.

Training records are maintained as quality records. Refer to GL-QS-E-008 for Quality Records Management and Disposition.

12.0 DOCUMENTATION OF DATA

Sample preparation data are recorded in AlphaLIMS.

13.0 SAFETY PRECAUTIONS AND HAZARD WARNINGS

WARNING

SODIUM HYDROXIDE AND SULFURIC ACID ARE HIGHLY CORROSIVE.

PREVENT SKIN AND EYE CONTACT BY USING SPECIFIED PERSONAL PROTECTIVE EQUIPMENT WHEN MAKING STOCK REAGENTS.

WORK UNDER A HOOD TO PREVENT INHALATION WHEN MAKING STOCK REAGENTS.

MAKE SURE THAT ADEQUATE VENTILATION IS USED ALONG WITH PERSONAL PROTECTIVE EQUIPMENT.

13.1 Wear eye protection with side shields while in the laboratory.

13.2 All chemicals and samples should be treated as potential health hazards, and exposure to these chemicals must be reduced to the lowest level possible. GEL maintains a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals in the laboratory as well as a reference file of Material Safety Data Sheets (MSDS). These documents are maintained in the laboratory. Individual sample MSDS forms provided by the clients are kept in Login.

13.3 Personal protective equipment

13.3.1 Gloves are required when handling the chemicals in this procedure. The gloves approved for this procedure are nitrile gloves for concentrated acids and bases.

13.3.2 Work under a hood when using concentrated acids and bases.

13.3.3 Wear heat resistant gloves when removing the distilling tubes from the unit.

13.4 Prior to handling radioactive samples, analysts must have had radiation safety training and must understand their full responsibilities in radioactive sample handling. Some general guidelines follow:

13.4.1 Wear a plastic apron over lab coat when working with radioactive samples.

13.4.2 Protect counter tops with counter paper or work from radioactive sample handling trays.

13.4.3 Prohibit admittance to immediate work area.

13.4.4 Post signs indicating that radioactive samples are in the area.

13.4.5 Take swipes of the counter tops upon completion of work. Deliver those swipes to the designated swipe count box.

13.4.6 Segregate radioactive wastes. Radioactive waste containers are obtained from Waste Management.

13.5 All samples, chemicals, extracts, and extraction residues must be transferred, delivered, and disposed of safely according to all related SOPs.

13.5.1 Segregate solid wastes from liquid wastes in the satellite area containers.

13.5.2 Segregate oil wastes from water-soluble wastes in the satellite area containers.

13.6 Never leave gas cylinders unchained or untied, including when they are on the moving carts.

14.0 SAMPLE RECEIPT FOR ANALYSIS

14.1 The analyst/technician gives the list of samples needed to the sample custodian. The list is typically in the form of a computer-generated batch sheet and is placed on the clipboard outside of the main cooler. The sample custodian removes the appropriate samples from the cooler and scans them using a bar code scanner to "DELV" status. The analyst then takes custody of the samples and scans the batch to the code for the area where the samples are to be prepared or analyzed.

14.2 Analysts and technicians are responsible for retrieving their own samples when the sample custodian is unavailable.

15.0 INSTRUMENTATION/EQUIPMENT/GLASSWARE

15.1 Equipment

15.1.1 Air Displacement Pipets

5 to10 mL with tips

10 to 100 µL with tips

100 to 1000 µL with tips

NOTE: All pipets must be calibrated in accordance with GL-LB-E-010.

15.1.2 Stir plate

15.1.3 Stirring magnets

15.1.4 Analytical balance capable of reading to second decimal place

NOTE: The balance must be calibrated in accordance with the procedure outlined in GL-LB-E-002 for Balances.

15.1.5 Labconco Protector laboratory hood

15.1.6 Water resistant lab marker

15.1.7 500 mL Nalgene squirt bottle

15.1.8 pH strips, range 8.0 to 9.7

15.1.9 Potassium Iodide (KI) starch paper

15.1.10 20 mL scintillation vials

15.1.11 50 mL graduated cylinders

15.1.12 Ammonium test strips with sodium hydroxide (NaOH) solution (purchased from vendor)

15.1.13 1 oz. portion cups

15.1.14 Glass beads

15.1.15 10 mL Luer-lock syringes

15.1.16 Acrodisk, 0.45 µm pore syringe filters

15.1.17 Parafilm

15.1.18 Vacuum source

15.2 Glassware

15.2.1 Various sizes of volumetric flasks for reagent preparation

15.2.2 Simple ammonia distillation system with all related glassware, as shown in Appendix I.

16.0 REAGENTS

NOTE: Unless otherwise specified, all chemicals should be of ACS grade or equivalent.

16.1 Ammonia standard: Stock standards may be purchased from an approved vendor.

16.2 Sodium hydroxide, NaOH

16.3 Sulfuric acid, concentrated H_2SO_4

16.4 Sodium tetraborate decahydrate, $Na_2B_4O_7 \cdot 10H_2O$

16.5 Sodium thiosulfate, $Na_2S_2O_3 \cdot 5H_2O$

16.6 Reagent preparation

NOTE: Unless otherwise stated, all reagents are usable for 6 months from date of sample preparation.

16.6.1 Sodium hydroxide solution (50% NaOH): In a 1 L Nalgene container, slowly add 500 mL DI water to 500 g sodium hydroxide pellets. Mix well and store in a covered Nalgene container.

CAUTION: This reaction is exothermic and will need a significant amount of time to cool prior to use.

16.6.2 Borate buffer solution: Partially fill a 2 L volumetric flask with DI water. Add 0.70 g NaOH pellets and 19.00 g sodium tetraborate decahydrate and dissolve. Bring up to volume with DI water and store in a suitable container.

16.6.3 Sulfuric acid solution (0.04 N H_2SO_4): Partially fill a 2 L volumetric flask with DI water. Pipet 2.24 mL concentrated sulfuric acid to the flask. Dilute to the mark with DI water and store in a suitable labeled container.

NOTE: The concentration of this solution may be increased for samples that have distillates with a pH > 2.

16.6.4 Sulfuric solution (0.08 N H_2SO_4): Partially fill a 2 L volumetric flask with DI water. Pipet 4.48 mL concentrated sulfuric acid to the flask. Dilute to the mark with DI water and store in a suitable labeled container.

16.6.5 Sodium thiosulfate solution: In a 1 L volumetric flask dissolve 3.5 g sodium thiosulfate using DI water. Bring up to volume with DI water and store in a suitable Nalgene or glass container. Prepare fresh weekly.

17.0 PREPARATION OF SAMPLES

17.1 Distillation procedure using a simple ammonia distillation unit

17.1.1 The unit should be assembled as described in the simple ammonia instructions manual. Turn the hot block on and set to 130° C.

17.1.2 Pour25 mL of sample into a 50 mL graduated cylinder. If sample is diluted, fill to 25 mL with 0.04 N sulfuric acid solution. Using the sodium thiosulfate solution in 16.6.5, remove any residual chlorine. The sample is first tested with KI starch paper. The presence of residual chlorine is verified by a color change from white to black. Sodium thiosulfate solution is not required if the sample tests negative for chlorine.

NOTE: For solid samples, place a recorded mass (typically 0.5 g) into the reflux tube and add 25 mL of 0.04N sulfuric acid solution.

17.1.3 For method blanks, use 25 mL of 0.04 N sulfuric acid solution.

17.1.4 For LCS, use 25 mL of 0.04 N sulfuric acid solution and pipet 0.250 mL of 100 mg/L standard into 25 mL of sample.

17.1.5 For matrix spikes, pipet 0.250 mL of 100 mg/L standard into 25 mL of sample.

NOTE: If sodium thiosulfate is required to remove chlorine, equal amounts should be added to the MB and LCS.

17.1.6 Add 50% sodium hydroxide solution drop wise until the pH reaches 9.5.

17.1.7 Cover the mouth of the cylinder with parafilm and shake to mix the sample. Check the pH using pH strips.

17.1.8 Add 1.25 mL of borate buffer to the sample.

17.1.9 Add 15 mL of 0.04 N sulfuric acid to the 25 mL collection traps.

17.1.10 Fill reflux tubes with samples. Add several glass beads to each reflux tube.

17.1.11 Assemble all components of the simple ammonia unit and make sure all connections are tight.

17.1.12 Turn on vacuum source. Adjust each vacuum valve to the rate of at least three bubbles per second is observed in each reflux tube.

17.1.13 Turn the heating block off after 60 minutes.

17.1.14 Disconnect the collection trap from the two port cap insert, then disconnect the vacuum line. Allow the vacuum to continue to bubble in the remaining traps

17.1.15 Using a wash bottle filled with 0.04 N sulfuric acid, bring the volume to the 25 mL fill line in the collection trap. Swirl to mix and transfer to a labeled 20 mL scintillation vial and refrigerate until time of analysis.

17.1.16 Check the pH of the distillate using pH strips. If the pH is >2, redo the distillation using a diluted portion of the sample, normally ½, ¼, or 1/10 .

17.1.17 Once all collection traps are removed and transferred, turn off vacuum source.

18.0 PREPARATION OF STANDARDS

Documentation of standards and their preparation is maintained in AlphaLIMS in accordance with GL-LB-E-007 for Laboratory Standards Documentation.

19.0 INSTRUMENT/EQUIPMENT START-UP PROCEDURE

Instrument start-up procedures are included in section 17 of this procedure.

20.0 QUALITY CONTROL (QC) REQUIREMENTS

20.1 Frequency of QC

20.1.1 A matrix duplicate and matrix spike are prepped for every batch of ≤ 10 samples and for each set of ten samples in batches with > 10 samples.

20.1.2 Prep a LCS and MB once for every batch of twenty or fewer samples.

20.2 Makeup of QC samples

20.2.1 Sample duplicate is a separate aliquot of a sample taken through the prep process exactly the same as the original sample.

20.2.2 Matrix spike is a separate 25 mL aliquot of the sample to which 0.25 mL of 100 mg/L standard is added. This gives the spike a nominal concentration of 1.00 mg/L.

20.2.3 Method blank is a 25 mL aliquot of 0.04 N sulfuric acid solution that is taken through the same prep process as the samples.

20.2.4 The LCS is a 25 mL aliquot of 0.04 N sulfuric acid solution spiked with the same amount of stock standard used in the matrix spikes.

20.3 Handling out-of-control situations

If the pH of the sample distillate is > 2, the sample must either be redistilled using a collection solution of greater acid concentration or be diluted using 0.08 N sulfuric acid solution before distillation.

21.0 RUN SEQUENCE

For Ammonia Determination by the Lachat QuikChem FIA+ 8000 Series, refer to GL-GC-E-106.

22.0 PROCEDURE

For Ammonia Determination by the Lachat QuikChem FIA+ 8000 Series, refer to GL-GC-E-106.

23.0 INSTRUMENT/EQUIPMENT SHUT-DOWN PROCEDURE

After the samples have been distilled, disassemble glassware and transfer to wash station.

24.0 DATA REVIEW, VALIDATION, AND APPROVAL PROCEDURE

24.1 While preparing a batch of samples, enter all information into the appropriate areas of the ammonia prep logbook in AlphaLIMS.

24.2 Enter the preparation data in the “Prep Data” screen of AlphaLIMS.

24.3 Print out a copy of the prep data report in AlphaLIMS.

25.0 DATA TRANSMITTAL

When a batch is issued a status of "DONE," the data are automatically made available to report personnel.

26.0 RECORDS MANAGEMENT

Records generated as a result of this procedure are maintained in accordance with GL-QS-E-008 for Quality Records Management and Disposition.

27.0 LABORATORY WASTE HANDLING AND DISPOSAL

For the proper disposal of sample and reagent wastes from this procedure, refer to the Laboratory Waste Management Plan, GL-LB-G-001.

28.0 METHOD VARIATION

28.1 Sulfuric acid (0.04 N) is used as the collection solution instead of boric acid. Standard Methods recommends the use of sulfuric acid as the collection solution when samples are analyzed by the phenate method.

28.2 GEL Laboratories, LLC developed the processes described in Section 17 from the referenced methods. Reduced volumes/masses of samples and reagents are used for the simple distillation unit. Validation of the process included initial demonstrations of capability, detection limit studies, and acceptable performance testing (PT) samples.

29.0 REFERENCES

29.1 Methods for Chemical Analysis of Water and Wastes. March 1979, EPA-600/4-79-020, "NITROGEN, AMMONIA," Method 350.1 (Colorimetric, Automated Phenate) Storet No. 00610.

29.2 Standard Methods for the Examination of Water and Wastewater, 22nd Edition. Method 4500-NH_3 B-2011. Preliminary Distillation Step.

30.0 HISTORY

Revision 18: Added Appendix 2 for Standard Concentrations.

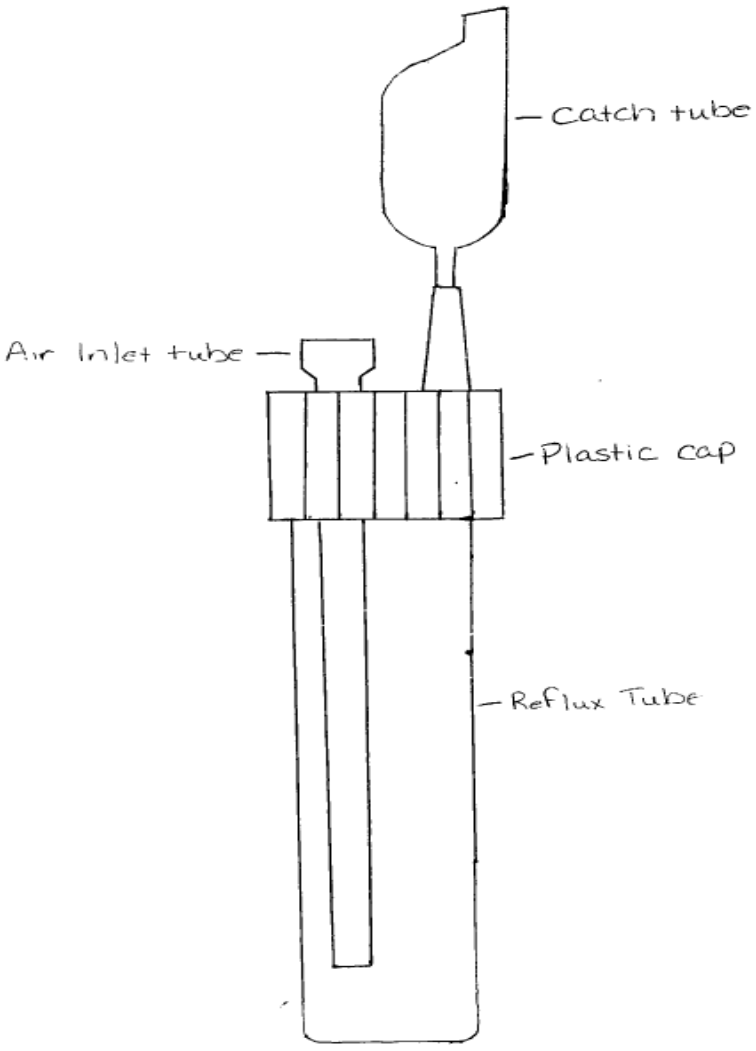
Revision 17: Updated standard method reference.

Revision 16: Revised to include preparation of solid samples and updated method reference.

Revision 15: Sodium thiosulfate added to reagents and neutralization requirements.

APPENDIX 1

SIMPLE AMMONIA DISTILLATION GLASSWARE



APPENDIX 2

Concentration of Standard	Volume of Stock	Final Volume of Standard
2 ppm	2 mL of 100 ppm std	100 mL 0.04 N H_2SO_4
1 ppm	1 mL of 100 ppm std	100 mL 0.04 N H_2SO_4
0.5 ppm	0.5 mL of 100 ppm std	100 mL 0.04 N H_2SO_4
0.1 ppm	0.1 mL of 100 ppm std	100 mL 0.04 N H_2SO_4
0.05 ppm	0.05 mL of 100 ppm std	100 mL 0.04 N H_2SO_4

Ammonia, Nitrogen

VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE FOR ION CHROMATOGRAPHY (IC)

(GL-GC-E-086 REVISION 28)

APPLICABLE TO METHODS:
EPA Method 300.0
SW-846 Method 9056A

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1.0 STANDARD OPERATING PROCEDURE FOR ION CHROMATOGRAPHY (IC)

2.0 METHOD CODES

2.1 EPA Method 300.0

2.2 SW-846 Method 9056A

3.0 METHOD OBJECTIVE/PURPOSE

3.1 This standard operating procedure (SOP) provides necessary instructions for the determination of the following inorganic anions by ion chromatography: bromide, chloride, fluoride, nitrate-N, nitrite-N, sulfate, orthophosphate-P, and iodide.

NOTE: For EPA Method 314.0 (Perchlorates by IC) refer to GL-GC-E-096.

3.2 The applicable matrixes to this method are as follows:

3.2.1 Drinking water

3.2.2 Surface water

3.2.3 Groundwater

3.2.4 Wastewater

3.2.5 Reagent waters

3.2.6 Aqueous extracts of solids

3.2.7 Leachates (when no acetic acid is used)

3.2.8 Collected solutions from bomb combustion of solid waste (Method 5050)

3.3 A small volume of sample is introduced into the ion chromatograph (IC). The anions of interest are separated and measured after traveling through a series of columns and a suppressor device. The sample passes through a conductivity detector (CD).

3.4 An extraction procedure must be performed to use this method for solids.

3.5 Approximately 4.9 mL of sample is flushed through the sample loop prior to injecting the sample. The large amount of sample flushing prevents sample cross-contamination.

4.0 METHOD APPLICABILITY

4.1 Method Detection Limit: The MDL is updated and verified every six months. For DoD QSM, LOD is verified quarterly.

NOTE: Because iodide reference Method 300.0 when analyzed, these MDLs are determined semiannually.

4.2 Method Precision: The limits are static at 100 ±10%.

4.3 Method Accuracy: Refer to the current SPC limits for these methods.

4.4 Technicians and analysts do not analyze client samples without supervision until they have been fully trained and have demonstrated the ability to generate acceptable data. Training records are maintained as quality records.

5.0 DEFINITIONS

5.1 Calibration Blank: A volume of reagent water without the analytes, internal standards or surrogates used within the calibration curve.

5.2 Calibration Standard (CAL): A solution prepared from the primary stock solution. The CAL solutions are used to calibrate the instrument.

5.3 Continuing Calibration Verification (CCV): A solution of the method analytes used to evaluate the performance of the instrument with respect to a defined set of criteria. The CCV concentration is varied throughout the calibration range.

5.4 Instrument Calibration Verification (ICV): A solution of the method analytes used to evaluate the performance of the instrument with respect to a defined set of criteria. The ICV concentration is at or near the calibration midpoint and is prepared from a different source than the calibration standards. A synonym for ICVs is an Instrument Performance Check (IPC).

5.5 Laboratory Control Sample (LCS): An aliquot of reagent water to which known quantities of the method analytes are added in the laboratory. The LCS is analyzed exactly like a sample, and its purpose is to determine whether methodology is in control. A synonym for LCS is a Lab Fortified Blank (LFB).

5.6 Linear Calibration Range (LCR): The concentration range over which the instrument response is linear.

5.7 Matrix Spike (MS): An aliquot of a known sample to which known quantities of the method analytes are added in the laboratory. The MS is analyzed like a sample and its purpose is to determine whether the sample matrix contributes to the analytical results. distribute

5.8 Method Blank (MB): An aliquot of reagent water that is treated exactly as a sample including exposure to all glassware, equipment, solvents and reagents which are used with other samples. The Method Blank is used to determine if method analytes or other interferences are present in the laboratory environment.

5.9 National Institute of Standards and Technology (NIST): For the purpose of this method, the national scientific body responsible for the standardization and acceptability of analyte solutions.

5.10 Refer to GL-QS-B-001 the Quality Assurance Plan for additional lab-wide used definitions.

6.0 METHOD VARIATIONS

6.1 Iodide is analyzed using a different instrument setup than that used for normal anions in accordance with SW846 9056A and EPA 300.0. These differences are described in section 14.3.

6.2 Retention Time study is done over a 3 day period instead of a 24-hour period.

7.0 SAFETY PRECAUTIONS AND HAZARD WARNINGS

WARNING

HYDROCHLORIC ACID IS A CORROSIVE AND A POISON.

SODIUM HYDROXIDE IS A CORROSIVE AND A POISON.

WORK UNDER A HOOD TO PREVENT INHALATION WHEN MAKING STOCK REAGENTS.

7.1 Wear eye protection with side shields while in the laboratory.

7.2 All chemicals and samples should be treated as potential health hazards, and exposure to these chemicals must be reduced to the lowest level possible. GEL maintains a current awareness file of OSHA regulations regarding the safe handling of the chemicals in the laboratory as well as a reference file of Material Safety Data Sheets (MSDS). Individual sample MSDS forms provided by the clients are also maintained.

7.3 Personal protective equipment

7.3.1 Gloves are required when handling the chemicals in this procedure. The gloves approved for this procedure are:

7.3.1.1 Nitrile gloves for concentrated acids and bases.

7.3.1.2 Non-sterile ambidextrous gloves for sample prep and standard handling.

7.3.2 Work under a hood when using concentrated acids and bases, and acetonitrile.

7.4 Prior to handling radioactive samples, analysts must have had radiation safety training and understand their full responsibilities in radioactive sample handling. Some general guidelines follow:

7.4.1 Protect counter tops with counter paper or work from radioactive sample handling trays.

7.4.2 Prohibit admittance to immediate work area.

7.4.3 Post signs indicating radioactive samples are in the area.

7.4.4 Take swipes of the counter tops upon completion of work. Deliver those swipes to the designated swipe count box.

7.4.5 Segregate radioactive wastes in radioactive waste containers are obtained from Waste Management.

7.5 All samples, chemicals, extracts, and extraction residues must be transferred, delivered, and disposed of safely according to all related SOPs.

7.5.1 Segregate solid wastes from liquid wastes in the satellite area containers.

7.5.2 Segregate oil wastes from water-soluble wastes in the satellite area containers.

7.6 Never leave gas cylinders unchained or untied, including when they are on the moving carts.

7.7 In the event of an accident or medical emergency, call for help immediately. When time and safety permit, an accident report form should be completed and turned in to the safety committee.

7.8 Fire escape routes are posted in the lab, and all personnel should be familiar with them. In addition, fire safety equipment such as fire extinguishers is located in the lab. Training is available on the proper operation of this equipment.

8.0 INTERFERENCES/LIMITATIONS

8.1 Interferences may be caused by substances with similar retention times which overlap those of the anions of interest. High concentrations of one analyte may interfere with the detection of another analyte. Usually a sample dilution and/or a gradient elution will overcome interferences evidenced by retention time problems.

8.2 Limitations may occur if particulates remain in a sample after filtration, or if the client insists that the sample is analyzed without filtration.

8.3 Many interferences are caused by contaminants in the reagent water, reagents, glassware, etc., which lead to elevated baselines or artifacts on the chromatograms.

8.4 Any anion that is not retained by the column or just slightly retained will elute in the area of fluoride and interfere. Known coelution is caused by carbonate and other small organic anions. At concentrations of fluoride above 1.5 mg/L, this interference may not be significant.

8.5 Because nitrite, orthophosphate, and bromide elute very closely together, they are potential interferants. When any of these anions are to be quantified in the presence of others, dilutions may be necessary to completely resolve the analytes.

9.0 APPARATUS, MATERIALS, REAGENTS, EQUIPMENT AND INSTRUMENTATION

9.1 Apparatus and Equipment for Dionex IC:

9.1.1 Dionex 5 mL sample vials and filter caps or equivalent

9.1.2 Dionex 5 mL automated sampler cassettes or equivalent

9.1.3 4 L reservoirs

9.1.4 Helium tanks (minimum purity, 99.995%) or equivalent

9.1.5 1-5 mL Oxford, 10-100 µL, and 100-1000 µL Eppendorf pipets or equivalent

9.1.6 0.20 µm Acrodisc syringe filters or equivalent

9.1.7 10 cc disposable syringes or equivalent

9.1.8 10, 50, 100, 250 and 500 mL volumetric flasks

9.1.9 25, 50 and 100 mL graduated cylinders

9.2 Analyte Reagents: All reagents must be American Chemical Society certified or equivalent.

9.2.1 Sodium chloride (NaCl)

9.2.2 Sodium hydroxide (NaOH)

9.2.3 Acetonitrile

9.2.4 Hydrochloric acid (HCl), concentrated

9.2.5 Sodium carbonate

9.2.6 Sodium bicarbonate

9.2.7 Stock standards for anions of interest at concentration of 1000 ppm or greater. These are usually purchased as certified solutions either in individual standards or in mixes of anions.

9.2.8 Teflon chip, glass beads or sand.

9.3 Eluent for IC methods 9056A/300.0 performed on the Dionex ICs

9.3.1 Eluent stock: 225 mM sodium carbonate/40 mM sodium bicarbonate.

9.3.1.1 Add 47.7 g of sodium carbonate and 6.72 g of sodium bicarbonate to a 2000 mL volumetric flask and fill to volume with DI water.

9.3.1.2 Working eluent: Dilute 80 mL stock (from 9.3.1.1) per four liters of DI water to make 4.5 nM sodium carbonate/0.8 sodium bicarbonate.

9.3.1.3 Sparge with helium to degas, if required.

9.3.2 Eluent for Iodide: 50 mM sodium hydroxide

9.3.2.1 Add 16.0 g of sodium hydroxide 50/50% solution to a 4L flask and bring to volume with DI water.

9.3.2.2 Sparge with helium to degas, if required.

9.4 Column Cleaning Solution: Combine 800 mL acetonitrile with 20 mL of concentrated hydrochloric acid and 180 mL DI water for the Dionex ICs.

9.5 Instrumentation:

9.5.1 Dionex ICS 1600 Ion Chromatography instrument consisting of the following components:

9.5.1.1 SP Isocratic pump

9.5.1.2 Autosampler Models AS40 or better

9.5.1.3 CD Conductivity Detector

9.5.1.4 DC separation Center with Rheodyne valve and suppressor

9.5.1.5 Computer Pentium IV, 1.0 G RAM, or comparable Windows 2000 (SP4), Windows 7

9.5.1.6 Instrument Operation Software: Chromeleon 7.2

9.5.1.7 50 µL sample loop (used for anion method)

9.5.2 Dionex ICS 5000 Ion Chromatography instrument consisting of the following components:

9.5.2.1 SP Isocratic pump

9.5.2.2 Autosampler Models AS40 or better

9.5.2.3 CD Conductivity Detector

9.5.2.4 DC separation Center with Rheodyne valve and suppressor

9.5.2.5 Computer Pentium IV, 1.0 G RAM, or comparable Windows 2000 (SP4), Windows 7

9.5.2.6 Instrument Operation Software: Chromeleon 6.80 SP2

9.5.2.7 50 µL sample loop (used for anion method)

9.5.3 Dionex ICS 3000 Ion Chromatography instrument consisting of the following components:

9.5.3.1 SP Isocratic pump

9.5.3.2 Autosampler Models AS40 or better

9.5.3.3 CD Conductivity Detector

9.5.3.4 DC separation Center with Rheodyne valve and suppressor

9.5.3.5 Computer Pentium IV, 1.0 G RAM, or comparable Windows 2000 (SP4), Windows 7

9.5.3.6 Instrument Operation Software: Chromeleon 6.80 SP2

9.5.3.7 50 µL sample loop (used for anion method)

9.5.4 Dionex Aquion I Ion Chromatography instrument consisting of the following components:

9.5.4.1 IC separation Center with Rheodyne valve and suppressor and CD.

9.5.4.2 Autosampler Model AS-DV

9.5.4.3 Computer Microsoft Windows 8.1 or Windows 7 or better.

9.5.4.4 Instrument Operation Software: Chromeleon 7.2 SR4.

9.5.4.5 50 µL sample loop (used for iodide method)

9.5.5 Dionex columns for anions by 9056A/300.0

9.5.5.1 Analytical Column; AS23

9.5.5.2 Guard Column; AG23

9.5.6 Dionex columns for iodide by 9056/300.0

9.5.6.1 Analytical Column; Dionex AS16

9.5.6.2 Guard Column; Dionex AG16

10.0 SAMPLE HANDLING AND PRESERVATION

10.1 Sample containers can be either glass or plastic. Samples should have no chemical additives for preservation purposes. All samples should be stored at an approximate temperature range of $0° \leq 6°C$ from the time of collection until the time of analysis.

10.2 All anions and iodide have 28-day holding times except nitrate (NO_3), nitrite (NO_2) and o-phosphate (PO_4), which have 48 hours. Holding times are calculated from the time and date of collection until the start of analysis, unless otherwise specified by the contract.

NOTE: The 48-hour hold time for solid samples (nitrate, nitrite, and o-phosphate) are calculated from the time of extraction.

10.3 If sample analysis is not performed before the holding time expires, the Project Manager will be notified. The Project Manager will determine if analysis should continue. If for any reason the Project Manager is unavailable, analysis will be performed and appropriate people notified as soon as possible.

10.4 Sample retrieval

10.4.1 The analyst/technician gives the list of samples needed to the sample custodian. The sample custodian removes the appropriate samples from the cooler and scans them in AlphaLIMS to the appropriate code for the area delivers. The analyst takes custody of the samples and scans them in AlphaLIMS to the Batch number and area where the samples are to be prepared and analyzed.

10.4.2 Analysts and technicians are responsible for retrieving their own samples when the sample custodian is not available.

11.0 PREPARATION OF SAMPLES

11.1 All IC samples are marked in login as "UNPRESERVED." Only unpreserved samples should be run on the IC. Any sample preparation is done only with the consent of the analyst. Most sample preparations are in the form of filtrations and can be performed by using 0.2 µm acrodisc filters and a syringe.

NOTE: It is assumed that material within the sample container is considered the sample. Removal of any extraneous material (leaves, twigs, large rocks, etc.) must be documented in the case narrative.

11.2 Extractions: Most extractions are performed on soil samples where a known weight of approximately 4.0 g of sample is diluted with DI water to a volume of 40 mL. Mix thoroughly for a minimum 5-10 minutes. The sample is then allowed to settle for a minimum of 1 hour. After 1 hour, if necessary, a centrifuge may be used. The extract is then filtered prior to injecting on the instrument. All prep data are recorded in AlphaLIMS prep logbooks.

11.3 Bomb Prep: Bomb preps are performed on sample matrices such as coal or oil. Here a known weight of approximately 0.5 g of sample is used and after being bombed, the residuals are filtered into a 100 mL volumetric flask and brought to volume with DI water. For instructions on performing a bomb prep, refer to GL-GC-E-079 for Bomb Preparation Method for Solid Waste. This prep procedure will not involve the various reagents from ASTM D 808-91 since the final analysis will be done by ion chromatography rather than gravimetrically. All prep data are recorded in the AlphaLIMS Prep logbook.

12.0 PREPARATION OF STANDARDS SOLUTIONS AND QUALITY CONTROL STANDARDS

12.1 Vendor-prepared stock standards are used to prepare calibration and check standard solutions for this analysis. Both individual analyte solutions and mixes of analytes may be purchased for use as standards. All stock standards are stable for at least one month when stored at $0° \leq 6°$C. Unopened stock solutions may be stored at ambient temperatures and their expiration is 1 year from receipt or the manufacturer's expiration date, whichever is sooner. There must be at least two independent sources of standards materials for calibration and calibration verification standard preparation.

12.2 Calibration standards are prepared from stock standards at a minimum of five concentration levels per analyte. The working standards for anions that include nitrate, nitrite and orthophosphate are prepared fresh daily and expire 48 hours after preparation. The working standards for iodide are prepared weekly (7 days). Refer to Appendix 2 for standard concentration.

12.3 Standard preparations are recorded in the AlphaLIMS system for documentation and recording of standards and reagents. Refer to GC-LB-E-007 for details on assigning unique standard IDs and establishing traceability.

12.4Laboratory control sample(s) (LCS) may be prepared from the calibration standard source.

12.5 ICVs are separate sources from the standards used in the calibration curve.

12.6 CCVs are varied in concentration and may be from the same source as the calibration standards.

12.7 Spiking compounds are added from commercially prepared standards solutions.

13.0 INSTRUMENT CALIBRATION AND PERFORMANCE

13.1 Establish ion chromatographic operating conditions for the determination of target ions. An external calibration is then performed.

13.1.1 For each analyte of interest, prepare a minimum of five calibration standards and a blank by adding accurately measured volumes of source standards to a known volume of reagent water. One of the calibration standards shall be at the PQL/LOQ.

13.1.2 The standards are placed on the instrument for analysis from highest to lowest. To automatically update the calibration curve enter AUTOCAL in the sample ID, followed by the level number.

13.1.3 Tabulate each concentration against area count. Include the blank in the linear calibration curve. Do not force the points through zero. The results determine a calibration curve.

13.1.4 The final calibration curve must have a correlation coefficient of 0.995 or better. If one standard is inconsistent with the rest of the standards it is acceptable to rerun the outlying standard and replace it in the curve.

13.1.5 If a sample analyte concentration is greater than the highest standard in the calibration range, the sample will be diluted with reagent water to approximately mid-range and reanalyzed.

13.1.6 The analyte's retention time is updated from the last standard run. A typical retention window size is 5-10%. This can be modified based on the analyst's experience.

13.1.7 Retention time must be updated after method set-up and after major maintenance. The acceptance criteria for RT width is ± 3 times standard deviation for each analyte over a 24-hour period. The RT window width position must be established once per multipoint calibration and position shall be at midpoint of initial calibration curve.

13.2 Calibration is verified daily or with every batch of samples. If the concentration or retention time window shifts by more than 10% from the expected value, a new calibration must be performed prior to the analysis of any samples.

13.3 Linear Calibration Range (LCR): The LCR is determined initially by acceptance criteria of the curve (0.995 correlation coefficient or better for each analyte). It is verified by using a blank and at least 3 standards. The ICV which is run daily may be used as a LCR verification, and the alternating concentrations of CCVs may also be considered for the same purpose. Acceptance criteria are 90-110% for all of these standards.

14.0 ANALYSIS AND INSTRUMENT OPERATION

14.1 Typical Run Sequence

14.1.1 ICV/IPC

14.1.2 ICB

14.1.3 MB

14.1.4 LCS (and LCS Duplicate if required)/LFB

14.1.5 Sample 1 through 8

14.1.6 CCV

14.1.7 CCB

NOTE: One LCS (and LCS duplicate, if applicable) per batch of 20 samples, and one matrix duplicate, matrix spike, and matrix spike duplicate (if applicable) for every 10 samples in a batch.

NOTE: A CCV and CCB are to be run after every 10 samples and at the end of the analysis sequence.

14.2 Analysis by Dionex ICS 3000

14.2.1 Fill the eluent reservoir as necessary with the proper eluent for the method that will be analyzed. This will be performed at the start of the working day. This will allow the daily checks to verify the calibration curve due to changing eluent.

14.2.2 All components should be turned on and the AS-DV autosampler should be in local position.

14.2.3 On the computer go to Chromeleon icon. ICS 3000 Panel will come up. Click on startup button. Make sure all switches turn green except the prime off button it should remain red.

14.2.4 Go to window tab on toolbar and go to Browser window. This will pull the method and sequence.

14.2.5 Sequence: It is good practice to create a new sequence daily. Rename previous sequence the new date and edit accordingly.

14.2.6 Go to batch tab on toolbar, on dropdown go to edit and a window will open go to Batch list tab and add the days sequence. Click on Ready check button. If everything is good click ok. Go to AS40 autosampler and hit load button. When plunger is all the way down hit start button in window.

14.2.7 Shutdown: The shutdown method is the loaded from the sequence.

14.3 Analysis by Dionex Aquion for Iodide Only

14.3.1 Fill the eluent reservoir as necessary with the proper eluent for the method that will be analyzed. This will be performed at the start of the working day. This will allow the daily checks to verify the calibration curve due to the changing eluent.

14.3.2 To connect the computer to components open icon Chromeleon 7. Click on Instruments (to turn to blue) go to Home tab. On the instrument images (Pump_ECD+AS-DV) click the top left buttons to turn green. Go to Pump_ECD tab to turn on Pump and Suppressor. Make sure all switches are greens.

14.3.3 Click on DATA (to turn blue). Edit schedule accordingly. You may also access the instrument method and process method if needed.

14.3.4 After editing daily schedule go to Instruments (turn to blue) and go to Queue tab click on add button to instrument and data, go to IC10folder and double click on the sequence to be loaded.

NOTE: For the CD, the temperature compensation defaults to 1.7, and the output range is set to 10.0 µS. With the Sodium Hydroxide eluent, the pump will flow at 1.2 ml/min for, and the pump pressure should normally be between 1600 and 2000 psi when using Dionex columns.

14.3.6 Prepare the initial rack of samples according to the run sequence:

14.3.7 Place 5 mL of the samples to be analyzed in the sample vials and place in the Autosampler AS-DV and push button on autosample to reset to #1 spot.

14.3.8 To start your daily sequence you can either click start button in queue or click on the start or resume button on the sequence page.

14.3.9 Go to the computer terminal and pull up the main menu "Run" screen.

14.3.10 On the run screen, under "load" choose "schedule."

NOTE: A method should have been loaded in the start-up procedure. Sample, method, and datafile names are located in each schedule. Creating a schedule allows the analyst to designate which method to use for each sample in the run, as well as where and with what file name the analyst wishes to store the data. Remember, this run information should be entered in the main menu "Schedule" screen and saved with the appropriate naming convention.

14.3.11 At the load schedule screen, give the name of the schedule for the run. On the next screen, designate the injection number if other than 1 to start the run.

14.3.12 At this point (still under main menu run) make sure the auto sampler is in the run position and choose the "run" command.

14.3.13 Under run, select "start" and confirm at the OK prompt. The run will begin.

NOTE: Instrument shutdown is performed manually.

14.4 Manual Shutdown:

14.4.1 Go to instruments, then Pump_ECD tab turn off Pump and Suppressor.

14.4.2 Turn off pressure valve on eluent tank and the pressure valve on water bottle.

15.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

Routine maintenance for the IC most often includes the changing of eluent or of the helium tank, as well as, priming the pump and clearing the flow lines of any trapped air bubbles. It

may also include any column work, such as cleaning, or it may call for regenerating the suppressor or changing of filters. The column cleaning solution is made as detailed in section 8.4.

16.0 DATA RECORDING, CALCULATION, AND REDUCTION METHODS

16.1 The raw data are processed by the IC software system. The software creates a data file including the method, which is stored on the network drive. The software then provides a printout with the following information: sample name, method used to process data, date and time run, (if report time differs from collection time both will be listed), analytes found with concentration, analytes not found and the sample chromatography.

16.2 An external program is used to generate a runlog, which is placed in the runlog binder. The runlog contains the following information in summary format: sample name, date and time run, batch number, dilution, QC type and description.

16.3 The following calculations are used in processing the data.

CSTD % Recovery = Reported Value/Expected Value x100

RPD = Sample 1 - Sample 2/(Sample 1+ Sample 2)/2 x100

% Rec = Spike - Sample/Nominal Concentration x100

Bomb Prep = Sample x Final Volume/Sample Weight

Extraction = Sample x Final Volume/Sample Weight

16.4 For Bomb Calculation:

16.4.1 Calculate the concentrations of each element detected in the sample according to the following equation:

$$C_o = \frac{C_{com} \times V_{com} \times DF \times \%moisture \times 100}{W_o}$$

Where:

C_o = Concentration of element in the sample, mg/kg

C_{com} = Concentration of element in the combustate, mg/L

V_{com} = Total volume of combustate, mL

DF = Dilution Factor

W_o = Weight of sample combusted, g

16.4.2 Percentage by weight of sulfur in sulfate (SO_4):

Where:

S = 32.06 amu,

O= 15.9994 amu x 4 = 63.9976 amu

SO_4= 96.0576 total amu

32.06 amu (S)/96.0586 amu (SO_4) = 0.333758 or 33.37% S in SO_4

16.4.3 MDLs and PQLs are 10X for all bombs.

16.5 Each compound of interest must be within the acceptable range to be reported as a positive detection. If the instrument is re calibrated during the day, the retention windows will be reset after the multipoint is complete.

17.0 QUALITY CONTROL (QC) REQUIREMENTS

17.1 According to EPA Method 300.0, a formal Quality Control Program for this method must be maintained by each laboratory. The GEL QC Program for method 300.0 consists of 2 main components.

17.1.1 MDL Study: a new MDL study covering IC analytes under EPA 300.0 Part A is performed every 6 months, using the guidelines set forth in GL-LB-E-001. For DoD samples LOD is verified quarterly on each instrument used for DoD sample analyses.

17.1.2 Quality Control Sample (QCS) study: A commercially available standard (traceable to NIST standards when possible) is run each time a client, state, or federal performance evaluation study is done. The results of this known must be within the vendor guidelines for the PE sample result to be considered acceptable. If the result is not acceptable the analyst must troubleshoot the problem immediately and reanalyze both the vendor known and the PE sample.

18.0 INSTRUMENT AND BATCH QC REQUIREMENTS

18.1 Instrument QC

18.1.1 A calibration curve is run any time the values or retention times of the check standards shift by more than 10%.

18.1.2 An initial calibration verification (ICV) is run as the first sample in each day's run.

18.1.3 An initial calibration blank (ICB) is run following the ICV.

18.1.4 A continuing calibration verification (CCV) of varying concentration is run after every 10 analytical samples and after the last analytical samples in the run.

18.1.5 A continuing calibration blank (CCB) is run after every CCV.

18.1.6 The ICV, ICB, CCVs of varying concentrations, and CCBs are used throughout the analysis to constantly check the linear range of the instrument. The ICB and CCB must not exceed the concentration of the low standard in the calibration curve. The ICV and CCVs of the varying concentrations must recover within 10% of the true value of the standard.

18.2 Batch QC

18.2.1 A sample spike and sample duplicate are run for every batch of <10 samples and for each set of ten samples in batches with >10 samples.

18.2.2 A Method Blank (MB) and Laboratory Control Sample (LCS) are run at least once for every batch of 20 samples or less. For solid batches, 4g of Teflon chips or sand is used.

18.2.3 For solid batches where the sample is prepped using DI water extraction, the LCS is made by spiking the aliquot of the Source standard onto 4 g of Teflon chips or sand to dilute to 40 mL with DI water.

18.2.4 For nonaqueous liquids or solids not easily DI extracted, the LCS and method blank are bomb prepped in the same manner as the samples.

NOTE: Some clients have statements of work that call for additional QC such as LCS Duplicates or Matrix Spike Duplicates.

18.3 Acceptance limits:

18.3.1 Correlation coefficient must be 0.995 or greater.

18.3.2 ICV recovery must be 90-110% for all batches.

18.3.3 CCV recovery must be 90-110% recovery for all batches.

18.3.4 Matrix relative percent difference (RPD): Determined yearly by the Statistical Process Control (SPC) system of AlphaLIMS. The limits are given in spreadsheet form to each analyst.

NOTE: DoD requires RPD limits of 0 < 10% for sample DUPs and 0 ≤ 15% for MS/MSD.

18.3.5 Matrix spike recovery: The Limits are static at 90-110% recovery for samples analyzed by EPA 300.0. Matrix spike limits for samples analyzed by SW-846 9056 are determined yearly by the Statistical Process Control (SPC) system of AlphaLIMS.

18.3.6 Method Blank: Must be less than the CRDL or Practical Quantitation Limits (PQL). DoD QSM requires the method blank to be < ½ RL.

18.3.7 LCS: Determined yearly by the Statistical Process Control (SPC) system of AlphaLIMS. The limits are given in spreadsheet form to each analyst. If the % Recovery falls outside these limits, the batch must be reanalyzed. For Method 300.0 the LCS acceptance limits are set at 90-110% for all liquid matrix batches.

18.3.8 LCS RPD: Determined yearly by the Statistical Process Control (SPC) system of AlphaLIMS. If the RPD falls outside these limits, the batch must be reanalyzed.

19.0 DATA REVIEW, VALIDATION, AND APPROVAL PROCEDURE

19.1 Before submitting results for data entry into AlphaLIMS, check the following items:

19.1.1 The run is dated and initialed.

19.1.2 All cross-outs are dated and initialed.

19.1.3 Standard preparation is documented in the AlphaLIMS standards logbook and standard identification numbers are recorded with the run in AlphaLIMS.

19.1.4 All dilution factors are clearly marked.

19.1.5 Sample prep logbook in AlphaLIMS has been filled out completely (if applicable).

19.1.6 Correlation coefficient is recorded accurately and is $\geq$ 0.995.

19.1.7 All quality control samples such as blanks, duplicates, and spikes are clearly identified and fall within current acceptance limits.

19.1.8 The correct units are recorded for the results.

19.1.9 Each nonconformance has been noted in the Case Narrative.

19.1.10 All manual reprocessing of chromatograms must have a reason for the reprocess. The changes must be signed by the person doing the reprocessing and a qualified peer.

19.2 After ensuring that the above items are properly documented, print the batch report. (Data are automatically transferred to AlphaLIMS.)

19.3 Review data report to ensure that all data are in, then status the batch to "Review." This will trigger the qualifiers.

19.4 The batch sheet, data report, and case narrative are stapled together. This is the package that enters the main data review process.

19.5 The data review is done by the analyst who performed the analysis. The analyst completes the review, noting and correcting any discrepancies. When the analyst has a correct report of the data, the package is passed on for peer review.

19.6 When the data package associated with a given batch satisfactorily passes review, the batch status is updated to "DONE" in AlphaLIMS.

20.0 RECORDS MANAGEMENT

All data associated with the activity described in this procedure, including relevant logbooks, are maintained as quality records in accordance with GL-QS-E-008 for Quality Records Management and Disposition.

21.0 LABORATORY WASTE HANDLING AND DISPOSAL

For the proper disposal of sample and reagent wastes from this procedure, refer to the Laboratory Waste Management Plan, GL-LB-G-001.

22.0 REFERENCES

22.1 EPA Method 300.0, "The Determination of Inorganic Anions in Water by Ion Chromatography."

22.2 SW-846, Method 9056A, Rev.1, "Determination of Inorganic Anions by Ion Chromatography," February 2007.

22.3 Dionex Chromeleon software

22.4 Dionex ICS 3000 Ion Chromatography Instruction Manual

22.5 Dept. of Defense (DOD), Dept. of Energy (DOE) Consolidated Quality Systems Manual (QSM) Version 5.3, July 2019.

23.0 HISTORY

Revision 28: Added Appendix 3: 10% Acceptable Reporting Ranges for Anion Standards.
Revision 27: Update to include the use of Teflon chips or sand for solid batches and removed bomb prep standards (which are found in GL-GC-E-079).
Revision 26: Updated for new instrument. Update the reference section for current DoD QSM. Version 5.2, December 2018.
Revision 25: Updated for current concentration of standards and dilutions used. Corrected typos
Revision 24: Added clarification to section 6.0 and 13.1.7.

APPENDIX 1: ACCEPTANCE LIMITS

Acceptance Criteria	GEL Standard	DoD QSM
CCV/ICV	90% - 110%	90% - 110%
CCB/ICB	± RL	± RL
Method Blank	<CRDL	± ½ RL, reprep failures, B flag for continued failures
LCS - liquid LCS - soil	90% - 110%	90% - 110% Glass beads/Teflon chips/Sand for solids, flag failures with Q if reprep not possible.
Matrix Spikes/ Matrix Spike Duplicates	90% - 110%	90% - 110%, when applicable. Flag failures with J.
Sample Duplicates	0% </= 20% when greater than 5X RL, +/-RL when less than 5X RL	0% </= 10% Flag failures with J.
MS/MSD RPD	RPD 0% </= 20% between MS/MSD	RPD 0% </= 15% between MS/MSD, J flag failures.
Qualifiers	COA = U/J/B for samples or MB. * for LCS, MS, MSD, DUP.	U for non detects, J for values estimated between LOD and LOQ
MDL/MDLV	MDL and MDLV are performed bi-annually per the methods	LODV performed quarterly on all instruments used for DoD analysis.

APPENDIX 2: CALIBRATION STANDARDS FOR IC

(For Illustrative Purposes Only)

Autocal 6
20 mg/L SO_4
10 mg/L Cl
5 mg/L Fl, NO_2, NO_3, I
3 mg/L Br, PO_4

Autocal 5
4 mg/L SO_4
2 mg/L Cl
1 mg/L Fl, NO_2, NO_3
2.5 mg/L Br, PO_4, I

Autocal 4
2 mg/L SO_4, I
1 mg/L Cl, Br, PO_4
0.5 mg/L Fl, NO_2, NO_3

Autocal 3
1 mg/L SO_4, I
0.5 mg/L Cl, Br, PO_4
0.25 mg/L Fl, NO_2, NO_3

Autocal 2
0.5 mg/L I
0.4 mg/L SO_4
0.2 mg/L Cl, Br, PO_4
0.1 mg/L Fl, NO_2, NO_3

Autocal 1
DI water

APPENDIX 3: 10% ACCEPTABLE REPORTING REANGES FOR ANION STANDARDS (FOR ILLUSTRATIVE PURPOSES)

	10% RANGE FOR ANION STANDARDS						
ICV/LCS/CCV	Flouride	Chloride	Nitrite	Bromide	Nitrate	Ortho-phosphate	Sulfate
low value	2.250	4.500	2.250	1.125	2.250	1.125	9.000
nominal	2.500	5.000	2.500	1.250	2.500	1.250	10.000
high value	2.750	5.500	2.750	1.375	2.750	1.375	11.000
CVH	Flouride	Chloride	Nitrite	Bromide	Nitrate	Ortho-phosphate	Sulfate
low value	3.375	6.750	3.375	1.688	3.375	1.688	13.500
nominal	3.750	7.500	3.750	1.876	3.750	1.876	15.000
high value	4.125	8.250	4.125	2.064	4.125	2.064	16.500

VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE FOR TOTAL PHOSPHORUS BY THE LACHAT QUIKCHEM FIA + 8000 SERIES INSTRUMENT

(GL-GC-E-103 REVISION 11)

APPLICABLE TO METHOD:
EPA 365.4
Standard Methods 22nd Edition, 4500 P H-2011

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1.0 STANDARD OPERATING PROCEDURE FOR TOTAL PHOSPHORUS BY THE LACHAT QUIKCHEM FIA+ 8000 SERIES INSTRUMENT

2.0 METHOD CODE

EPA Method 365.4 (Colorimetric, Automated, Block Digester AAII) and Standard Methods 22nd Edition 4500 P H-2011

3.0 METHOD OBJECTIVE/PURPOSE

This Standard Operating Procedure (SOP) provides the necessary instructions to determine the concentration of total phosphorus (PO_4-Total) by flow injection analysis colorimetry using the Lachat QuikChem FIA+ 8000 Series instrument.

4.0 METHOD SUMMARY

4.1 Total phosphorus is first converted to orthophosphorus by hydrolysis with sulfuric acid, mercuric oxide, and potassium sulfate. The determination of orthophosphorus is then based on the colorimetric method in which a blue color is formed by the reaction of orthophosphate, ammonium molybdate, and antimony potassium tartrate followed by reduction with ascorbic acid at an acidic pH. This complex is then read at 880 nm.

4.2 Samples are digested prior to analysis using a block digestor.

5.0 APPLICABLE MATRICES

5.1 Groundwater

5.2 Drinking water

5.3 Domestic and industrial wastewater

NOTE: Clients may request that this analysis be performed on miscellaneous liquid or solid samples. In these cases, the procedure is modified as necessary.

6.0 HOLDING TIME

Holding time for non-South Carolina samples is 28 days from the time and date of collection until the start of analysis unless otherwise specified by contract. Samples reported for compliance in South Carolina must be analyzed within 24 hours of sample preparation.

7.0 SAMPLE CONTAINER/COLLECTION/PRESERVATION/STORAGE REQUIREMENTS

7.1 Samples may be collected in plastic or glass containers.

7.2 Samples are preserved at time of collection with sulfuric acid to a pH less than 2.

7.3 Samples are stored at $0 \leq 6$ °C until analyzed.

8.0 INTERFERENCES

8.1 High concentrations of iron may cause the phosphorus in samples to precipitate out.

8.2 If samples contain arsenate in concentrations higher than phosphorus, the arsenate could serve as a positive interference.

8.3 Silica forms a pale blue complex that also absorbs at 880 nm.

8.4 Sample color that absorbs in the wavelength range used for analysis will interfere.

9.0 PERFORMANCE CHARACTERISTICS

9.1 Method concentration range: 0.01 to 20 mg/L

9.2 Calibration range: 0.05 to 3.0 mg/L

9.3 Method detection limit (MDL): Refer to current MDL studies.

9.4 Method precision: Refer to current Statistical Process Control (SPC) limits.

9.5 Method accuracy: Refer to current SPC limits.

10.0 DEFINITIONS

10.1 AlphaLIMS: The Laboratory Information Management System used at GEL Laboratories, LLC.

10.2 Calibration Standard (CAL): A solution prepared from the primary dilution standard solution or stock standard solutions and the internal standards and surrogate analytes. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

10.3 Continuing Calibration Blank (CCB): An aliquot of reagent water or other blank matrix that is analyzed after each CCV. The CCB is used to determine whether the analytical sequence is in control during sample analysis.

10.4 Continuing Calibration Verification (CCV) Standard: An aliquot of reagent water or other blank matrix to which known quantities of the method analytes are added in the laboratory. The CCV is analyzed exactly like a sample, periodically throughout the run sequence. Its purpose is to determine whether the analytical sequence is in control during sample analysis. It may be prepared from the same source as the calibration standards, and is usually of varied concentration.

10.5 Independent Calibration Blank (ICB): An aliquot of reagent water or other blank matrix that is analyzed after each ICV. The ICB is used to determine whether there is carryover contamination after injection of the mid-level ICV.

10.6 Independent Calibration Verification (ICV): A solution of method analytes of known concentrations that is used to fortify an aliquot of blank or sample matrix. The ICV is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check laboratory performance with externally prepared test materials.

10.7 Laboratory Control Standard (LCS): An aliquot of reagent water or other blank matrix to which known quantities of the method analytes are added in the laboratory. The LCS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

10.8 Laboratory Duplicate (DUP, LCSD, MSD or PSD): Aliquots of a sample taken from the same container and processed in the same manner under identical laboratory conditions. The aliquot is analyzed independently from the parent sample and the results are compared to measure precision and accuracy.

10.9 Linear Calibration Range (LCR): The concentration range over which the instrument response is linear.

10.10 Method Blank (MB): An aliquot of reagent water or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The MB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

10.11 Method Detection Limit (MDL): The minimum concentration of an analyte that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero.

10.12 Spike (Matrix Spike or Post Spike): An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS or PS is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS or PS corrected for background concentrations.

10.13 Statistical Process Control (SPC) Limits: Statistically derived limits that establish acceptable ranges for recoveries of analytes of interest, including LCS, MS, MSD, PS, PSD and internal standards.

10.14 Stock Standard Solution: A concentrated solution containing one or more method analytes prepared in the laboratory using certified reference materials or purchased from a reputable commercial source.

10.15 Total Phosphorus: Refers to the concentration of all phosphorus present in the sample, regardless of form. This includes phosphorus in orthophosphate, $[(PO_4)^{3-}]$, polyphosphate $[(P_2O_7)^{4-}]$, and organic phosphorus forms.

10.16 Refer to GL-QS-B-001 the Quality Assurance Plan for additional lab-wide used definitions.

11.0 ANALYST VERIFICATION

Before a technician/analyst is allowed to analyze samples without supervision, he/she is trained by qualified personnel and will be required to successfully analyze a proficiency sample. Training records are maintained as quality records.

12.0 DOCUMENTATION OF DATA

12.1 As data are acquired, computer printouts of the data are generated. These dated hard copies of the data are kept in the PO_4-Total binder in the Lachat lab. When the analysis is complete, the results are submitted for data entry into AlphaLIMS.

12.2 Documentation of standards preparation is maintained in AlphaLIMS.

12.3 Prep data are entered in AlphaLIMS.

13.0 SAFETY PRECAUTIONS AND HAZARD WARNINGS

WARNING

SULFURIC ACID IS AN EXTREME CORROSIVE.

PREVENT SKIN AND EYE CONTACT BY USING SPECIFIED PERSONAL PROTECTIVE EQUIPMENT WHEN MAKING STOCK REAGENTS.

WORK UNDER A HOOD TO PREVENT INHALATION WHEN MAKING STOCK REAGENTS.

WARNING

ANTIMONY POTASSSIUM TARTRATE IS TOXIC IF INGESTED AND HARMFUL IF INHALED.

PREVENT SKIN AND EYE CONTACT BY USING SPECIFIED PERSONAL PROTECTIVE EQUIPMENT.

WORK UNDER A HOOD TO AVOID INHALATION.

13.1 Wear safety glasses while in the laboratory.

13.2 Treat all chemicals and samples as potential health hazards, and limit exposure to these chemicals to the lowest level possible. GEL maintains a current awareness file of OSHA regulations regarding the safe handling of the chemicals in the laboratory as well as a reference file of Material Safety Data Sheets (MSDS). Individual sample MSDS forms provided by the clients are also maintained.

13.3 Personal protective equipment

13.3.1 Gloves are required when making reagents in this procedure. The gloves approved for this procedure are nitrile gloves for sulfuric acid and antimony potassium tartrate.

13.3.2 Work under a hood when using the concentrated acids in this procedure.

13.3.3 A protective apron over a lab coat is required when working with sulfuric acid.

13.4 Prior to handling radioactive samples, analysts must have had radiation safety training and must understand their full responsibilities in radioactive sample handling. Some general guidelines follow:

13.4.1 Protect counter tops with counter paper or work from radioactive sample handling trays.

13.4.2 Post signs indicating radioactive samples are in the area.

13.4.3 Take swipes of the counter tops upon completion of work. Deliver those swipes to the designated swipe count box.

13.4.4 Segregate radioactive wastes. Radioactive waste containers are obtained from Waste Management.

13.5 All samples, chemicals, extracts, and extraction residues must be transferred, delivered, and disposed of safely according to all related SOPs.

13.5.1 Segregate solid wastes from liquid wastes in the satellite area containers.

13.5.2 Segregate oil wastes from water-soluble wastes in the satellite area containers.

13.6 Never leave gas cylinders unchained or untied, including when they are on the moving carts.

14.0 SAMPLE RECEIPT FOR ANALYSIS

14.1 The analyst/technician gives the list of samples needed to the sample custodian. The sample custodian removes the appropriate samples from the cooler and either delivers them to the analyst/technician or places them on the pickup shelf in the main cooler.

14.2 Analysts and technicians are responsible for retrieving their own samples when the sample custodian is not available.

14.3 Previously digested samples are stored in the General Chemistry refrigerator until they are retrieved by the analyst, just prior to analysis.

15.0 INSTRUMENTATION/EQUIPMENT/GLASSWARE

15.1 Instrumentation: Lachat QuikChem FIA+ 8000 Series analyzer.

15.1.1 Autosampler

15.1.2 Multichannel proportioning pump
15.1.3 Reaction unit or manifold
15.1.4 Colorimetric detector
15.1.5 Data system

15.2 Equipment
15.2.1 Air Displacement Pipets
15.2.1.1 10-100 µL with tips
15.2.1.2 100-1000 µL with tips
15.2.1.3 1.00-5.00 mL with tips

NOTE: Pipets are calibrated in accordance with GL-LB-E-012.

15.2.2 Carboy for disposal of sample reagents and residue.
15.2.3 Helium degassing tube

16.0 REAGENTS

NOTE: Unless otherwise specified, all chemicals should be of ACS grade or equivalent.

16.1 Helium gas
16.2 Red Mercuric Oxide (HgO)
16.3 Potassium Sulfate (K_2SO_4)
16.4 Ammonium Molybdate Tetrahydrate [$(NH_4)_6Mo_7O_{24}•4H_2O$]
16.5 Antimony Potassium Tartrate (potassium antimonyl tartrate trihydrate $C_8H_4O_{12}K_2Sb_2•3H_2O$)
16.6 Ascorbic Acid ($C_6H_8O_6$)
16.7 Sulfuric Acid, concentrated (H_2SO_4)
16.8 Sodium Dodecyl Sulfate (SDS), [$CH_3(CH_2)_{11}OSO_3Na$]
16.9 Sodium Chloride (NaCl)
16.10 Sodium Hydroxide (NaOH)
16.11 Type I water: Deionized (DI) water (See GL-LB-E-016)
16.12 1000 mg/L Total Phosphorus standard (2 separate independent sources)
16.13 Reagent Preparation

16.13.1 Stock Mercuric Sulfate Solution

To a 50 mL volumetric flask, add 20 mL of DI Water, 5 mL of concentrated sulfuric acid, and 4 g of red mercuric oxide. Stir with a magnetic stirrer at low heat until dissolved, dilute to the mark, and invert to mix. Use solution immediately after preparation.

16.13.2 Digestion Solution

In a 1 L flask, add approximately 700 mL of DI Water, then add 200 mL of concentrated sulfuric acid. Add 133 g of potassium sulfate. Add 25 mL of stock mercuric sulfate solution and dilute to the mark. Mix with a magnetic stirrer and allow the solution to cool. Dilute to the mark after the solution has cooled. This reagent can be stored at room temperature for one month.

16.13.3 Diluent / Carrier

In a 1 L volumetric flask containing approximately 600 mL of DI water, add 200 mL of the digestion solution, dilute to the mark and invert to mix. This reagent can be stored at room temperature and must be prepared fresh weekly.

16.13.4 Stock Ammonium Molybdate Solution

In a 1 L volumetric flask, dissolve 40.0 g of ammonium molybdate tetrahydrate in approximately 800 mL of DI water. Dilute to the mark with DI water and mix with a magnetic stirrer for at least four hours. This reagent must be refrigerated in a plastic container and is good for six months.

16.13.5 Stock Antimony Potassium Tartrate Solution

In a 1 L volumetric flask, dissolve 3.22 g of antimony potassium tartrate in approximately 800 mL of DI water. Dilute to the mark with DI water and mix with a magnetic stirrer until dissolved. This reagent can be stored for up to two months in a dark bottle and refrigerated.

16.13.6 Molybdate Color Reagent

To a 1 L volumetric flask, add about 500 mL of DI water and then add 213 mL of ammonium molybdate solution and 72 mL of antimony potassium tartrate solution. Dilute to the mark with DI water and invert to mix. Degas this reagent with helium. This reagent may be stored at room temperature for up to six months.

16.13.7 Ascorbic Acid Reducing Solution

In a 1 L volumetric flask, dissolve 60.0 g of ascorbic acid in about 700 mL of DI water. Add 1.0 g of sodium dodecyl sulfate. Dilute to the mark with DI water and mix with a magnetic stirrer. Degas this reagent with helium. This reagent may be stored at room temperature and must be prepared fresh every two days.

16.13.8 Sodium Chloride/Sodium Hydroxide Solution

In a 1 L volumetric flask, dissolve 160 g of sodium chloride and 20 g of sodium hydroxide in about 600 mL of DI water. Dilute to the mark with DI water and mix with a magnetic stirrer. Degas this reagent with helium. This reagent may be stored at room temperature and must be prepared fresh weekly.

17.0 PREPARATION OF SAMPLES

Refer to GL-GC-E-071 for Total Phosphorus and Total Kjeldahl Nitrogen Sample Preparation.

18.0 PREPARATION OF STANDARDS

18.1 Documentation of standards and their preparation is maintained in AlphaLIMS in accordance with GL-LB-E-007 for Laboratory Standards Documentation.

18.2 All standards are prepared from traceable solutions according to the protocol found in Appendix I.

18.3 All total phosphorous standards and blanks are prepared exactly as samples and are digested.

18.4 All total phosphorous standards are prepared fresh daily.

19.0 INSTRUMENT/EQUIPMENT START-UP PROCEDURE

19.1 Set up the Phosphorus Manifold as shown in Appendix I.

19.2 Refer to the Lachat QuikChem Method 10-115-01-1-C: Determination of Total Phosphorus by Flow Injection Analysis Colorimetry for instrument start-up procedures.

19.3 Refer to the Lachat QuikChem FIA+ 8000 Series User Manual for instrument start-up procedures.

20.0 QUALITY CONTROL (QC) REQUIREMENTS

20.1 Instrument QC

20.1.1 An Initial Calibration Verification (ICV) is run immediately after the calibration curve. This standard must be made from a different source than the calibration standards.

20.1.2 An Initial Calibration Blank (ICB) is run following the ICV.

20.1.3 A Continuing Calibration Verification (CCV) is run after every 10 analytical samples and after the last analytical sample is run.

20.1.4 A Continuing Calibration Blank (CCB) is run after every CCV.

20.1.5 MDL and/or MDL verifications are performed in accordance with GL-LB-E-001 for the determination of method detection limits and method quantitation limits.

20.1.6 Linear calibration range (LCR) checks are performed on a 6-month basis. The high standard of the curve is read back against the calibration and must recover ± 10%. If this procedure fails, the problem must be identified and rectified.

20.1.7 Calculated curve readbacks are: 50%-150% for the first point (low-end) and 90%-110% for all the other points. The calculated zero point must be ± ½ RL. Any failure will require re-calibration of the instrument.

20.2 Batch QC

20.2.1 A matrix spike and matrix duplicate are run for every 10 samples in a batch.

20.2.2 A MB and an LCS are run at least once for every batch of 20 or less samples.

20.2.3 For liquid samples, the LCS is normally a 1 mg/L standard taken through the same process as the samples.

20.3 Acceptance limits

20.3.1 Correlation coefficient (r) must be 0.995 or greater

20.3.2 ICV recovery must be 90-110% for all batches. If the ICV fails, the calibration curve must be reanalyzed. If the ICV fails again, new standards must be made and a new calibration conducted.

20.3.3 CCV recovery must be 90-110% for all batches. If the CCV fails, all samples bracketed by the out-of-control CCV must be reanalyzed.

20.3.4 Matrix Relevant Percent Difference (RPD): Refer to current SPC limits.

20.3.5 Matrix spike recovery: Refer to current SPC limits.

20.3.6 Method Blank: - PQL < MB < PQL

NOTE: Some clients may require MB ≤ ½ PQL.

20.3.7 LCS: Refer to current SPC limits. If the percent recovery falls outside these limits, the batch must be reanalyzed.

20.3.8 LCS RPD: Refer to current SPC limits.

20.3.9 Due to the scope of the test and method, peaks are verified visually. These peaks should have a steady rise and fall. Any peak that is sharp with jagged edges must be re-analyzed for verification.

20.4 Handling out-of-control situations:

20.4.1 If a sample result exceeds the highest calibration standard, the sample must be diluted and reanalyzed.

20.4.2 The correlation coefficient must be a value of at least 0.995. If it is less than 0.995, reanalyze the calibration standards. If this does not bring the correlation coefficient to a value greater than 0.995, all calibration standards must be re-made and a new calibration conducted.

NOTE: Refer to GL-QS-E-014 for Quality Assurance Measurement Calculations and Processes. The intercept should be evaluated as stated in Section 9.2. The absolute value of the intercept should be less than 3 times the MDL.

NOTE: If the batch must be redigested, notify the Group Leader.

21.0 TYPICAL RUN SEQUENCE

21.1 Calibration Curve (analyzed from high standard to blank)
21.2 ICV (Initial Calibration Verification standard)
21.3 ICB (Initial Calibration Blank)
21.4 Method Blank
21.5 LCS
21.6 Sample
21.7 Sample duplicate (if required)
21.8 Matrix spike of the sample (if required)
21.9 Up to 5 more samples
21.10 CCV (Continuing Calibration Verification standard)
21.11 CCB (Continuing Calibration Blank)
21.12 Sample
21.13 Sample duplicate (if required)
21.14 Matrix spike of the sample (if required)
21.15 Up to 7 more samples
21.16 CCV

21.17 CCB
21.18 Remaining samples (a maximum of 20 samples is allowed per batch)
21.19 CCV
21.20 CCB

22.0 PROCEDURE

22.1 Create Sample Table:
22.1.1 Click on 'Open' and choose file to open.
22.1.2 Fill in the Sample table according to the Run Sequence 21.0 and change dates on calibration standards sample names.
22.1.3 Enter the sample ID and dilution factor.
22.1.4 Check to ensure the cup location is incremented correctly.
22.1.5 Load samples onto the autosampler as listed on the printed sample table.
22.2 Check to make sure all reagents and lines are pumping correctly.
22.3 Click on 'Start'.
22.4 Refer to the Lachat QuikChem Method 10-115-01-1-C: Determination of Total Phosphorous by Flow Injection Analysis Colorimetry.
22.5 Refer to the Lachat QuikChem FIA + 8000 Series User Manual.

23.0 INSTRUMENT/EQUIPMENT SHUT-DOWN PROCEDURE

23.1 Place the reagent lines in DI water and allow DI water to pump through the system for 5 minutes.
23.2 Remove the reagent tubes from the DI water and allow air to pump through the system for 5 minutes, or until water no longer appears in the lines.
23.3 Turn off power, and release platen tension.
23.4 Refer to the Lachat QuikChem FIA + 8000 Series User Manual for instrument shutdown procedure(s).

24.0 DATA REVIEW, VALIDATION, AND APPROVAL PROCEDURE

24.1 Refer to GL-GC-E-092 for General Chemistry Data Review Packaging and GL-LB-E-005 for Data Review and Validation.
24.2 Data Reduction
24.2.1 Linear regression is used for calibration. The analyst must not force the calibration line through the origin.
24.2.1.1 Make certain that the instrument response is treated as the dependent variable (y) and the concentration as the independent variable (x). The regression will produce the slope and intercept terms for a linear equation in the form:

$$y = ax + b$$

Where:
y = instrument response
a = slope of line (also called the "coefficient of x")
x = concentration of the calibration standard

b = the intercept

24.2.1.2 The analyst should not force the line through the origin, but have the intercept calculated from the standard data points.

24.2.1.3 The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be ≥ 0.995. The calculated intercept value needs to be evaluated before reporting sample results.

24.2.1.4 In calculating the sample concentrations, the regression equation is rearranged to solve for the concentration (x) as shown below:

$$x = \frac{(y - b)}{a}$$

24.2.1.5 The curve must meet all criteria set forth in section 20.0.

25.0 DATA TRANSMITTAL

Data are entered in AlphaLIMS as they are obtained.

26.0 RECORDS MANAGEMENT

All data associated with the performance of this procedure, including relevant logbooks, are maintained as quality records in accordance with GL-QS-E-008 for Quality Records Management and Disposition.

27.0 ROUTINE INSTRUMENT/EQUIPMENT MAINTENANCE

27.1 Refer to the Lachat QuikChem FIA+ 8000 Series User Manual for routine instrument maintenance.

27.2 The Lachat QuikChem FIA+ 8000 Series instrument maintenance logbook is maintained in the Lachat lab.

28.0 LABORATORY WASTE HANDLING AND DISPOSAL

For the proper disposal of sample and reagent wastes from this procedure, refer to the Laboratory Waste Management Plan, GL-LB-G-001.

29.0 METHOD VERIFICATION

Method detection limit studies are performed in accordance with GL-LB-E-001.

30.0 REFERENCES

30.1 "Phosphorous, Total," EPA Method 365.4 (Colorimetric, Automated, Block Digester AA II) Storet No. 00665. Issued 1996.

30.2 Lachat QuikChem Method 10-115-01-1-C: Determination of Total Phosphorus by Flow Injection Analysis Colorimetry. Lachat Instruments 6645 West Mill Rd. Milwaukee, WI 53218.

30.3 Lachat QuikChem FIA+ 8000 Series User Manual. Lachat Instruments 6645 West Mill Rd. Milwaukee, WI 53218.

30.4 Standard Methods for the Examination of Water and Wastewater, 22nd Edition, 4500 P H-2011, Manual Digestion and Flow Injection Analysis for Total Phosphorus.

31.0 HISTORY

Revision 8: Updates made to Appendix I.
Revision 9: Updated DI water type in accordance with GL-LB-E-016, updated Standard Methods reference per MUR II.
Revision 10: Revised reference for Std Methods reference method.
Revision 11: Added readback requirements for calibration curve.

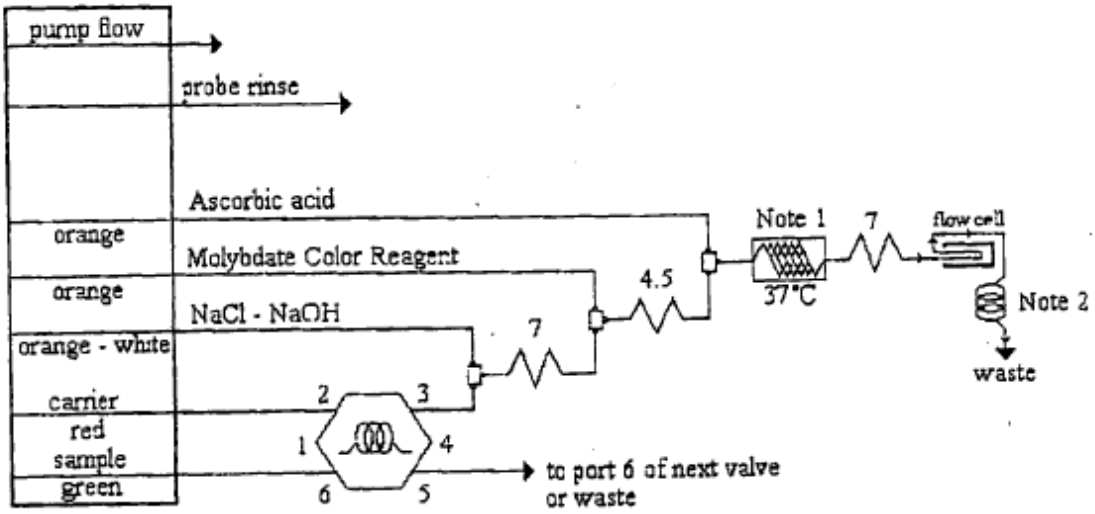
APPENDIX I

Conc of STD	Vol of Stock	Final Vol of Std
3 ppm	0.6 mL of 100 ppm std	20 mL 0.04 N H_2SO_4
1 ppm	0.2 mL of 100 ppm std	20 mL 0.04 N H_2SO_4
0.5 ppm	0.1 mL of 100 ppm std	20 mL 0.04 N H_2SO_4
0.1 ppm	0.02 mL of 100 ppm std	20 mL 0.04 N H_2SO_4
0.05 ppm	0.01 mL of 100 ppm std	20 mL 0.04 N H_2SO_4

Total phosphorous

APPENDIX II

PHOSPHORUS MANIFOLD DIAGRAM



Carrier: TKN/PO_4 Diluent/Carrier
Manifold Tubing: 0.8 mm (0.032 in) i.d. This is 5.2 µL/cm.
AE Sample Loop: 25 cm
QC8000 Sample Loop: 30.5 cm
Interference Filter: 880 nm

Apparatus: An injection valve, a 10 mm path length flow cell , and a colorimetric detector module is required.

4.5: 70 cm of tubing on a 4.5 cm coil support
7: 135 cm of tubing on a 7 cm coil support

Note 1: 175 cm of tubing on the heater.
Note 2: 200 cm restrictor coil, 0.52 mm (0.022 in.) i.d.

VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE

FOR

NITRATE/NITRITE (NO_3+NO_2) ANALYSIS USING THE LACHAT QUIKCHEM FIA+ 8000 SERIES INSTRUMENT

(GL-GC-E-128 REVISION 10)

APPLICABLE TO METHODS:

EPA 353.2
Standard Methods 22nd, 4500 NO_3 F-2011

PROPRIETARY INFORMATION

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1.0 STANDARD OPERATING PROCEDURE FOR NITRATE/NITRITE (NO_3+NO_2) ANALYSIS USING THE LACHAT QUIKCHEM FIA+ 8000 SERIES INSTRUMENT

2.0 METHOD CODE

2.1 EPA Method 353.2 (Determination of Nitrate+Nitrite Nitrogen by Semi-Automated Colorimetry)

2.2 Standard Methods 22nd Edition, 4500 NO_3^- F-2011

3.0 METHOD OBJECTIVE/PURPOSE

The purpose of this SOP is to provide the necessary instructions to determine the concentration of total nitrate+nitrite nitrogen in samples using the Lachat QuikChem FIA+ 8000 Series Instrument.

4.0 METHOD SUMMARY

Nitrate is quantitatively reduced to nitrite by passage of the sample through a copperized cadmium column. The nitrite (reduced nitrate plus original nitrite) is then determined by diazotizing with sulfanilamide followed by coupling with N-(1-naphthyl) ethylenediamine dihydrochloride. The resulting water soluble dye has a magenta color which is read at 520 nm. Nitrite alone also can be determined by removing or circumventing the cadmium column if holding time of 48 hours is observed.

5.0 APPLICABLE MATRICES

5.1 Groundwater

5.2 Drinking water

5.3 Domestic and industrial wastewater

NOTE: Clients may request that this analysis be performed on soils, sludge, miscellaneous liquid or solid samples. In these cases, the procedure is modified as necessary. Modification is not permitted for samples being reported to SC DHEC for regulatory monitoring or NPDES.

6.0 HOLDING TIME

Holding time is 28 days from the time and date of collection until the start of analysis unless otherwise specified by contract.

7.0 SAMPLE CONTAINER/PRESERVATION/COLLECTION/STORAGE REQUIREMENTS

7.1 Samples may be collected in plastic or glass containers.

7.2 Samples are preserved at time of collection with sulfuric acid to a pH less than 2.

7.3 Samples are stored at 0° $\leq$ 6° C until analyzed.

8.0 INTERFERENCES/LIMITATIONS

8.1 Residual chlorine can interfere by oxidizing the cadmium column.

8.2 Low results would be obtained for samples that contain high concentrations of iron, copper, or other metals. In this method, EDTA is added to the buffer to reduce this interference.

8.3 Samples that contain large concentrations of oil and grease will coat the surface of the cadmium. This interference is eliminated by pre-extracting the sample with an organic solvent.

8.4 Sample turbidity may interfere. Turbidity can be removed by filtration through a 0.45 µm pore diameter membrane filter prior to analysis.

9.0 PERFORMANCE CHARACTERISTICS

9.1 Method concentration range: 0.01 to 2.00 mg/L

9.2 Calibration range: 0.02 to 1.50 mg/L

9.3 Method detection limit (MDL): Refer to current MDL study.

9.4 Method precision: Refer to current SPC limits.

9.5 Method accuracy: Refer to current SPC limits.

10.0 DEFINITIONS

10.1 AlphaLIMS: The Laboratory Information Management System used at GEL Laboratories, LLC.

10.2 Calibration Standard (CAL): A solution prepared from the primary dilution standard solution or stock standard solutions and the internal standards and surrogate analytes. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

10.3 Continuing Calibration Blank (CCB): An aliquot of reagent water or other blank matrix that is analyzed after each CCV. The CCB is used to determine whether the analytical sequence is in control during sample analysis.

10.4 Continuing Calibration Verification (CCV) Standard: An aliquot of reagent water or other blank matrix to which known quantities of the method analytes are added in the laboratory. The CCV is analyzed exactly like a sample, periodically throughout the run sequence. Its purpose is to determine whether the analytical sequence is in control during sample analysis. It may be prepared from the same source as the calibration standards, and is usually of varied concentration.

10.5 Independent Calibration Blank (ICB): An aliquot of reagent water or other blank matrix that is analyzed after each ICV. The ICB is used to determine whether there is carryover contamination after injection of the mid-level ICV.

10.6 Independent Calibration Verification (ICV): A solution of method analytes of known concentrations that is used to fortify an aliquot of Blank or sample matrix. The ICV is obtained from a source external to the

laboratory and different from the source of calibration standards. It is used to check laboratory performance with externally prepared test materials.

10.7 Instrument Performance Check Solution (IPC): A solution of one or more method analytes, surrogates, internal standards, or other test substances used to evaluate the performance of the instrument system with respect to a defined set of criteria.

10.8 Laboratory Control Standard (LCS): An aliquot of reagent water or other blank matrix to which known quantities of the method analytes are added in the laboratory. The LCS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

10.9 Laboratory Duplicate (DUP, LCSD, MSD or PSD): Aliquots of a sample taken from the same container and processed in the same manner under identical laboratory conditions. The aliquot is analyzed independently from the parent sample and the results are compared to measure precision and accuracy.

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10.11 Method Blank (MB): An aliquot of reagent water or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The MB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

10.12 Method Detection Limit (MDL): The minimum concentration of an analyte that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero.

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10.14 Statistical Process Control (SPC) Limits: Statistically derived limits that establish acceptable ranges for recoveries of analytes of interest, including LCS, MS, MSD, PS, PSD and internal standards.

10.15 Stock Standard Solution: A concentrated solution containing one or more method analytes prepared in the laboratory using certified reference materials or purchased from a reputable commercial source.

10.16 Refer to GL-QS-B-001 the Quality Assurance Plan for additional lab-wide used definitions.

11.0 ANALYST VERIFICATION

Technicians and analysts are not allowed to analyze samples without supervision until trained by qualified personnel and upon successful analysis of a proficiency sample. Training records are maintained as quality records.

12.0 DOCUMENTATION OF DATA

12.1 As data are acquired, computer printouts of the data are generated. These dated hardcopies of the data are kept in the NO_3+NO_2 binder in the Lachat lab. Results are uploaded into AlphaLIMS as data are obtained.

12.2 Documentation of standards preparation is maintained in AlphaLIMS.

12.3 Prep data are entered in AlphaLIMS.

13.0 SAFETY PRECAUTIONS AND HAZARD WARNINGS

WARNINGS

HYDROCHLORIC ACID, PHOSPHORIC ACID, AND SODIUM HYDROXIDE ARE HIGHLY CORROSIVE.

POTASSIUM NITRITE IS TOXIC AND A POSSIBLE MUTAGEN.

N-(1-NAPHTHYL) ETHYLENEDIAMINE DIHYDROCHLORIDE IS IRRITATING TO MUCOUS MEMBRANES AND UPPER RESPIRATORY TRACT.

COPPER SULFATE MAY BE TOXIC UPON INGESTION. DEATH COULD OCCUR FROM SHOCK OR RENAL FAILURE.

WORK UNDER A HOOD TO PREVENT INHALATION WHEN MAKING STOCK REAGENTS.

PREVENT SKIN AND EYE CONTACT BY USING SPECIFIED PERSONAL PROTECTIVE EQUIPMENT WHEN MAKING STOCK REAGENTS.

PREVENT INHALATION AND SKIN AND EYE CONTACT BY USING SPECIFIED PERSONAL PROTECTIVE EQUIPMENT.

13.1 Wear eye protection with side shields while in the laboratory.

13.2 Treat all chemicals and samples as potential health hazards and limit exposure to these chemicals to the lowest level possible. GEL maintains a current awareness file of OSHA regulations regarding the safe handling of the chemicals in the laboratory as well as a reference file of Material Safety Data Sheets (MSDS). These documents and individual sample MSDS forms are maintained in the laboratory and on AlphaLIMS.

13.3 Personal protective equipment: Gloves are required when handling the chemicals in this procedure.

13.4 Prior to handling radioactive samples, analysts must have had radiation safety training and understand their full responsibilities in radioactive sample handling. Some general guidelines follow:

13.4.1 Wear a plastic apron over lab coat when working with radioactive samples.

13.4.2 Protect counter tops with counter paper or work from radioactive sample handling trays.

13.4.3 Prohibit admittance to immediate work area.

13.4.4 Post signs indicating radioactive samples are in the area.

13.4.5 Take swipes of the counter tops upon completion of work. Deliver those swipes to the designated swipe count box.

13.4.6 Segregate radioactive wastes. Radioactive waste containers are obtained from Waste Management.

13.5 All samples, chemicals, extracts, and extraction residues must be transferred, delivered, and disposed of safely according to all related SOPs.

13.5.1 Segregate solid wastes from liquid wastes in the satellite area containers.

13.5.2 Segregate oil wastes from water-soluble wastes in the satellite area containers.

14.0 SAMPLE RECEIPT FOR ANALYSIS

14.1 The analyst/technician gives the list of samples needed to the sample custodian. The sample custodian removes the appropriate samples from the cooler and either delivers them to the analyst/technician or places them on the pickup shelf in the main cooler.

14.2 Analysts and technicians are responsible for retrieving their own samples when the sample custodian is not available.

15.0 INSTRUMENT/EQUIPMENT/GLASSWARE

15.1 Instrumentation: Lachat QuikChem FIA+ 8000 Series

15.1.1 Autosampler

15.1.2 Multichannel proportioning pump

15.1.3 Reaction Unit or Manifold

15.1.4 Colorimetric detector

15.1.5 Data System

15.2 Equipment

15.2.1 Air Displacement Pipets

15.2.1.1 10-100 µL with tips

15.2.1.2 100-1000 µL with tips

15.2.1.3 1.00-5.00 mL with tips

NOTE: Pipets must be calibrated in accordance with GL-LB-E-010.

15.2.2 Carboy for disposal of sample reagents and residue

15.2.3 Analytical balance capable of accurately weighing to the nearest

0.0001 g.

NOTE: The balance must be calibrated in accordance with the procedure outline in GL-LB-E-002 for Balances.

15.2.4 PVC pump tubes

15.2.5 Nitrite test strips

15.2.6 10 mL Luer-lock syringe

15.2.7 Acrodisk, 0.45 µm pore syringe filters

15.2.8 40 mL centrifuge tubes with lids

16.0 REAGENTS

NOTE: Unless otherwise specified, all chemicals should be of ACS grade or equivalent.

16.1 ASTM Type I deionized (DI) water (see GL-LB-E-016)

16.2 0.04 N Sulfuric Acid (0.04 N H_2SO_4): Carefully add 22.4 mL of concentrated sulfuric acid to 20 L of DI water.

16.3 Dilute Hydrochloric Acid (HCl): Carefully add 500 mL concentrated HCl to 500 mL DI water.

16.4 Sodium Hydroxide (NaOH)

16.5 Ammonium Chloride (NH_4Cl)

16.6 Disodium Ethylenediamine Tetraacetic acid dihydrate ($Na_2EDTA•2H_2O$)

16.7 85% Phosphoric Acid (H_3PO_4)

16.8 Sulfanilamide ($C_6H_8N_2O_2S$)

16.9 N-(1-naphthyl) ethylenediamine dihydrochloride (NED)

16.10 Copper Sulfate ($CuSO_4•5H_2O$)

NOTE: All reagents are good for six months unless otherwise noted.

16.11 15 N Sodium Hydroxide

CAUTION! This solution will get very hot!

Add 150 g of NaOH very slowly to 250 mL of DI water. Swirl until dissolved. Cool and store in a plastic bottle.

16.12 Ammonium Chloride buffer, pH 8.5

In a 1 L volumetric flask, dissolve 85.0 g of NH_4Cl and 1.0 g of $Na_2EDTA•2H_2O$ in about 800 mL of DI water. Adjust the pH to 8.5 with 15 N NaOH solution. Dilute to the mark and mix.

16.13 Sulfanilamide color reagent

To a 1 L volumetric flask, add about 600 mL of DI water. Then add 100 mL of 85% phosphoric acid, 40.0 g of sulfanilamide, and 1.0 g of NED. Shake to wet and stir for 30 minutes to dissolve. Bring to volume. Store in a dark bottle. This solution is stable for one month.

16.14 Granulated cadmium: 40-60 mesh (CASRN 7440-43-9). Other mesh sizes may be used.

16.15 2% Copper Sulfate Solution

Dissolve 20.0 g of $CuSO_4 \cdot 5H_2O$ in 500 mL of DI water and dilute to 1 L.

16.16 Copper-cadmium: The cadmium granules (new or used) are cleaned with dilute HCl and copperized with 2% solution of copper sulfate in the following manner (also known as re-generating the cadmium column):

16.16.1 Wash the cadmium with dilute hydrochloric acid and rinse with distilled water. The color of the treated cadmium should be silver.

16.16.2 Swirl 10 g cadmium in 100 mL portions of 2% solution of copper sulfate for five minutes or until blue color partially fades, decant and repeat with fresh copper sulfate until a brown colloidal precipitate forms.

16.16.3 Wash the copper-cadmium with reagent water (at least 10 times) to remove all the precipitated copper. The color of the treated cadmium should be black.

NOTE: Typically, the cadmium columns are purchased new and are not regenerated.

16.17 100 mg/L Stock Standard (NO_3+NO_2), certified standard purchased from vendor (2 independent sources are required.)

16.18 100 mg/L Nitrate Standard (Nitrate as N), certified standard purchased from vendor. May require refrigerated storage, per the manufacturer.

16.19 100 mg/L Nitrite Standard (Nitrite as N), certified standard purchased from vendor. This standard material must be stored under refrigeration.

17.0 PREPARATION OF SAMPLES

17.1 All samples are tested for approximate nitrate concentration with test strips.

17.2 DI water extraction for solid samples:

17.2.1 Weigh out 4.0 g or other recorded weight of sample into a 40 mL centrifuge tube. Be careful to get a portion homogeneous to the entire sample.

17.2.2 Fill centrifuge tube to 40 mL with 0.04 N sulfuric acid.

17.2.3 Spike MS/MSD with 0.4 mL stock standard.

17.2.4 Cap the tube and shake several times every few minutes for approximately 30 minutes.

17.2.5 Let sample sit and allow all solid matter to settle out.

17.2.6 Decant the liquid off the top for analysis. The liquid may need to be filtered through an Acrodisk or similar disposable filter to remove all solid particles.

17.2.7 The initial sample weight and the volume of 0.04 N sulfuric acid used to extract is recorded in AlphaLIMS.

17.3 No preparation is required for water matrix samples.

18.0 PREPARATION OF STANDARDS

Documentation of standards and their preparation is maintained in AlphaLIMS in accordance with GL-GC-E-007 for Laboratory Standards Documentation. Each standard is prepared fresh daily.

18.1 100 mg/L primary stock NO_3+NO_2 standard purchased commercially

18.2 100 mg/L secondary stock NO_3+NO_2 standard purchased commercially

Dilute 1.0 mL of the 100 mg/L secondary stock NO_3+NO_2 standard in DI water to a volume of 100 mL using a 100 mL volumetric flask.

18.3 100 mg/L NO_3 stock standard purchased commercially

18.4 100 mg/L NO_2 stock standard purchased commercially

18.5 1.0 mg/L NO_3 standard

Dilute 1.0 mL of the 100 mg/L NO_3 stock standard in DI water to a volume of 100 mL using a 100 mL volumetric flask.

18.6 1.0 mg/L NO_2 standard

Dilute 1.0 mL of the 100 mg/L NO_2 stock standard in DI water to a volume of 100 mL using a 100 mL volumetric flask

18.7 Calibration curve standards

18.7.1 1.5 mg/L working standard by diluting 1.5 mL of the 100 mg/L primary stock NO_3+NO_2 standard in DI water to a volume of 100 mL using a 100 mL volumetric flask.

18.7.2 1.0 mg/L working standard by diluting 1.0 mL of the 100 mg/L primary stock NO_3+NO_2 standard in DI water to a volume of 100 mL using a 100 mL volumetric flask. This standard also serves as the CCV.

18.7.3 0.5 mg/L working standard by diluting 0.5 mL of the 100 mg/L primary stock NO_3+NO_2 standard in DI water to a volume of 100 mL using a 100 mL volumetric flask.

18.7.4 0.1 mg/L working standard by diluting 0.1 mL of the 100 mg/L primary stock NO_3+NO_2 standard in DI water to a volume of 100 mL using a 100 mL volumetric flask.

18.7.5 0.05 mg/L working standard by diluting 0.05 mL of the 100 mg/L primary stock NO_3+NO_2 standard in DI water to a volume of 100 mL using a 100 mL volumetric flask.

18.7.6 0.02 mg/L working standard by diluting 0.02 mL of the 100 mg/L primary stock NO_3+NO_2 standard in DI water to a volume of 100 mL using a 100 mL volumetric flask. This standard is used only

for SC samples.

19.0 INSTRUMENT/EQUIPMENT START-UP PROCEDURE

19.1 Set up the Manifold as shown in Appendix 1.

19.2 DI water should never be run through the cadmium column. Turn switch to off to rinse lines.

19.3 Refer to the Lachat QuikChem Method 10-107-04-1-C: Determination of Nitrate/Nitrite in Surface and Wastewaters by Flow Injection Analysis for instrument start-up procedures.

19.4 Refer to the Lachat QuikChem FIA+ 8000 Series User Manual for instrument start-up procedures.

20.0 QUALITY CONTROL (QC) REQUIREMENTS

20.1 Instrument QC

20.1.1 An initial calibration verification (ICV) is run immediately after the calibration curve. This standard must be made from a different source than the calibration standards. The concentration is 1.0 mg/L. If the ICV fails, the calibration curve must be reanalyzed See section 20.3.2. If the ICV fails again, new standards must be made and a new calibration conducted.

20.1.2 An initial calibration blank (ICB) is run following the ICV.

20.1.3 A 1.0 mg/L nitrate (NO_3) check standard is analyzed after the ICB to verify the reduction of nitrate by the cadmium column.

20.1.4 A 1.0 mg/L nitrite (NO_2) check standard is analyzed after the 1.0 mg/L nitrate (NO_3) standard.

20.1.5 A continuing calibration verification (CCV 1.0 mg/L) is run after every 10 analytical samples and after the last analytical sample in the run. If the CCV fails, all samples bracketed by the out of control CCV must be reanalyzed. See section 20.3.3.

20.1.6 A continuing calibration blank (CCB) is run after every CCV as prepared in 18.7.2.

20.1.7 MDL and/or MDL verifications are performed every 6 months. These values are submitted for Quality review.

20.1.8 Linear Calibration Range (LCR) checks are performed every 6 months. The high standard of the curve is read back against the calibration and must recover ± 10%. If this procedure fails, the problem must be identified and rectified.

20.1.9 Calculated curve readbacks are: 50%-150% for the first point (low-end) and 90%-110% for all the other points. The calculated zero point must be ± ½ RL. Any failure will require re-calibration of the instrument.

20.2 Batch QC

20.2.1 For liquid batches, a matrix duplicate (DUP) and post spike (PS) are run for every batch of ≤ 10 samples and for each set of ten samples in batches with > 10 samples.

20.2.2 A LCS and method blank (MB) are run at least once for every batch of 20 samples or less.

20.2.3 For liquid samples, the LCS is normally a 1 mg/L standard taken through the same process as the samples. For solid samples, 1.0 g of sand is weighed out and spiked with an appropriate amount of secondary stock standard.

20.2.4 The post spike is normally prepared by adding 0.05 mL of 100 mg/L stock standard solution to 5 mL of sample.

20.2.5 Linear Calibration Range (LCR) checks are performed on a 6 month basis. The high standard of the curve is read back against the calibration and must recover ± 10 %. If this procedure fails, the problem must be investigated and rectified.

20.2.6 For solid batches a DUP, MS, and MSD are run for every batch of ≤ 10 samples and for each set of 10 samples in batches with > 10 samples.

20.3 Acceptance limits

20.3.1 Coefficient of determination (r^2) must be 0.995 or greater.

20.3.2 ICV recovery must be 90-110% for all batches unless otherwise directed by project specifications.

20.3.3 CCV recovery must be 90-110% for all batches unless otherwise directed by project specifications.

20.3.4 Matrix relative percent difference (RPD): Refer to current SPC limits.

20.3.5 Matrix spike recovery: Refer to current SPC limits.

20.3.6 Method Blank: MB<PQL for most clients depending on client contract. If the MB fails, the analyst must reanalyze the MB with the entire batch.

20.3.7 LCS: Refer to current SPC limits. If the %Recovery falls outside these limits or other limits directed by project specifications, the batch must be reanalyzed.

20.3.8 LCS RPD: Refer to current SPC limits. If the RPD falls outside these limits or other limits directed by project specifications, the batch must be reanalyzed.

20.3.9 The 1.0 mg/L NO_3 standard and the 1.0 mg/L NO_2 standard must be within ± 10% of each other and of their true value.

20.4 Handling out-of-control situations:

20.4.1 If a sample result exceeds the highest calibration standard, the

sample must be diluted with the diluents reagent and reanalyzed.

NOTE: QC sets must be manually diluted.

20.4.2 The coefficient of determination must be a value of at least 0.995. If it is less than 0.995, reanalyze the calibration standards. If this does not bring the correlation coefficient to a value $\geq$ 0.995, then the reagents must be made up fresh and the calibrants reanalyzed. If these steps do not correct the problem, the calibration standards must be remade.

NOTE: Refer to GL-QS-E-014 for the Quality Assurance Measurement Calculations and Processes. The absolute value of the intercept should be less than 3 times the MDL.

20.4.3 If the 1.0 mg/L NO_3 standard does not recover within 10% of its true value or within 10% of the NO_2 standard, the cadmium column must be replaced with a new column.

20.4.4 Due to the scope of the test and method, peaks are verified visually. They should have a steady rise and fall. Any peak that is sharp in nature with jagged edges must be reanalyzed for verification.

20.4.5 Client samples that are less than the negative PQL are analyzed for verification. If the reanalysis is still more negative, a dilution is performed until the matrix suppression is less than the absolute value of the PQL.

21.0 RUN SEQUENCE

21.1 Calibration Standards (analyzed from high standard to blank)
21.2 ICV (Initial Calibration Verification standard)
21.3 ICB (Initial Calibration Blank)
21.4 1.0 mg/L NO_3 standard
21.5 1.0 mg/L NO_2 standard
21.6 Method blank
21.7 LCS
21.8 Sample
21.9 Sample duplicate (if required)
21.10 Post spike (if required)
21.11 Up to 7 samples
21.12 CCV
21.13 CCB
21.14 Up to 8 samples
21.15 Sample duplicate (if required)

21.16 Post spike (if required)

21.17 CCV

21.18 CCB

21.19 Remaining samples in batch (a batch may have no more than 20 samples)

21.20 CCV

21.21 CCB

22.0 PROCEDURE

22.1 Create Sample Table:

22.1.1 Click on "Open" and choose file to open.

22.1.2 Fill in the Sample table according to the Run Sequence 21.0 and change dates on calibration standards sample names.

22.1.3 Enter the sample ID and dilution factor.

22.1.4 Check to ensure the cup location is incremented correctly.

22.1.5 Load samples onto the autosampler as listed on the printed sample table.

22.2 Check to make sure all reagents and lines are pumping correctly.

NOTE: Allow reagents to pump through reagent lines for approximately two minutes before turning cadmium column to "in-line."

22.3 Click on "Start."

22.4 Refer to the Method 10-107-04-1-C: Determination of Nitrate/Nitrite in Surface and Wastewaters by Flow Injection Analysis for the analytical procedures.

22.5 Refer to the Lachat QuikChem FIA+ 8000 Series User Manual for analytical procedures.

23.0 INSTRUMENT/EQUIPMENT SHUT-DOWN PROCEDURE

23.1 Turn the cadmium column to "off-line."

23.2 Place the reagent lines in DI water and allow for DI water to pump through the system for 5 minutes.

23.3 Remove the reagent tubes from the DI water and allow air to pump through the system for 5 minutes, or until water no longer appears in the lines.

23.4 Turn off power, and release platen tension.

23.5 Refer to the Lachat QuikChem FIA+ 8000 Series User Manual for instrument/equipment shut-down procedures.

24.0 DATA REVIEW, VALIDATION, AND APPROVAL PROCEDURE

24.1 Refer to GL-GC-E-092 for General Chemistry Data Review and Packaging and GL-LB-E-005 for Data Review and Validation.

24.2 Data Reduction

24.2.1 Linear regression is used for calibration. The analyst must not

force the calibration line through the origin.

24.2.1.1 Make certain that the instrument response is treated as the dependent variable (y) and the concentration as the independent variable (x). The regression will produce the slope and intercept terms for a linear equation in the form:

$$y = ax + b$$

Where:

y = instrument response

a = slope of line (also called the "coeffiecient of x")

x = concentration of the calibration standard

b = the intercept

24.2.1.2 The analyst should not force the line through the origin, but have the intercept calculated from the standard data points.

24.2.1.3 The regression calculation will generate a correlation coefficient (r^2) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r^2 must be $\geq$ 0.995. The calculated intercept value needs to be evaluated before reporting sample results.

24.2.1.4 In calculating the sample concentrations, the regression equation is rearranged to solve for the concentration (x) as shown below:

$$x = \frac{(y - b)}{a}$$

24.2.1.5 The curve must meet all criteria set forth in Section 20.0 and GL-QS-E-014 for Quality Assurance Measurement Calculations and Processes.

25.0 DATA TRANSMITTAL

Data are uploaded in AlphaLIMS as they are obtained.

26.0 RECORDS MANAGEMENT

All data associated with the performance of this procedure, including relevant logbooks, are maintained as quality records in accordance with GL-QS-E-008 for Quality Records Management and Disposition.

27.0 ROUTINE INSTRUMENT/EQUIPMENT MAINTENANCE

27.1 Refer to the Lachat QuikChem FIA+ 8000 Series User Manual for routine instrument maintenance.

27.2 The Lachat QuikChem FIA+ 8000 Series instrument maintenance logbook is maintained in the Lachat lab.

28.0 LABORATORY WASTE HANDLING AND DISPOSAL

For the proper disposal of sample and reagent wastes from this procedure, refer to the Laboratory Waste Management Plan, GL-LB-G-001.

29.0 METHOD VERIFICATION

Method detection limit studies are performed every six months in accordance with GL-LB-E-001 for The Determination of Method Detection Limits.

30.0 REFERENCES

30.1 EPA Method 353.2, Revision 2.0, August 1993. Determination of Nitrate+Nitrite Nitrogen by Semi-Automated Colorimetry.

30.2 EPA Method 353.2, Revision 1.0, 1978. Determination of Nitrate+Nitrite Nitrogen by Semi-Automated Colorimetry.

30.3 EPA Method 353.2, Revision 0, 1974. Determination of Nitrate+Nitrite Nitrogen by Semi-Automated Colorimetry.

30.4 Method 10-107-04-1-C: Determination of Nitrate/Nitrite in Surface and Wastewaters by Flow Injection Analysis. Lachat Instruments 6645 West Mill Rd., Milwaukee, WI 53218.

30.5 Lachat QuickChem FIA+ 8000 Series User Manual. Lachat Instruments 6645 West Mill Rd., Milwaukee, WI 53218.

30.6 Standard Methods, 22nd Edition, 4500 NO_3^- F-2011

31.0 HISTORY

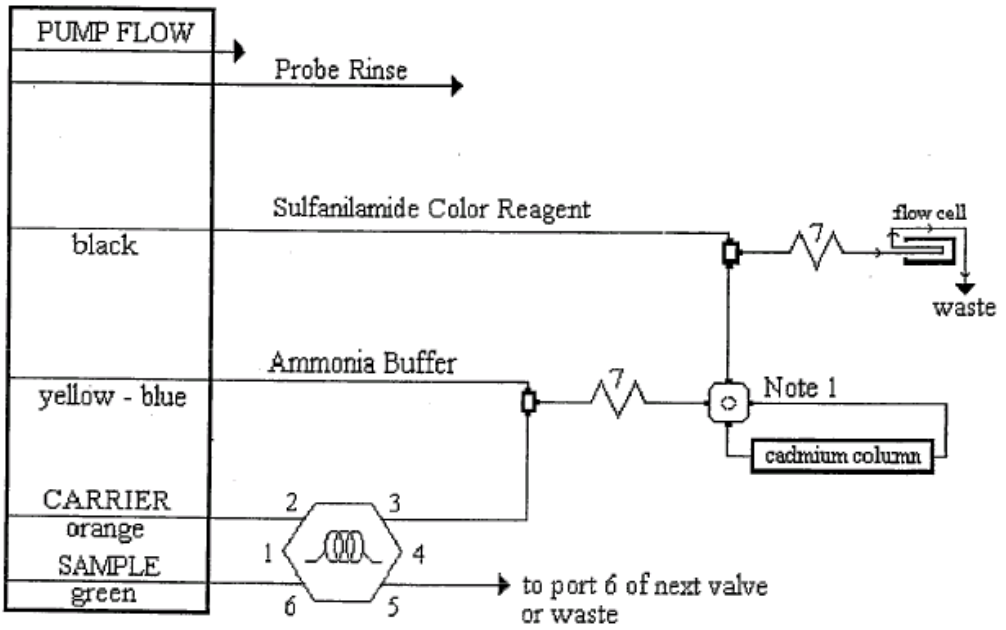
Revision 7: Deleted "The intercept should be evaluated as stated in section 7.2.2.5.3." from section 20.4.2.

Revision 8: Updated Standard Methods reference to 22nd Edition in accordance with MUR II, updated DI Water in accordance with GL-LB-E-016.

Revision 9: Added refrigeration requirements for single component standards.

Revision 10: Added readback requirements for calibration curve standards.

APPENDIX 1: NITRATE/NITRITE MANIFOLD DIAGRAM

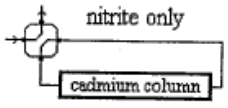


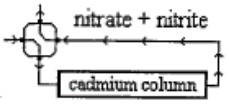
Carrier: Helium Degassed DI water
Manifold Tubing: 0.8 mm (0.032 in) i.d. This is 5.2 µL/cm.
AE Sample Loop: 17 cm x 0.8 mm i.d.
QC8000 Sample Loop: 22.5 cm x 0.8 mm i.d.
Interference Filter: 520 nm

Apparatus: An injection valve, a 10 mm path length flow cell , and a colorimetric detector module is required.

7: **135** cm of tubing on a 7 cm coil support

Note 1: This a 2 state switching valve used to place the cadmium column in-line with the manifold





APPENDIX 2

Conc of STD	Vol of Stock	Final Vol of Std
1.5 ppm	1.5 mL of 100 ppm std	100 mL DI H_2O
1.0 ppm	1.0 mL of 100 ppm std	100 mL DI H_2O
0.5 ppm	0.5 mL of 100 ppm std	100 mL DI H_2O
0.1 ppm	0.1 mL of 100 ppm std	100 mL DI H_2O
0.05 ppm	0.05 mL of 100 ppm std	100 mL DI H_2O
0.02 ppm	0.02 mL of 100 ppm std	100 mL DI H_2O

NO_x (Nitrate/Nitrite)

VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE

FOR

TOXICITY CHARACTERISTIC

LEACHING PROCEDURE PREPARATION

(GL-LB-E-006 REVISION 22)

APPLICABLE METHOD:
SW-846 1311

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1.0 STANDARD OPERATING PROCEDURE FOR TOXICITY CHARACTERISTIC LEACHING PROCEDURE PREPARATION

2.0 PURPOSE

This Standard Operating Procedure (SOP) provides the necessary instructions to prepare Toxicity Characteristic Leaching Procedure (TCLP) samples for extraction to determine the mobility of inorganic and organic contaminants present in liquid, solid, and multiphase wastes, in accordance with SW-846 Method 1311. See appropriate individual analytical standard operating procedure for the necessary instructions for subsequent analysis of prepared TCLP extracts.

3.0 DISCUSSION

3.1 TCLP is used to determine whether or not a waste passes or fails the toxicity characteristic by simulating leaching that would occur in a landfill. The TCLP leachate may be analyzed for the parameters listed in Appendix 1.

3.2 For liquid wastes (those containing less than 0.5% solid material), the wastes after filtration through a 0.6 to 0.8 µm glass fiber filter are defined as the TCLP extract.

3.3 TCLP extracts are analyzed for any combination of semivolatiles, volatiles, and/or metals. Once extracted, the metals fraction is spiked in the TCLP laboratory. This spike addition occurs before preservation with nitric acid to a pH<2.

3.4 For wastes containing greater than or equal to 0.5% solids, the liquid is separated from the solid phase and stored for later analysis; the particle size of the solid phase is reduced, if necessary. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. Zero-Head Space Extraction Vessel (ZHE) is used when testing for the volatile analytes. Following extraction, the liquid extract is separated from the solid phase by filtration through a 0.6 to 0.8 µm glass fiber filter.

3.5 If compatible, the initial liquid phase of the waste is added to the liquid extract, and these are analyzed together. If incompatible, the liquid is analyzed separately and the results are mathematically combined.

3.6 Analysts must be familiar with the reference method and understand how to refer to it.

3.7 Preservatives shall not be added to samples before extraction.

3.8 Samples may be refrigerated unless refrigeration results in irreversible physical change to the waste. If precipitation occurs, the entire sample including precipitate should be extracted.

3.9 TCLP extracts should be prepared for analysis and analyzed as soon as possible following extraction. Extracts or portions of extracts for metallic analyte determinations must be acidified with nitric acid to a pH <2, unless precipitation

occurs. If a precipitation occurs then a non-preserved portion of the extract shall be analyzed as soon as possible after filtration.

4.0 DEFINITIONS

4.1 Solid: yields no liquid upon filtration through a 0.6 to 0.8 µm glass fiber filter at a maximum pressure of 50 psi.

4.2 Liquid: yields <0.5% solid when filtered through a 0.6 to 0.8 µm glass fiber filter at a maximum pressure of 50 psi.

4.3 Matrix Spike and Matrix Spike Duplicate (MS and MSD): An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS corrected for background concentrations.

4.4 Semi-solid: Yields 0.5% or greater of solid when filtered through a 0.6 to 0.8 µm glass fiber filter at a maximum pressure of 50 psi.

4.5 Refer to GL-QS-B-001 the Quality Assurance Plan for additional lab-wide used definitions.

5.0 EQUIPMENT

5.1 Agitation Apparatus: capable of39 rotating extraction vessels in an end-over-end fashion at 30 ± 2 rpm.

5.2 2 L High density polyethylene (HDPE), polypropylene (PP) or polyvinyl chloride (PVC) may be used to tumble sample for analysis of metals. Glass, polytetrafluoroethylene (PTFE) or stainless steel equipment may be used when evaluating the mobility of both organic and inorganic components.

5.3 ZHE: used when waste is being tested for the mobility of volatile analytes. Should have an internal volume of 500 - 600 mL and be equipped to accommodate a 90 - 110 mm filter.

5.4 Positive Pressure Filtration Device and Glass Fiber Filter: used to filter semivolatile and metals samples. It should be capable of reaching 50 psi and supporting a glass fiber filter.

5.5 pH paper and pH meter. The pH meter should be accurate to ± 0.05 units at 25°C.

5.6 40 mL volatile vials are used to collect volatile extracts upon filtration.

5.7 Laboratory Balance: Minimum accuracy 0.01 g.

5.8 Glass beaker (150 mL) used to do pH test.

5.9 Watch glass: covers beaker during pH test.

5.10 Magnetic stirrer: stirs sample during pH test.

5.11 Deionized Water (DI).

5.12 Sieve: 9.5 mm mesh for use in particle size reduction for extraction.
5.13 Sieve: 1 mm mesh for use in particle size reduction for pH test.
5.14 Hotplate: Heats sample during pH test.
5.15 Linear pump: used to transfer extraction fluid into the ZHE
5.16 Nalgene tubing
5.17 Gas tight syringe
5.18 hypodermic needle
5.19 syringe
5.20 Crimpable glass 2.5mL vials
5.21 syringe filters (0.7µm)

6.0 REAGENTS, CHEMICALS, AND STANDARDS

NOTE: All reagents are purchased from an approved vendor. The reagents are screened before use to verify the integrity of the reagent. Logbooks are kept on all reagents.

6.1 1N Hydrochloric Acid (HC1) made from American Chemical Society (ACS) reagent grade HC1. Add 41.7 mL of concentrated HCl acid to approximately 400 mL of DI water. Stir. Cool to room temperature and dilute to total volume of 500 mL.

6.2 1N HNO_3 made from ACS reagent grade HNO_3. Add 31.3 mL of concentrated HNO_3 to 400 mL of DI water. Stir. Cool to room temperature and dilute to total volume of 500 mL.

6.3 1N NaOH made from ACS reagent grade NaOH. Add 40 g NaOH to a 1L glass Erlenmeyer flask. Bring up to 1L with DI water. Stir.

6.4 TCLP Extraction Fluid #1 (this may be ordered ready for use):

6.4.1 Add 30L of DI water into a carboy. Add 285 mL of Glacial Acetic Acid, ACS reagent grade.

6.4.2 Weigh 128.7 g NaOH in 1L plastic beaker. Add 500 mL DI water. Stir and pour the NaOH solution into the carboy. Repeat adding DI water, stirring and pouring until all NaOH pellets are dissolved.

6.4.3 Add DI water to 50L. Shake the carboy well.

6.4.4 Check the pH of this fluid. When accurately prepared, the pH will be 4.93 ± 0.05. Record all information in AlphaLIMS.

6.5 TCLP Extraction Fluid #2 (this may be ordered ready for use):

6.5.1 Add 30L of DI water into a carboy. Add 285 mL of Glacial Acetic Acid.

6.5.2 Add DI water to 50L. Shake the carboy well.

6.5.3 Check the pH of this fluid. When accurately prepared, the pH will be 2.88 ± 0.05. Record all information in AlphaLIMS.

6.6 Metals Spike

6.6.1 The TCLP spiking solutions for metals are ordered from approved vendors. The solutions typically contain multiple analytes in varied concentrations. Analytes in the TCLP spike mixes are at concentrations appropriate for RCRA level analysis.

6.6.2 All reagents, chemicals and standard spike solutions are recorded in electronic logbooks, according to related laboratory standard operating procedures.

7.0 ANALYST VERIFICATION

Technicians and analysts do not analyze client samples without supervision until trained by qualified personnel and upon the successful analysis of a proficiency sample. Training records are maintained as quality records. See SOP GL-QS-E-011 for initial demonstration of proficiency protocols.

8.0 PRELIMINARY EVALUATIONS

NOTE: Preliminary TCLP evaluations are performed on a minimum of 100 grams of waste. This aliquot may not actually undergo TCLP extraction. These preliminary evaluations include: (1) determination of the percent solids (section 9.0); (2) determination of whether the waste contains insignificant solids and is therefore, its own extract after filtration (section 9.13); (3) determination of whether the solid portion of the waste requires particle size reduction (section 9.14); and (4) determination of which of the two extraction fluids are to be used for the nonvolatile TCLP extraction of the waste (Section 9.15).

9.0 DETERMINATION OF THE PERCENT SOLIDS

9.1 If the waste will obviously yield no liquid when subjected to pressure filtration (i.e., is 100% solids) proceed to section 9.14. Record this observation on the TCLP preliminary evaluation queue sheet.

9.2 If the sample is liquid or multi-phasic, liquid/solid separation to make a preliminary determination of percent solids is required. This involves using the filtration device.

9.3 Insert filter into filtration device. Thoroughly wet filter by adding >20 mL of DI water. Securely close device. Evacuate water by injecting 20 psi of compressed nitrogen for 10 seconds. Disassemble unit and weigh moist filter. Record as pre-filter weight on the TCLP preliminary evaluation queue sheet.

9.4 Reassemble filtration device with moist filter. Pre-weigh container to catch filtrate (i.e. 1 L polypropylene, etc.). Record weight on preliminary queue sheet. Place the pre-weighed container under the filtration device to catch the filtrate. Try to get the bottle as close to the bottom of the device as possible to avoid lost volume through splattering.

9.5 Tare balance. Place beaker/container on the balance. If a) limited sample is available and no VOAs are needed, quantitatively transfer entire contents to the beaker/container; b) limited sample is available and VOAs are needed, quantitatively transfer two-thirds of entire contents to the beaker/container; c) sample volumes are adequate and solids are minimal, quantitatively transfer contents to the beaker/container; d) sample volumes are adequate and solids are relatively high, quantitatively transfer enough sample to give ample solids to work with, but never too much to clog filter or give over 100 g of solid material. For examples c) and d), be sure to set aside enough volume for VOAs, preferably up to 500 g (~ 0.5 L).

9.6 Record the weight to the nearest 0.1 g unit on the TCLP preliminary evaluation queue sheet under the "Beaker with aliquot weight" column.

9.7 Quantitatively transfer the waste sample to the filter holder (liquid and solid phases). When possible, spread the waste sample evenly over the surface of the filter. If filtration of the waste at 0 to </= 6° C reduces the amount of expressed liquid over what would be expressed at room temperature then allow the sample to warm up to room temperature in the device before filtering. Gradually apply a vacuum or gentle pressure of 1-10 psi until air or pressurizing gas moves through the filter. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2 minute interval, proceed to the next 10 psi increment. When the pressurizing gas begins to move through the filter, or when liquid flow has ceased at 50 psi, stop the filtration.

NOTE: If the filter is clogged, testing will need to be repeated at a lesser volume.

9.8 Weigh beaker/container containing sample residue on tared balance. Enter this information on the queue sheet under the "Beaker with residue weight" column. The sample sub-aliquot is defined as "Beaker with aliquot weight" minus "Beaker with residue weight." This value is recorded in the data entry column "Sub-sample aliquot" in LIMS.

9.9 The material in the filter holder is defined as the solid phase of the waste and filtrate is defined as the liquid phase.

NOTE: Some wastes such as oily wastes and some paints will obviously contain some material that appears to be a liquid. If after applying pressure filtration, as described in 9.7 and the material does not filter, then the material within the filtration device is defined as a solid. Do not replace the original filter with a fresh filter under any circumstances. If after applying pressure, as described in 9.7 and the material does filter, then the waste liquid will be treated as its own extract. Due to the saturated nature of the filter when filtering these matrices, percent solids are nearly impossible to determine. If

no visible solids are noted, the filter will be discarded. If solids are noted, the filter will be kept for tumbling. The weight used for tumbling will be calculated using:

$$(\text{Post-filter oily weight})^{9.11} - (\text{Pre-filter weight})^{9.3}$$

This value is used as the wet solid weight in section 10.4.3. For all oily waste extractions that do not require tumbling of the filter, document in the batch narrative.

9.10 Weigh the container used to collect the filtrate on a tared balance. Record this value in the "post-container weight" column. The difference between the pre- and post-container weights is the filtrate weight, entered as "Filtrate weight."

9.11 Weigh the moist filter with solids on a tared balance. Enter this weight as "Post-filter weight." The difference between the post-filter and pre-filter weights is recorded as the "wet solid weight." If <0.5 % TCLP solids, discard filter and proceed to step 10.5. The filtrate is its own extract.

9.12 Calculate the % TCLP solids as follows:

$$\frac{(\text{Post-filter weight})^{9.11} - (\text{Pre-filter weight})^{9.3}}{(\text{Sub-sample aliquot})^{9.8}} \times 100 = \%\ \text{TCLP solids}$$

9.13 Determination of insignificant solids

9.13.1 If the percent TCLP solids determined in Section 9.12 is equal to or greater than 0.5 %, proceed to Section 9.14. Retain the liquid fraction to combine with the solid portion in step 10.4.6.2. Store the liquid fraction at 0 to </= 6 °C until ready to combine.

9.13.2 If the percent TCLP solids determined in Section 9.12 is less than 0.5%, then the liquid phase is its own extract. Proceed to step 10.5 for preservation of TCLP extracts.

9.14 Determination of the necessity of particle size reduction

9.14.1 Using the solid portion of the waste, evaluate the solid for particle size. Particle size reduction is required, unless the solid has a surface area per gram of material equal to or greater than 3.1 cm2, or is smaller than 1 cm in its narrowest dimension (i.e., is capable of passing through a 9.5 mm (0.375 inch) standard sieve). If the surface area is smaller or the particle size larger than described above, prepare the solid portion of the waste for extraction by crushing, cutting, or grinding the waste to a surface area or particle size as described above. Special precautions, as outlined in Method 1311 section 15.3, must be followed when performing particle size reduction for volatile constituents.

NOTE: To meet method requirements, the object must fit through the sieve with its narrowest dimension, i.e. a pencil would fit yet a dime would not.

9.15 Determination of appropriate extraction fluid:

NOTE: If the solid content of the waste is greater than or equal to 0.5% and the waste will be extracted for volatile constituents, use extraction fluid #1 and proceed to section 11.0 for extraction of volatile constituents. If the solid content of the waste is greater than or equal to 0.5% and the waste will be extracted for nonvolatile (metals and organics) constituents, determine the appropriate extraction fluid by the following steps.

9.15.1 Weigh out a suitable glass beaker. If conditions 9.5 a) or 9.5 b) exist, there will not be enough solid sample to do the pH determination. In this case, extraction fluid #2 will be utilized for the tumble. If conditions 9.5 c) or 9.5 d) exists, reassemble filtration device with a fresh filter and filter enough sample to obtain 5.0 grams of solid material. Dispose of the liquid phase from this filtration.

9.15.2 For conditions 9.5 c) or 9.5 d), add 96.5 mL of DI water to the beaker and cover with a watch glass. Stir vigorously for 5 minutes using magnetic stirrer. Turn magnetic stirrer to low, so that it remains spinning slowly. Measure and record the initial pH on TCLP prep queue sheet. If the pH$\leq$ 5.0 use extraction fluid #1. Proceed to section 10.0 for extraction of non-volatile organics.

9.15.3 If the pH from section 9.15.2 is >5.0 add 3.5 mL 1N HCl. Slurry briefly. Cover with a watch glass, heat to 50°C and hold for 10 minutes.

9.15.4 Let the solution cool to room temperature. The final pH is recorded on TCLP prep queue sheet. If the final pH is $\leq$5.0, use extraction fluid #1. If the final pH is >5.0, use extraction fluid #2. Proceed to section 10.0 for extraction of non-volatile constituents.

NOTE: Due to ALARA (As Low As Reasonably Achievable), reduced sample volumes or difficult sample matrices the more aggressive fluid #2 may be used. For these conditions, the client must be contacted via the Project Manager, and approval must be received prior to proceeding. The deviation from the method will be documented via the batch narrative.

10.0 NON-VOLATILES PROCEDURE

10.1 A minimum sample size of 100 grams (solid and liquid phases) is recommended. If 100 grams is not available the client must be contacted via the project manager, and approval must be received prior to proceeding. If the analysis is continued with less than 100 grams the comment is added to the batch narrative. In some cases a larger sample size may be necessary. This is especially true when the % solids is near 0.5%.

NOTE: For ALARA (As Low As Reasonably Achievable) purposes, RAD 2 and RAD 3 samples may use a reduced volume. This also applies to difficult sample matrices (metal, construction debris, etc). The volume of extraction fluid will be proportioned at 20:1 with the weight of the sample. For these conditions, the client must be contacted via the Project Manager, and approval must be received prior to proceeding. The deviation from the method will be documented via the batch narrative.

10.2 If the aliquot of the waste used for the preliminary evaluation was determined to be 100% solid at step 9.1, that aliquot may be used for extraction in this section. At least 100 grams should remain for non-volatile extractions. Proceed to step 10.4.1 for determinations on the amount of the extraction fluid to use.

10.3 If the aliquot of the waste required liquid/solid separation as determined by the preliminary evaluations proceed to step 10.4 with the solid fraction from step 9.8. Enough solids should be generated by filtration to support the analyses required of the TCLP extract. Use percent solids information to determine the optimum sample size for filtration.

10.4 Solid TCLP extraction for non-volatile constituents.

10.4.1 If not already performed prepare the solid portion of the waste for extraction by crushing, cutting, or grinding the waste to a surface area or particle size as described in Step 9.14.

10.4.2 Transfer the solid material into an extractor bottle. Record the net weight of the solid sample on the TCLP preparation queue sheet. For samples <100% solid, the filter used to separate the liquid and solid phases will also be included in the extractor bottle. (Do not include the weight of the filter in the aliquot weight).

10.4.3 Determine the amount of extraction fluid to add to the extractor vessel by multiplying (20 X wet solid weight).

Example Calculations:

Where waste is a dry solid and

Aliquot size = 100.0 g

Extraction fluid is calculated as 20 X aliquot size

(20 X 100.0 = 2000 mL)

10.4.4 Slowly add the calculated amount of extraction fluid to the extractor vessel. Close the extractor bottle tightly and secure in the rotary agitation device (TCLP Tumbler). Prepare required tumble blanks at this time and tumble with the samples being analyzed.

Rotate at 30 +/- 2 rpm for 18 +/- 2 hours. The ambient temperature shall be maintained at 23 +/- 2°C during the extraction period.

NOTE: As tumbling occurs, pressure may build up with in the extractor bottle for some types of wastes. To relieve excess pressure, the extractor bottle may be periodically opened and vented into a hood.

10.4.5 Following the 18 +/- 2 hour extraction, separate the material in the extractor vessel into its component liquid and solid phases by filtering through a new glass fiber filter. For final filtration the filter may be changed to facilitate filtration. Discard the solid portion and retain the liquid portion. The liquid portion is the extract.

10.4.6 Prepare the TCLP extract as follows:

10.4.6.1 If the initial waste was 100% solid, the filtered extraction liquid from step 10.4.5 is the only extraction liquid. This liquid fraction is defined as the TCLP extract. Proceed to sample preservation with the liquid fraction to step 10.5.

10.4.6.2 If the initial waste was greater than 0.5% solid but less than 100% solid, determine if the filtered extraction fluid from step 10.4.5 and the initial separated liquid fraction from step (9.7) are miscible. If the two extracts are not miscible, contact the project manager. The extract liquid from the solid fraction and the initial liquid fraction will be prepared and analyzed separately and their results will be mathematically combined. Together, each separate extract is defined as the TCLP extract. Proceed to sample preservation with each fraction to step 10.5.

10.4.6.3 If the two extracts are miscible, combine the two extracts into a single suitable container. This combined liquid is defined as the TCLP extract. Proceed to sample preservation with the liquid fraction to step 10.5.

10.5 Preservation of TCLP extracts.

10.5.1 Measure the pH of each liquid fraction and record the value on the TCLP data entry screen.

10.5.2 The TCLP extracts for organic analyses are not to be preserved with nitric acid and are chilled at 0 to </=6°C until analyzed or prepped by the Organics laboratory.

10.5.3 The TCLP extracts for metals analysis shall have a fraction set aside for matrix spike and spike duplicate if necessary. After the spike solution is added to the spike sample, each sample and spike sample shall be preserved to a pH of < 2.0 with nitric acid. If precipitation is observed after adding nitric acid to a small aliquot of the filtrate then the remaining filtrate will not be preserved, a comment will be added to the batch narrative, and the extract will be analyzed as soon as possible.

NOTE: If the sample matrix is oil that passes through the glass fiber filter then spiking and preservation will occur in the inorganic prep lab.

10.6 Once the extracts are labeled and entered into LIMS they are delivered to the laboratory for further fractional prep.

11.0 VOLATILES PROCEDURE

11.1 The ZHE device is used for volatiles extraction. Volatiles samples should not be exposed to the air for more time than is absolutely necessary. Any manipulation of volatiles samples should be performed at 0 to </= 6ºC to minimize loss of volatiles. Perform all necessary particle size reduction following step 9.14 at this time.

11.2 Assemble the ZHE device.

11.2.1 Place the ZHE piston within the body of ZHE. Make sure gas inlet/outlet valve is closed.

11.2.2 Place a glass fiber filter between two support screens.

11.3 If the sample is 100% solid, weigh out a 25 g subsample of the waste in to the ZHE body. Record weight on the TCLP Prep Queue Sheet.

11.3.1 Secure filter and support screens onto the top flange of the device and secure the top flange to the ZHE body in accordance with the manufacturer's instructions.

11.3.2 Tighten all ZHE fittings and place the device in a vertical position with liquid inlet/outlet valve on top. Open SLOWLY. Apply pressure of 1 - 10 psi or more when needed up to 50 psi to force out any headspace.

11.3.3 Vent the compressed air side of the ZHE. Add 500 mL of extraction fluid #1 with a gas tight syringe or linear pump. Proceed to section 11.12.

NOTE: Prepare an empty ZHE for preparation blank. 500 mL of extraction fluid #1 is used as the preparation blank unless the sample is < 0.5 solids in which case Deionized water is used.

11.4 If the sample contains < 0.5% solids, the liquid portion of the sample after filtration, is defined as the TCLP extract. Tumbling is not required for samples classified as liquids.

11.4.1 Weigh at least 50 mL of liquid sample into the ZHE body. Record the weight in the data entry screen.

11.4.2 Proceed to 11.10 immediately.

11.5 If the sample contains ≥0.5% solids, but ≤5%, weigh out 500 g of sample into ZHE body. Proceed to Section 11.8.

11.6 If the sample contains ≥5% solids, but less than 100% solids, use the percent wet solids information recorded in 9.12 to determine the amount of sample needed in ZHE. Recommended sample size:

$$\text{Wt. of waste needed} = \frac{25}{\%solids} \times 100 \text{ (volume should never exceed 500 g)}$$

Example: % solids = 10%; Wt. needed = (25/10) x 100 = 250 g

11.6.1 Weigh out sample (liquid and solid phases) into ZHE body and record the weight on the TCLP data entry screen.

11.7 Secure filter and support screens onto the top flange of the device and secure the top flange to the ZHE body in accordance with the manufacturer's instructions.

11.7.1 Tighten all ZHE fittings and place the device in a vertical position with liquid inlet/outlet valve on top. Open SLOWLY. Apply pressure of 1 - 10 psi or more when needed up to 50 psi to force out any headspace. When adding pressure, check the ZHE vessel and piston O-rings for leakage.

11.7.2 At the first appearance of liquid, quickly close the valve.

11.8 Attach male fitting to inlet/outlet valve on top of ZHE. Attach precut Nalgene tubing to male fitting, and place labeled volatiles vial or other glass container at end of tubing to collect extract. Make sure whatever container used is large enough to handle the volume anticipated with the filtering process.

11.8.1 Attach gas line to gas inlet/outlet valve on bottom.

11.8.2 Apply gas pressure of 1-10 psi and open liquid inlet/outlet valve on top so liquid will flow through Nalgene tubing and into volatiles vial or glass container.

11.8.3 Increase pressure at 10 psi increments until no more liquid is expelled or you reach a maximum of 50 psi.

11.8.4 Close liquid inlet/outlet valve.

11.8.5 Turn off gas pressure and release pressure at gas inlet/outlet valve on the bottom of the ZHE.

11.8.6 Store the liquid filtered off at 0 to </= 6°C until the solid portion is extracted.

11.9 If the sample is <0.5% solids, this filtrate is considered the TCLP extract. Make sure the 40 ml volatile vial is labeled properly. Proceed to step 11.16.

11.10 Using a gas tight syringe or linear pump, add an amount of extraction fluid #1 equal to 20 times the weight of the solid phase remaining in the ZHE unit. Extraction fluid #1 is used in all cases. Record the volume of extraction fluid on the data entry screen.

Volume of extraction fluid #1 = [(Wt. of waste used x % solids)/100] x 20

Example: % solid = 10%, Wt. used = 250 g; Volume of Extraction fluid #1 = [(250 x 10)/100] x 20 = 500 ml

NOTE: Prepare an empty ZHE for preparation blank. 500 mL of extraction fluid #1 is used as the preparation blank unless the sample is < 0.5% solids in which case Deionized water is used.

11.11 Place the ZHE in the rotary agitation apparatus and rotate at 30 ± 2 rpm for 18 ± 2 hours. Ambient temperature shall be maintained at 23 ± 2° C during agitation.

11.12 After the sample has completed the tumbling process, attach male fitting to inlet/outlet valve on top of ZHE. Attach precut Nalgene tubing to male fitting, and place labeled volatiles vial (if samples was 100% solid) or other glass container (if less than 100% solid samples was used) at end of tubing to collect extract. Make sure the glass container used for the samples that were less than 100% solid is large enough to handle the volume anticipated with the filtering process.

11.12.1 Attach gas line to gas inlet/outlet valve on bottom.

11.12.2 Apply gas pressure of 1-10 psi and open liquid inlet/outlet valve on top so liquid will flow through Nalgene tubing and into volatiles vial or glass container.

11.12.3 Increase pressure at 10 psi increments until no more liquid is expelled or you reach a maximum of 50 psi.

11.12.4 Close liquid inlet/outlet valve.

11.13 Turn off gas pressure and release pressure at gas inlet/outlet valve on the bottom of the ZHE.

11.14 For samples that were less than 100%, combine the entire tumbled extracted sample liquid with the liquid collected from Section 11.9. After mixing thoroughly, but not so vigorous as to liberate volatile compounds, filled a labeled 40 ml volatile vial. For samples that were 100% solid, the tumbled extracted sample liquid is considered the TCLP extract liquid and a 40 ml labeled vial is filled.

NOTE: If the initial liquid filtrate and the TCLP extract do not combine, then these two layers must be prepared and analyzed as two separate samples. Project Management will be notified and the final results mathematically combined according to the ratio of the two original layers.

11.15 Place each labeled volatile vial in a vial rack and transfer custody to the VOAs area.

11.16 For ALARA (As Low As Reasonably Achievable) purposes RAD category 2 and 3 samples will use a reduced volume procedure for TCLP volatiles. Difficult organic and potential highly contaminating matrices (i.e. greases, oils, paints, solvents, etc.) will also use a reduced volume procedure for TCLP volatiles. Place 2.25 grams of sample in a 45 mL glass vial. Fill the vial with 45 mL of TCLP fluid #1, making sure to eliminate the presence of air bubbles from the vial. Seal the samples and place them on the tumbler for 18 ± 2 hours.

11.17 Remove samples from the tumbler after allotted time. Place a hypodermic needle on a 10 mL Luer-lock syringe. Insert needle into septum of 45 mL glass vial and withdraw 10 mL extract. Replace needle with filter cartridge, and divide extract taken in 3 glass vials 2.5 mL each. Cap and crimp vials placing them in centrifuge tubes with sample ID. After extraction the plastic bags or centrifuge tubes containing sample extracts should be stored in the areas of required tests.

12.0 HOLDING TIMES

12.1 Samples must undergo TCLP extraction with the following holding times:

Sample Maximum Holding Times (Days)				
	From:	From:	From:	
	Field Coll.	TCLPP Extract.	Prep Extract	
	To:	To:	To:	Total Elapsed
	TCLP Extract	Prep Extract.	Analysis	Time (Days):
Volatiles	14	N/A	14	28
Semivolatiles	14	7	40	61
Mercury	28	N/A	28	56
Metals (exc. Hg)	180	N/A	180	360

N/A = Not applicable

If the sample holding times are exceeded, the values obtained will be considered minimum concentrations. Exceeding holding time is not acceptable in establishing that a waste does not exceed the regulatory level. Exceeding the holding time does not invalidate characterization if the waste exceeds the regulatory level.

13.0 QUALITY ASSURANCE

13.1 A minimum of one blank must be analyzed for every 20 samples that have been extracted. Use the same extraction fluid that is used for samples. If both types are used make a separate blank for fluid #1 and fluid # 2.

13.2 A matrix spike shall be performed for each waste type (e.g., wastewater treatment sludge, contaminated soil, etc.) unless the result exceeds the regulatory level and the data are being used solely to demonstrate that the waste property exceeds the regulatory level.

13.3 Matrix spike solutions for metals are added after filtration of the TCLP extract and before acid preservation. Organic TCLP extracts are spiked according to the prep and analytical method requirements and spiking is performed by the organic laboratory responsible for preparation or analysis of the TCLP extract.

NOTE: Some organic analytes have shown degradation when spiking materials are added to the TCLP matrix and a delay occurs between spiking and completing the preparation of the samples. Spiking solutions for organics analyses should be added during the extraction/sample prep procedure, as with prep of non-TCLP sample extracts.

13.4 Tumbler rotation shall be checked on a weekly basis and documented in LIMS.

13.5 The temperature in the tumbler room is monitored constantly and is recorded in LIMS.

13.6 Prior to accepting extraction fluid #1 for use it is screened in the organics labs. If methanol or volatile compounds are detected at levels > PQL, the extraction fluid is not logged for use in TCLP. Records of the screens are maintained with the COA for the extraction fluid.

14.0 EQUIPMENT MAINTENANCE

14.1 ZHEs are rotated so that varying ZHE vessels will be used for the blank quality control sample for each batch tumbled. A ZHE must be used as a tumble blank with 5% frequency. This is tracked via AlphaLIMS system. This will monitor cleanliness of the ZHEs and expose any possible contamination problems.

14.2 For the ZHE to be acceptable for use the piston within the ZHE should be able to move, using approximately 15 psi or less. If it takes more pressure to move the piston, the O-rings in the device should be replaced. If this does not solve the problem, the ZHE is unacceptable for TCLP analysis and the manufacturer is contacted.

14.3 The ZHE should be checked for leaks after every extraction. If the device contains a built-in pressure gauge, pressurize the device to 50 psi, allow it to stand unattended for one hour, and recheck the pressure. If the device does have a built-in gauge, pressurize the device to 50 psi, submerge it in water, and check for the presence of air bubbles escaping. If pressure is lost, check all fittings and inspect and replace O-rings, if necessary. Retest the device. If leakage problems cannot be resolved, contact the manufacturer.

14.4 Before using TCLP extraction vessels, separate all parts and clean. Bake at 180° C for one hour, if required.

15.0 DETECTION LIMITS, ANALYSIS AND CALCULATIONS

15.1 Method Detection Limits will be adjusted for masses/volumes used in TCLP leachate preparation. See the limits associated with each chemistry being performed for details. Limits are adjusted by AlphaLIMS.

15.2 Analytical calculations are verified according to the procedures associated with the analytical test being performed.

16.0 INTERFERENCES

16.1 Any interferences in the method are discussed in the analytical procedures for which the TCLP extracts are prepared.

17.0 CALIBRATION AND STANDARDIZATION

17.1 pH meters are calibrated in accordance with GL-GC-E-008. When pH outside the range of pH 4 to pH 10 are read, additional buffer solutions are used to verify proper calibration. These additional buffer checks are recorded in LIMS.

17.2 Balances are calibrated in accordance with GL-LB-E-002.

18.0 SAFETY, HEALTH AND ENVIRONMENTAL HAZARDS

18.1 Personnel performing this analytical procedure are trained in and follow the safe laboratory practices outlined in the Safety, Health and Chemical Hygiene Plan, GL-LB-N-001.

18.2 Personnel handling radioactive materials are trained in and follow the procedures outlined in GL-RAD-S-004 for Radioactive Material Handling.

18.3 Personnel handling biological materials are trained in and follow the procedures outlined in GL-RAD-S-010 for Handling Biological Materials.

18.4 If there is any question regarding the safety of any laboratory practice, stop immediately, and consult qualified senior personnel such as a Group or Team Leader.

19.0 RECORDS MANAGEMENT

19.1 Record data in AlphaLIMS extraction logbook for each sample prepared.

19.2 Test each extraction fluid for correct pH and impurities. TCLP extraction fluids are logged in LIMS after being created.

19.3 Spiking standards are recorded in the appropriate standard logbooks.

20.0 CORRECTIVE ACTIONS

20.1 Limited volume/mass of sample is the most frequent cause for departure from the method required minimum sample size of 100 grams (solid and liquid phases). When 100 grams is not available or the sample is determined to be highly contaminated, a lesser volume/mass is used as discussed in section 10.1.

21.0 REFERENCES

21.1 USEPA SW-846, Method 1311, July 1992.

21.2 USEPA, Federal Register 40 CFR Parts 261, 264, 265, 268, 271 and 302. Vol. 55, no. 126, Friday, June 29, 1990, Rules and Regulations.

21.3 USEPA, Federal Register 40 CFR Parts 261, Appendix II. (7-1-92 edition), 1992.

21.4 Larry P. Jackson, Environmental Testing and Analysis, Pp. 18-25, 9, 1993.

21.5 Operation Maintenance Instruction OM-149, Millipore Corporation, Nov. 1986.

21.6 Instruction for Use Model 3775-GTS Gas-Tight Syringe, Associated Design and Manufacturing Company, February 1990.

21.7 Nuclear Regulatory Commission, Joint NRC/EPA Guidance on Testing Requirements for Mixed Radioactive and Hazardous Waste, [7590-01], July, 1997.

22.0 HISTORY

Revision 18: Clarified spiking practices
Revision 19: Updated preparation of TCLP Extraction Fluid #1 and #2 to eliminate overnight standing. Added ZHE must be used as a tumble blank with a 5% frequency.
Revision 20: Added a NOTE to 9.14.1 for clarification sieve size method requirement.
Revision 21: Added clarification statement about difficult matrices.
Revision 22: Updated to included clarification ALARA statement for difficult sample matrices.

APPENDIX 1

Metals	Volatile Organics	Acid Extractables	Base /Neutral Extractables	Pesticides	Herbicides
Arsenic	Benzene	o-cresol	2,4 Dinitrotolune	Chlordane	2,4-D
Barium	Carbon Tetrachloride	m-cresol	Hexchloro benzene	Endrin	2,4,5-TP (Silvex)
Cadmium	Chlorobenzene	p-cresol	Hexachloro butadiene	Heptachlor	
Lead	Chloroform	Pentachloro-Phenol	Hexachloroethane	Heptachlor Epoxide	
Mercury	1,4-dichlorobenzene	2,4,5-trichlorophenol	Nitrobenzene	Lindane	
Selenium	1,2-dichloroethane	2,4,6-trichlorophenol	Pyridine	Methoxychlor	
Silver	1,1-dichloroethlyene			Toxaphene	
Chromium	Methyl ethyl Ketone Tetrachloro Ethylene Triochloro ethylene Vinyl chloride				
	1,4-Dichlorobenzene		1,4-Dichlorobenzene		

VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE FOR DETERMINATION OF METALS BY ICP

(GL-MA-E-013 REVISION 31)

APPLICABLE TO METHODS:
EPA Method 200.7
EPA SW-846 Method 6010B
EPA SW-846 Method 6010C
EPA SW-846 Method 6010D

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1.0 STANDARD OPERATING PROCEDURE FOR DETERMINATION OF METALS BY ICP

2.0 METHOD CODES

2.1 EPA SW-846 Method 6010B

2.2 EPA SW-846 Method 6010C

2.3 EPA SW-846 Method 6010D

2.4 EPA Method 200.7

3.0 METHOD OBJECTIVE AND PURPOSE

This standard operating procedure (SOP) describes the procedure for the determination of metals with the Perkin Elmer (PE) Optima ICPs.

4.0 METHOD APPLICABILITY AND METHOD SUMMARY

4.1 Analytes: Ag, Al, As, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, Pb, Sb, Se, Si, SiO_2, Sn, Sr, Ti, Tl, U, V, Zn, P, and S.

4.2 Applicable Matrices: These methods are applicable to the determinations of any of the analytes listed above for various matrices including waters, oils, soils, sludges, Toxicity Characteristic Leachate Procedure (TCLP) extracts and other more unusual types of sample which are generally classified as a miscellaneous matrix.

4.3 General Method Summary: After samples are prepared in accordance with the sample preparation SOP, they are analyzed by Inductively Coupled Plasma (ICP) as follows:

4.3.1 The instrument is calibrated with a minimum of two calibration points for each element to be analyzed. The points consist of a calibration blank solution to define the lower calibration point and at least one standard calibration solution at the analyte concentrations to define the higher calibration point(s). A correlation coefficient of 0.995 or better (0.998 or better for SW-846 Method 6010C) is required for each analyte if multiple standards are used or the instrument is recalibrated for the analyte of interest.

4.3.2 Prepared client samples, check standards, and quality control samples identified in section 21.1 are then analyzed. The check standards and quality control samples are used to determine the quality and acceptability of the analytical data.

4.3.3 Continuing Calibration Verification Samples (CCV) are analyzed a minimum of every 10 samples to ensure that the instrument is continuing to perform correctly.

5.0 METHOD SCOPE AND PERFORMANCE CHARACTERISTICS

5.1 Calibration Range: The calibration range consists of the concentrations between the calibration blank and that of the highest calibration standard for each analyte.

5.2 Tested Concentration Range: For ICP analyses, the upper limit of the linear range is determined by analyzing a linear range verification check standard, near the

expected linear range limit. The analytically determined concentration of this standard shall be within ± 10% of the true value.

5.3 Method Detection Limit (MDL) studies for each analyte are performed and/or verified at least annually. These studies are conducted and calculated in accordance with SW-846, Chapter 1, Section 5.0, and GL-LB-E-001 for The Determination of Method Detection Limits. The Reported Quantitation Limit (RQL) reflects the current MDL study. For 6010D mean recovery of the seven replicates is ± 35% of the known value with an RSD of ≤ 20%.

5.4 Instrument Detection Limits (IDLs) are useful means to evaluate the instrument noise level and response changes over time for each analyte from a series of reagent blanks analyses to obtain a calculated concentration. IDLs are determined by the mean of blank results plus three times the standard deviation of 10 replicate analyses of the reagent blank solution. (Use zero for the mean if the mean is negative). Each measurement should be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure normally performed between the analysis of separate samples). IDLs should be determined at least once using new equipment, after major instrument maintenance such as changing the detector, and/or at a frequency designated by the project.

5.5 Method Precision: To assure analytical precision of methods used, MS/MSD or sample DUP, or LCS/LCSD are analyzed with each batch or as method requires.

5.6 Method Bias (Accuracy): Determined by calculating recoveries of LCS of a similar matrix.

6.0 DEFINITIONS

6.1 Continuing Calibration Blank (CCB): An aliquot of reagent water or other blank matrix that is analyzed after each CCV. The CCB is used to determine whether the analytical sequence is in control during sample analysis.

6.2 Continuing Calibration Verification (CCV) Standard: An aliquot of reagent water or other blank matrix to which known quantities of the method analytes are added in the laboratory. The CCV is analyzed exactly like a sample, periodically throughout the run sequence. Its purpose is to determine whether the analytical sequence is in control during sample analysis. It may be prepared from the same source as the calibration standards, and is usually of varied concentration.

6.3 Independent Calibration Blank (ICB): An aliquot of reagent water or other blank matrix that is analyzed after each ICV. The ICB is used to determine whether there is carryover contamination after injection of the mid-level ICV.

6.4 Independent Calibration Verification (ICV): A solution of method analytes of known concentrations that is used to fortify an aliquot of Blank or sample matrix. The ICV is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check laboratory performance with externally prepared test materials.

6.5 Laboratory Control Standard (LCS): An aliquot of reagent water or other blank matrix to which known quantities of the method analytes are added in the laboratory. The LCS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

6.6 Linear Calibration Range (LCR): The concentration range over which the instrument response is linear.

6.7 Method Detection Limit (MDL): The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero.

6.8 Statistical Process Control (SPC) Limits: Statistically derived limits that establish acceptable ranges for recoveries of analytes of interest, including LCS, MS, MSD, PS, PSD and internal standards.

6.9 Stock Standard Solution: A concentrated solution containing one or more method analytes prepared in the laboratory using certified reference materials or purchased from a reputable commercial source.

6.10 Limit of Detection (LOD): An analyte, method and matrix specific estimate3 of the minimum amount of a substance that can be reliably detected. GEL has established LOD = 2 x MDL.

6.11 Limit of Quantitation (LOQ): An analyte, method and matrix specific estimate of the minimum amount of a substance that can be reported with a specific estimate of confidence. LOQ is set at or above the concentration of the lowest initial calibration standard. The laboratory must empirically demonstrate precision and bias at the LOQ. The LOQ and associated precision and bias must meet client requirements and must be reported. GEL uses the following guidance (LOD < LOQ):

When LOD < PQL, PQL =LOQ

When LOD > PQL, LOQ is raised to next lowest calibration standard

6.12 Practical Quantitation Limit (PQL): The PQL is typically at or above the lowest point on an acceptable initial calibration curve. It may also be determined by multiplying the MDL by approximately 2 to 10. Concentration of a target analyte determined to be greater than its PQL are defined as quantitative results. This limit is not used in DoD ELAP reporting.

6.13 Refer to GL-QS-B-001 the Quality Assurance Plan for additional lab-wide use definitions.

7.0 INTERFERENCES TO THE METHOD

ICP analysis is subject to three types of interferences:

7.1 Physical interferences are those physical properties of a sample solution that prevent their introduction to the plasma with an efficiency equal to that of the calibration standards. This type of interference can be reduced through the use of an internal

standard in accordance with the instrument operating manual, or by diluting the sample in reagent blank solution until the percent recovery is acceptable.

7.2 Chemical interferences are minimal in ICP Spectroscopy because the extremely high energy of the plasma breaks chemical bonds.

7.3 Interelement Spectral Interference can be overcome by use of Interelement Correction (IEC) factors. IECs are initially calculated when a new instrument or method is brought on-line and are continually calculated on a biannual basis. To calculate IECs, single element standards are analyzed at their respective linear ranges. IEC coefficients are calculated if the element interfered with is outside ± PQL. Further calculations may be required if tighter criteria is required. IECs may be checked using single element standards (analyzed at a minimum of every 6 months) as often as needed to ensure the correct factors are being used.

7.4 The IEC coefficients are calculated if the element interfered with is outside ± PQL when analyzing a single element standard at its linear range:

7.4.1 Perkin Elmer Optima ICPs: The interfering element uses the mg/L concentration. The IEC for Cd via PE software is ¯ 18/490, or ¯ 0.03673. When verifying the IEC by analyzing the ICSA standard or the single element analysis, the non-spiked analyte concentration should be within ± 2x the PQL (6010B and 6010C), ± PQL (6010D), or ± 2x MDL for DoD QSM. IECs are calculated for all minerals and for commonly seen analytes that occur at high concentrations in environmental samples, i.e., manganese and zinc.

8.0 SAFETY PRECAUTIONS AND HAZARD WARNINGS

WARNING

PREVENT SKIN AND EYE CONTACT BY USING SPECIFIED PERSONAL PROTECTIVE EQUIPMENT WHEN MAKING STOCK REAGENTS.

WORK UNDER A HOOD TO PREVENT INHALATION WHEN MAKING STOCK REAGENTS.

8.1 Sample digestates are not extremely volatile or spontaneously combustible, but they are normally acidic and should be handled with care. Small spills may generally be wiped up with paper towels that can be disposed of in the trash. Larger spills may require the use of a mop. The mop head may have to be disposed of as potentially hazardous waste in accordance with the Laboratory Waste Management Plan, GL-LB-G-001. If the spilled digestates begin any obvious fuming or reacting, pour a generous amount of the acid neutralizer, located in each lab, onto the spill before attempting to clean it up.

8.1.1 Approved gloves will be worn to avoid skin contact with the digestate during clean-up.

8.1.2 Wear eye protection with side shields while performing procedures in the laboratory. An eyewash station is located in each analysis lab.

8.1.3 Do not persist in cleaning up a spill in the presence of strong fumes. Move out of the area, attempt to isolate the area, and notify your supervisor immediately.

8.2 These instruments use high voltage electricity and therefore should be shut completely down any time electronic components may be exposed to personnel or any liquids.

8.3 Prior to handling radioactive samples, analysts must have radiation safety training and must understand their full responsibilities in radioactive sample handling. Some general guidelines follow:

8.3.1 Protect counter tops with counter paper, or work from radioactive sample handling trays.

8.3.2 Prohibit admittance to immediate work area.

8.3.3 Post signs indicating radioactive samples are in the area.

8.3.4 Take swipes of the counter tops upon completion of work. Deliver those swipes to the designated swipe count box.

8.3.5 Segregate radioactive wastes. Radioactive waste containers are obtained from Waste Management.

8.3.6 For additional guidance, refer to GL-RAD-S-004 for Radioactive Material Handling, and GL-LB-G-001, the Laboratory Waste Management Plan.

8.4 Treat all chemicals and samples as potential health hazards, and limit exposure to these chemicals to the lowest level possible. GEL maintains a current awareness file of OSHA regulations regarding the safe handling of the chemicals in the laboratory as well as a reference file of Material Safety Data Sheets (MSDS). Individual sample MSDS forms provided by the clients are also maintained.

8.5 All samples, chemicals, extracts, and extraction residues must be transferred, delivered, and disposed of safely according to all related SOPs.

8.5.1 Segregate solid wastes from liquid wastes in the satellite area containers.

8.5.2 Segregate oil wastes from water-soluble wastes in the satellite area containers.

8.6 Never leave gas cylinders unchained or untied, even when they are on moving carts.

8.7 In the event of an accident or medical emergency, call for help immediately. When time and safety permit, an accident report form should be completed and turned in to the safety committee.

8.8 Fire escape routes are posted in the lab, and all personnel should be familiar with them. In addition, fire safety equipment such as fire extinguishers is located in the lab. Training is available on the proper operation of this equipment.

9.0 CAUTION WARNINGS

Special care should be taken when handling hydrofluoric acid to ensure that the acid does not come into contact with the skin.

10.0 APPARATUS AND MATERIALS; REAGENTS; EQUIPMENT AND INSTRUMENTS

10.1 Apparatus and Equipment

10.1.1 Replacement glass parts for the ICP such as quartz torch bodies, purge extension windows, injector tips, nebulizers, and spray chambers may be ordered from an approved vendor through GEL's Purchasing Agent.

10.1.2 High purity argon gas is provided to the ICP instruments, via a network of pipelines, into the lab from liquid storage tanks located outside the building. The argon is purchased from a GEL approved vendor.

10.1.3 Consumable materials such as tubing are often attainable from various scientific product companies. The GEL Purchasing Agent can help to find the best prices from an approved vendor. All orders are placed through GEL's Purchasing Agent.

10.2 Reagents, Chemicals, and Standards

10.2.1 Nitric acid, reagent grade and ultra pure

10.2.2 Hydrochloric acid, reagent grade and ultra pure

10.2.3 Lithium solution, 5% or 1%

10.2.4 Scandium solution, 1000 µg/mL

10.2.5 5% Cesium

10.2.6 Yttrium solution,1000 µg/mL

10.2.7 Other Chemicals: Additional compounds, surfactants, oils, cleaning agents, etc. may be routinely ordered through GEL's Purchasing Agent.

10.3 Instrumentation

10.3.1 PE AVIO 500 with compatible PC and printer

10.3.2 PE Optima 8300DV with compatible PC and printer

10.3.3 PE Optima 7300DV with compatible PC and printer

10.3.4 ESI Autosampler

10.3.5 Polyscience6105 PE Chillers or equivalent

10.3.6 Appropriate Nebulizer (i.e., Meinhard or Burgener Mira Mist)

11.0 SAMPLE HANDLING AND PRESERVATION REQUIREMENTS

11.1 Aqueous samples should be preserved with nitric acid to a pH of < 2 prior to receipt by the analyst. Sediment samples should be kept at 0° ≤ 6° C prior to analysis.

11.2 Refer to GL-SR-E-001 for Sample Receipt, Login, and Storage.

12.0 SAMPLE PREPARATION TECHNIQUES

12.1 All samples, except drinking water with Turbidity <1 NTU and samples specifically exempted by contract, are prepared in accordance with the SOPs for Metals

Digestion: GL-MA-E-006 (USEPA SW-846 Method 3005A), GL-MA-E-008 (USEPA SW-846 Method 3010A), GL-MA-E-009 (USEPA SW-846 Method 3050A), and GL-MA-E-020 (NIOSH 7303). Other digestion procedures may also be used.

12.2 Additional filtration may be required to prevent clogging of sample introduction system. The method blank (MB) and laboratory control sample (LCS) must be filtered to check for contamination from the filtering process.

12.3 Sample spills should be handled as stated in Section 8.1.

13.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

Routine Preventative and Special Operational (Failure)

13.1 Routine Preventative Maintenance (PM) is recommended as follows:

Frequency	Procedure
When Needed	Oil the peristaltic pump with silicon spray
	Replace peripump sample introduction tubing
	Change pump hoses on drain systems
	Check drain waste collection containers and empty as needed
	Clean/replace nebulizer
	Clean/replace torch. Align according to manufacturer's specifications
	Clean/replace air filters

13.2 Non-Routine Maintenance Procedures (Special, Operational or Failure Mode Maintenance)

13.2.1 If the instrument will not function properly, refer to the troubleshooting section in the appropriate operator's manual.

13.2.2 If the analyst is unable to fix the instrument, call the GEL Instrument Technician, and if needed, the manufacturer's Service Department.

13.3 Refer to the ICP Maintenance Logbook for routine records. Preventative maintenance and service should be recorded in the maintenance logbook.

14.0 PREPARATION OF STANDARD SOLUTION AND QUALITY CONTROL SAMPLES

14.1 Source standards records are recorded in the Metals Source Standards Logbook, which is maintained in the Metals laboratory. The preparation of working standards is fully described in the "Maintain Reference Materials" application in AlphaLIMS.

14.2 Standards are receipted, labeled, prepared and stored in accordance with GL-LB-E-007 for Laboratory Standards Documentation.

15.0 INSTRUMENT CALIBRATION

15.1 Calibration and sample analysis are conducted automatically using the autosampler. (Samples can also be analyzed manually.)

15.1.1 Profiling: All analytical channels were aligned within specifications during initial installation or realigned after relocation of the instrument by profiling

daily, all elements will be peak-on-center. Profiling should be done after plasma warm-up prior to sample analysis and verified as needed during the day (more frequently on days with high temperature and humidity fluctuations).

15.1.1.1 Operate/Profile instrument using the procedures found in the corresponding ICP Operator's or Hardware Manual.

15.1.1.2 The PE Optima ICPs are auto-profiled using a mercury lamp.

15.1.1.3 When an acceptable profile is attained, print the graphical profile and display. An acceptable profile is defined as having the mercury peak being perfectly centered on the 253.652 nm axis. If the peak is off-centered, run the diagnostic check or have service look at the instrument.

15.1.2 Standardization: Standardization is required every twenty-four when the instrument is in use. Additionally, restandardization and/or recalibration is required when calibration checks fail.

15.2 Calculations are described in the instrument manuals.

15.3 Calibration standards may vary according to method. A minimum of a blank and standard are required, but a typical calibration uses a blank and three standards of varying concentration. For the multiple standard curve, the correlation coefficient (r) must be equal to or greater than 0.995 (0.998 for 6010C).

15.4 For typical (recommended) calibration standards, refer to Appendix 2.

15.5 For continuing calibration requirements, refer to Section 21.

15.6 For actions concerning a calibration verification failing to meet requirements, refer to Section 21.3.

16.0 INSTRUMENT PERFORMANCE REQUIREMENTS

16.1 Before samples may be analyzed, the instrument must be calibrated and verified using an Initial Calibration Verification (ICV) which is prepared from an independent source, an Initial Calibration Blank (ICB), Interference Check Sample(s) (ICS-A, ICS-AB), Practical Quantitation Limit (PQL), and a Linear Range Verification (LR). These must meet the requirements stated in Section 21.2 and Appendix 4 for each analyte being reported, unless specified otherwise by the client or contract.

16.2 The instrument calibration and all continuing verification data are maintained on the printed hard copy and electronically saved onto the computer network. The printouts are kept in chronological order by instrument. Recent files are stored in the respective metals laboratories and older records are archived in the warehouse.

17.0 ANALYST AND METHOD VERIFICATION REQUIREMENTS

17.1 Analyst training is conducted in accordance with GL-HR-E-002 for Employee Training.

17.2 Method performance is verified by conducting MDL studies in accordance with GL-LB-E-001 for The Determination of Method Detection Limits, and by the evaluation of laboratory proficiency samples (audits).

18.0 ANALYSIS PROCEDURES AND INSTRUMENTAL OPERATION

18.1 All samples are introduced to the instrument via a peristaltic pump. The amount of sample entering the instrument is dependent on the pump speed, the diameter of the pump tubing, and the nebulizer efficiency.

18.2 Run Sequence

18.2.1 Calibration sequence (may vary slightly under different autosampler files/see Appendix 2 also).

18.2.1.1	S0	(reagent blank, 3% HCl/1%HNO_3)
18.2.1.2	S0.1	(100 ppb standard)
18.2.1.3	S0.5	(500 ppb standard)
18.2.1.4	SCAL	(1000 ppb standard)
18.2.1.5	S10	(20 to 50 ppm standard)

18.2.2 The calibration verification steps include:

18.2.2.1 ICV
18.2.2.2 ICB
18.2.2.3 PQL (low level ICV for 6010C)
18.2.2.4 ICS-A
18.2.2.5 ICS-AB
18.2.2.6 LR1
18.2.2.7 LR2
18.2.2.8 CCV
18.2.2.9 CCB

18.2.3 Samples (10 samples or less)

18.2.4 Continuing Calibration Verification (CCV), Low Level Calibration Verification (PQL) required for SW-846 6010C, and Continuing Calibration Blank (CCB)

18.2.5 Repeat steps 18.2.3 and 18.2.4 through the end of the run or until applicable verification is out of specifications.

18.2.6 Final verification steps in order:

18.2.6.1 ICS-A (required for 200.7)
18.2.6.2 ICS-AB (required for 200.7)
18.2.6.3 CCV
18.2.6.4 PQL (required for SW-846 6010C)
18.2.6.5 CCB

NOTE: 6010C only requires there to be a closing low level continuing calibration standard (PQL). For ease of analysis, the PQL standard can be analyzed every 10 samples.

18.2.7 ICS-A and ICS-AB are analyzed at least every 8 hours or after the analytical batch for 200.7 analyses.

18.3 For data storage see section 23 and 24.

18.4 General operation of the instrument

The Perkin Elmer Optima 7300V, 8300DV and AVIO500 and are simultaneous, dual view, inductively coupled, argon plasma emission spectrometers. As configured for GEL, it is capable of analyzing 32 substances: Ag, Al, As, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, P, Pb, S, Sb, Se, Si, SiO_2, Sr, Ti, Sn, Tl, U, V, and Zn. The operating software of the system is a menu driven type.

18.4.1 Typical Analytical Challenges

18.4.1.1 Sample Over-range: If an analyte is above the working linear range of the instrument, even if this element is not required, dilute the sample to an acceptable range and reanalyze, especially if the over ranged element is an interferant of the reported analyte. Dilutions are required if the value of an analyte exceeds 100% of the working linear range when the element of interest is being reported, or is interfering with an element that will be reported. Dilution factors must be taken into account when reporting final values. Additionally, further rinse and/or an acidified rinse solution may be required before continuing with the next sample.

18.4.1.2 Torch/Sample Introduction Drift: Various changes in torch plasma conditions, sample introduction, or other external factors can have a great effect on the analysis.

18.4.1.2.1 If sample introduction drift is suspected several items should be reviewed. Check the peristaltic pump tubing for wear and replace as needed. Check the internal standard to make sure it is being introduced to the instrument at a constant rate. Check the nebulizer to ensure that it's free of clogs and that the spray is a continuous mist.

18.4.1.2.2 Profile drift may mimic torch/sample introduction drift. Re-profiling should be done routinely. Profile drift is a function of temperature and should be checked more frequently when large temperature swings are observed. During an analytical run profile drift is evident when a clean

blank shows significant deviation from zero. As suggested by the manufacturer, two elements to track for this problem are Na and Al.

18.4.1.2.3 Tubing connections may become loose, allowing air to bubble into the sample path. Tighten or replace tubing connection as necessary.

18.4.1.3 Sample Matrix: The internal standard technique is used to correct for physical interferences. Each ICP has a Scandium (Sc) internal standard that is continuously fed through the sample introduction system via a peristaltic pump. Scandium is used as the internal standard. It is used at 10 ppm both radially and axially on the Optimas. In addition, the Optimas run yttrium at 2 ppm to reprocess as an internal standard if Sc is suspected in the sample.

18.5 Power switches and auxiliaries are addressed in detail in the appropriate operator's manual and/or hardware guide.

18.6 Start-up and shutdown procedures are addressed in detail in the appropriate operator's manual and/or hardware guide.

18.7 Sample quantity requirements vary based on several different factors such as nebulizer type, diameter of tubing, and pump rate.

18.8 Autosampler can be used with various nebulizers by attaching the sample introduction tube to the autosampler sample tube. The autosampler rinse should be setup by connecting the autosampler rinse tubing to the peristaltic pump in the instrument.

19.0 CALCULATIONS AND DATA REDUCTION METHODS

19.1 Any dilutions, concentrations, or preparation factors must be taken into account prior to reporting data to a client. The appropriate factors are entered into AlphaLIMS.

19.2 Care must be taken that the correct units are being employed. Analytical results originating from the ICPs are reported in ppb or µg/L.

20.0 DATA RECORDING

20.1 The Optima data are stored on the Network as a CSV file and printed at the instrument as each sample is analyzed.

20.2 ICP samples and standards are generally analyzed in three replicates (minimum of 2), and the reported value is the average of the replicates. At times, one replicate may be removed due to an attributable cause. This attributable cause should be documented on the hard data (i.e. third replicate ran out of volume). The average of the two remaining replicates is subsequently recalculated prior to reporting the data to the client.

20.3 Data are retrieved by the data entry personnel where unused data can be eliminated, dilution factors entered, and conversions made prior to uploading to AlphaLIMS.

21.0 QUALITY CONTROL REQUIREMENTS

21.1 Frequency of Quality Control Activities (refer to Appendix 3)

21.1.1 The Initial Calibration Verification (ICV) is performed following each calibration. The Continuing Calibration Verification (CCV) is performed following every ten or fewer samples. Any quality control samples excluding the CCV and CCB are counted as samples no more than two hours should pass between analyzing successive CCVs that bracket reportable data.

21.1.2 An Initial Calibration Blank (ICB) is performed immediately following the ICV, and Continuing Calibration Blanks (CCB) must follow each CCV.

21.1.3 The PQL standard is analyzed after each calibration and recommended for analysis at least every 10 samples, between the CCV and CCB analyses for SW-846 Method 6010C. Method 6010C requires analysis at the end of every batch and only recommends analysis every 10 samples. This standard may also be labeled as a CRDL standard.

21.1.4 An Interference Check Standard is analyzed after the calibration. For 200.7 analysis, an ICSA and ICSAB must be performed before the final CCV, CCB. Interference check standards are analyzed at least every 8 hours per Method 200.7.

21.1.5 A method blank (MB) is performed for each batch of twenty or fewer samples, or more frequently per client contract requirement.

21.1.6 A matrix spike (MS) and a sample duplicate (DUP), or a matrix spike (MS) and a matrix spike duplicate (MSD) are analyzed for each batch of 20 or fewer samples or per client or method requirements. For a matrix spike recovery to be applicable the sample value may not exceed four times the nominal concentration of the spike.

21.1.7 A laboratory control sample (LCS) is analyzed with each batch of twenty or fewer samples. A LCS Duplicate (LCSD) is performed at the same frequency as the LCS when required by a specific program or client.

21.1.8 Serial dilutions are analyzed to indicate the presence or absence of interferences. The analyte concentrations must be greater than 50 times the detection limit for 200.7, 6010B, and 6010C. The analyte concentrations must be greater than 25 times the reporting limit for 6010D for a serial dilution to be applicable. The serial dilution is generally performed at a 5x dilution of the test sample or requested by client contracts.

21.1.9 A serial dilution will be performed for each SDG of up to twenty samples.

21.1.10 When performing 6010C and 6010D analyses, a post-spike must be performed on the original sample when the matrix spike recovery falls outside the control limits and the sample result does not exceed 4x the spike added. Otherwise, post spikes are analyzed per client request.

21.1.11 Linear Range standards are analyzed after each calibration. These standards are labeled LR1 and LR2.

21.2 Acceptance Limits (refer to Appendix 4)

ICV and CCV results must fall between 90% to 110% of the true values for work under EPA SW-846 Method 6010B/6010C/6010D. ICV must be between 95% to 105% of the true value for work under EPA Method 200.7. The ICV/CCV Relative Standard Deviation (RSD) (minimum of 2 replicates) must be < 5% for all methods, **NOTE:** The ICV is a second source standard and may be used as the CCV to show calibration verification.

21.2.1 ICV and/or CCV failure requires a recalibration of the instrument and/or repreparation of standard solutions. Samples bracketed by calibration verifications that are not acceptable for required analytes must be reanalyzed. An ICV or CCV that has failed may be reanalyzed provided there is an attributable cause known to have affected the check standard only and not the previous samples. Examples of an acceptable cause may be a sample tip out of solution during analysis, an incorrectly prepared CCV, or obvious carryover in the CCV from a very high sample immediately prior to the CCV. If a CCV is reanalyzed, the data must be lined through, initialed and dated, and the reason for the reanalysis must be documented on the raw data. In addition, corrective action should be taken to eliminate the cause of the initial CCV failure to prevent future occurrence.

21.2.2 The ICB and CCB results for the various methods are displayed in Appendix 4. ICB and CCB failure requires recalibration of the instrument and/or repreparation of the calibration blank. When analyzing under SW-846, a CCB may be accepted above the PQL level under two conditions: 1) the CCB is acceptable if the sample value is below the PQL and 2) the CCB is acceptable if the level of analyte in the CCB is less than 1/10 the analyte value in the lowest reported sample. For DoD QSM, the ICB and CCB must have an absolute value less than ½ LOQ or less than 10x value of the samples bracketed by the blank.

21.2.3 The PQL recovery requirements are displayed in Appendix 4. This includes the low level continuing calibration verification standard analyzed every 10 samples or at the end of the analytical batch. For all methods, sample results can be evaluated for reporting if they are at least 2x the PQL for a

given analyte. For 6010B or 200.7 analysis no specific acceptance criteria have been established for the PQL, however an unofficial range of 70% to 130% has been set for guidance purposes. PQL failure should be evaluated, but no action is required if the results are outside of the 70% to 130% range for all methods unless otherwise specified by the client. DoD QSM analysis must have passing PQL recoveries or the instrument needs recalibration. Further failures need to be communicated to the client for guidance.

21.2.4 The analytes for the ICS-AB results typically must fall between 80% and 120% of the true value. The requirements for analytes for the ICS-A are displayed in Appendix 4.

21.2.5 Method blank results should be less than the PQL but no less than the negative PQL or ± ½ LOQ for DOD QSM. Additionally, the method blank may exceed the PQL provided that the blank value is insignificant relative to the lowest value of the samples in the batch. Specific method requirements are displayed in Appendix 4. All failures require reprep of the samples in the batch..

21.2.6 The LCS and LCSD are used for indicators of sample digestion validity. LCS and LCSD recoveries and RPDs must fall within process control limits as established by Statistical Process Control, manufacturer's certification, or method requirements (See Appendix 4). LCS and/or LCS duplicate results outside of established acceptance limits require the batch to be redigested and reanalyzed.

21.2.7 Matrix spike and matrix spike duplicates recoveries are applicable to those sample values that do not exceed four times the nominal concentration of the spike. Recoveries of one or more of the matrix spikes that fall outside recommended ranges (as displayed in Appendix 4) may indicate an improper sample digestion, sample in homogeneity, or matrix interference.

21.2.8 The relative percent difference (RPD) between an MS and MSD or a sample and DUP must fall within ≤ 20% if the analyte level is sufficiently high (5x the PQL). See Appendix 4 for method requirements for the DUP.

21.2.9 Serial dilution results that agree within the method limits displayed in Appendix 4 suggest the absence of interference. Failures of a serial dilution should be noted, but are not considered pass/fail criteria. A criterion of greater than 50 times the MDL may be requested by some clients. The criterion of greater than 50 times the LOQ is used for DoD QSM.

21.2.10 The LR1 and LR2 standards must fall within 90-110% of the known value. If any analyte fails to meet the 10% criteria, the working linear range is then defined as the high standard concentration used in the calibration curve.

21.2.11 Post spike recovery requirements are displayed in Appendix 4. Acceptable recoveries suggest the absence of interferences.

21.3 Out-of-Control situations

21.3.1 ICS failure requires that the interferences be corrected, by recalculation, of Interelement Correction Factors or selection of background correction factors so that the ICS can be read within the required limits before samples are analyzed. ICS failure at the end of an analysis period will require that the samples run for the affected analyte(s) during that period to be reanalyzed

21.3.2 Matrix spikes, duplicates, post spikes, and spike duplicates are used only as indicators of method effectiveness on that sample and will not be used as acceptability criteria for the process, unless this is a client-specific requirement.

21.3.3 When analytical results suggest the presence of interference, the serial dilution test discussed in Section 21.1.8 must be employed.

21.3.4 The intensity and percent recovery of the internal standard are monitored per sample. There is no acceptance criterion per SW-846 and 200.7; however, the laboratory has adopted a ± 50% recovery criterion. If the internal standard fails to meet the criteria, reprocess the data using a different internal standard if available or dilute the sample within acceptable parameters.

21.4 Analytical data are evaluated for conformance with the requirements stated in Section 21.2 by the analyst during and/or after the analysis but before the data are entered into AlphaLIMS. Data may be accepted or rejected by the analyst at this point or by the data reviewer(s) as stated in Section 22.

21.5 Corrective actions taken for data not conforming to the requirements in Section 21.2 are stated in Section 21.3. If these corrective actions can be taken by the analyst prior to the acceptance of the data, then no nonconformance documentation is required. However, if these corrective actions include redigestion of the batch or sample and if the data have already been accepted, then a nonconformance/ corrective action report should be completed. This report includes the date, person requesting the action, sample(s) or batch(es) affected, and action requested, all provided by the requester. The person taking the action will provide any pertinent comments, a signature, and the date the action is completed. The disposition of the nonconformance will then be verified by the Group Leader and Quality Assurance Officer if pertinent. These reports will be kept on file in AlphaLIMS. Refer to GL-QS-E-004 for Documentation of Nonconformance Reporting and Dispositioning and Control of Nonconforming Items.

22.0 DATA REVIEW, VALIDATION, AND APPROVAL PROCEDURES

22.1 The AlphaLIMS Data Report and the original batch sheet are provided to the ICP reviewer.

22.2 A knowledgeable person other than the originating analyst shall review the data within the day or specified time limit. When this review is completed and all of the data are found to be acceptable, the reviewer signs and dates the batch data report. If the reviewer determines that the reviewed data are not acceptable and requires additional work or correction, they are returned to the Group Leader or representative with an appropriate explanation.

22.3 Listed below are the data review responsibilities at the Analyst, Peer Review and Validator levels:

22.3.1 Analyst Review is performed by the analyst who generated the data before the data are submitted for entry into AlphaLIMS. The analyst uploads the data to AlphaLIMS and completes the batch data report.

22.3.2 Peer Analyst Review is performed by an individual who did not perform the analysis but is familiar with the analytical method used and the reporting requirements. This person reviews the complete data report after all data are entered and ensures that any data entry corrections are made before the data are approved, and then signs the batch data report. If the data do not meet the necessary quality requirements and need further analysis, they are returned to the analyst.

22.3.3 Data Validation: For data requiring forms packages, the forms are generated per contract requirements and validated per GL-MA-E-017.

22.4 Specific items that are reviewed at each level include the following:

22.4.1 Analyst Review: Before data are submitted for entry into AlphaLIMS.

22.4.1.1 All analyses in the batch are completed with explanations of exceptions.

22.4.1.2 Any corrections and comments on the data are properly initialed and dated.

22.4.1.3 Proper standard identification numbers appear on the runlog to ensure traceability from the data to original source standards.

22.4.1.4 Data acceptance limit criteria identified in Section 21.2 and Appendix 4 are met or an explanation given.

22.4.2 Peer Analyst Review: before Data are statused to DONE in AlphaLIMS:

22.4.2.1 All data are complete and accurate in the AlphaLIMS Data Report.

22.4.2.2 Any exceptions or shortcomings have been sufficiently explained or corrected.

22.4.2.3 Data are reported in the proper units, or an explanation is given.

22.4.2.4 Prep factor and dilution calculations by AlphaLIMS are present in the data report and AlphaLIMS calculations are correct.

22.4.2.5 LCS and Spike Recoveries and RPD calculations by AlphaLIMS are correct and are within control limits.

22.4.3 Validation: Before data are reported out of the lab:

22.4.3.1 Analyst and Peer Level reviews have been completed and any corrections or comments made.

22.4.3.2 Preparation data have been entered and reviewed by a peer.

22.4.3.3 Analytical data are present for all analyses.

22.4.3.4 Weight/Volume, dilution, spike recovery, RPD, and LCS recoveries appear to be calculated correctly.

22.4.3.5 All forms, raw data, and log sheets requested by the client are present.

22.4.3.6 Results are accurate on forms (refer to GL-MA-E-017).

22.5 Documentation and review process are reflected in the Data Review Flow Chart in Appendix 1.

23.0 DATA REPORT

23.1 To report data after the initial review process has been completed:

23.1.1 Enter the AlphaLIMS program through GEL home page and click on AlphaLIMS application.

23.1.2 Click on MAINTAIN BATCHES and type in the queue at SELECT A QUEUE (ICP for the analytical).

23.1.3 Select batch number at SAMPLE ID column, and the run status will show whether data are ready to calculate.

23.1.4 When data are ready to calculate, click on CAL.

23.1.5 Click on SAVE to save the data.

23.1.6 Send data to review at STATUS column for quality purposes.

23.1.7 Print data, and initial and date for final review.

23.2 The Final Reviewer is responsible for changing the batch status to DONE in AlphaLIMS and initialing and dating the data report.

24.0 RECORDS MANAGEMENT

All data associated with the performance of this procedure, including relevant logbooks, are maintained as quality records in accordance with GL-QS-E-008 for Quality Records Management and Disposition.

25.0 LABORATORY WASTE HANDLING AND DISPOSAL

All Laboratory wastes are handled and disposed in accordance with the Laboratory Waste Management Plan, GL-LB-G-001.

26.0 METHOD VARIATION

SW-846 requires that ICB and CCB results must agree within three standard deviations of the mean blank value. GEL uses the absolute value of the PQL for evaluating ICB and CCB results. The acceptance limits are further discussed in Appendix 4.

27.0 REFERENCES

27.1 Perkin Elmer Optima 7300DV, 8300DV and AVIO500 Operator's Manual

27.2 Test Methods for Evaluating Solid Waste: Laboratory Manual Physical/ Chemical Methods, Volume 1A, SW-846, 3rd Edition, Nov. 1986. Method 6010B, "Inductively Coupled Plasma-Atomic Emission Spectroscopy," Rev 2, Sept. 1994. USEPA Office of Solid Waste and Emergency Response, Washington, DC 20460.

27.3 Test Methods for Evaluating Solid Waste: Laboratory Manual Physical/ Chemical Methods, Volume 1A, SW-846, 3rd Edition, Nov. 1986. Method 6010C, "Inductively Coupled Plasma-Atomic Emission Spectroscopy," Rev 3, February. 2007. USEPA Office of Solid Waste and Emergency Response, Washington, DC 20460.

27.4 Test Methods for Evaluating Solid Waste: Laboratory Manual Physical/Chemical Methods, Volume 1A, SW-846, Update V, Revision 4, Method 6010D "Inductively Coupled Plasma - Optical Emission Spectrometry"

27.5 EPA Method 200.7, Revision 4.4.

27.6 Department of Defense (DoD), Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories, Version DoD 5.1 and DOE 3.1, January 2017.

28.0 HISTORY

Revision 31: Updated to remove retired and add new instrumentation.

Revision 30: Removed readback statements for methods no longer performed, clarified should vs. must in several sections.

Revision 29: Clarification of DOD QSM criteria and method acceptance criteria.

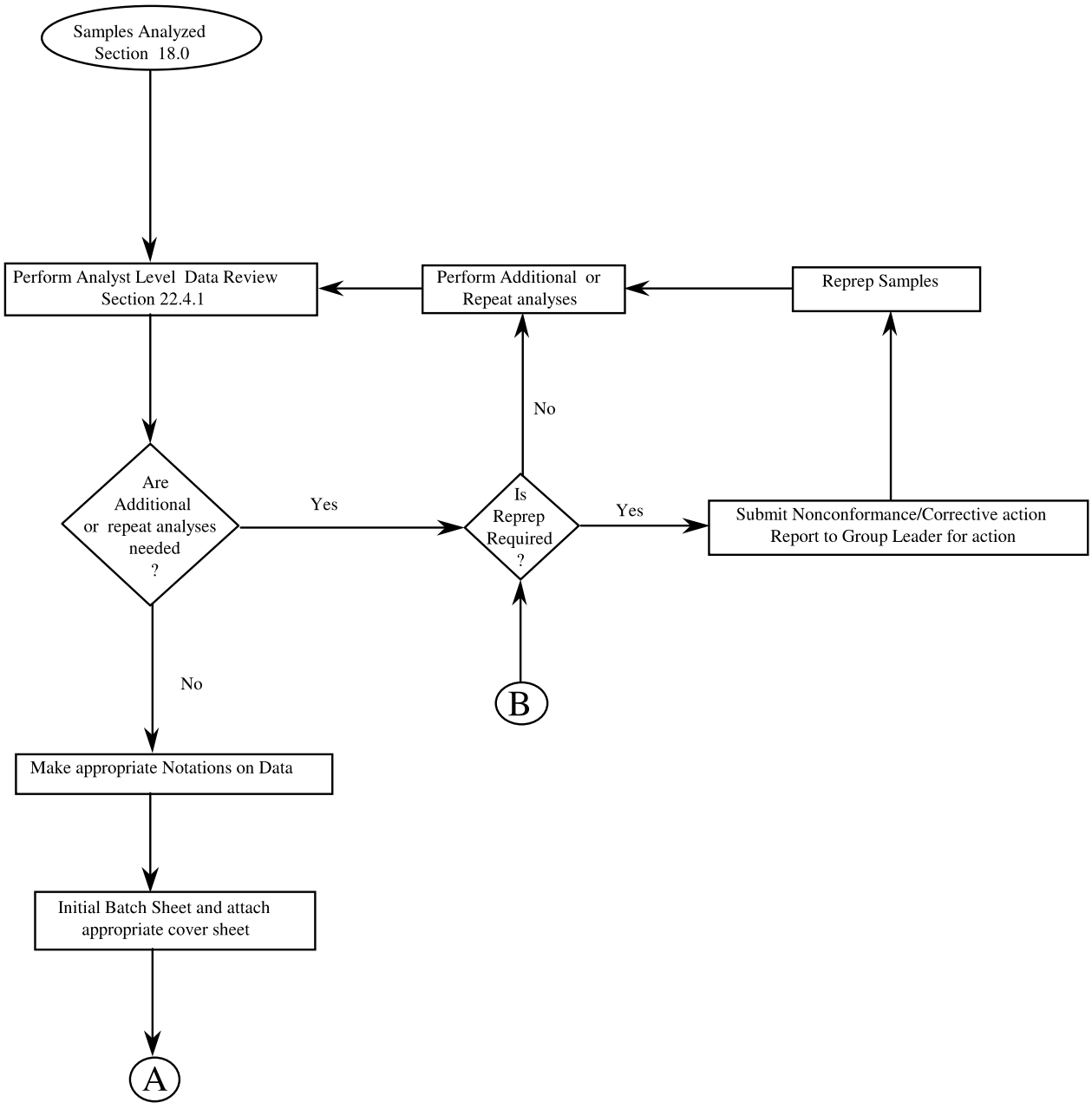
Revision 28: Add statements to clarify the IEC verifications.

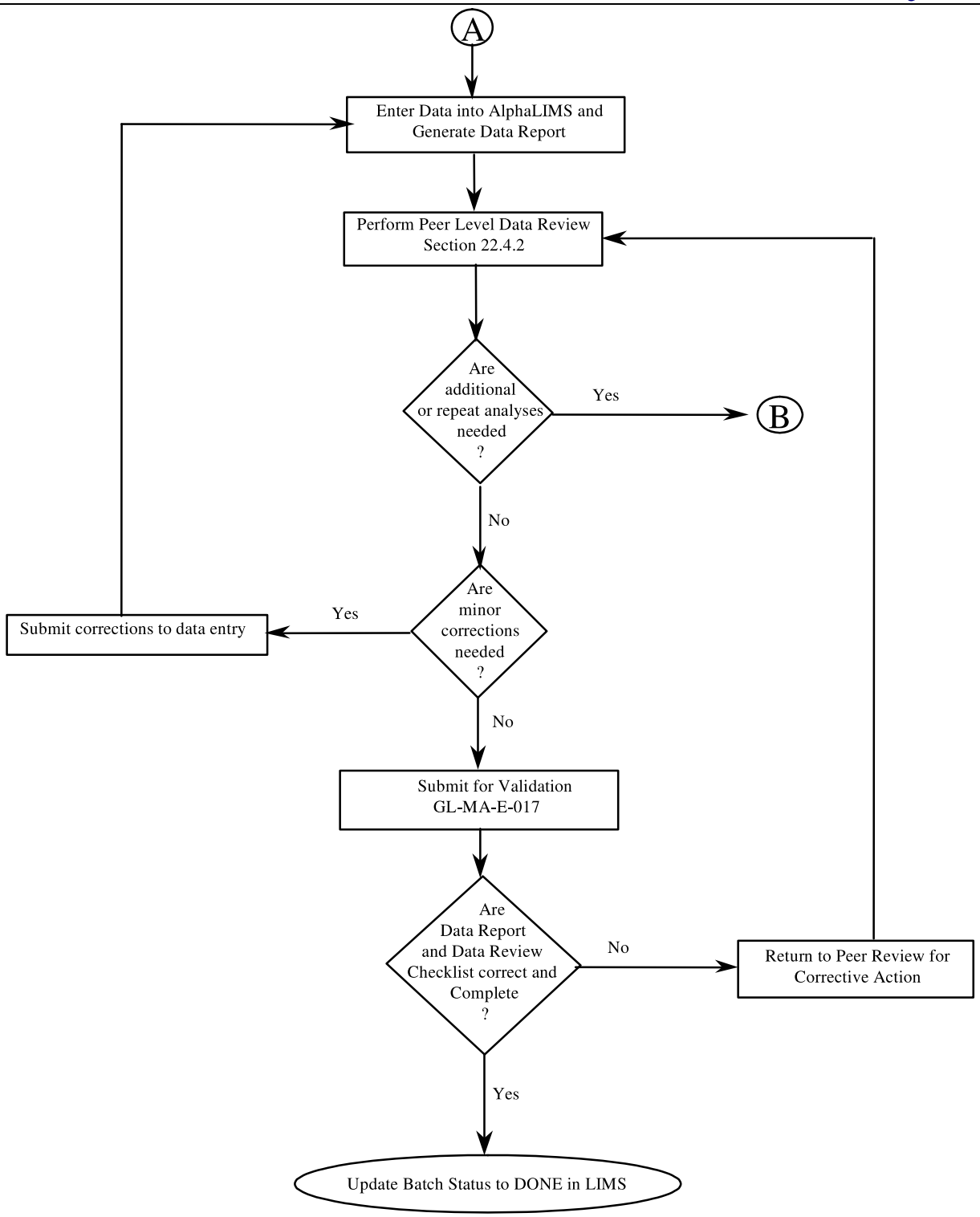
Revision 27: Added method requirement for IEC verification performed on a quarterly basis.

APPENDIX 1: PRODUCTION FLOW CHART

(For Illustrative Purposes Only)

Data Review Flow Chart




A
Enter Data into AlphaLIMS and Generate Data Report
Perform Peer Level Data Review Section 22.4.2
Are additional or repeat analyses needed ?
Yes
B
No
Are minor corrections needed ?
Yes
Submit corrections to data entry
No
Submit for Validation GL-MA-E-017
Are Data Report and Data Review Checklist correct and Complete ?
No
Return to Peer Review for Corrective Action
Yes
Update Batch Status to DONE in LIMS

APPENDIX 2: CALIBRATION STANDARDS FOR TRACE

(For Illustrative Purposes Only)

S0 reagent blank (3% HCl / 1% HNO_3)

S0.1 0.1 ppm (100 ppb) As, Ba, Be, B, Cd, Cr, Co, Cu, Pb, Mn, Ni, Se, Sr, Tl, U, V, Zn Sb, Mo, Ag, Sn, Ti

0.2 ppm (200 ppb) S

0.5 ppm (500 ppb) Si, P

1 ppm (1000 ppb) Al, Ca, Mg, K, Na

S0.5 0.5 ppm (500 ppb) As, Ba, Be, B, Cd, Cr, Co, Cu, Pb, Mn, Ni, Se, Sr, Tl, U, V, Zn Sb, Mo, Ag, Sn, Ti

1 ppm (1000 ppb) S

2.5 ppm (2500 ppb) Si, P

5 ppm (5000 ppb) Al, Ca, Mg, K

SCAL 1 ppm (1000 ppb) As, Ba, Be, B, Cd, Cr, Co, Cu, Pb, Mn, Ni, Se, Sr, Tl, U, V, Zn Sb, Mo, Ag, Sn, Ti

2 ppm (2000 ppb) S

5 ppm (5000 ppb) Si, P

10 ppm (10000 ppb) Al, Ca, Mg, K, Na

S10 50 ppm (50000 ppb) Ca, Al, Mg
20 ppm (20000 ppb) Fe, Na

(Analytes and concentrations may change with further method development)

APPENDIX 3: FREQUENCY OF QUALITY CONTROL ACTIVITIES

(For Illustrative Purposes Only)
Frequency of Quality Control Activities

Method/ Frequency	**SW-846 6010B and 6010D**	**EPA 200.7**	**SW-846 6010C**
Linear Range Std	Per calibration	Per calibration	Per calibration
ICV	Per calibration	Per calibration	Per calibration
ICB	Per calibration	Per calibration	Per calibration
PQL/CRI (CLP)	Per calibration	Per calibration	Per calibration and at end of each analytical batch.
ICSA	Per calibration	Per calibration; every 8 hours after or end of analytical run	Per calibration
ICSAB	Per calibration	Per calibration; every 8 hours after or end of analytical run	Per calibration
CCV	Every 10 instrument runs	Every 10 instrument runs	Every 10 instrument runs
CCB	Every 10 instrument runs	Every 10 instrument runs	Every 10 instrument runs
Method Blank	5% or per batch	5% or per batch	5% or per batch
LCS - liquid LCS - soil	5% or per batch	5% or per batch	5% or per batch
Matrix Spikes	5% or per request	10% or per request	5% or per request
Sample Duplicates	5% or per request	5% or per request	5% or per request
Serial Dilutions	5% or per request	5% or per request	5% or per request
Matrix Spike Duplicates	5% or per request	10% or per request	5% or per request
Post Digestion Spikes	5% or per request	5% or per request	5% or per request

APPENDIX 4: ACCEPTANCE LIMITS

Method/ Acceptance Criteria	SW-846 6010B	EPA 200.7	DOD QSM Version 5.1	SW-846 6010C	SW-846 6010D
Linear Range Std	± 10% of true value	± 10% of true value	± 10% of the true value	± 10% of true value	± 10% of true value
ICV	90% - 110%	95% - 105%	90%-110%	90% - 110%	90% - 110%
ICB	< absolute value of PQL	< absolute value of PQL *	< ± ½ LOQ	< absolute value of PQL	<absolute value of ½ PQL
PQL	70% - 130% advisory limits only	70% - 130% advisory limits only	80%-120% or investigate and recalibrate	70% - 130% or investigate and recalibrate	80%-120% or investigate and recalibrate
ICSA	80-120% for major components, ±2XPQL for non-spiked	80-120% for major components, ±2XPQL for non-spiked	80%-120% for major components, < ± ½ LOQ for non-spiked	80-120% for major components, ±2XPQL for non-spiked	80%-120% for major components, ±PQL for non-spiked
ICSAB	80%-120%	80%-120% (may be requested)	80%-120%	80%-120%	Not Required
CCV	90% - 110%	90% - 110%	90%-110%	90% - 110%	90% - 110%
CCB	± PQL	±PQL *	± ½ LOQ	± PQL	± PQL
Method Blank	± PQL	± PQL *	± ½ LOQ , except for Al, Fe, Ca, Mg, Na and K	± PQL	± ½ PQL
LCS - liquid LCS - soil	80% - 120% current SPC limits	85% - 115% current SPC limits	Use QSM Specified limits	80% - 120% current SPC limits	80% - 120% current SPC limits
Matrix Spikes	75% - 125%, when applicable	75% - 125%, when applicable	Use QSM Specified limits	75% - 125%, when applicable	75% - 125%, when applicable
Sample Duplicates	0% - 20% when greater than 5X PQL, +/-PQL when less than 5X PQL	0% - 20% when greater than 5X PQL, +/-PQL when less than 5X PQL	0% - 20% when greater than 5X PQL, +/-PQL when less than 5X PQL	0% - 20% when greater than 5X PQL, +/-PQL when less than 5X PQL	0% - 20% when greater than 5X PQL, +/-PQL when less than 5X PQL
Serial Dilutions	0% - 10% of initial raw value, when applicable	0% - 10% of initial raw value, when applicable	0% - 10% of initial raw value, when > 50X LOQ	0% - 10% of initial raw value, when applicable	0% - 20% of initial raw value, when applicable
Post Digestion Spikes	75%-125%, when applicable	75%-125%, when applicable	80%-120%	80%-120%, for all	75%-125%; when applicable
Internal Standards	50%-150%, then evaluate dilution	50%-150%, then evaluate dilution	50%-150%, then evaluate dilution	50%-150%, then evaluate dilution	50%-150%, then evaluate dilution
Matrix Spike Duplicate	0% - 20%	0% - 20%	0% - 20%	0% - 20%	0% - 20%

*North samples require ½ the PQL

VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE
FOR
ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS
BY
GAS CHROMATOGRAPHY/MASS SPECTROMETRY

(GL-OA-E-009 REVISION 45)

APPLICABLE TO METHODS:
EPA SW-846 Methods 625.1, 8000D, 8270C, 8270D and 8270E

PROPRIETARY INFORMATION

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1.0 STANDARD OPERATING PROCEDURE FOR THE ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY

2.0 METHOD CODES

2.1 EPA Method 625.1

2.2 EPA SW-846 Methods 8270C, 8270D, 8270E

2.3 EPA SW-846 Methods 8000D

3.0 METHOD OBJECTIVE AND PURPOSE

This standard operating procedure (SOP) covers the determination of semivolatile organic compounds in a wide variety of solid waste matrices, soil, and water according to USEPA methods 8270C, 8270D, 8270E and 625.1. Other analytical protocols such as Department of Defense (DOD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental laboratories are also covered. Please refer to Appendix 8 for a summary of these requirements.

4.0 METHOD APPLICABILITY AND METHOD SUMMARY

4.1 Applicable matrices include, but are not limited to, groundwater, wastewater, surface water, leachate, soil, oil, and other miscellaneous matrices.

4.2 Methods 8270C, 8270D, 8270E and 625.1 may be used to quantitate many neutral, acidic, and basic organic compounds that are soluble in methylene chloride. The compounds are separated using a gas chromatograph (GC) and detected using a mass selective detector (MSD). Such compounds include polynuclear aromatic hydrocarbons (PAH), chlorinated hydrocarbons, phthalate esters, pesticides, nitrosamines, haloethers, aldehydes, ketones, pyridines, and phenols. Appendix 1 lists the analytes typically analyzed using these methods. Please note that the list may change as new compounds are added or existing compounds are removed. Appendix 1 includes internal standards and surrogates.

4.3 The samples are prepared for analysis by gas chromatography/mass spectrometry (GC/MS) using the appropriate sample preparation method. The semivolatile compounds are introduced into a GC equipped with a narrow-bore fused silica capillary column. The GC column is temperature programmed to separate the analytes, which are then detected by the MSD. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact spectra of standards. Quantitation is accomplished by comparing the response of the quantitation ion relative to an internal standard using an appropriate calibration curve.

4.4 Appendix 5 outlines the procedure for dioxin screen analysis.

4.5 Appendix 6 outlines the procedure for SIM (Selective Ion Monitoring) analysis. Please note that SIM analysis is performed for some PAH analytes and the list may change as new compounds are added or existing compounds are removed.

4.6 Methods associated with this SOP may include USEPA SW-846 methods 8270C, 8270D, 8270E and USEPA method 625.1, as well as others not listed.

4.7 The practical quantitation limit (PQL) is the lowest level in the calibration curve. The PQL is the lowest level at which compounds may be accurately quantitated and is compound dependent. The calibration curve typically ranges from 10 µg/mL to 120 µg/mL. These ranges reflect instrument readings which are in µg/mL (ppm). The required detection limit (RDL) may be different from the PQL if a prep procedure is performed on the sample. It should be noted that the calibration range may vary between calibrations and instruments. Some compounds may have PQLs that are slightly higher or lower than those listed above. Additionally, newer instrumentation may make it possible to achieve even lower quantitation limits in the future.

4.8 Method detection limit studies (MDLs) are performed on an annual basis. MDLs are done for liquid and solid matrices. For more information regarding MDLs, refer to The Determination of Method Detection Limits, GL-LB-E-001.

5.0 METHOD SCOPE AND PERFORMANCE CHARACTERISTICS

Various performance characteristics associated with this method should be given special consideration. Benzidine and 3,3'-dichlorobenzidine can be subject to oxidative losses during solvent concentration. In addition, these two compounds often break down quickly when prepared in a mixture with other compounds. Hence, they may need to be prepared separately. Some of the pesticides are particularly prone to decomposition in the injection port if the temperature is too high or the liner becomes dirty. Various substituted phenols, in particular 4-nitrophenol, 2,4-dinitrophenol, and 4,6-dinitro-2-methylphenol are susceptible to breakdown and erratic chromatography. 2-nitroaniline, 3-nitroaniline, and 4-chloroaniline should be watched closely for poor responses and decomposition. Care should be taken when installing a new guard column to look for degradation and subsequent loss of pentachlorophenol. The guard column may need to be rinsed or cut prior to installation to prevent this from happening. Please see Appendix 7 for additional information on poor responders.

6.0 DEFINITIONS

6.1 Definitions specific to this SOP include:

6.2 Continuing Calibration Verification (CCV) Standard: An aliquot of reagent water or other blank matrix to which known quantities of the method analytes are added in the laboratory. The CCV is analyzed exactly like a sample periodically throughout the run sequence. Its purpose is to determine whether the analytical sequence is in control during sample analysis. It may be prepared from the same source as the calibration standards and is usually of varied concentration.

6.3 Limit of Detection (LOD): The lowest concentration level that can be determined by a single analysis and with a defined level of confidence to be statistically different from a blank. The LOD verification is typically spiked at two times the MDL.

6.4 Limit of Quantitation (LOQ): The lowest concentration level of the initial calibration curve used to quantitate an analyte. The LOQ verification is typically spiked at the PQL.

6.5 Lower Limit of Quantitation (LLOQ): The lowest concentration at which a target analyte can be reliably measured and reported. The LLOQ is ≥ the lowest point in the

calibration curve and represents a concentration at which both quantitative and qualitative requirements can be consistently demonstrated. The LLOQ is verified at least annually, but typically quarterly, as the LOQ verification. The verifications performed by extracting and analyzing an LCS spiked at 0.5 to 2 times the LOQ. The LLOQ verification is carried through the same preparation and analytical procedures as environmental samples and QC. It is recommended to analyze the LLOQ on every instrument where data are reported and this is the laboratory's normal protocol. Recovery of target analytes in the LLOQ are compared to in-house-statistically-derived limits. Concentrations in samples reported below the LLOQ and above the MDL are qualified as estimated.

6.6 Practical Quantitation Limit (PQL): The lowest level in the calibration curve. With the prep factor applied, the PQL is referred to as the effective PQL. The PQL is equivalent to the LOQ and the LLOQ.

6.7 Lab-wide used definitions can be found in GL-QS-B-001 the Quality Assurance Plan.

7.0 INTERFERENCES TO THE METHOD

7.1 Contaminants found in extraction glassware, solvents, and other sample processing hardware may jeopardize the integrity of this method.

7.2 Glassware must be scrupulously cleaned as soon as possible after extraction.

7.3 Contamination may also occur in the GC/MS system. High boiling materials tend to build up in the injection port and the front end of the column. The analyst should maintain a thorough working knowledge of keeping the injection port free of contamination, including changing out the septum, injection port liner, O-ring, ferrule, and gold seal. To eliminate build-up of high boiling material in the front end of the column, a guard column can be connected between the injection port and the analytical column and cut periodically. Guard columns typically have no phase and are typically 5 to 10 meters long.

7.4 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed between samples with solvent. If carryover is suspected, potentially impacted samples should be re-analyzed after any needed maintenance or cleaning has been done.

8.0 SAFETY PRECAUTIONS AND HAZARD WARNINGS

WARNING

METHYLENE CHLORIDE IS A SUSPECTED CARCINOGEN AND A KNOWN SKIN IRRITANT.

CONTACT WITH OXIDIZERS MAY GENERATE EXPLOSIVE MIXTURES.

PYRIDINE IS A FLAMMABLE COMPOUND AND IS TOXIC UPON INHALATION. PREVENT INHALATION BY USING PYRIDINE UNDER A HOOD.

PREVENT SKIN AND EYE CONTACT BY USING SPECIFIED PERSONAL PROTECTIVE EQUIPMENT WHEN MAKING STOCK REAGENTS.

WORK UNDER A HOOD TO PREVENT INHALATION WHEN MAKING STOCK REAGENTS.

8.1 Eye protection should be worn when handling samples, reagents, or standards.

8.2 Treat all chemicals and samples as potential health hazards and reduce exposure to these chemicals to the lowest level possible. GEL maintains a current awareness file of OSHA regulations regarding the safe handling of the chemicals in the laboratory as well as a reference file of Material Safety Data Sheets (MSDS). These documents and individual sample MSDS provided by clients are maintained in the laboratory.

8.3 Personal Protective Equipment (PPE)

8.3.1 Gloves should be worn when handling chemicals, solvents, and samples.

8.3.2 Analysts should prepare samples and standards under the hood.

8.4 Prior to handling radioactive samples, analysts must have had radiation safety training and must understand their full responsibilities in radioactive sample handling. Some general guidelines follow:

8.4.1 Proper PPE should be worn at all times when handling radioactive samples. Gloves, safety glasses, and a lab coat should be worn when handling radioactive samples. In addition, a disposable lab apron may be worn over the lab coat.

8.4.2 Protect counter tops with counter paper, or work from radioactive sample handling trays.

8.4.3 Post signs indicating radioactive samples are in the area.

8.4.4 Swipes of the counter tops should be taken upon completion of work. Deliver those swipes to the designated swipe count box.

8.4.5 Segregate radioactive wastes. Radioactive waste containers are obtained from Waste Management.

8.5 All samples, chemicals, extracts, and extraction residues must be transferred, delivered, and disposed of safely according to all related SOPs.

8.6 Radioactive and non-radioactive wastes are segregated in the waste satellite area.

8.7 Never leave gas cylinders unchained or untied.

8.8 In the event of an accident or medical emergency, call for help immediately. When time and safety permit, an accident report form should be completed.

8.9 Fire escape routes are posted in the lab, and all personnel should be familiar with them. In addition, fire safety equipment such as fire extinguishers is located in the lab. Training is available on the proper operation of this equipment.

8.10 Refer to SOP GL-LB-N-001 the Safety, Health and Chemical Hygiene Plan for additional general safety and health information pertaining to the laboratory.

9.0 CAUTION WARNINGS

The analyst must use care when operating and assembling instrumentation. Use caution when handling samples and reagents. Check to see that the gas chromatograph equipment is properly assembled and hooked up to the proper gas cylinder and power, by referencing appropriate reference manual.

10.0 APPARATUS AND MATERIALS; REAGENTS; EQUIPMENT AND INSTRUMENTS

10.1 Equipment associated with this method includes:

- 10.1.1 Gas tight syringes
- 10.1.2 Volumetric flasks
- 10.1.3 40 mL vials
- 10.1.4 Refrigerator and freezer
- 10.1.5 Mininert vials
- 10.1.6 2 mL autosampler vials
- 10.1.7 250 µL clear glass vial inserts
- 10.1.8 Teflon crimp tops
- 10.1.9 Crimper
- 10.1.10 Columns
- 10.1.11 Glass injection port liners
- 10.1.12 O-rings
- 10.1.13 Ferrules
- 10.1.14 Column cleaving tool
- 10.1.15 Septa
- 10.1.16 Guard column connectors

10.2 Reagents, chemicals, and standards

- 10.2.1 Source Standards: Source Standards are purchased directly from vendors and may be diluted to make stock, intermediate, or working standards. Source standards expire per the vendor expiration date or after one year from the date opened, whichever is shorter. Please reference GL-LB-E-007 for further information regarding standards and their preparation.
- 10.2.2 Internal Standard Solutions: The recommended internal standards are: 1,4-dichlorobenzene-d4, naphthalene-d8, acenaphthene-d10, phenanthrene-d10, chrysene-d12, and perylene-d12. Internal standard solutions are prepared from pure standard materials or purchased as certified solutions.
- 10.2.3 GC/MS Tuning Standard: A methylene chloride solution containing 50 ng/µL of decafluorotriphenylphosphine (DFTPP) should be prepared. The standard should also contain 50 ng/µL of pentachlorophenol, benzidine and 4,4'-DDT to verify injection port inertness and GC column performance.
- 10.2.4 Calibration Standards: Calibration standards are prepared at a minimum of five concentration levels. One of the calibration standards should be at a concentration near, but above, the method detection limit; the others should correspond to the expected range of compounds found in samples. Calibration standards may be purchased pre-mixed as source standards or may be prepared in the laboratory from certified source standards.

Calibration standards expire after a maximum of six months or the shortest vendor expiration date (whichever is sooner) and should be monitored frequently for signs of degradation.

10.2.5 Surrogate Standards: Surrogate recoveries are used to monitor unusual matrix effects, sample processing errors, and extraction efficiency. The recommended base/neutral surrogates are: 2-fluorobiphenyl, nitrobenzene-d5, and terphenyl-d14. These surrogates are added at a concentration of 50 µg/mL. The recommended acid surrogates are: 2-fluorophenol, 2,4,6-tribromophenol, and phenol-d5. These surrogates are added at a concentration of 100 µg/mL. Surrogates are added to each blank, laboratory control sample (LCS), matrix spike (MS), matrix spike duplicate (MSD) and sample during the extraction. Surrogate concentrations may vary, depending on the prep method. For example, method 3580 (waste dilution) requires surrogates at ten times the normal concentration. The surrogate typically used for the SIM PAH analysis is 5-alpha-androstane.

10.2.6 Laboratory Control Sample (LCS) and Matrix Spike (MS) Standards: The LCS and MS standards contain a representative list of target compounds. Depending on client and contract requirements, some or all of the spiking compounds may be monitored for recovery. In addition, some analytical protocols such as QSM, require that every target analyte be spiked and monitored. For TCLP samples, the matrix spiking standards are: hexachloroethane, nitrobenzene, hexachlorobutadiene, 2,4-dinitrotoluene, hexachlorobenzene, pyridine, 2-methylphenol, 3-methylphenol, 4-methylphenol, 2,4,6-trichlorophenol, 2,4,5-trichlorophenol, and pentachlorophenol, and 1,4-dichlorobenzene. When method 625.1. is referenced, the matrix spike should contain all target 625.1 compounds of interest. Both the acid and base compounds are typically added at 50 µg/mL, however spike concentrations may vary, depending on the prep method. Waste dilution prep (method 3580) requires elevated spike and surrogate concentrations to account for the dilution in the prep step.

10.2.7 Calibration Verification Standards (CCV): The CCV is prepared at a concentration that is near to the midpoint of the calibration curve. A CCV may be purchased as a source standard or prepared from other source standards.

10.3 Instrumentation

10.3.1 Gas Chromatograph: A gas chromatograph (GC) should be capable of temperature programming and suitable for split/splitless injections. Electronic pressure control (EPC) is recommended but not required. In most cases, an autosampler is used for injections. The laboratory utilizes Agilent 6890 and 7890 GCs and 5973, 5975 and 5977 MSDs. Agilent Chemstation/Enviroquant software is used for data processing and evaluation.

10.3.2 Suggested parameters for the autosampler:

Sample volume – 0.5 µL to 1 µL

Number of sample washes - 1

Solvent washes - 6

Sample viscosity wait - 0

Number of sample pumps - 6

Injection mode is 'Fast'.

10.3.3 The mass selective detector (MSD) should be capable of scanning from 35 to 500 AMU every 1 second or less, using a recommended electron energy of 70 volts in the electron impact ionization mode. The MSD must be capable of producing a mass spectrum that meets the criteria established by the EPA. A slightly polar fused silica capillary column, such as a J&W DB-5MS column or its equivalent, is recommended for use with this method.

A suggested temperature program follows:

Temperature 1	50° C
Time	1 min.
Rate	15° C/min.
Temperature 2	120° C
Rate	20°/min
Temperature 3	280° C
Rate	2° C/min
Temperature 4	295 ° C
Rate	20° C/min
Final Temperature	325° C
Time	4.5 min

Run Time:	Approximately 27 minutes or until Benzo (a,e) pyrene elutes.
Scan Start Time:	2.0 (or prior to 2-ethoxyethanol)
Splitless Valve Time:	1.0
Number of A10 Samples:	2
MS Threshold:	100 counts
Mass Range:	35 - 500 AMU

NOTE: These instrument conditions and rates are guidelines which may change.

11.0 SAMPLE HANDLING AND PRESERVATION REQUIREMENTS

11.1 Sample extracts have a 40-day holding time from the date of extraction.

11.2 Sample extracts are delivered from the prep lab to the semivolatiles lab and are stored in a freezer at approximately -10° C or less. QSM requires -10° to -20° C. The extracts are usually grouped according to batches and are accompanied by the batch pull sheet and other pertinent paperwork.

11.3 Custody of samples is monitored using the AlphaLIMS sample tracking system. Each analyst should scan the samples he/she plans to run into his/her custody prior to analysis.

11.4 All sample extracts should be treated with caution as potential health hazards. Refer to Section 8.0 on safety.

12.0 SAMPLE PREPARATION TECHNIQUES

12.1 Before extracts can be analyzed on the instrument, they must first be transferred to an autosampler vial and have internal standards added. A determination must also be made as to whether the extract should be diluted. The decision to dilute a sample extract is based on a number of factors: sample screening, historical data about the sample or sample site, the appearance of the extract (color, viscosity, odor, turbidity, etc.), or regulatory considerations. The experience of the analyst is invaluable in making this determination.

NOTE: Sample extracts may contain extraneous material, multiple layers or sediment. Sediment or other extraneous material cannot be injected into a GC column, and therefore, is excluded from the aliquot taken from the vial. Multiple layers are treated on a case-by-case basis. If the extract can be homogenized, then a uniform sample is achieved. If the extract remains bi-phasic, the PM and client are contacted for further guidance.

12.2 If a sample is to be analyzed without dilution ('neat'), internal standard solution is added to the autosampler vial with sample at a ratio of 1:50. For example, if 5 µL of a 2000 µg/mL internal standard solution is added to an autosampler vial using a syringe, 250 µL of the sample extract is then added to the same vial. A cap is then placed on the vial and secured by crimping. This procedure may vary slightly depending on the specific analytical protocol being used (i.e. CLP).

12.3 If samples are diluted, the dilution is made using methylene chloride or appropriate solvent. Again, 5 µL of the 2000 µg/mL internal standards solution is added to the autosampler vial. If the sample were being diluted 1:5, 50 µL of the sample extract would be added along with 200 µL of methylene chloride.

12.4 Once samples are prepped, they are ready to be injected onto the GC/MS. Usually, an autosampler, such as the Agilent 7683, is used to inject standards and extracts on the instrument.

12.5 Some samples cannot be extracted and concentrated using typical prep procedures. Oils and solvents that are miscible in methylene chloride are common examples. In these cases, method 3580 (waste dilution) may be used.

13.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

13.1 Preventative maintenance on a GC/MS system involves the following basic areas:

13.1.1 Vacuum pumps for the analyzer need a change of oil about every year or when system performance indicates it is needed.

13.1.2 The GC injection port is cleaned as needed, usually every day or every other day. It is recommended that the septum (if there is one) and injection port liner be replaced at the time of cleaning. A guard column is often used to prevent high boiling contaminants from reaching the analytical column. A loop or two of the guard column should be cleaved off when cleaning. Additionally, the gold plated seal should be cleaned.

13.1.3 Analyzer maintenance is usage dependent. The type and quantity of samples that have been injected determine the frequency of ion source cleaning and electron multiplier replacement.

13.1.4 Autosampler maintenance is primarily that of cleanliness. Most autosamplers need their moving parts to be clean and lightly lubricated. The most frequent corrective maintenance is that of changing the syringe.

13.1.5 Instrument maintenance logs are kept with each instrument and serve as a record of all the maintenance that has been done on the instrument.

13.2 Non-Routine Maintenance Procedures (Special, Operational or Failure Mode Maintenance)

13.2.1 Service is provided to the instrument via the analyst, the in-house instrument service engineer, or a technical support specialist from the manufacturer. When instrument failure occurs, different parts of the instrument are isolated to determine the root cause. For example, the injection port may be capped off if a leak is suspected to prove the leak is/is not coming from that source. Instrument maintenance logbooks are kept for each instrument detailing the type of maintenance performed on the instrument and when it was performed.

13.2.2 Analytical columns are replaced when the existing column shows signs of excessive degradation or the inability to properly resolve chromatographic peaks. Excessive peak tailing, poor responses, and baseline disturbances may also indicate that the column needs to be replaced. When the analytical column is replaced, the instrument is recalibrated.

14.0 PREPARATION OF STANDARD SOLUTIONS AND QUALITY CONTROL SAMPLES

14.1 Source standards are purchased as certified mixtures or as neat chemicals. Documentation of the standard's quality and traceability should be provided from the vendor. This documentation is scanned and linked to the standard in the LIMS database. Standards may be purchased from certified vendors.

14.2 Source standards are assigned a unique code number for the purposes of traceability. The standard, along with its code, is recorded in AlphaLIMS. AlphaLIMS can be used to generate a label that is affixed to the standards container. Source standards are good for one year after opening or from the vendor’s expiration date, whichever is shorter.

14.3 Intermediate, and working standards are likewise assigned a unique code number and recorded in AlphaLIMS. These standards are valid for six months, or from the vendor's expiration date, whichever is shorter.

15.0 INSTRUMENT CALIBRATION

15.1 Initial Calibration. Prior to running a multi-level calibration, take precautions to ensure that the instrument is clean and functioning properly. An instrument blank should be analyzed with each ICAL tune window. A minimum of five calibration levels is run for each analyte. Typically, the calibration levels range from 10 µg/mL to 120 µg/mL. However, these levels may vary among compounds. Special care is taken to ensure that saturation or overloading is not taking place in the higher level standards. If saturation is noted, take appropriate corrective action, such as manually re-tuning the instrument or lowering the multiplier voltage.

15.2 In some cases the upper level(s) of the calibration may be removed in order to meet method criteria for single compounds. This practice results in a narrower calibration range. Individual calibration levels may be analyzed within the same tune window if no samples have yet been analyzed. Target analytes detected above the highest calibration level are re-analyzed at a dilution to bring the elevated concentrations within the instrument's calibration range. The low standard representing the PQL is not dropped. Please note that this practice does not represent "cherry picking," which is acknowledged as an unacceptable laboratory practice.

15.3 Because some of the target analytes may decompose or react when mixed with other analytes, several calibration mixes are used. These mixes are prepared at differing concentrations. The various mixes are used to prevent chemical degradation. All of the mixes are analyzed at the same time. The analytical sequence may lapse over two days.

15.4 Each instrument should be hardware-tuned to meet EPA criteria for DFTPP before any analyses, including the initial calibration, can begin.

15.4.1 Calculate response factors (RF) for each compound relative to one of the internal standards. The internal standard selected for the calculation of the RF for a compound is the internal standard that has a retention time closest to the compound being measured. The RF is calculated as follows:

$$RF = (A_X C_{IS}) / (A_{IS} C_X)$$

Where:

A_X = Area of the characteristic ion for the compound being measured.

A_{IS} = Area of the characteristic ion for the specific internal standard.

C_{IS} = Concentration of the specific internal standard.

C_X = Concentration of the compound being measured.

15.4.2 The average RF must be calculated for each compound. A system performance check should be made before this calibration curve is used. Four compounds

(System Performance Check Compounds, or SPCCs) are checked for a minimum average response factor. These compounds are N-nitroso-di-n-propylamine, hexachlorocyclopentadiene, 2,4-dinitrophenol, and 4-nitrophenol. The minimum acceptable average RF for these compounds is 0.0500. These compounds typically have RFs of 0.05 to 0.10. These compounds are used to check for standard degradation, system cleanliness, and active sites in the system. Methods 8270D and 8270E do not designate SPCCs. Rather, it is recommended that a minimum RF for the most common target analytes be demonstrated for each calibration level (refer to Appendix 9 for a list of RFs) as a means to ensure that these compounds are recovering as expected. These methods also note that meeting the minimum RF criteria for the lowest calibration standard is critical to demonstrating appropriate sensitivity. Due to the large number of analytes that may be analyzed by these method, some analytes will fail to meet these criteria. In those cases, data may still be reported if the compounds are not project-critical. Otherwise, the data are qualified as estimated or the sample is re-analyzed on an instrument that passes the response factor criteria. Results should not be reported if any compounds of interest for a project fail the minimum response factor criteria for South Carolina samples. All compounds should have a minimum RF of at least 0.01 and the failures should be addressed in the case narrative.

15.5 The percent relative standard deviation (%RSD) should be less than 15% for each compound in method 8270C and 35% for method 625.1. However, the %RSD for each individual Calibration Check Compound (CCC) must be less than 30%, in method 8270C.

$$\%RSD = \frac{SD}{\bar{x}} \times 100$$

Where:

RSD = relative standard deviation

$\bar{x}$ = mean of 5 or more initial RFs for a compound

SD = standard deviation of average RFs for a compound

$$SD = \sqrt{\sum_{c=1}^{n} \frac{\left(x_1 - \bar{x}\right)^2}{N-1}}$$

The following compounds are CCCs: phenol; 1,4-dichloro-benzene; 2-nitrophenol; 2,4-dichlorophenol; hexachlorobutadiene; 4-chloro-3-methylphenol; 2,4,6-trichlorophenol; acenaphthene; diphenylamine; pentachlorophenol; fluoranthene; di-n-octylphthalate; and benzo(a)pyrene. Methods 8270D and 8270E do not designate CCCs. Rather, all target analytes should have calculated RSDs of ≤ 20%. If more than 10% of the compounds included in the initial calibration exceed the 20% RSD limit and do not meet the minimum correlation coefficient (0.99), then the chromatographic system is

considered too reactive for analysis to begin and maintenance and re-calibration may be required.

15.6 If the %RSD of any compound is 15% or less (20% for 8270D and 8270E), then the relative response factor is assumed to be constant over the calibration range and the average relative response factor may be used for quantitation. If the %RSD for any compound is greater than 15% (20% for 8270D and 8270E), a calibration curve of area ratio (A/A_{IS}) versus concentration using the first regression fit should be constructed. Certain compounds are known poor responders and may prove to be difficult to calibrate. Please refer to Appendix 7 for a list of these analytes.

15.7 If the analyst chooses to use linear regression, he/she must not force the calibration line through the origin.

15.7.1 Make certain that the instrument response is treated as the dependent variable (y) and the concentration as the independent variable (x). The regression will produce the slope and intercept terms for a linear equation in the form:

$$y = ax + b$$

Where:

y = instrument response

a = slope of line (also called the "coefficient of x")

x = concentration of the calibration standard

b = the intercept

15.7.2 The analyst should not force the line through the origin, but have the intercept calculated from the five or more data points. Otherwise, the problems noted with the RSD value will occur, i.e., a line through the origin will not meet the QC specifications. In addition, do not include the origin (0, 0) as a sixth calibration point.

15.7.3 The regression calculation will generate a coefficient of determination (r^2) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r^2 should be not less than 0.99. (The correlation coefficient is r and must be greater than 0.990.) The calculated intercept value needs to be evaluated before reporting sample results.

15.7.4 A positive value for the intercept indicates that there is some threshold instrument response that is the limiting factor in establishing linearity. A negative intercept value can be transformed into an x-intercept value that represents a threshold concentration that is the limitation. If the intercept is positive, then as a general rule, results where the instrument response is less than three times (3x) the intercept value may be unreliable. This will afford some protection against false positive results. If the intercept is negative, results below the concentration of the lowest concentration calibration standard may be unreliable.

Some clients require further evaluation when an analyte is calibrated using a linear regression curve. In these cases, the intercept value is multiplied by the ISTD concentration. This value is then compared to the MDL (the lower of the soil and water MDLs is used). If the calculated value is > 3x the MDL, then the curve is changed to weighted linear; r^2 should still be > 0.99.
When calculating the calibration curves using the linear regression model in 8270D and 8270E, a minimum quantitation check of the lowest calibration standard should be performed. The lowest calibration standard is re-quantitated as sample and evaluated. The re-calculated concentration of the low calibration point should be within 30% of the standard's true concentration for method 8270D and 50 % for method 8270E. Other recovery criteria may be applicable depending on the project-specific criteria or QAP. If the minimum quantitation check criteria are not met the analyst may take corrective action by re-defining the lower limit of quantitation and/or reporting the "out of control" target analytes as estimated when the concentration is at or near the lowest calibration point. The laboratory must be aware that raising the lower limit of quantitation may not be suitable to meet the project data quality objectives or permit limits where applicable. Other corrective actions, such as instrument maintenance and re-calibrating may be used instead of raising the quantitation limit or qualifying data for all samples analyzed after the calibration curve.

15.7.5 In calculating the sample concentrations, the regression equation is rearranged to solve for the concentration (x) as shown below:

$$x = \frac{(y - b)}{a}$$

15.7.6 Method 8000D outlines two procedures that may be used to determine calibration function acceptability for linear and non-linear curves. The calibration data are refitted back to the calibration model. % Error and Relative Standard Error (RSE) evaluate the difference between the measured amount and the true amount (or concentration). % Error is determined as follows:

$$\%\,Error = \frac{x_i - x'_i}{x_i} \times 100$$

Where:

x'_i = Measured amount of analyte at calibration level *i*, in mass or concentration units

x_i = True amount of analyte at calibration level *i*, in mass or concentration units.

15.7.7 Percent error between the calculated and expected amounts of an analyte should be ≤30% for all standards and ≤ 50% for the lowest calibration level.

15.7.8 Relative Standard Error is calculated as follows:

$$RSE = 100 \times \sqrt{\sum_{i=1}^{n} \left[\frac{x_i' - x_i}{x_i} \right]^2 \Big/ (n - p)}$$

Where:

x_i = True amount of analyte in calibration level i, in mass or concentration units.

x'_i = Measured amount of analyte in calibration level i, in mass or concentration units.

p = Number of terms in the fitting equation
(average = 1, linear 2, quadratic =3, cubic =4)

n = Number of calibration points

15.7.9 The RSE acceptance limit criterion is the same as the RSD Limit for CF or RF in the determinative method. If the RSD limit is not defined in the determinative method, the limit should be set at ≤20% for well performing compounds and ≤30% for poor responders. Please see the Appendix for a list of demonstrated poor responders.

15.8 Following calibration, an initial calibration verification (ICV) standard is analyzed. If available, the ICV should be made using standards from a vendor other than the one used for the calibration standards. If not available, a second lot from the same vendor may be used for verification. The criteria for the ICV are the same in 8270C and 625.1 as for the daily CCV (refer to section below). 8270D and 8270E requires that the ICV should recover within 30% of the expected concentration. An alternative recovery limit may be appropriate based on the project-specific data quality objectives. Quantitative sample analysis should not proceed for those analytes that fail the second source standard ICV. Any results reported for these compounds would be considered estimated values.

15.9 Calibration Check Standards

15.9.1 The initial calibration curve for each compound of interest must be checked and verified once every 12 hours of analysis time for all methods. This is accomplished by analyzing a calibration standard that is at a concentration near the midpoint concentration for the working range of the GC/MS.

15.9.2 System Performance Check Compounds (SPCCs): A system performance check for 8270C must be made every 12 hours. If the SPCC criteria are met, a comparison of response factors is made for all compounds. This is the same

check that is applied during the initial calibration. If the minimum response factors are not met, the system must be evaluated and corrective action must be taken before sample analysis begins. The minimum response factor for semivolatile SPCCs is 0.05. Some possible problems are standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. Methods 8270D and 8270E do not designate SPCCs. Rather, minimum response factor (RF) criteria should be met. See Appendix 9 for RF criteria.

15.9.3 Calibration Check Compounds (CCCs): After the system performance check is met, CCCs are used to check the validity of the initial calibration in method 8270C. Calculate the percent difference using:

$$\% \text{ Difference} = \frac{\left|\overline{RF}_i - RF_c\right|}{\overline{RF}_i} \times 100$$

Where:

$\overline{RF}_i$ = average response factor from initial calibration

RF_c = response factor from current verification check standard

15.9.3.1 If the percent difference for each CCC is less than 20%, the initial calibration is assumed to be valid. If the criterion is not met (>20% difference), for any one CCC, corrective action should be taken. Problems similar to those listed under SPCCs could affect this criterion. If no source of the problem can be determined after corrective action has been taken, a new calibration may need to be generated. This criterion must be met before quantitative sample analysis begins. For method 625.1 analyses, the %D for all requested compounds of interest listed in Table 6 of the method must be 20% or less.

15.9.3.2 If one or more of the CCCs fail to meet criteria corrective action should be taken. This may require analyzing another calibration verification standard, or the instrument may require cleaning or additional maintenance. If the CCV continues to fail, a new calibration may have to be analyzed. When CCC criteria are not met, samples may still be analyzed, providing that all of the target compounds have a %D value $\leq$ 20%. Target analytes that are not CCCs should have a %D of $\leq$ 60 % in the daily check standard. If these criteria are not met, another CCV should be analyzed. In some cases, client specific criteria may be used provided they are tighter than the %D of $\leq$ 60% criteria. Exceptions may be taken to this and include cases where the analyte is biased high and there are no detects in the associated samples. Also, some analytes are known poor responders and may have to be evaluated on a case by

case basis. These analytes include benzoic acid, 2,4-dinitrophenol, the substituted anilines, benzidine, 3,3'-dichlorobenzidine, p-phenylenediamine, hexachlorophene, p-benzoquinone, n-decane, n-octadecane, atrazine, and others. In these cases, the high %D values need to be clearly documented and communicated to the client.

15.9.3.3 For 8270D and 8270E evaluation of the CCV, each of the target analytes in the calibration verification standard should meet the minimum response factor criteria as noted in Appendix 9. These criteria are particularly important when the common target analytes are also project-required compounds. This is the same check that is applied during the initial calibration.

15.9.3.4 If the minimum response factors are not met, the system should be evaluated, and corrective action should be taken before sample analysis begins. Possible problems include standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system.

15.9.3.5 Problems similar to those listed under initial calibration could affect the ability to pass the calibration verification standard analysis. If the problem cannot be corrected by other measures, a new initial calibration should be generated. The calibration verification criteria must be met before sample analysis begins.

15.9.4 The internal standard (ISTD) responses and retention times in each calibration check verification standard (CCV) should be evaluated against the mid-level standard from the calibration curve of the corresponding mix. The responses in the CCV should be within +100% and -50% of the responses in the mid-level calibration standard. The retention times should be within a half-minute of those in the mid-level standard.

15.10 If continuing calibration fails, the analyst determines why the check failed (i.e., instrument or standards) and makes the appropriate corrective action. Samples cannot be analyzed until the validity of the calibration has been verified.

15.11 For method 8270D and 8270E, if the %D (difference or drift) for a target analyte is less than or equal to 20%, then the initial calibration for that compound is assumed to be valid. Due to the large number of compounds that may be analyzed by this method, it is expected that some compounds will fail to meet the criterion. If more than 20% of the compounds included in the initial calibration do not meet the criterion, then corrective action must be taken prior to analysis of samples. In cases where compounds fail (total less than 20% in the calibration) they may still be reported as nondetects provided that it can be demonstrated that there was adequate sensitivity to detect the compound at the applicable quantitation limit. Low level sensitivity may be evaluated by examining the lowest calibration level and the daily

CCV response factors. Depending on client requirements and regulatory agencies, the data may be reported as estimated or the sample may need to be re-analyzed for those compounds not meeting the 20 % criteria on a passing instrument. It is not acceptable to report estimated results for compliance samples.

15.12 The GC/MS should meet certain performance requirements, including DFTPP tune, initial calibration, calibration check standard, internal standard response factors, retention times, surrogate recoveries, and correct ion spectra, as well as others. These criteria are outlined in other parts of this SOP. When samples or QC fail to pass, it is important to verify whether the instrument has successfully passed all of the performance criteria. In this way, it can be determined if the samples or QC in question need to be re-extracted or if corrective action needs to be taken with the instrument.

15.13 Resolution of closely eluting structural isomers with similar mass spectra should be evaluated for the mid-point calibration standard and subsequent continuing calibration standards. The height of the valley between two isomer peaks must be less than 50% of the average of the two peak's heights (i.e. percent valley greater than 50%) for Method 8270C if closely eluting isomers are to be reported. For Method 8270D and 8270E, the percent valley must be greater than 75%.

16.0 ANALYST AND METHOD VALIDATION REQUIREMENTS

16.1 To establish the ability to generate acceptable accuracy and precision, the analyst should perform an "analyst validation study", Initial Demonstration of Proficiency, or QC Startup. Four LCS standards are extracted and analyzed. Calculate the average recovery and the standard deviation of the recovery for each analyte of interest using the four results. Table 6 of method 8270C (Appendix 2) may be used for comparison. If the validation study fails for one or more of the compounds, then the study must be repeated for those compounds which failed. For method 8270D and 8270E, SPC derived limits will be used.

16.2 For method 625.1, a quality control check sample concentrate is required containing each parameter of interest at a concentration of 100 ug/mL. Multiple solutions may be required. Calculate the average recovery in ug/L and the standard deviation of the recovery in ug/L and the standard deviation of the recovery(s) in ug/L for each parameter using the four results. For each parameter compare s and x with the corresponding acceptance criteria precision and accuracy, respectively, found in Table 6. If s and x meet the acceptance criteria, analysis of samples can begin. If an individual analyte does not meet the acceptance limits, the test must be repeated for that analyte.

17.0 ANALYSIS PROCEDURES AND INSTRUMENTAL OPERATION

17.1 DFTPP tune.

During each tune period, the GC/MS system is checked to see if acceptable performance criteria are achieved. For methods 8270C, 8270D and 625.1 the tune frequency is defined per 12 hours. Method 8270E requires that a tune be analyzed

with each calibration. A sample of 50 ng of decafluorotriphenylphosphine (DFTPP) is injected into the GC/MS system. (Refer to Appendix 3.) Compounds benzidine and pentachlorophenol are included in the DFTPP tune, and the tailing factor is calculated and checked daily. For method 8270C, DDT breakdown should be ≤ 20% and the tailing factors for pentachlorophenol and benzidine should be < 5 and < 3 respectively. The tailing factor criteria for 625.1 is <2 benzidine and pentachlorphenol. For Methods 8270D and 8270E, DDT breakdown should be ≤ 20% and the tailing factors for both pentachlorophenol and benzidine should be < 2.

$$\% \text{ breakdown of DDT} = \frac{\text{sum of degradation peak areas (DDD + DDE)}}{\text{sum of all peak areas (DDT + DDE + DDD)}} \times 100$$

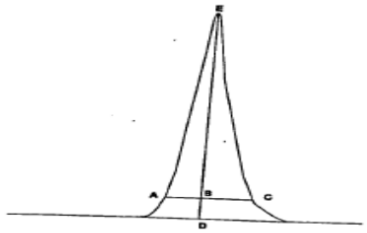
Tailing = BC/AB

Peak Height = DE

10% Peak Height = BD

Peak Width at 10% Peak Height = AC

Apex = E



17.1.1 If the tune does not pass, the GC/MS may need to be manually tuned to achieve the appropriate abundance. The suggested relative abundances are:

Mass 69 100%

Mass 219 35-45%

Mass 502 > 0.8%, but < 2%

These suggestions are meant to serve as guidelines only. Analysts should consult the manufacturer's guidelines and specifications.

17.2 Data Interpretation

17.2.1 Qualitative Analysis

17.2.1.1 The qualitative identification of compounds using this SOP is based on retention time and the comparison of the sample mass spectrum with the mass spectrum of a standard reference of the suspected compound. The characteristic ions from the reference mass

spectrum are defined as the three ions of greatest relative intensity or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. At least two ions (m/z) must be present to qualitatively identify a compound.

17.2.1.2 The relative retention time (RRT) of the sample component should be within ± 0.06 RRT units of the RRT of the standard component. Particular attention should be paid to the following compounds for possible coelution and misidentification: 1,2-, 1,3-, 1,4-dichloro-benzene, 2,4,5- ,2,4,6- trichlorophenol , benzo(b)fluoranthene, and benzo(k)fluoranthene, phenanthrene and anthracene, fluoranthene, benzo(a) anthracene, chrysene, and pyrene, as well as others.

17.2.1.3 The relative intensities of the characteristic ions should agree within 30% of the relative intensities of these ions in the reference spectrum. When analytes of different m/z ratios coelute, the identification criteria can be met, but each analyte spectrum will contain extraneous ions contributed by the coeluting compound.

17.2.1.4 For samples containing components not associated with the calibration standards, a library search may be performed for the purpose of tentative identification. **NOTE:** Non-requested calibrated analytes detected in a client sample may be reported on the Form 1 or certificate of analysis as TICs. A library search of a blank for that day must be performed for contamination check. Guidelines for tentative identification procedures are found in Appendix 4.

18.0 CALCULATIONS AND DATA REDUCTION METHODS

18.1 Quantitative Analysis

18.1.1 When a compound has been identified, the quantitation of that compound will be based on the integrated abundance from the EICP of the primary characteristic ion. Quantitation will take place using the internal standard technique. The internal standard used shall be the one nearest the retention time of that of a given compound.

18.1.2 The concentration of each identified compound ion in the sample is calculated as follows if using a response factor:

Liquid Matrix:

Concentration

$$(\mu g/L) = \{[(Ax)(Is)(Vt)]/[(Ais)(\overline{RF})(Vo)(Vi)]\}$$

Where:

Ax = Area of characteristic ion for compound being measured

Is = Amount of internal standard injected (ng).

Vt = Volume of total extract, taking into account dilutions (i.e., a 1 to 10 dilution of a 1 mL extract will mean Vt = 10,000 µL.)

Ais = Area of characteristic ion for the internal standard.

RF = Average response factor for compound being measured.

Vo = Volume of water extracted (mL).

Vi = Volume of extract injected (µL).

Sediment/Soil Sludge (on a dry-weight basis) and Waste (normally on a wet-weight basis):

Concentration

$$(\mu g/kg) = \{[(Ax)(Is)(Vt) / [(Ais)(RF)(Vi)(Ws)(D)]\}$$

Where:

Ax, Ix, Vt, Ais, RF, Vi = Same as for water matrix

Ws = Weight of sample extracted or diluted in grams.

D = (100% moisture in sample)/100, or 1 for wet-weight basis.

18.1.3 Where applicable, an estimate of concentration for noncalibrated components in the sample should be made. The formulas given above should be used with the following modifications: the areas Ax and Ais should be from the total ion chromatograms and the RF for the compound should be assumed to be 1. The concentration obtained should be reported indicating (1) that the value is an estimate and (2) which internal standard was used to determine concentration. Use the nearest internal standard free of interferences.

18.1.4 Quantitation of multicomponent compounds (i.e. Aroclors, toxaphene, and chlordane) is beyond the scope of methods associated with this SOP.

18.2 When a target analyte is detected at a concentration above the calibration range, the sample is diluted to bring the concentration within the calibration range. All efforts are made to analyze samples undiluted or at the lowest possible dilutions. If a sample is initially diluted and no target analytes are detected, the lab will try to re-analyze the sample at a lower dilution or undiluted. In some cases, matrix interference or the presence of non-target analytes may prevent analysis at low or undiluted concentrations.

19.0 DATA RECORDING

19.1 Data are evaluated qualitatively and quantitatively as above using analytical software program such as Chemistation/Environquant.

19.2 Data are reviewed initialed, and annotated electronically in LIMS. Reasons for manual integrations or removal if false positives are documented as well.

19.3 Additional supporting documentation, LCS, MS/MSD, and surrogate recovery reports; ISTD recovery report; run logs; calibration summary; calibration history; and TIC search data are with the data.

20.0 QUALITY CONTROL REQUIREMENTS

20.1 Blanks:

20.1.1 A blank is extracted with each analytical batch or every 20 samples to demonstrate that interferences from the analytical system, glassware, and reagents are under control. Blanks are carried through all stages of sample preparation and analysis.

20.1.2 Typically, a blank, laboratory control sample (LCS), matrix spike (MS), and matrix spike duplicate (MSD) are extracted and analyzed with each prep batch. However, this may vary depending on such factors as sample availability and client requirements. Other client requirements may include a laboratory control sample duplicate (LCSD).

20.2 Laboratory Control Samples and Matrix Spikes

20.2.1 The lab typically spikes the analytes listed in the TCL list. In other cases, clients may require that every target analyte be spiked. Usually compounds are spiked at a concentration of 50 µg/mL. Concentrations may vary depending on the specific method or client requirements. Method 625.1 requires that all analytes of interest are spiked. Refer to Method 625.1 for a complete list of these compounds and concentrations. TCLP spiking compounds are:

1,4-dichlorobenzene
pyridine
o-cresol
hexachloroethane
m,p-cresols
nitrobenzene
hexachlorobutadiene
2,4,6-trichlorophenol
2,4,5-trichlorophenol
2,4-dinitrotoluene
hexachlorobenzene
pentachlorophenol

20.2.2 To determine recovery limits for LCSs, MSs, and MSDs, the following procedure is used. For each LCS, MS, and MSD, the percent recovery is calculated. Once a minimum of twenty data points is obtained, the average percent recovery (P) and the standard deviation (s) are calculated for each

spiking analyte. For a given matrix, upper and lower control limits are calculated as follows:

Upper Control Limit (UCL) = P + 3s

Lower Control Limit (LCL) = P - 3s

Control limits, also referred to as SPC or Statistical Process Control limits are typically generated semi-annually. For method 625.1, the LCS, MS/MSD recoveries must pass the "P1", "P2" and "RPD" criteria found in table 6 of that method.

20.2.3 If recovery is not within these limits, the data may need to be re-checked for errors, or the LCS, MS, MSD may be re-analyzed or re-extracted. In addition, the instrumentation may be checked for performance problems or the QC extracts may be checked to insure that the final volume was accurately measured and recorded. If the LCS fails to meet acceptance criteria due to low recovery, the associated samples may have to be re-extracted and re-analyzed when possible. If one or more recoveries are high in the LCS and these analytes are not detected in the samples, the event should be documented and data may be reported. If the MS and MSD both fail due to matrix interference and/or dilution, data may be reported provided the associated LCS passes acceptance criteria.

NOTE: Many clients have contract specific criteria that must be considered when evaluating recovery of the Quality Control samples.

20.2.4 The recovery limits are entered into the laboratory information management system (AlphaLIMS) at the time of certification.

20.2.5 Surrogates are added to all samples and QC at the time of prep. The method recommended surrogates are:

2-fluorophenol

phenol-d6

nitrobenzene-d5

2-fluorobiphenyl

2,4,6-tribromophenol

p-terphenyl-d14

Acid surrogates are usually spiked at a concentration of 100 µg/mL and base surrogates at a concentration of 50 µg/mL. The concentration may vary depending on the matrix and historical information of samples. Other analytical protocols, such as CLP, may require the use of different or additional surrogates.

20.2.6 The percent recovery of each surrogate is calculated. In cases where a sample or QC sample is diluted, the surrogates may be diluted out of the calibration range, and a calculation need not be performed. Periodically, a minimum of

twenty data points is collected in order to trend and update surrogate recovery limits. The average percent recovery (P) and the standard deviation of the percent recovery (s) are calculated for each of the surrogates. For solid and liquid matrices, the upper and lower control limits are calculated as follows:

Upper control limit (UCL) = P + 3s

Lower control limit (LCL) = P - 3s

20.2.7 The internal standard responses (ISTD) and retention times in the daily check standard are compared with those in the midpoint standard of the most recent calibration curve. ISTD responses must agree within + 100% and – 50% of those found in the midpoint standard. The retention times must agree within ± 0.5 minutes. Subsequent daily runs (check standards, QC and samples) are compared to the daily CCV and must meet the same criteria. For QSM samples, compare the ISTD for samples and QC to the mid-level calibration standard.

20.3 Handling Non-Compliant Data

While every attempt is made to satisfy all method and client requirements, instances when non-compliant data are reported. For example, it may not be possible to re-extract a sample due to lack of availability or a re-extraction may yield non-compliant results or holding times may be exceeded. Samples analyzed by methods 8270C, 8270D, 8270E and 625.1, in particular, are subject to non-compliance due to the extensive list of possible target analytes and the great variability in their chemistries. These methods, along with other analytical protocols and programs, such as QSM describe how to handle, qualify and report non-compliant data. Appendix 8 of this SOP outline some of the more common methodologies and their requirements for reporting non-compliant data. Analysts should approach these appendices for guidance, along with the body of this SOP, client-specific contracts, QSM, as well as the methods (such as SW-846). Analysts are also encouraged to seek guidance from other analysts, senior chemists, data validators, group leaders and the quality department as circumstances may dictate.

21.0 DATA REVIEW, VALIDATION, AND APPROVAL PROCEDURES

21.1 A review process is used to insure the quality of the data. Raw data are peer reviewed by a second analyst or a validator. When the analyst is satisfied that the data have been entered correctly, a data report is generated from AlphaLIMS. The AlphaLIMS report along with the raw data and supporting documentation, such as a run log and case narrative, are submitted for review to the data validator or another experienced analyst. The reviewer goes through the raw data as if he/she was working it up for the first time and verifies that they are correct. In addition, he/she must make sure that the data have been correctly entered into AlphaLIMS. AlphaLIMS reports may be self-reviewed. If errors are discovered in either the raw data or the AlphaLIMS report, then the two analysts should discuss the differences and how best to resolve them. Other analysts may be called upon to offer their advice or opinions as well. In some cases, the peer review

process may uncover errors that lead to a sample being re-extracted or re-run. All anomalies must be discussed in the case narrative.

21.2 Once the data review has been completed by the reviewer, the batch is returned to the analyst for corrections (if applicable) and the status is updated from REVW to DONE in AlphaLIMS.

22.0 DATA TRANSMITTAL

Data may be transmitted automatically to AlphaLIMS. This automatic "upload" procedure may be activated prior to data review or after data review is complete. In either case, the data recorded in AlphaLIMS are checked by the analyst for accuracy and completeness.

23.0 RECORDS MANAGEMENT AND DOCUMENT CONTROL

23.1 Run logs are generated for each instrument each day that the instrument is run. These run logs serve as records of what is run on the instrument, including samples, QC, calibrations, tunes, etc. Additional information is provided in the run log, including the analyst's initials, run date and time, and file name.

23.2 Raw data are stored electronically. Data are archived daily onto a serve.

24.0 LABORATORY WASTE HANDLING AND DISPOSAL

Sample extracts that have been run are temporarily stored in case they have to be reanalyzed. Once space is no longer available to keep them in the lab, they are moved to Waste Disposal where they are handled and disposed in accordance with the Laboratory Waste Management Plan, GL-LB-G-001.

25.0 REFERENCES

25.1 Test Methods for Evaluating Solid Waste: Laboratory Manual Physical/ Chemical Methods, Volume 1B, SW-846, 3rd Edition, Nov. 1986. Method 8270C, "Semivolatile Organic Compounds by Gas Chromatograph/ Mass Spectroscopy (GC/MS): Capillary Column Technique," Rev. 3, Dec. 1996. USEPA, Office of Solid Waste and Emergency Response, Washington, DC 20460.

25.2 Federal Register, 40 CFR Part 136, December 22, 2000, 81295, contains Attachment 1 to Method 625.1.

25.3 Test Methods for Evaluating Solid Waste: Laboratory Manual Physical/ Chemical Methods, Volume 1B, SW-846, 3rd Edition, Feb 2007. Method 8270D, "Semivolatile Organic Compounds by Gas Chromatograph/ Mass Spectroscopy (GC/MS): Capillary Column Technique," Rev. 4, Feb. 2007. USEPA, Office of Solid Waste and Emergency Response, Washington, DC 20460.

25.4 Test Methods for Evaluating Solid Waste: Laboratory Manual Physical/ Chemical Methods, Volume 1B, SW-846, 3rd Edition, Feb 2007. Method 8270E, "Semivolatile Organic Compounds by Gas Chromatograph/ Mass Spectroscopy (GC/MS): Capillary Column Technique," Rev 6, June 2018. USEPA, Office of Solid Waste and Emergency Response, Washington, DC 20460.

25.5 Dept. of Defense (DOD), Dept. of Energy (DOE) Consolidated Quality Systems Manual (QSM) Version 5.3, May 2019.

25.6 Test Methods for Evaluating Solid Waste: Laboratory Manual Physical/Chemical Methods, Volume 1B, SW846 Update V, Revision 4, Method 8000D, July 2014.

26.0 HISTORY

Revision 45: Prometon and Sulfolane were added to the compounds in Appendix 1
Revision 44: Updated DoD QSM to version 5.3, July. Added Appendix 11.
Revision 43: Updated for clarification of methods.
Revision 42: Updated to include new updated SW-846 Method 8270E. Corrected grammatical errors. Updated to DoD QSM Version 5.2, December 2018.
Revision 41: deleted sentence for standard used for 625.1.
Revision 40: Update for new 600 methods.

APPENDIX 1: METHOD ANALYTES

Analyte	CAS Number	Analyte	CAS Number
1,1'-Biphenyl	92-52-4	4-Bromophenylphenylether	101-55-3
1,2,4,5-Tetrachlorobenzene	95-94-3	4-Chloro-3-methylphenol	59-50-7
1,2,4-Trichlorobenzene	120-82-1	4-Chloroaniline	106-47-8
1,2-Dichlorobenzene	95-50-1	4-Chlorophenylphenylether	7005-72-3
1,2-Diphenylhydrazine	122-66-7	4-Nitrophenol	100-02-7
1,3,5-Trinitrobenzene	99-35-4	4-Nitroquinoline-1-oxide	56-57-5
1,3-Dichlorobenzene	541-73-1	5-Methylchrysene	3697-24-3
1,4-Dichlorobenzene	106-46-7	5-Nitro-o-toluidine	99-55-8
1,4-Dinitrobenzene	100-25-4	7,12Dimethylbenz(a)anthracene	57-97-6
1,4-Dioxane	123-91-1	Acenaphthene	83-32-9
1,4-Naphthoquinone	130-15-4	Acenaphthylene	208-96-8
1-Hexanol	111-27-3	Acetophenone	98-86-2
1-Methylnaphthalene	90-12-0	Aniline	62-53-3
1-Naphthylamine	134-32-7	Anthracene	120-12-7
2,2'-Dichlorobenzil	21854-95-5	Aramite	140-57-8
2,3,4,6-Tetrachlorophenol	58-90-2	Atrazine	1912-24-9
2,3-Dichloroaniline	608-27-5	Benzaldehyde	100-52-7
2,4,5-Trichlorophenol	95-95-4	Benzidine	92-87-5
2,4,6-Tribromophenol (surr)	118-79-6	Benzo(a)anthracene	56-55-3
2,4,6-Trichlorophenol	88-06-2	Benzo(a)pyrene	50-32-8
2,4-Dichlorophenol	120-83-2	Benzo(b)fluoranthene	205-99-2
2,4-Dimethylphenol	105-67-9	Benzo(ghi)perylene	191-24-2
2,4-Dinitrophenol	51-28-5	Benzo(k)fluoranthene	207-08-9
2,4-Dinitrotoluene	121-14-2	Benzoic acid	65-85-0
2,6-Dichlorophenol	87-65-0	Benzyl alcohol	100-51-6
2,6-Dinitrotoluene	606-20-2	Biphenyl	
2-Acetylaminofluorene	53-96-3	Butylbenzylphthalate	85-68-7
2-Butoxyethanol	111-76-2	Caprolactam	105-60-2
2-Chloronaphthalene	91-58-7	Carbazole	86-74-8
2-Chlorophenol	95-57-8	Chlorobenzilate	510-15-6
2-Ethoxyethanol	110-80-5	Chrysene	218-01-9
2-Fluorobiphenyl (surr)	321-60-8	Cresols (total)	1319-77-3
2-Fluorophenol (surr)	367-12-4	Di-n-butylphthalate	84-74-2
2-Methyl-4,6-dinitrophenol	534-52-1	Di-n-octylphthalate	117-84-0
2-Methylnaphthalene	91-57-6	Diallate	2303-16-4
2-Naphthylamine	91-59-8	Dibenzo(a,e)pyrene	192-65-4
2-Nitrophenol	88-75-5	Dibenzo(a,h)anthracene	53-70-3
2-Picoline	109-06-8	Dibenzo(a,h)pyrene	189-64-0
3,3'-Dichlorobenzidine	91-94-1	Dibenzofuran	132-64-9
3,3'-Dimethylbenzidine	119-93-7	Diethylphthalate	84-66-2
3-Methylcholanthrene	56-49-5	Dimethoate	60-51-5
4,4'-Methylenebis(2-chloroaniline)	101-14-4	Dimethylphthalate	131-11-3
4-Aminobiphenyl	92-67-1	Dinoseb	88-85-7

Analyte	CAS Number
Diphenylamine	122-39-4
Disulfoton	298-04-4
Ethyl Methanesulfonate	62-50-0
Ethyl methacrylate	97-63-2
Famphur	52-85-7
Fluoranthene	206-44-0
Fluorene	86-73-7
Hexachlorobenzene	118-74-1
Hexachlorobutadiene	87-68-3
Hexachlorocyclopentadiene	77-47-4
Hexachloroethane	67-72-1
Hexachlorophene	70-30-4
Hexachloropropene	1888-71-7
Indeno(1,2,3-cd)pyrene	193-39-5
Isodrin	465-73-6
Isophorone	78-59-1
Isosafrole	120-58-1
Kepone	143-50-0
Methapyrilene	91-80-5
Methoxychlor	72-43-5
Methyl methacrylate	80-62-6
Methyl methanesulfonate	66-27-3
Methyl parathion	298-00-0
N-Methyl-N-nitrosomethylamine	62-75-9
N-Nitrosodi-n-butylamine	924-16-3
N-Nitrosodiethylamine	55-18-5
N-Nitrosodipropylamine	621-64-7
N-Nitrosomethylethylamine	10595-95-6
N-Nitrosomorpholine	59-89-2
N-Nitrosopiperidine	100-75-4
N-Nitrosopyrrolidine	930-55-2
Naphthalene	91-20-3
Nitrobenzene	98-95-3
Nitrobenzene-d5 (surr)	4165-60-0
Parathion	56-38-2
Pentachlorobenzene	608-93-5
Pentachloroethane	76-01-7
Pentachloronitrobenzene	82-68-8
Pentachlorophenol	87-86-5
Phenacetin	62-44-2
Phenanthrene	85-01-8
Phenol	108-95-2
Phenol-d5 (surr)	4165-62-2

Analyte	CAS Number
Phorate	298-02-2
Prometon	1610-18-0
Pronamide	23950-58-5
Pyrene	129-00-0
Pyridine	110-86-1
Sulfolane	126-33-0
Safrole	94-59-7
Sulfotepp	3689-24-5
Thionazin	297-97-2
Tributylphosphate	126-73-8
Triethylphosphorothioate	126-68-1
a,a-Dimethylphenethylamine	122-09-8
alpha-Terpineol	98-55-5
bis(2-Chloro-1-methylethyl)ether	108-60-1
bis(2-Chloroethoxy)methane	111-91-1
bis(2-Chloroethyl) ether	111-44-4
bis(2-Ethylhexyl)phthalate	117-81-7
m,p-Cresols	65794-96-9
m-Dinitrobenzene	99-65-0
m-Nitroaniline	99-09-2
m-Toluidine	108-44-1
n-Decane	124-18-5
n-Octadecane	593-45-3
o-Cresol	95-48-7
o-Nitroaniline	88-74-4
o-Toluidine	95-53-4
p-(Dimethylamino)azobenzene	60-11-7
p-Benzoquinone	106-51-4
p-Nitroaniline	100-01-6
p-Phenylenediamine	106-50-3
p-Terphenyl-d14 (surr)	1718-51-0
p-Toluidine	106-44-1

APPENDIX 2: QC ACCEPTANCE CRITERIA

Table 6 – QC Acceptance Criteria – Method 625.1

Analyte	Range for Q (%)	Limit for s (%) [3]	Range for $\bar{X}$ (%)[3]	Range for P_s (%) [3]	Limit for RPD (%)
Acenaphthene	70-130	29	60-132	47-145	48
Acenaphthylene	60-130	45	54-126	33-145	74
Aldrin	7-152	39	7-152	D-166	81
Anthracene	58-130	40	43-120	27-133	66
Benzo(a)anthracene	42-133	32	42-133	33-143	53
Benzo(b)fluoranthene	42-140	43	42-140	24-159	71
Benzo(k)fluoranthene	25-146	38	25-146	11-162	63
Benzo(a)pyrene	32-148	43	32-148	17-163	72
Benzo(ghi)perylene	13-195	61	D-195	D-219	97
Benzyl butyl phthalate	43-140	36	D-140	D-152	60
beta-BHC	42-131	37	42-131	24-149	61
delta -BHC	D-130	77	D-120	D-120	129
bis(2-Chloroethyl)ether	52-130	65	43-126	12-158	108
bis(2-	52-164	32	49-165	33-184	54
bis(2-Chloroisopropyl)	63-139	46	63-139	36-166	76
bis(2-Ethylhexyl)	43-137	50	29-137	8-158	82
4-Bromophenyl phenyl	70-130	26	65-120	53-127	43
2-Chloronaphthalene	70-130	15	65-120	60-120	24
4-Chlorophenyl phenyl	57-145	36	38-145	25-158	61
Chrysene	44-140	53	44-140	17-168	87
4,4'-DDD	D-135	56	D-135	D-145	93
4,4'-DDE	19-130	46	19-120	4-136	77
4,4'-DDT	D-171	81	D-171	D-203	135
Dibenz(a,h)anthracene	13-200	75	D-200	D-227	126
Di-*n*-butyl phthalate	52-130	28	8-120	1-120	47
3,3'-Dichlorobenzidine	18-213	65	8-213	D-262	108
Dieldrin	70-130	38	44-119	29-136	62
Diethyl phthalate	47-130	60	D-120	D-120	100
Dimethyl phthalate	50-130	110	D-120	D-120	183
2,4-Dinitrotoluene	53-130	25	48-127	39-139	42
2,6-Dinitrotoluene	68-137	29	68-137	50-158	48
Di-*n*-octyl phthalate	21-132	42	19-132	4-146	69
Endosulfan sulfate	D-130	42	D-120	D-120	70
Endrin aldehyde	D-189	45	D-189	D-209	75
Fluoranthene	47-130	40	43-121	26-137	66
Fluorene	70-130	23	70-120	59-121	38
Heptachlor	D-172	44	D-172	D-192	74

Heptachlor epoxide	70-130	61	71-120	26-155	101
Hexachlorobenzene	38-142	33	8-142	D-152	55
Hexachlorobutadiene	68-130	38	38-120	24-120	62
Hexachloroethane	55-130	32	55-120	40-120	52
Indeno(1,2,3-cd)pyrene	13-151	60	D-151	D-171	99
Isophorone	52-180	56	47-180	21-196	93
Naphthalene	70-130	39	36-120	21-133	65
Nitrobenzene	54-158	37	54-158	35-180	62
N-Nitrosodi-*n*-	59-170	52	14-198	D-230	87

Analyte	**Range for Q (%) [2]**	**Limit for s (%) [3]**	**Range for $\bar{X}$ (%) [3]**	**Range for P, P_s (%) [3]**	**Limit for RPD (%)**
PCB-1260	19-130	77	19-130	D-164	128
Phenanthrene	67-130	24	65-120	54-120	39
Pyrene	70-130	30	70-120	52-120	49
1,2,4-Trichlorobenzene	61-130	30	57-130	44-142	50
4-Chloro-3-methylphenol	68-130	44	41-128	22-147	73
2-Chlorophenol	55-130	37	36-120	23-134	61
2,4-Dichlorophenol	64-130	30	53-122	39-135	50
2,4-Dimethylphenol	58-130	35	42-120	32-120	58
2,4-Dinitrophenol	39-173	79	D-173	D-191	132
2-Methyl-4,6-	56-130	122	53-130	D-181	203
2-Nitrophenol	61-163	33	45-167	29-182	55
4-Nitrophenol	35-130	79	13-129	D-132	131
Pentachlorophenol	42-152	52	38-152	14-176	86
Phenol	48-130	39	17-120	5-120	64
2,4,6-Trichlorophenol	69-130	35	52-129	37-144	58

1 Acceptance criteria are based upon method performance data in Table 7 and from EPA Method 625.1. Where necessary, limits for recovery have been broadened to assure applicability to concentrations below those used to develop Table 7.
2 Test concentration = 100 µg/mL
3 Test concentration = 100 µg/L

Q = Calibration verification (Sections 7.3.1 and 13.4)
s = Standard deviation for four recovery measurements in the DOC test (Section 8.2.4).
$\bar{X}$ = Average recovery for four recovery measurements in the DOC test (Section 8.2.4).
P, Ps = MS/MSD recovery (Section 8.3.2, Section 8.4.2).
RPD = MS/MSD relative percent difference (RPD; Section 8.3.3).
D = Detected; result must be greater than zero.

APPENDIX 3: ABUNDANCE CRITERIA

DFTPP KEY IONS AND ION ABUNDANCE CRITERIA (8270C, 625.1)

MASS	ION ABUNDANCE CRITERIA
51	30 - 60% of mass 198
68	< 2% of mass 69
70	< 2% of mass 69
127	40 - 60% of mass 198
197	< 1% of mass 198
198	Base peak, 100% of mass 198
199	5 - 9% of mass 198
275	10 - 30% of mass 198
365	> 1% of mass 198
441	Present but less than mass 443
442	> 40% of mass 198
443	17 - 23% of mass 442

DFTPP KEY IONS AND ION ABUNDANCE CRITERIA (8270D)

MASS	ION ABUNDANCE CRITERIA
51	10 - 80% of Base Peak
68	< 2% of mass 69
70	< 2% of mass 69
127	10 - 80% of Base Peak
197	< 2% of mass 198
198	Base peak or > 50% of mass 442
199	5 - 9% of mass 198
275	10 - 60% of Base Peak
365	> 1% of mass 198
441	Present but < 24% of mass 442
442	Base peak or > 50% of mass 198
443	15 - 24% of mass 442

APPENDIX 3: ABUNDANCE (continued)

DFTPP KEY IONS AND ION ABUNDANCE CRITERIA (8270E)

MASS	ION ABUNDANCE CRITERIA
68	<2% of m/z 69
69	Present
70	<2% of m/z 69
197	<2% of m/z 198
198	Base peak or present
199	5-9% of m/z 198
365	>1% of Base Peak
441	<150% of m/z 443
442	Base peak or present
443	15-24% of m/z 442

APPENDIX 4: TENTATIVE IDENTIFICATION PROCEDURES

1. Relative intensities of major ions (> 10%) in the reference spectrum should be present in sample spectrum. This equates to detects of TICs $\geq$ 4 ppb concentration (10% of 40 ppb internal standard concentration).

2. Relative intensities of the major ions should agree within $\pm$ 30% (i.e., for an ion with an abundance of 50% of the standard spectra, the corresponding sample ion abundance must be between 30 and 70 percent.)

3. Molecular ion present in reference spectrum should be present in sample spectrum.

4. Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

5. If no valid identification can be made, the compound should be reported as "unknown." If an additional classification can be given to the unknown (unknown hydrocarbon, aromatic, chlorinated, etc.) it should be listed as such.

6. Peaks that are detected in the sample and analytical blank should be flagged as such on the report.

7. TIC detects with “fit” value < 85 will be identified with J qualifier. Detects with “fit” value > 85 will be identified with NJ qualifier and parameter name.

8. Peaks that are suspected as aldol-condensation reaction products should be flagged as such on the report.

9. The CAS for calibrated compounds not included on the client requested list of parameters will be the true CASRN, while the CASRN for non-calibrated compounds will be reported with leading zeros (000) in front of the probable CASRN.

10. GEL’s policy for reporting calibrated compounds when requested by the client as TICs only is to follow TIC reporting from National Functional Guidelines and report only $\geq$ 4 ppb for SVOA with J or NJ qualification.

APPENDIX 5: DIOXIN SCREENS

Dioxin Screens may be analyzed as a part of EPA Method 625.1. This appendix outlines the steps for preparing extracts and conducting the analysis. A dioxin screen is used for qualitative characterization only and may not be used to generate quantitative results. The presence of 2,3,7,8-TCDD is indicated as either 'yes" or "no."

Liquid or solid samples are extracted and concentrated according to routine prep procedures (refer to applicable SOPs). The sample extracts and the associated blank extract are evaporated to dryness under a hood. The extracts are then re-constituted with 100 µL of methylene chloride. Approximately 50 µL of the extract is then placed in an autosampler vial and injected on the GC/MS.

Prior to analyzing samples and blanks, an appropriate 2,3,7,8-TCDD standard is analyzed on the instrument. This standard is used for qualitative purposes and to determine the retention time. No acceptance criteria apply. The compound merely needs to be detected.

Dioxin screens are analyzed using a Selective Ion Monitoring (SIM) method. One window is used for the duration of the entire run and three ions are monitored: m/z 257, 320, and 322.

After a samples is analyzed, the analyst checks to see if a peak is present at the appropriate retention time and exhibiting all three ions. If such a peak is detected, a "yes" result is reported.

APPENDIX 6: SIM PAH ANALYSIS

Selected ion monitoring (SIM) allows for specific ion fragments to be monitored and detected by the mass spectrometer. Lower detection limits can be achieved with SIM since the instrument is only looking at a small number of mass fragments during each scan and more scans can take place each second. Since only a few mass fragments of interest are being monitored, matrix interferences can also be minimized.

SIM is ideally suited for smaller target compound lists when lower detection limits are critical. The analysis of polynuclear aromatic hydrocarbons (PAHs) is therefore a common application of SIM.

The criteria outlined in this SOP for tuning, initial calibrations, calibration verification standards, and quality control samples also apply to SIM analyses. Please refer to those sections for acceptance criteria and guidance. This appendix provides an overview of SIM and SIM PAH in particular.

SIM PAH samples are extracted and prepped for analysis in the same manner as regular 8270C or 8270D, 8270E samples using methods 3510C, 3550C and 3541. Quality control samples include a method blank (MB), laboratory control sample (LCS), laboratory control sample duplicate (LCSD) if appropriate, matrix spike (MS), and matrix spike duplicate (MSD). All samples and QC are spiked with a surrogate standard as well. For SIM PAH, the surrogate is 5-alpha-Androstane (CAS 438-22-2).

The SIM PAH initial calibration is subject to the same acceptance criteria as an 8270C or 8270D, 8270E calibration (See pertinent sections in this SOP). The typical SIM PAH calibration range is from 0.1 ng/uL to 20 ng/uL (on-column concentration), although this may vary depending on project requirements.

Some clients may request SIM analysis for additional compounds for liquid matrices using prep method 3510C (sometimes referred to as SIM PLUS).

The following compounds may be analyzed using SIM PAH:

Compound	Target Ions (m/z)
Naphthalene	128, 129
2-Methylnaphthalene	142, 141
1-Methylnaphthalene	142, 141, 115
2-Chloronaphthalene	162, 164
Acenaphthylene	152, 151, 153
Acenaphthene	154, 153, 152
Fluorene	166, 165
Phenanthrene	178, 179
Anthracene	178, 179
Fluoranthene	202, 203
Pyrene	202, 200
Benzo(a)anthracene	228, 226, 229
Chrysene	228, 229, 226
Benzo(b)fluoranthene	252, 253
Benzo(k)fluoranthene	252, 253

Benzo(a)pyrene	252, 253
Indeno(1,2,3-cd)pyrene	276, 138
Dibenzo(a,h)anthracene	278, 138
Benzo(ghi)perylene	276, 138
N-Methyl-N-nitrosomethylamine	74, 42
Bis(2-Chloroethyl)ether	93, 63
N-Nitrosodipropylamine	70, 42
N-Nitrosodiethylamine	102, 42, 57
N-Nitrosoprrolidine	101, 41, 68
N-Nitrosidi-n-butylamine	84, 57, 41
Benzidine	184, 92
3,3'-Dichlorobenzidine	252, 254

APPENDIX 7: POOR RESPONDERS

The following compounds may require special treatment when being determined by this method. As documented poor responders, these compounds do not extract or recover well. As a result, low recoveries are to be expected in LCS, MS, and MSD.

Benzidine may be subject to oxidative losses during the solvent concentration.

p-Phenylenediamine belongs to a class of amine compounds that are very unstable at higher pHs and will readily oxidize under basic conditions. p-Phenylenediamine has been a historically difficult compound to extract in water matrices due to the solubility and the solvent extraction efficiency.

N-nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be separated from Diphenylamine.

1,2-Diphenylhydrazine is unstable even at ambient temperature and readily converts to Azobenzene.

Benzoic acid may recover poorly in spiked samples if the pH is not acidic enough (liquid) and is subject to erratic chromatography.

Pentachlorophenol, 2,4-Dinitrophenol, 4-Nitrophenol, 4,6-Dinitro-2-methylphenol, 4-Chloro-3-methylphenol, 2-Nitroaniline, 3-Nitroaniline, 4-Nitroaniline and Benzyl alcohol are subject to erratic chromatography behavior, especially if the GC system is contaminated.

p-Benzoquinone is a poor responder and typically has a very low RF.

Kepone is susceptible to degradation associated with active sites and GC system contamination.

Hexachlorocyclopentadiene is subject to thermal decomposition and chemical reaction in acetone solution.

APPENDIX 8: BNA Method Comparison

SVOA Method Comparison Table
Last Updated: July 2019

	8270C	8270D	8270E	QSM 5.3	625.1
IDOC	Analyze four LCS replicates. Evaluate average and standard deviation of replicates with method limits (Table 6 for 8270C and 625). Generate QC Summary and training certificate.	Analyze four LCS replicates. Evaluate average and standard deviation of replicates with method limits (Table 6 for 8270C and 625). Generate QC Summary and training certificate.	Analyze four LCS replicates. Evaluate average and standard deviation of replicates with method limits (Table 6 for 8270C and 625). Generate QC Summary and training certificate.	Analyze four LCS replicates. Evaluate average and standard deviation of replicates with method limits (Table 6 for 8270C and 625). Generate QC Summary and training certificate.	Analyze four LCS replicates. Evaluate average and standard deviation of replicates with method limits (Table 6 for 8270C and 625). Generate QC Summary and training certificate.
MDL LOD/LOQ LLOQ	Following initial set-up, MDLs are determined using points from each quarter. Q1: 1/2x, 1x, LOD, LOQ, MDL. Q2, Q3, Q4: LOD, LOQ, MDL. Analyzed on every instrument. LOD = 2x MDL LOQ = LLOQ=PQL	Following initial set-up, MDLs are determined using points from each quarter. Q1: 1/2x, 1x, LOD, LOQ, MDL. Q2, Q3, Q4: LOD, LOQ, MDL. Analyzed on every instrument. LOD = 2x MDL LOQ = LLOQ=PQL	Following initial set-up, MDLs are determined using points from each quarter. Q1: 1/2x, 1x, LOD, LOQ, MDL. Q2, Q3, Q4: LOD, LOQ, MDL. Analyzed on every instrument. LOD = 2x MDL LOQ = LLOQ=PQL	Following initial set-up, MDLs are determined using points from each quarter. Q1: 1/2x, 1x, LOD, LOQ, MDL. Q2, Q3, Q4: LOD, LOQ, MDL. Analyzed on every instrument. LOD = 2x MDL LOQ = LLOQ=PQL .	Following initial set-up, MDLs are determined using points from each quarter. Q1: 1/2x, 1x, LOD, LOQ, MDL. Q2, Q3, Q4: LOD, LOQ, MDL. Analyzed on every instrument. LOD = 2x MDL LOQ = LLOQ=PQL
Tune	Analyze every 12 hours. Criteria in method 8270C. Breakdown ≤ 20% for DDT; PCP tailing factor < 5; Benzidine tailing factor < 3	Analyze every 12 hours. Criteria in method 8270D. Breakdown ≤ 20% for DDT; PCP tailing factor < 2; Benzidine tailing factor < 2	Analyze with ICAL. Daily tune is optional. Meet ion ratio criteria for DFTPP in Table 3. Breakdown ≤ 20% for DDT; PCP tailing factor < 2; Benzidine tailing factor < 2	Analyze every 12 hours. Use 8270C or 8270D criteria depending on how samples are logged. Breakdown ≤ 20% for DDT. PCP tailing factor < 2; Benzidine tailing factor < 2.	Analyze every 12 hours. Criteria in method 8270C. Breakdown ≤ 20% for DDT; PCP tailing factor < 2; Benzidine tailing factor < 2
ICAL	Minimum of five levels. SPCCs: minimum RF is 0.05. CCCs: RSD ≤ 30%.	Minimum of five levels. RSD ≤ 20% for all targets or linear regression R^2 ≥ 0.99. Due to large number of diverse analytes, 8270D allows up to	Minimum of five levels. RSD ≤ 20% for all targets or linear regression R^2 ≥ 0.99. Due to large number of diverse analytes, 8270E allows up to	Minimum of five levels. There are no CCCs or SPCCs. RSD ≤ 15% for all targets or linear regression R^2 ≥ 0.99.	Minimum of three levels. RSD for each target analyte ≤ 35%. No linear curves.

	RSD for each target analyte ≤ 15% or linear regression for each target analyte R^2 ≥ 0.99. Some documented poor responders may not meet criteria. Qualitative analysis may proceed, but if these compounds are detected, re-analysis on a passing ICAL is required. %Error: ± 50% for low level and ± 30% for all other levels. RSE: criteria same as RSD for the method. If %Error or RSE fail, consult validator or GL. If adequate sensitivity has been established, ICAL may be acceptable for qualitative analysis.	10% outliers. If there are > 10% outliers, then re-calibration is required. Recommended minimum RFs (see Table 4) should be met for each level (With new ISTD approach, RF failures should be few if any). For outliers, non-detects do not require qualification, providing adequate sensitivity has been demonstrated with LLOQ level check standard (see CCV section). Analytes using a linear curve require evaluation of the intercept. The low level standard is re-evaluated as a sample. Recoveries must be within ± 30% of the true concentration. %Error: ± 50% for low level and ± 30% for all other levels. RSE: criteria same as RSD for the method. If %Error or RSE fail, consult validator or GL. If adequate sensitivity has been established, ICAL may be acceptable for qualitative analysis.	10% outliers. If there are > 10% outliers, then re-calibration is required. Recommended minimum RFs (see Table 4) should be met for each level (With new ISTD approach, RF failures should be few if any). For outliers, non-detects do not require qualification, providing adequate sensitivity has been demonstrated with LLOQ level check standard (see CCV section). Analytes using a linear curve require evaluation of the intercept. ICAL standards are re-evaluated as samples. Recoveries must be within ± 50% of the true concentration for LLOQ and ± 30% for other levels. Or, RSE ≤ 20%.	Analytes that do not meet either option may not be analyzed per QSAS. If criteria are not met, analyze sample(s) and QC on passing instrument for those compound(s).	
ICV	Second source. Same acceptance criteria as CCV.	Second source. All targets should recover within ± 30%.	Second source. All targets should recover within ± 30%.	Second source. All targets should recover within ± 20%.	Second source. All targets should recover within ± 20%.

		Minimum RFs should be met as outlined in Table 4 of the method. Per 8270D, *quantitative* analysis should not proceed for those analytes that fail ICV. Qualitative analysis may still be allowed.	Minimum RFs should be met as outlined in Table 4 of the method. Per 8270E, *quantitative* analysis should not proceed for those analytes that fail ICV. Qualitative analysis may still be allowed.	If a compound fails with a positive bias and there are no detects, results may be reported (with Q qualifier). Otherwise, re-analyze failing target compound(s) on a passing instrument.	
CCV	Analyze every 12 hours. CCC %D or %Drift ≤ 20%. SPCC RF ≥ 0.05. All targets RF ≥ 0.01. All targets %D or %Drift within ± 60% (source: DOE-AL SOW). Some exceptions may be made for documented poor performing compounds (see SOP) or target compounds with a positive bias if there are no detects. Note exceptions in narrative and DER. If the CCCs are not included in the target list, all analytes must pass 20% (section 7.4.5.2 of method).	Analyze every 12 hours. No CCCs or SPCCs. All targets should recover within ± 20% of the expected value. Method 8270D allows for up to 20% outliers (compared to target list of analytes). If more than 20% of analytes do not meet recovery criteria, then corrective action is required prior to analysis of samples. Recommended minimum RFs should be met as outlined in Table 4 of the method. If an outlier target compound is detected, the sample should be re-analyzed on a passing instrument. Non-detects may be reported, provided adequate sensitivity is demonstrated via a passing LLOQ standard. If a compound fails ± 20% in the CCV low, a LLOQ standard is analyzed. If the compound	Analyze every 12 hours. No CCCs or SPCCs. All targets should recover within ± 20% of the expected value. Method 8270E allows for up to 20% outliers (compared to target list of analytes). If more than 20% of analytes do not meet recovery criteria, then corrective action is required prior to analysis of samples. Recommended minimum RFs should be met as outlined in Table 4 of the method. If an outlier target compound is detected, the sample should be re-analyzed on a passing instrument. Non-detects may be reported, provided adequate sensitivity is demonstrated via a passing LLOQ standard. If a compound fails ± 20% in the CCV low, a LLOQ standard is analyzed. If the compound	Analyze every 12 hours and at end of tune window. All targets within ± 20%. All targets within ± 50% for ending CCV(s). For ending CCV(s), the first CCV must be injected within tune window. If target(s) fail, inject two additional CCVs within one hour. If both CCVs pass for the failing target(s), data may be reported. If either fails, corrective action must be taken (i.e. rerun with passing CCV). If a compound fails with a positive bias and there are no detects, results may be reported (with Q qualifier). Otherwise, re-analyze failing target compound(s) on a passing instrument.	Analyze every 12 hours. All targets within ± 20%.

		recovers within ± 30% (or high), the non-detect may be reported with no qualification. If it does not pass, the sample should be rerun.	recovers within ± 30% (or high), the non-detect may be reported with no qualification. If it does not pass, the sample should be rerun.		
ISTDs	Samples and QC: RT within ± 30 seconds from RT in CCV. EICP areas within -50% to +100% of the CCV. ISTD failures must be confirmed by re-analysis.	Samples and QC: RT within ± 30 seconds from RT in CCV. EICP areas within -50% to +100% of the CCV. ISTD failures must be confirmed by re-analysis.	Samples and QC: RT within ± 30 seconds from RT in CCV. CCV is compared to mid-level of ICAL. EICP areas within -50% to +100% of the CCV. ISTD failures must be confirmed by re-analysis.	CCV, Samples, and QC: RT within ± 10 seconds from RT in the midpoint of the ICAL. EICP areas within -50% to +100% of the midpoint in the ICAL. ISTD failures must be confirmed by re-analysis.	Samples and QC: RT within ± 30 seconds from RT in CCV. EICP areas within -50% to +100% of the CCV. ISTD failures must be confirmed by re-analysis.
MB	Detects of target analytes in MB acceptable if not detected in samples or if detects in samples are 10x higher than in MB. Apply B qualifier.	Detects of target analytes in MB acceptable if not detected in samples or if detects in samples are 10x higher than in MB. Apply B qualifier.	Detects of target analytes in MB acceptable if not detected in samples or if detects in samples are 10x higher than in MB. Apply B qualifier.	No target analytes detected >1/2 LOQ or >1/10 the amount measured in any sample or >1/10 the regulatory limit, whichever is greater. Common contaminants must not be detected > LOQ.	Detects of target analytes in MB acceptable if not detected in samples or if detects in samples are 10x higher than in MB. Apply B qualifier.
LCS	Evaluate with SPC limits. Re-extract samples if LCS fails (can fail high if there are no detects of those analytes in samples). Over-range analytes should be diluted to bring them within the calibration range. When samples are out of holding: consult with validator or GL for industrial clients (re-extraction may not be required); for others, re-extract if within 2x holding. Narrate and DER any failures.	Evaluate with SPC limits. Re-extract samples if LCS fails (can fail high if there are no detects of those analytes in samples). Over-range analytes should be diluted to bring them within the calibration range. When samples are out of holding: consult with validator or GL for industrial clients (re-extraction may not be required); for others, re-extract if within 2x holding. Narrate and DER any failures.	Evaluate with SPC limits. Re-extract samples if LCS fails (can fail high if there are no detects of those analytes in samples). Over-range analytes should be diluted to bring them within the calibration range. When samples are out of holding: consult with validator or GL for industrial clients (re-extraction may not be required); for others, re-extract if within 2x holding. Narrate and DER any failures.	Evaluate with QSM 5.0 limits in Appendix C. If analyte is not listed in the table, use SPC limits. If LCS fails, re-extract samples (can fail high if there are no detects of these analytes). Spike with ALL target analytes. If there are failures and samples cannot be re-extracted, narrate and DER. Identify compounds and validator will apply Q flag.	Evaluate with SPC limits (DHEC requires 625 method limits). Re-extract samples if LCS fails (can fail high if there are no detects of those analytes in samples). Over-range analytes should be diluted to bring them within the calibration range. When samples are out of holding: consult with validator or GL for industrial clients (re-extraction may not be required); for others, re-extract if within 2x holding. Narrate and DER any failures.

MS/MSD	Evaluate with SPC limits for recovery and RPD. Confirmed failures may be attributed to matrix interference.	Evaluate with SPC limits for recovery and RPD. Confirmed failures may be attributed to matrix interference.	Evaluate with SPC limits for recovery and RPD. Confirmed failures may be attributed to matrix interference.	Evaluate with QSM 5.0 limits in Appendix C for recovery and RPD (same limits used for LCS). Confirmed failures may be attributed to matrix interference.	Evaluate with SPC limits (DEHEC requires method 625 limits) for recovery and RPD. Confirmed failures may be attributed to matrix interference.
Surrogates	Evaluate with SPC limits. If surrogates fail low, re-extract the sample for confirmation. If there is obvious matrix interference, dilution and re-analysis may be appropriate and re-extraction may not be required. If surrogates fail high with no detects, data may be reported.	Evaluate with SPC limits. If surrogates fail low, re-extract the sample for confirmation. If there is obvious matrix interference, dilution and re-analysis may be appropriate and re-extraction may not be required. If surrogates fail high with no detects, data may be reported.	Evaluate with SPC limits. If surrogates fail low, re-extract the sample for confirmation. If there is obvious matrix interference, dilution and re-analysis may be appropriate and re-extraction may not be required. If surrogates fail high with no detects, data may be reported.	Evaluate with QSM 5.0 limits. If surrogates fail low, re-extract the sample for confirmation. If there is obvious matrix interference, re-extraction may not be required. Failures are Q qualified.	Evaluate with SPC limits. If surrogates fail low, re-extract the sample for confirmation. If there is obvious matrix interference, dilution and re-analysis may be appropriate and re-extraction may not be required. If surrogates fail high with no detects, data may be reported.

APPENDIX 9: 8270D MINIMUM RFs

RECOMMENDED MINIMUM RESPONSE FACTOR CRITERIA FOR INITIAL AND CONTINUING CALIBRATION VERIFICATION USING THE SUGGESTED IONS FROM TABLE 1

Semivolatile Compounds	Minimum Response Factor (RF)
Benzaldehyde	0.010
Phenol	0.800
Bis(2-chloroethyl)ether	0.700
2-Chlorophenol	0.800
2-Methylphenol	0.700
2,2'-Oxybis-(1-chloropropane)	0.010
Acetophenone	0.010
4-Methylphenol	0.600
N-Nitroso-di-n-propylamine	0.500
Hexachloroethane	0.300
Nitrobenzene	0.200
Isophorone	0.400
2-Nitrophenol	0.100
2,4-Dimethylphenol	0.200
Bis(2-chloroethoxy)methane	0.300
2,4-Dichlorophenol	0.200
Naphthalene	0.700
4-Chloroaniline	0.010
Hexachlorobutadiene	0.010
Caprolactam	0.010
4-Chloro-3-methylphenol	0.200
2-Methylnaphthalene	0.400
Hexachlorocyclopentadiene	0.050
2,4,6-Trichlorophenol	0.200
2,4,5-Trichlorophenol	0.200
1,1'-Biphenyl	0.010
2-Chloronaphthalene	0.800

Semivolatile Compounds	Minimum Response Factor (RF)
2-Nitroaniline	0.010
Dimethyl phthalate	0.010
2,6-Dinitrotoluene	0.200
Acenaphthylene	0.900
3-Nitroaniline	0.010
Acenaphthene	0.900
2,4-Dinitrophenol	0.010
4-Nitrophenol	0.010
Dibenzofuran	0.800
2,4-Dinitrotoluene	0.200
Diethyl phthalate	0.010
1,2,4,5-Tetrachlorobenzene	0.010
4-Chlorophenyl-phenyl ether	0.400
Fluorene	0.900
4-Nitroaniline	0.010
4,6-Dinitro-2-methylphenol	0.010
4-Bromophenyl-phenyl ether	0.100
N-Nitrosodiphenylamine	0.010
Hexachlorobenzene	0.100
Atrazine	0.010
Pentachlorophenol	0.050
Phenanthrene	0.700
Anthracene	0.700
Carbazole	0.010
Di-n-butyl phthalate	0.010
Fluoranthene	0.600
Pyrene	0.600
Butyl benzyl phthalate	0.010
3,3'-Dichlorobenzidine	0.010
Benzo(a)anthracene	0.800

Semivolatile Compounds	Minimum Response Factor (RF)
Chrysene	0.700
Bis-(2-ethylhexyl)phthalate	0.010
Di-n-octyl phthalate	0.010
Benzo(b)fluoranthene	0.700
Benzo(k)fluoranthene	0.700
Benzo(a)pyrene	0.700
Indeno(1,2,3-cd)pyrene	0.500
Dibenz(a,h)anthracene	0.400
Benzo(g,h,i)perylene	0.500
2,3,4,6-Tetrachlorophenol	0.010

APPENDIX 10: CALIBRATION CONCENTRATIONS

	Level 1 (ng/uL)	Level 2 (ng/uL)	Level 3 (ng/uL)	Level 4 (ng/uL)	Level 5 (ng/uL)	Level 6 (ng/uL)	Level 7 (ng/uL)	Level 8 (ng/uL)	Level 9 (ng/uL)	Level 10 (ng/uL)
1,4-Dichlorobenzene-d4 (INTERNAL STANDARD)										
Naphthalene-d8 (INTERNAL STANDARD)										
Acenaphthene-d10 (INTERNAL STANDARD)										
Phenanthrene-d10 (INTERNAL STANDARD)										
Chrysene-d12 (INTERNAL STANDARD)										
Perylene-d12 (INTERNAL STANDARD)										
2-Fluorophenol (SURROGATE)		10	20	40	50	80	100	120	30	60
Phenol-d5 (SURROGATE)		10	20	40	50	80	100	120	30	60
Nitrobenzene-d5 (SURROGATE)		10	20	40	50	80	100	120	30	60
2-Fluorobiphenyl (SURROGATE)		10	20	40	50	80	100	120	30	60
2,4,6-Tribromophenol (SURROGATE)		10	20	40	50	80	100	120	30	60
p-Terphenyl-d14 (SURROGATE)		10	20	40	50	80	100	120	30	60
N-Nitrosodimethylamine	1**	10	20	40	50	80	100	120	30	60
Pyridine		10	20	40	50	80	100	120	30	60
Aniline		10	20	40	50	80	100	120	30	60
Phenol		10	20	40	50	80	100	120	30	60
bis(2-Chloroethyl)ether		10	20	40	50	80	100	120	30	60
2-Chlorophenol		10	20	40	50	80	100	120	30	60
n-Decane		10	20	40	50	80	100	120	30	60
1,3-Dichlorobenzene		10	20	40	50	80	100	120	30	60
1,4-Dichlorobenzene		10	20	40	50	80	100	120	30	60
Benzyl Alcohol		10	20	40	50	80	100	120	30	60
1,2-Dichlorobenzene		10	20	40	50	80	100	120	30	60
bis(2-Chloro-1-methylethyl)ether		10	20	40	50	80	100	120	30	60

o-Cresol (2-Methylphenol)		10	20	40	50	80	100	120	30	60
N-Nitrosodipropylamine		10	20	40	50	80	100	120	30	60
m,p-Cresols (3-Methylphenol & 4-Methylphenol)		10	20	40	50	80	100	120	30	60
Hexachloroethane		10	20	40	50	80	100	120	30	60
Nitrobenzene		10	20	40	50	80	100	120	30	60
Isophorone		10	20	40	50	80	100	120	30	60
2-Nitrophenol		10	20	40	50	80	100	120	30	60
2,4-Dimethylphenol		10	20	40	50	80	100	120	30	60
bis(2-Chloroethoxy)methane		10	20	40	50	80	100	120	30	60
2,4-Dichlorophenol		10	20	40	50	80	100	120	30	60
Benzoic Acid			20	40	50	80	100	120	30	60
1,2,4-Trichlorobenzene		10	20	40	50	80	100	120	30	60
Naphthalene	1	10	20	40	50	80	100	120	30	60
alpha-Terpineol		10	20	40	50	80	100	120	30	60
4-Chloroaniline		10	20	40	50	80	100	120	30	60
Hexachlorobutadiene		10	20	40	50	80	100	120	30	60
4-Chloro-3-methylphenol		10	20	40	50	80	100	120	30	60
2-Methylnaphthalene	1	10	20	40	50	80	100	120	30	60
1-Methylnaphthalene	1	10	20	40	50	80	100	120	30	60
Hexachlorocyclopentadiene		10	20	40	50	80	100	120	30	60
2,3-Dichloroaniline		10	20	40	50	80	100	120	30	60
2,4,6-Trichlorophenol		10	20	40	50	80	100	120	30	60
2,4,5-Trichlorophenol		10	20	40	50	80	100	120	30	60
2-Chloronaphthalene	1	10	20	40	50	80	100	120	30	60
o-Nitroaniline		10	20	40	50	80	100	120	30	60
m-Nitroaniline		10	20	40	50	80	100	120	30	60
Dimethylphthalate	1	10	20	40	50	80	100	120	30	60
2,6-Dinitrotoluene		10	20	40	50	80	100	120	30	60
Acenaphthylene	1	10	20	40	50	80	100	120	30	60

Acenaphthene	1	10	20	40	50	80	100	120	30	60
2,4-Dinitrophenol			20	40	50	80	100	120	30	60
Dibenzofuran		10	20	40	50	80	100	120	30	60
2,4-Dinitrotoluene		10	20	40	50	80	100	120	30	60
Diethylphthalate	1	10	20	40	50	80	100	120	30	60
4-Nitrophenol		10	20	40	50	80	100	120	30	60
Fluorene	1	10	20	40	50	80	100	120	30	60
4-Chlorophenyl phenyl ether		10	20	40	50	80	100	120	30	60
2-Methyl-4,6-dinitrophenol		10	20	40	50	80	100	120	30	60
p-Nitroaniline		10	20	40	50	80	100	120	30	60
Diphenylamine		10	20	40	50	80	100	120	30	60
1,2-Diphenylhydrazine		10	20	40	50	80	100	120	30	60
4-Bromophenyl phenyether		10	20	40	50	80	100	120	30	60
Hexachlorobenzene		10	20	40	50	80	100	120	30	60
Pentachlorophenol		10	20	40	50	80	100	120	30	60
n-Octadecane		10	20	40	50	80	100	120	30	60
Phenanthrene	1	10	20	40	50	80	100	120	30	60
Anthracene	1	10	20	40	50	80	100	120	30	60
Di-n-butylphthalate	1	10	20	40	50	80	100	120	30	60
Fluoranthene	1	10	20	40	50	80	100	120	30	60
Pyrene	1	10	20	40	50	80	100	120	30	60
Butylbenzylphthalate	1	10	20	40	50	80	100	120	30	60
Benzo(a)anthracene	1	10	20	40	50	80	100	120	30	60
Chrysene	1	10	20	40	50	80	100	120	30	60
bis (2-Ethylhexyl) phthalate	1	10	20	40	50	80	100	120	30	60
Di-n-octylphthalate	1	10	20	40	50	80	100	120	30	60
Benzo(b)fluoranthene	1	10	20	40	50	80	100	120	30	60
Benzo(k)fluoranthene	1	10	20	40	50	80	100	120	30	60
Benzo(a)pyrene	1	10	20	40	50	80	100	120	30	60
Indeno-(1,2,3-cd)pyrene	1	10	20	40	50	80	100	120	30	60

Dibenzo(a,h)anthracene	1	10	20	40	50	80	100	120	30	60
Benzo(ghi)perylene	1	10	20	40	50	80	100	120	30	60
m-Dinitrobenzene		10	20	40	50	80	100	120	30	60
2,3,4,6-Tetrachlorophenol		10	20	40	50	80	100	120	30	60
Dinoseb		10	20	40	50	80	100	120	30	60
Carbazole	1	10	20	40	50	80	100	120	30	60
p-Benzoquinone		10	20	40	50	80	100	120	30	60
Methoxychlor		10	20	40	50	80	100	120	30	60
p-Toluidine		10	20	40	50	80	100	120	30	60
m-Toluidine		10	20	40	50	80	10	120	30	60
1,4-Dinitrobenzene		10	20	40	50	80	100	120	30	60
2-Ethoxyethanol		10	20	40	50	80	100	120	30	60
Phthalic anhydride		10	20	40	50	80	100	120	30	60
Methylenebis(2-chloroaniline)		10	20	40	50	80	100	120	30	60
Dibenzo(a,e)pyrene		10	20	40	50	80	100	120	30	60
Benzaldehyde		10	20	40	50	80	100	120	30	60
Acetophenone		10	20	40	50	80	100	120	30	60
Caprolactam		10	20	40	50	80	100	120	30	60
1,1'-Biphenyl		10	20	40	50	80	100	120	30	60
Atrazine		10	20	40	50	80	100	120	30	60
Benzidine		10	20	40	50	80	100	120	30	60
3,3'-Dichlorobenzidene		10	20	40	50	80	100	120	30	60
1,4-Dioxane		10	20	40	50	80	100	120	30	60
Methyl methacrylate		10	20	40	50	80	100	120	30	60
Ethyl methacrylate		10	20	40	50	80	100	120	30	60
2-Picoline		10	20	40	50	80	100	120	30	60
N-Nitrosomethylethylamine		10	20	40	50	80	100	120	30	60
Methyl methanesulfonate		10	20	40	50	80	100	120	30	60
N-Nitrosodiethylamine		10	20	40	50	80	100	120	30	60
Ethyl methanesulfonate		10	20	40	50	80	100	120	30	60

Pentachloroethane		10	20	40	50	80	100	120	30	60
N-Nitrosopyrrolidine		10	20	40	50	80	100	120	30	60
N-Nitrosomorpholine		10	20	40	50	80	100	120	30	60
o-Toluidine		10	20	40	50	80	100	120	30	60
N-Nitrosopiperidine		10	20	40	50	80	100	120	30	60
a,a-Dimethylphenethylamine		10	20	40	50	80	100	120	30	60
2,6-Dichlorophenol		10	20	40	50	80	100	120	30	60
Hexachloropropene		10	20	40	50	80	100	120	30	60
N-Nitrosodi-n-butylamine		10	20	40	50	80	100	120	30	60
Safrole		10	20	40	50	80	100	120	30	60
1,2,4,5-Tetrachlorobenzene		10	20	40	50	80	100	120	30	60
Isosafrole		10	20	40	50	80	100	120	30	60
1,4-Naphthoquinone		10	20	40	50	80	100	120	30	60
Pentachlorobenzene		10	20	40	50	80	100	120	30	60
1-Naphthylamine		10	20	40	50	80	100	120	30	60
2-Naphthylamine		10	20	40	50	80	100	120	30	60
5-Nitro-o-toluidine		10	20	40	50	80	100	120	30	60
1,3,5-Trinitrobenzene		10	20	40	50	80	100	120	30	60
Phenacetin		10	20	40	50	80	100	120	30	60
Diallate		10	20	40	50	80	100	120	30	60
cis-Diallate		1.5	3	6	7.5	12	15	18	4.5	9
trans-Diallate		8.5	17	34	42	68	85	102	25.5	51
4-Aminobiphenyl		10	20	40	50	80	100	120	30	60
Pentachloronitrobenzene		10	20	40	50	80	100	120	30	60
Pronamide		10	20	40	50	80	100	120	30	60
4-Nitroquinoline-1-oxide		10	20	40	50	80	100	120	30	60
Methapyrilene		10	20	40	50	80	100	120	30	60
Isodrin		10	20	40	50	80	100	120	30	60
Aramite		10	20	40	50	80	100	120	30	60
Kepone		10	20	40	50	80	100	120	30	60

p-(Dimethylamino)azobenzene		10	20	40	50	80	100	120	30	60
Chlorobenzilate		10	20	40	50	80	100	120	30	60
3,3'-Dimethylbenzidine		10	20	40	50	80	100	120	30	60
2-Acetylaminofluorene		10	20	40	50	80	100	120	30	60
7,12-Dimethylbenz(a)anthracene		10	20	40	50	80	100	120	30	60
3-Methylcholanthrene		10	20	40	50	80	100	120	30	60
Hexachlorophene		500	1000	1250	1500	1750	2000			
p-Phenylenediamine		500	1000	1250	1500	1750	2000			
bis(Chloromethyl)ether		10	20	40	50	80	100	120	30	60
Tributylphosphate		10	20	40	50	80	100	120	30	60
Triethylphosphorothioate		10	20	40	50	80	100	120	30	60
Thionazin		10	20	40	50	80	100	120	30	60
Sulfotepp		10	20	40	50	80	100	120	30	60
Phorate		10	20	40	50	80	100	120	30	60
Dimethoate		10	20	40	50	80	100	120	30	60
Disulfoton		10	20	40	50	80	100	120	30	60
Methyl parathion		10	20	40	50	80	100	120	30	60
Famphur		10	20	40	50	80	100	120	30	60
Parathion		10	20	40	50	80	100	120	30	60
bis(Chloromethyl)ether		10	20	40	50	80	100	120	30	60
4-Chlorothiophenol		10	20	40	50	80	100	120	30	60
4-Chlorothioanisole		10	20	40	50	80	100	120	30	60
Phthalic acid		10	20	40	50	80	100	120	30	60
Hydroxymethyl phthalimide		10	20	40	50	80	100	120	30	60
Diphenyl sulfide		10	20	40	50	80	100	120	30	60
Diphenyl disulfide		10	20	40	50	80	100	120	30	60
Phenyl sulfone		10	20	40	50	80	100	120	30	60
Octachlorostyrene		10	20	40	50	80	100	120	30	60
Thiophenol		10	20	40	50	80	100	120	30	60
2,2'-Dichlorobenzil		10	20	40	50	80	100	120	30	60

bis(p-Chlorophenyl)disulfide		10	20	40	50	80	100	120	30	60
bis(p-Chlorophenyl)sulfone		10	20	40	50	80	100	120	30	60
SIM PLUS PAH										
5-alpha-Androstane (SURROGATE)	0.1	0.2	0.5	1	2	5	10	20		
N-Methyl-N-nitrosomethylamine		0.2	0.5	1	2	5	10	20		
bis(2-Chloroethyl)ether	0.1	0.2	0.5	1	2	5	10	20		
N-Nitrosodipropylamine	0.1	0.2	0.5	1	2	5	10	20		
Naphthalene	0.1	0.2	0.5	1	2	5	10	20		
2-Methylnaphthalene	0.1	0.2	0.5	1	2	5	10	20		
1-Methylnaphthalene	0.1	0.2	0.5	1	2	5	10	20		
2-Chloronaphthalene	0.1	0.2	0.5	1	2	5	10	20		
Acenaphthylene	0.1	0.2	0.5	1	2	5	10	20		
Acenaphthene	0.1	0.2	0.5	1	2	5	10	20		
Fluorene	0.1	0.2	0.5	1	2	5	10	20		
Phenanthrene	0.1	0.2	0.5	1	2	5	10	20		
Anthracene	0.1	0.2	0.5	1	2	5	10	20		
Fluoranthene	0.1	0.2	0.5	1	2	5	10	20		
Pyrene	0.1	0.2	0.5	1	2	5	10	20		
Benzo(a)anthracene	0.1	0.2	0.5	1	2	5	10	20		
Chrysene	0.1	0.2	0.5	1	2	5	10	20		
Benzo(b)fluoranthene	0.1	0.2	0.5	1	2	5	10	20		
Benzo(k)fluoranthene	0.1	0.2	0.5	1	2	5	10	20		
Benzo(a)pyrene	0.1	0.2	0.5	1	2	5	10	20		
Indeno-(1,2,3-cd)pyrene	0.1	0.2	0.5	1	2	5	10	20		
Dibenzo(a,h)anthracene	0.1	0.2	0.5	1	2	5	10	20		
Benzo(ghi)perylene	0.1	0.2	0.5	1	2	5	10	20		

APPENDIX 11: 1,4-Dioxane Analysis in Water Samples by 8270 SIM

1. Due to the high solubility and polarity of 1,4-Dioxane, multiple analytical approaches may be used for this compound. These include Method 8270 Full Scan Mode, Method 522, and Method 8270 SIM Mode. This appendix addresses the unique method variations employed when analyzing 1,4-Dioxane using Method 8270 SIM Mode.
2. Many of the procedures used for extracting and analyzing samples by Method 522 are followed for 8270 SIM. As such, SOP GL-OA-E-073 should be consulted for details concerning this procedure. This appendix primarily addresses key differences when using Method 8270 instead of Method 522.
3. Method 8270 requires that water samples be extracted within seven days of collection and analyzed within 40 days. 100 mL of sample are extracted using solid phase extraction (SPE) by Method 3535 following the procedure outlined in section 15.4 of SOP GL-OA-E-073.
4. While Method 522 uses BFB for tuning the mass spec, Method 8270 uses DFTPP. As such, for this analysis, 10 uL of a 5 ng/uL DFTPP tuning solution are used to tune the instrument. Samples must then be analyzed within twelve hours of the DFTPP standard.
5. The analytical column used with this analysis is a Restek Rtx-624, 30 m x 0.25 mm x 1.4 um film thickness (catalog# 10968) or equivalent. Other columns may be used, such as a DB-5MS column. However, MDL studies and other method performance evaluations must be analyzed with the new column prior to submitting sample results.
    a. Calibration standards are made to encompass the desired calibration range of 1,4-dioxane and the surrogate, 1,4-dioxane-d8. A minimum of five calibration levels are used. The typical calibration range for 1,4- dioxane is from 10 ug/L to 500 ug/L (on-column). Using a standard SPE extraction (100 mL to 2 mL), the final reporting concentration range is from 0.2 ug/L to 10 ug/L. Some states, such as North Carolina, have an action level of 0.4 ug/L.

6. Per the recommendation in Method 8270E, 1,4-Dioxane-d8 is used as the surrogate standard to monitor extraction efficiency. The surrogate recovery limits are set at 70-130% until statistically derived control limits can be generated. The internal standard employed in this SIM analysis is Tetrahydrofuran-d8 (THF-d8). The peak area of the IS must be monitored in all injections during each analysis day. The IS response must not deviate from the response in the most recent CVS by more than 30%. All samples and quality control samples are fortified with surrogate and internal standard.
    a. The specifications and run conditions described in this appendix were used during method development and may be changed if QC criteria outlined in the method are met. Note that a large volume injection (LVI) is used in order to achieve maximum sensitivity.

APPENDIX 11: 1,4-Dioxane Analysis in Water Samples by 8270 SIM (Continued)

Injection: 10 uL
Inlet: large volume splitless, 120 ºC, split delay of 0.5 min Pressure pulse: 30 psi, 0.4 min
MS Conditions: This SOP is written for SIM analysis, although method 522 does allow for SCAN analysis.
Window 1: m/z 46, 78, and 80 are scanned for the ISTD (tetrahydrofuran-d8) at a rate of 19.27 cycles/sec.
Window 2: m/z 58 and 88 are scanned for the target (1,4-dioxane) as well as m/z 62, 64, and 96 for the SURR (1,4-dioxane-d8) at 13.6 cycles/sec.
The SIM windows should be set so that no peak elutes within 5 seconds of the beginning or end of the window.

VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE FOR

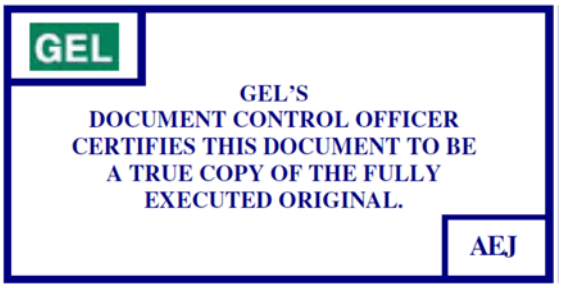
ANALYSIS OF CHLOROPHENOXY ACID HERBICIDES BY ECD

(GL-OA-E-011 REVISION 25)

APPLICABLE TO METHODS:
EPA SW-846 METHODS 8000D, 8151A

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1.0 STANDARD OPERATING PROCEDURE FOR ANALYSIS OF CHLOROPHENOXY ACID HERBICIDES BY ECD

2.0 METHOD CODES

EPA SW-846 Method 8000D, and 8151A

3.0 METHOD OBJECTIVE AND PURPOSE

This standard operating procedure provides the necessary instructions to conduct the analysis of samples for chlorophenoxy acid herbicides.

4.0 METHOD APPLICABILITY AND METHOD SUMMARY

4.1 This is a gas chromatographic procedure for quantitatively determining certain chlorophenoxy acid herbicides. The following compounds can be quantitatively and qualitatively determined by this procedure:

Compound	
Pentachlorophenol	
2,4-D	Dalapon
2,4-DB	Dicamba
2,4,5-T	Dichlorprop
2,4,5-TP(Silvex)	Dinoseb
MCPP	MCPA

4.2 This method describes the analysis of chlorophenoxy acid herbicides by gas chromatography. Samples are extracted with diethyl ether and then esterified with either diazomethane or pentafluoro-benzyl bromide. The derivatives are determined by gas chromatography with dual columns and dual electron capture detectors (ECD).

4.3 The method code for chlorophenoxy acid herbicides is SW-846 Method 8151A.

5.0 METHOD SCOPE AND PERFORMANCE CHARACTERISTICS

5.1 The calibration concentration ranges vary per analyte. The typical tested concentration ranges follow. These concentrations reflect on-column values and do not include prep factors.

Compound	Concentration Range µg/L
Pentachlorophenal	25-400
Dalapon	500 - 4000
Dicamba	25 - 400
MCPP	5000 - 40000
MCPA	5000 - 40000
Dichlorprop	25 - 400
2,4 - D	25 - 400
2,4,5-T	25 - 400
Dinoseb	25 - 400

2,4-DB	25 - 400
2,4-DCAA (surr)	62.5 – 1000
2,4,5-TP	25 - 400

5.2 Method Detection Limits (MDL) for herbicides are performed annually.

5.3 Precision is determined by the Relative Percent Difference (RPD) between the Laboratory Control Sample (LCS) and the Laboratory Control Sample Duplicate (LCSD) or a Matrix Spike (MS) and Matrix Spike Duplicate (MSD). The RPD is determined through Statistical Process Control (SPC).

5.4 Accuracy is determined by the percent recovery on LCSs, and the control ranges are determined through Statistical Process Control (SPC). For South Carolina samples, the LCS must pass within 70-130%.

6.0 DEFINITIONS

6.1 Definitions specific to this SOP include:

6.2 Limit of Detection (LOD): The lowest concentration level that can be determined by a single analysis and with a defined level of confidence to be statistically different from a blank. The LOD verification is typically spiked at two times the MDL.

6.3 Limit of Quantitation (LOQ): The lowest level in the calibration curve. With the prep factor applied, the LOQ is referred to as the effective LOQ. The LOQ is equivalent to the PQL and LLOQ.

6.4 Lower Limit of Quantitation (LLOQ): The lowest concentration at which a target analyte can be reliably measured and reported. The LLOQ is the lowest point in the calibration curve and represents a concentration at which both quantitative and qualitative requirements can be consistently demonstrated. The LLOQ is verified quarterly, as the LOQ verification. The verifications performed by extracting and analyzing an LCS spiked at the LOQ. The LLOQ verification is carried through the same preparation and analytical procedures as environmental samples and QC. The LLOQ is analyzed on every instrument where data are reported and this is the laboratory's normal protocol. Recovery of target analytes in the LLOQ are compared to in-house-statistically-derived limits. Concentrations in samples reported below the LLOQ and above the MDL are qualified as estimated.

6.5 Practical Quantitation Limit (PQL): The lowest level in the calibration curve. With the prep factor applied, the PQL is referred to as the effective PQL. The PQL is equivalent to the LOQ and the LLOQ.

6.6 Relative Percent Difference (RPD): The difference between two duplicate samples, such as a MS/MSD, LCS/LCSD, or sample/sample DUP. It is determined by taking the difference between the two results and dividing by the average.

6.7 Statistical Process Control (SPC) Limits: Statistically derived limits that establish acceptable ranges for recoveries of analytes of interest, including LCS, MS, MSD, PS, PSD and internal standards.

6.8 Lab-wide definitions can be found in GL-QS-B-001 the Quality Assurance Plan.

7.0 REFERENCES

7.1 Test Methods for Evaluating Solid Wastes: Laboratory Manual Physical/Chemical Methods, Volume 1B, SW-846, 3rd Edition, June 1997. Method 8151A, "Chlorinated Herbicides by GC Using Methylation or Pentafluorobenzylation Derivatization: Capillary Column Technique," Revision 1, December 1996.

7.2 Dept. of Defense (DOD), Dept. of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories DOD QSM Version 5.0, July 2013 and Version 5.1, January 2017; DOE QSAS 3.0, July 2013 and Version 3.1 January 2017.

8.0 INTERFERENCES TO THE METHOD

8.1 The interferences in industrial effluents are high and varied and often pose great difficulty in obtaining accurate and precise measurements of chlorinated acid herbicides. Sample clean-up procedures are generally required. However, they may result in the loss of certain herbicides.

8.2 Organic acids, especially chlorinated acids, cause the most direct interference with this determination process. Phenols, including chlorophenols, will also interfere with the analysis.

8.3 Alkaline hydrolysis and subsequent extraction eliminates many of the predominant chlorinated insecticides that may otherwise interfere with the analysis.

8.4 The herbicides, being strong organic acids, react readily with alkaline substances and may be lost during analysis. Glassware and glass wool should be acid rinsed, and the sodium sulfate should be muffled and acidified to avoid the possibility.

9.0 SAFETY PRECAUTIONS AND HAZARD WARNINGS

WARNING

HEXANE IS MODERATELY TOXIC. PREVENT SKIN AND EYE CONTACT BY USING SPECIFIED PERSONAL PROTECTIVE EQUIPMENT WHEN MAKING STOCK REAGENTS. WORK UNDER A HOOD TO PREVENT INHALATION WHEN MAKING STOCK REAGENTS.

9.1 Wear eye protection with side shields while performing procedures in the lab.

9.2 Treat all chemicals and samples as potential health hazards and limit exposure to these chemicals to the lowest level possible. GEL maintains a current awareness file of OSHA regulations regarding the safe handling of the chemicals in the laboratory as well as a reference file of Material Safety Data Sheets (MSDS). These documents and individual sample MSDSs are maintained in the laboratory.

9.3 Gloves are required when handling the chemicals and samples in this procedure. Latex or nitrile gloves may be used.

9.4 Prior to handling radioactive samples, analysts must have had radiation safety training and must understand their full responsibilities in radioactive sample handling. Follow these general guidelines:

9.4.1 A plastic apron may be worn over lab coat when working with radioactive samples.

9.4.2 Protect counter tops with counter paper or work from radioactive sampling trays.

9.4.3 Prohibit admittance to immediate work area.

9.4.4 Post signs indicating radioactive samples are in the area.

9.4.5 Take swipes of the counter tops upon completion of work. Deliver those swipes to the designated swipe count box.

9.4.6 Segregate radioactive wastes. Radioactive waste containers are obtained from Waste Management.

9.5 All samples, chemicals, extracts, and extraction residues must be transferred, delivered, and disposed of safely according to all related SOPs.

9.6 Never leave gas cylinders unchained or untied, including when they are on the moving carts.

9.7 In the event of an accident or medical emergency, call for help immediately. When time and safety permit, an accident report should be completed and turned in to the safety committee.

9.8 Fire escape routes are posted in the lab and all personnel should be familiar with them. In addition, fire safety equipment, such as fire extinguishers, is located in the lab. Training is available on the proper operation of this equipment.

10.0 CAUTION WARNINGS

Hexane is moderately toxic. It should be used in a well-ventilated area.

11.0 APPARATUS AND EQUIPMENT, REAGENTS, AND INSTRUMENTS

11.1 Apparatus and Equipment

11.1.1 2 mL amber autosampler screw cap vials with Teflon-lined septa

11.1.2 Supelco Thermogreen 12.5 mm GC septa or equivalent

11.1.3 Pasteur pipets

11.1.4 Microliter syringe

11.1.5 Diverter valve (8")

11.1.6 Capillary cleaving tool

11.1.7 Septum penetrating tool

11.1.8 0.32 mm ferrules

11.1.9 MXTY Connector

11.2 Reagents, Chemicals, and Standards

11.2.1 Standards can be bought from a certified vendor able to produce documentation tracing it to a certified source.

11.2.2 Hexane (C_6H_{14}), pesticide quality or equivalent

11.2.3 Diazomethane (CH_2N_2)

11.2.4 Silicic acid (H_2SiO_5)

11.3Instrumentation

GC: HP6890 (or equivalent)

HP7673 GC/SFC auto sampler (or equivalent)

Recommended Columns:

J&W Scientific DB-XLB	30 m x 0.25 mm x 0.2 µm
J&W Scientific DB17MS	30 m x 0.25 mm x 0.2 µm
Restek CLP Pest I	30 m x 0.25 mm x 0.2µm
Restek CLP Pest II	30 m x 0.25 mm x 0.2µm

It should be noted that the instrumentation and columns listed above are merely the recommended instrumentation and columns for use with this method.

12.0 SAMPLE HANDLING AND PRESERVATION REQUIREMENTS

12.1 Samples are collected in an amber glass bottle with a Teflon-lined cap. The collection containers are bought precleaned from a certified vendor.

12.2 All liquid samples have a holding date of seven days from the time of collection to be extracted, while all solid samples have fourteen days from the collection date to be extracted. Samples are protected from light and stored at 0° ≤ 6° C in amber vials with Teflon-sealed screw caps.

13.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

13.1 In order to maintain the gas chromatograph's columns and detectors, the gases should be changed when the tank pressure is below 250 psi. The gas chromatograph's septum in the injection port should be changed as needed. The splitter should be changed and column maintenance performed when baseline rise is present or the column becomes contaminated.

13.2 Routine Maintenance

Component	As Needed	Semi-Annually
Gases	X	
SiltekMXT or other connector	X	
Column Maintenance	X	
Detector Wipe Test		X
Detector Cleaning	X	
Injector Cleaning	X	

13.3 Non-routine Maintenance

13.3.1 When a check standard fails, column maintenance may need to be performed. One loop of each column should be cleaved and the Siltek MXT connector changed. The instrument is then baked out at 270º C overnight or until a straight baseline is obtained. If a straight baseline is

not obtained, the column is leaking or bleeding too badly to use and column maintenance must be performed again.

13.3.2 When contamination occurs, first replace/clean the autosampler syringe. If contamination is still present, column maintenance should be performed. If contamination is still present, replace the column. The column must then be leak checked and baked overnight. The instrument is then to be checked to determine if the problem is solved and if the instrument is stable. If not, the in-house service technician is called.

13.3.3 When maintenance is done on the instrument, it must be documented in the AlphaLIMS system.

14.0 PREPARATION OF STANDARD SOLUTION AND QUALITY CONTROL SAMPLES

14.1 Standard Solutions

14.1.1 Source standard solutions are purchased form Chem-Service, Protocol, Accu-Standard, and other certified vendors as organic acids and then esterified in the lab prior to use. The standards are traceable to National Institute of Standards and Technology (NIST) standards. The standards are given a unique identifying number for that day and is recorded in AlphaLIMS. Refer to Appendix 1 for detailed instructions on herbicide standards and ICAL preparations.

14.1.2 For guidance on standard documentation and traceability, refer to GL-LB-E-007 for Laboratory Standards Documentation.

15.0 INSTRUMENT CALIBRATION

15.1 The instrument should be checked for cleanliness and stability prior to analyzing calibration standards. The standards are loaded onto the auto sampler with the lowest standard being analyzed first to prevent carryover.

15.2 An external standard technique is used to calibrate the instrument. Calibration is obtained by analyzing the standards using the same method used for samples. Each standard must contain the same analytes, but at different concentration levels. The area counts of each peak along with the concentration of that particular analyte can then be used to plot a calibration curve. All standards are purchased in acid form and are esterified in the laboratory. (Herbicide standards in the acid form are good for two months after date opened. Esterified herbicide standards are good for six months). The typical calibration range is from 25 ug/L to 400 ug/L. MCPP and MCPA are calibrated from 5,000 ug/L to 40,000 ug/L and Dalapon is calibrated from 500 ug/L to 4,000 ug/L. The lowest calibration level corresponds to the LLOQ (PQL). Refer to Appendix 1 for calibration concentrations. The MDL, LOD, and LLOQ (LOQ) are verified quarterly. The MDL verification is spiked at the MDL concentration (approximately one third of the LLOQ). The LOD is spiked at two times the MDL. And the LLOQ is spiked at the lowest calibration level (the PQL) . Verification samples are extracted using the same methods and processes used for samples and analyzed on each

instrument used for that analysis. Statistical Process Limits (SPC) are calculated for the LLOQ using historical data from the lab and are used to evaluate the LLOQ recoveries.

15.3 The calibration standards are introduced into the gas chromatograph using a syringe. Calculate the Calibration Factor (CF) and the relative standard deviation for each analyte at each standard concentration using the formula below.

NOTE: Both columns should meet acceptance criteria for each analyte of interest before analyzing samples.

15.3.1 Calculate the calibration factor for each analyte at each concentration as:

$$CF = \frac{\text{Peak Area of the Compound in the Standard}}{\text{Mass of Compound injected (in nanograms)}}$$

The CF can also be calculated using the concentration of the standard rather than the mass in the denominator of the equation above. However, the use of the concentration will require changes to the equations that are used to calculate sample concentration.

15.3.2 Calculate the mean calibration factor for each analyte as:

$$\overline{CF} = \frac{\sum_{i-1}^{n} CF_i}{n}$$

Where n = the number of standards analyzed

15.3.3 Calculate the standard deviation and the RSD of the calibration factor for each analyte as:

$$SD = \sqrt{\frac{\sum_{I=1}^{N}(CF_i - \overline{CF})^2}{n-1}} \qquad RSD = \frac{SD}{CF} X\ 100$$

If the percent relative standard deviation (%RSD) of the calibration factor is $\leq$ 20% over the working range, linearity through the origin can be assumed, and the mean calibration factor can be used to quantitate sample results. When this is not the case, linearity cannot be assumed and the analyst must use a calibration curve.

15.4 If the analyst chooses to use linear regression, he/she must not force the calibration line through the origin.

15.4.1 Make certain that the instrument response is treated as the dependent variable (y) and the concentration as the independent variable (x). The regression will produce the slope and intercept terms for a linear equation in the form:

$$y = ax + b$$

Where:

y = instrument response

a = slope of line (also called the "coefficient of x")

x = concentration of the calibration standard

b = the intercept

15.4.2 The analyst should not force the line through the origin, but have the intercept calculated from the data points. Otherwise, the problems noted with the RSD value will occur, i.e., a line through the origin will not meet the QC specifications. In addition, do not include the origin (0,0) as a sixth calibration point.

15.4.3 The regression calculation will generate a correlation coefficient (R) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, R must be $\geq$ 0.99. The calculated intercept value needs to be evaluated before reporting sample results. If the system you are using calculates the coefficient of determination (R^2), the value must still meet the 0.99 criteria. Please note that second order curve types (i.e. quadratic fit) cannot be used to meet the acceptance criteria.

15.4.4 In calculating the sample concentrations, the regression equation is rearranged to solve for concentration (x) as shown below:

$$x = \frac{(y - b)}{a}$$

15.5 A minimum number of five calibration standards is required by the method.

15.5.1 Method 8000D outlines two procedures that may be used to determine calibration function acceptability for linear and non-linear curves. The calibration data are refitted back to the calibration model. % Error and Relative Standard Error (RSE) evaluate the difference between the measured amount and the true amount (or concentration).

15.5.2 % Error is determined as follows:

$$\%Error = \frac{x_i - x'_i}{x_i} x100$$

Where:

x'_i = Measured amount of analyte at calibration level *i*, in mass or concentration units

x_i = True amount of analyte at calibration level *i*, in mass or concentration units.

Percent error between the calculated and expected amounts of an analyte should be ≤30% for all standards and ≤50% for the lowest calibration level.

15.5.3 Relative Standard Error is calculated as follows:

$$RSE = 100 \times \sqrt{\sum_{i-1}^{n} \left[\frac{x'_i - x_i}{x_i} \right]^2 \Big/ (n - p)}$$

Where:

x_i = True amount of analyte in calibration level *i*, in mass or concentration units.

x'_i = Measured amount of analyte in calibration level *i*, in mass or concentration units.

p = Number of terms in the fitting equation

(average = 1, linear 2, quadratic =3, cubic =4)

n = Number of calibration points

15.5.4 The *RSE* acceptance limit criterion is the same as the RSD Limit for $\overline{CF}$ or $\overline{RF}$ in the determinative method. If the RSD limit is not defined in the determinative method, the limit should be set at ≤ 20% for well performing compounds.

15.6 Continuing Calibration

15.6.1 The initial calibration curve for each compound of interest must be checked and verified prior to conducting any sample analysis. This is accomplished by analyzing a calibration standard that is at a concentration near the midpoint concentration for the working range of the GC. The initial calibration verification standard must be from a second vender if available or a second lot from the same vendor. If a second lot is used, it should be from a different source than the ICAL. The standard must also be injected at intervals of once every ten samples and at the end of the analysis sequence. The continuing calibration check standard may be from the same source as the initial calibration. This will determine the validity of the initial calibration on a daily basis. These standards are prepared using acids which go through the same esterification process as the samples.

15.6.2 Calibration verification for linear curves involves the calculation of the percent drift or the percent difference of the instrument response between the initial calibration and each subsequent analysis of the verification standard. Use the equations below to calculate % Drift or % Difference.

$$\% \text{ Drift} = \frac{\text{Calculated Concentration - Theoretical Concentration}}{\text{Theoretical Concentration}} \text{ x } 100$$

Where the calculated concentration is determined using the calibration factor or response factor from the initial calibration and the theoretical concentration is the concentration at which the standard was prepared.

$$\% \text{ Difference} = \frac{\overline{CF} - CF_v}{\overline{CF}} \times 100$$

Where:

CF_v = response factor from current verification check standard

15.6.3 The CVS should recover within ± 15% (8151 A) or ± 20% (DoDQSM) of the known concentration. If this criterion is exceeded, inspect the gas chromatographic system to determine the cause and perform whatever maintenance is necessary before verifying calibration and proceeding with sample analysis. If no source of the problem can be determined after corrective action has been taken, a new five-point calibration will be generated. This criterion must be met before quantitative sample analysis begins. If a bracketing CVS recovers with a positive bias and there are no detects in the samples, the data may be reported. If there are detects, the samples must be reanalyzed for confirmation. If the bracketing CVS fails with a negative bias on both columns, the samples in the bracket must be reanalyzed. If the CVS fails low on only one column and there are no detects, data may be reported.

16.0 INSTRUMENT PERFORMANCE REQUIREMENTS

16.1 Gas Chromatography Recommended Conditions:

Detector Temperature: 325° C
Injector Temperature: 215° C
Column A: DB-17MS or CL Pest I
Column B: DB-XLB or CLP Pest II
Column Flow = 2.0 mL/minute
Carrier Gas: Helium or Hydrogen
Make-up Gas: Nitrogen
1st Temperature: 50° C
Hold = 30 sec.
1st Ramp = 25.00° C/minute
2nd Temperature = 190° C
Hold = 1 minute
2nd Ramp = 11.00 ° C/minute
3rd Temperature: 300° C
Hold = 0 minute
3rd Ramp = 20.00° C /minute

Final Temperature = 320° C

Hold = 3 minutes

Run Time = 21.10 minutes

NOTE: Slight variations may be needed as column length changes.

16.2 Before samples can be analyzed, the baseline must be stable with little or no background noise or baseline rise, and the continuing calibration check standard must be within ± 15% of the true value (± 20% for DoD QSM).

17.0 ANALYST AND METHOD VERIFICATION REQUIREMENTS

17.1 To establish that an analyst can perform the procedures in an acceptable manner and that the method generates data of acceptable bias and precision, the following operations are performed:

17.1.1 A Quality Control (QC) check standard must be prepared containing each analyte of interest. It must be prepared from pure standard material or purchased as a certified solution. It must be made from a source independent of that used for calibration.

17.1.2 Four aliquots should be prepared and analyzed by the same procedures used to prepare and analyze actual samples.

17.1.3 Calculate the average recovery in µg/L, and the standard deviation of the recovery (S) in µg/L, for each analyte of interest using the four results.

17.1.4 For each analyte compare S with the corresponding acceptance criteria for precision and accuracy, respectively, given in the quality control table at the end of the method. If the S for all analytes of interest meets the acceptance criteria, the systems performance is acceptable and analysis of actual samples can begin. If any individual S exceeds the precision limits or fall out of the range for accuracy, the system's performance is unacceptable for that analyte and a check standard for that analyte must be prepared and reanalyzed.

17.2 Method Detection Limits are also determined and documented annually.

17.3 Analysts are given continual performance evaluation samples as an ongoing assessment of their ability to perform the procedure.

18.0 ANALYSIS PROCEDURES AND INSTRUMENTAL OPERATION

18.1 Standards, samples, blanks, and Quality Control samples are introduced to the instrument via direct injection. Daily retention time windows must be centered for each analyte. Refer to GL-OA-E-001 for Establishing Retention Time Windows for GC and HPLC Analysis as to when and how often retention time windows should be established.

18.2 Samples are analyzed in a set referred to as analysis sequence or run sequence. The sequence begins with the instrument blank, followed by the sample extracts. A mid-level calibration check standard must be analyzed after every ten extracts

and at the end of each run sequence. The sequence ends when all extracts have been injected and analyzed.

18.3 The data are entered into the computer and a data report is generated. A run log is created by the analyst on the computer. It lists every injection made by the instrument in order, for a given date.

18.4 Before the instrument is used, a highly concentrated standard may be analyzed to prime the columns.

18.5 1.0 µl of the extract is injected into the instrument.

18.6 The autosampler is used to inject extracts sequentially. It can be set to run from any given starting position to a specific ending position. The digital integrator converts the analog signal from the instrument to digital information that is processed into a graphic format showing concentration based on peak area.

18.7 Samples containing target analyte concentrations that exceed the linear range of the analyte calibration curve must be diluted. The dilution level should be performed to place the highest analyte concentration between the middle and high points of the calibration curve (on column). If a sample is initially diluted, and target analytes are not detected and non-target analytes are not interfering with the analysis, the sample must be reanalyzed at a lower dilution. Analysts should be aware that diluting samples will increase the detection limits for undetected analytes. Random dilutions without due cause are not acceptable. Samples should undergo appropriate clean up methods prior to diluting for observed matrix problems.

18.8 The automated sequence is initiated by programming the autosampler to start at the first sample to be analyzed.

19.0 CALCULATIONS AND DATA REDUCTION METHODS

The concentration of each analyte in extract can be determined by comparing the response obtained from analyzing the extract to calibration curve.

20.0 DATA RECORDING

Data are recorded and calculated by the Chemstation data acquisition system. Data are stored on a chem server. The data are also input into AlphaLIMS.

21.0 QUALITY CONTROL REQUIREMENTS

21.1 Before analysis of any Quality Control samples, the instrument calibration must be verified. A continuing calibration check standard must be analyzed, before any QC or client samples, to verify instrument calibration and must be ± 15% of its true value.

21.2 A Method Blank (MB) is used to determine background concentrations of analytes of interest that have the potential to interfere with sample analysis. These MBs are analyzed with every analytical batch that has a maximum number of twenty samples. The criteria for acceptance are that there are no target analytes of interest present above the Lower Limit of Quantitation (LLOQ).

21.2.1 If the analyte of interest is present at a concentration between the MDL and LLOQ, all data are qualified with a “B” flag and reported. If the

analyte of interest is present at a concentration above the LLOQ and the samples contain the analyte of interest at a concentration of greater than 10 times the concentration found in the MB, the data are qualified with a "B" flag and reported. If the concentration found in the sample is less than 10 times that found in the MB and greater than the LLOQ, the samples must be re-extracted.

21.3 A Laboratory Control Sample (LCS) is analyzed with every batch. The accuracy and precision of the extraction and analysis are monitored with these samples. Sample spikes and sample duplicates are also used for this purpose. The criterion for acceptance is that the recoveries must be within the fixed range given for any given analyte for a specific matrix. Refer to sections 5.3 and 5.4. In addition to a LCS, a Matrix Spike (MS) and Matrix Spike Duplicate (MSD) are typically extracted and analyzed with each batch of samples. If not enough sample aliquot is available for a MS and MSD, a LCSD may be extracted and analyzed.

21.4 For Method 8151A, the surrogate 2,4-DCAA is used. Its acceptance is based on SPC limits and is matrix specific.

21.5 The Retention Time (RT) window should be established using the initial calibration check standard. Refer to GL-OA-E-001 for Establishing Retention Time Windows for GC and HPLC Analysis for instructions. In order to report a concentration from the external standards table, the retention time must be within the established window. If not, the concentration is not reported.

21.6 Nonconformance

21.6.1 If the continuing check standard fails any criteria in section 15.6, the analyst must take action to correct the situation. This may be performing any of the maintenance steps described in section 13.0 to get the instrument to meet its daily calibration. If all attempts fail, the analyst must analyze a new series of calibration standards, thus obtaining a new calibration curve.

21.6.2 If the percent recovery in the MS or MSD falls outside the established SPC limits for recovery, the analyst should evaluate the LCS recoveries and MB analyses. If the LCS and MB analyses do not indicate a problem with the preparation procedures, the MS recoveries may be attributed to matrix effect. Surrogate recovery data should also be used to evaluate the data. Recoveries of both MS compounds and surrogates that are outside the acceptance limits suggest more pervasive analytical problems than problems with the recoveries of either MSs or surrogates alone. Analysts are not required to reanalyze the MSs for failing recoveries; however they should seek additional technical support before deciding not to reanalyze MS samples. Acceptance limits are determined semi-annually using Statistical Process Control (SPC). The limits are maintained in the Quality Department. SPC limits are generated for surrogates, LCSs, MSs, MSDs, and RPDs.

21.6.3 When a Method Blank fails, the acceptance criteria as described in section 21.2, the analytical batch may be re-extracted.

21.6.4 If a surrogate in a sample falls outside of the acceptable range, the sample should be re-extracted. However, if the recovery is above the acceptance range and no target analytes are detected in the sample, the result may be reported. A DER must be generated and the failure documented in the Case Narrative. If the surrogate fails the second time, the failure is attributed to matrix interference.

21.6.5 If retention time is out of the window, the concentration for that analyte cannot be reported. However, if the continuing check standard falls outside the established window, all the samples bracketed by that standard must be re-injected.

21.7 Any positive identification and quantitation of an analyte of interest must be confirmed on a separate column. Qualitative confirmation is presence within the RT window on each column. Quantitation of an analyte of interest must be taken from a column whose daily check standards, bracketing that sample set, have met the acceptable Quality Control criteria.

21.7.1 Since the same method criteria and instrument calibration specified in section 15.0 are applied uniformly to both columns, either column can be selected to serve as the primary or confirmatory column. The lab's standard practice is to report the lower column result.

21.7.2 The determination of MDLs is also applied to both columns. Refer to The Determination of Method Detection Limits, GL-LB-E-001.

22.0 DATA REVIEW, VALIDATION, AND APPROVAL PROCEDURES

22.1 Upon completion of a batch, the analyst enters data. A data report is generated and is placed in a file folder along with the batch sheet and all the raw data (chromatograms). This folder is given to a Data Review person for reviewing.

22.2 Levels of Review

22.2.1 First level review: It is the responsibility of the analyst to ensure that the check standard passes and that the Quality Control results were within acceptance ranges. The analyst makes sure that all necessary paperwork is given to the data reviewer.

22.2.2 Second level review: The data reviewer reviews all of the raw data making sure that the check standard passed and that the QC results are within acceptance ranges. The data reviewer makes sure that the correct result, date, and time were uploaded by the analyst, as well as any dilutions or comments. He/she is to compare the data entry report with the raw data, initialing and dating each hard copy. He/she also ensures that the calibration curves being used are acceptable and that the standards have not expired. Once the reviewer determines that everything is correct or that corrections need to be made, he/she returns the data to

the analyst, who makes the necessary corrections and/or sends the batch to "Done" status.

23.0 DATA TRANSMITTAL

After the review process is complete, Data Management generates the appropriate report.

24.0 RECORDS MANAGEMENT AND DOCUMENT CONTROL

All raw data along with the run sequence, batch sheet, and data entry report must be stored together. After a time, it is boxed and stored off-site.

25.0 LABORATORY WASTE HANDLING AND DISPOSAL: SAMPLES, EXTRACTS, DIGESTATES AND REAGENTS

Samples, extracts, solvent wastes, and expired standards are stored in a hazardous waste can. Once the can is full, it is emptied by the waste disposal specialist.

26.0 HISTORY

Revision 21: Removed reference to retired procedure GL-OA-E-002.
Revision 22: Updated Section 21 for compliance with 8000C and 8000D.
Revision 23: Updated to include Method 8000D requirements.
Revision 24: Revised definitions and instrument calibration sections for clarification of LLOQ. Added DOD/DOE QSM version 5.1.
Revision 25: Added statement for SC Samples, the LCS must pass within 70-130%.

APPENDIX 1

Herbicide Standard

Need two **10 mL** volumetric flasks for Initial Calibration Curve (ICAL) and Check Standards. Rinse clean with ether.

ICAL Intermediate Standard

1. Add 500 µL of surrogate standard to ICAL flask.
2. Add 200 µL of herbicide mix #1 to ICAL flask.
3. Add 200 µL of herbicide mix #2 to ICAL flask.
4. Add 200 µL of herbicide mix #3 to ICAL flask.

Dilute to the 10 mL mark with Ether.

Continuing Calibration Intermediate Standard

Same as above, but different lot numbers, serving as a second source.

Initial Calibration Curve (ICAL) Preparation

1. Need 7- 15 mL centrifuge tubes, rinsed clean with hexane.
2. Label each tube with the different concentrations 25 ppb, 50 ppb, 100 ppb, 150 ppb, 200 ppb, 300 ppb, 400 ppb.
3. 25 ppb add 62.5 µL of ICAL standard
4. 50 ppb add 125 µL of ICAL standard
5. 100 ppb add 250 µL of ICAL standard
6. 150 ppb add 375 µL of ICAL standard
7. 200 ppb add 500 µL of ICAL standard
8. 300 ppb add 750 µL of ICAL standard
9. 400 ppb add 1000 µL of ICAL standard

Continuing Calibration Curve Preparation

1. Need 4- 15 mL centrifuge tubes
2. The check standard is the same as the 200 ppb level 5 ICAL
3. Add 500 µL of Check Standard to each tube

To each tube add **0.5 mL of isooctane** and **0.25 mL methanol** to extract. Dilute to a volume of **4 mL of hexane**.

Add 1 mL of **diazomethane** and swirl. Cover rack with aluminum foil. Extract should stay yellow after 30 minutes. If it does not stay yellow add more diazomethane.

Add approximately 10 mg silicic acid which will destroy excess diazomethane. This does not need to be weighed on a balance. Let stand for 15 minutes. Bring to a final volume of 10 mL with **hexane**.

Pipet the standard from the centrifuge tube, taking care to avoid the silicic acid, and transfer to a clean 10 mL volumetric flask. (Intermediate herbicide standards are good for two months. Esterified herbicide standards are good for six months).

NOTE: Too much diazomethane will cause low recoveries of Dinoseb.

VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE FOR THE EXTRACTION OF HERBICIDES FROM GROUNDWATER, WASTEWATER, AND OTHER AQUEOUS SAMPLES

(GL-OA-E-015 REVISION 20)

APPLICABLE TO METHOD:
EPA Method 8151A for Herbicides

PROPRIETARY INFORMATION

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1.0 STANDARD OPERATING PROCEDURE FOR THE EXTRACTION OF HERBICIDES FROM GROUNDWATER, WASTEWATER, AND OTHER AQUEOUS SAMPLES

2.0 METHOD REFERENCE

EPA SW-846 8151A

3.0 PURPOSE

To describe the manner in which aqueous samples are extracted for selected herbicides listed in EPA Method 8151A.

4.0 DISCUSSION

Analytes listed in the above-referenced method are partitioned from aqueous matrices using separatory funnel extraction. An acid-base cleanup is integrated into the procedure prior to concentration using Kuderna-Danish (K-D) techniques. This solvent concentrate is esterified so that analytes are in a form that is suitable for analysis, using a gas chromatography technique that employs an electron capture detector.

5.0 INTERFERENCE TO THE METHOD

5.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts or elevated baselines in gas chromatograms. All these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis, by analyzing reagent blanks.

5.2 Glassware must be scrupulously cleaned. Clean each piece of glassware as soon as possible after use by rinsing it with the last solvent used in it. This should be followed by detergent washing with hot water and rinses with tap water, then with organic-free reagent water. Glassware should be solvent-rinsed with acetone and pesticide-quality hexane. After rinsing and drying, glassware should be sealed and stored in a clean environment to prevent any accumulation of dust or other contaminants. Separatory funnels are kept in the open lab. Immediately prior to use, glassware and separatory funnels should be rinsed with the next solvent to be used. Refer to GL-LB-E-003 for instructions.

5.3 The use of high purity reagents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

5.4 Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from waste to waste, depending upon the nature and diversity of the waste being sampled.

5.5 Organic acids, especially chlorinated acids, cause the most direct interference with the determination by methylation. Phenols, including chlorophenols, may also interfere with this procedure.

5.6 Alkaline hydrolysis and subsequent extraction of the basic solution removes many chlorinated hydrocarbons and phthalate esters that might otherwise interfere with the electron capture analysis. However, hydrolysis may result in the loss of

dinoseb and the formation of aldol condensation products if any residual acetone remains from the extraction of solids.

5.7 The herbicides, being strong organic acids, react readily with alkaline substances and may be lost during analysis. Therefore, glassware must be acid washed and then rinsed with organic-free reagent water. Sodium sulfate must be acidified.

5.8 Sample extracts should be dry prior to methylation or else poor recoveries will be obtained.

5.9 Method Detection Limit studies are performed annually for each analytical method associated with this SOP. Refer to GL-LB-E-001 for instructions.

6.0 DEFINITIONS

6.1 AlphaLIMS: The Laboratory Information Management System used at GEL Laboratories, LLC.

*6.2 Herbicide Spike: A solution added to the laboratory control sample, laboratory control sample duplicate, matrix spike, and matrix spike duplicate that contains all compounds of interest. These standards expire two months from date prepared. Please refer to the Maintaining Reference Material section of AlphaLIMS for analyte names and concentrations.

*6.3 Herbicide Surrogate: A compound with properties that mimic the analytes of interest, but that is unlikely to be found in environmental samples. The surrogate is added to each sample and quality control sample to monitor the extraction efficiency. 2,4-Dichlorophenyl acetic acid is used as the surrogate for this method. This standard expires two month from date prepared or the expiration date of the parent, which ever comes first.

6.4 Laboratory Control Sample (LCS): Organic free reagent water containing herbicide spiking solution to indicate the process is in control and to indicate accuracy.

6.5 Laboratory Duplicate (DUP, LCSD, MSD or PSD): Aliquots of a sample taken from the same container and processed in the same manner under identical laboratory conditions. The aliquot is analyzed independently from the parent sample and the results are compared to measure precision and accuracy.

6.6 Matrix Spike and Matrix Spike Duplicate (MS and MSD): An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS or MSD is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS or MSD corrected for background concentrations.

6.7 Method Blank (MB): An aliquot of reagent water or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other

samples. The MB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

6.8 Method Detection Limit (MDL): The minimum concentration of an analyte that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero. Typically, the MDL is two to three times less than the PQL.

6.9 Sample Duplicate: A duplicate of a sample indicating reproducibility.

6.10 Stock Standard Solution: A concentrated solution containing one or more method analytes prepared in the laboratory using certified reference materials or purchased from a reputable commercial source.

6.11 Lower Limit of Quantitation (LLOQ): The lowest concentration at which a target analyte can be reliably measured and reported. The LLOQ is the lowest point in the calibration curve and represents a concentration at which both quantitative and qualitative requirements can be consistently demonstrated. The LLOQ is verified quarterly, as the LOQ verification. The verification is performed by extracting and analyzing an LCS spiked at the lowest level of initial calibration curve (see the appropriate analytical SOP for calibration concentrations). The LLOQ verification is carried through the same preparation and analytical procedures as environmental samples and QC. The LLOQ is analyzed on every instrument where data are reported and this is the laboratory's normal protocol. Recovery of target analytes in the LLOQ are compared to in-house-statistically-derived limits. Concentrations in samples reported below the LLOQ and above the MDL are qualified as estimated.

6.12 Refer to GL-QS-B-001 the Quality Assurance Plan for additional lab-wide definitions.

(*) All herbicide surrogate and spike compounds are added in the free acid form. The free acid is used so that not only are you monitoring extraction efficiency, but also the derivitization efficiency. All standards are screened prior to use in extractions.

7.0 SAFETY, HEALTH, AND ENVIRONMENTAL HAZARDS

WARNING

METHYLENE CHLORIDE IS A POSSIBLE CARCINOGEN.

DIETHYL ETHER IS EXTREMELY FLAMMABLE.

HEXANE IS FLAMMABLE AND TOXIC.

ISOOCTANE IS EXTREMELY FLAMMABLE.

METHANOL IS TOXIC.

SODIUM HYDROXIDE CAUSES SEVERE BURNS AND MAY BE FATAL IF SWALLOWED.

SULFURIC ACID IS CORROSIVE, CAUSES SERIOUS BURNS, AND MAY BE FATAL IF SWALLOWED.

DIAZOMETHANE IS A CARCINOGEN.

DIAZOMETHANE IS EXTREMELY EXPLOSIVE.

7.1 Wear eye protection with side shields while working in the laboratory.

7.2 Treat all chemicals as potential health hazards, and limit exposure to them to the lowest level possible. GEL maintains a current awareness file of OSHA regulations regarding the safe handling of the chemicals used in this SOP. A reference file of Material Safety Data Sheets (MSDS) is available to all personnel involved in this SOP via GEL's Intranet. If necessary, contact the safety coordinator for specific MSDS sheets.

7.3 Personal protective equipment

7.3.1 Gloves are required when working with solvents, standards and samples. Solvents, along with any solute in them, can absorb easily through the skin.

7.3.2 Work under a hood when using concentrated acids.

7.3.3 To protect clothes and skin from being exposed to corrosive material, wear a lab coat.

7.4 Prior to handling radioactive samples analysts must have had radiation safety training and understand their full responsibilities in radioactive sample handling. Some general guidelines follow:

7.4.1 Wear a dosimeter at all times while working in the lab to monitor radioactive exposure.

7.4.2 Protect counter tops with counter paper or work from radioactive sample handling trays.

7.4.3 Prohibit admittance to immediate work area.

7.4.4 Post signs indicating radioactive samples are in the area.

7.4.5 Take swipes of the counter tops upon completion of work. Deliver those swipes to the designated swipe count box.

7.4.6 Segregate radioactive wastes. Radioactive waste containers are obtained from Waste Management.

7.5 All samples, chemicals, extracts, and extraction residues must be transferred, delivered, and disposed of safely according to all related SOPs.

7.5.1 Segregate solid wastes from liquid wastes in the satellite area containers.

7.5.2 Segregate oil wastes from water soluble wastes in the satellite area containers.

7.6 Solvent in a closed separatory funnel creates excessive pressure, so always vent immediately when beginning to shake.

7.7 When venting a separatory funnel, point stopcock end toward fume hood to minimize solvent fumes and always away from other people working in the area.

7.8 Hood doors should be pulled down partially while K-D apparatus are on the bath. Snyder columns may shoot off due to pressure inside the K-D. Always wet

Snyder column with 1 to 2 mL of the extraction solvent to help minimize a pressure build up.

7.9 In the event of an accident or medical emergency, call for help immediately. When time and safety permit, an accident report form should be completed and turned in to the safety committee.

7.10 Fire escape routes are posted in the lab and all personnel should be familiar with them. In addition, fire safety equipment such as fire extinguishers is located in the lab. Training is available on the proper operation of this equipment.

8.0 APPARATUS AND MATERIALS

8.1 Glassware

8.1.1 Separatory funnel, 2000 mL with Teflon stopcock or equivalent.

8.1.2 Concentrator tube, Kuderna-Danish 10 mL graduated (Kontes K-570050-1025 or equivalent). Calibration must be checked at the volumes employed in the test.

8.1.3 Evaporative flask, Kuderna-Danish 500 mL (Kontes K-570001-0500 or equivalent). Attach to concentrator tube with clips.

8.1.4 Snyder column, Kuderna-Danish three ball macro (Kontes K-503000-0121 or equivalent). Rotary evaporation set-up may be used alternatively.

8.1.5 Two mL glass vials with Teflon-lined cap (autosampler vials).

8.1.6 Disposable pipettes, Pasteur

8.1.7 Graduated cylinders, 1000 mL

8.1.8 Glass or Teflon funnels.

8.1.9 Vials 10 mL, glass with PTFE-lined screw caps.

8.2 Volumetric flasks, Class A,10 mL to 1000 mL.

8.2.1 Glass wool, Pyrex, acid washed.

8.2.2 Beaker, 400mL, thick-walled.

8.2.3 Erlenmeyer flasks, 250 mL and 2000 mL with a ground-glass joint at the neck

8.2.4 Centrifuge

8.2.5 Balance, analytical, capable of accurately weighing to 0.0001 g.

8.2.6 Diazald kit, recommended for the generation of diazomethane (Aldrich Chemical Co., No. 201,025-0, or equivalent)

8.2.7 Boiling chips, solvent rinsed, approximately 10/40 mesh (silicon carbide or equivalent)

8.2.8 Water bath, heated with concentric ring cover, capable of temperature control (± 5° C). The bath should be used in a hood.

8.2.9 Nitrogen evaporator with high purity (grade 4.5 or equivalent) nitrogen gas source.

9.0 REAGENTS AND STANDARDS

9.1 Reagents should meet ACS analytical criteria. Reagents should not be stored in plastic containers. Refer to GL-OA-E-065 for instructions.

9.1.1 Organic-free reagent water

9.1.2 12 N Sulfuric acid (H_2SO_4): Slowly add 335 mL of sulfuric acid to 665 mL of organic free reagent water in Erlenmeyer flask.

9.1.3 6 N Sodium hydroxide: Dissolve 216 g NaOH in 900 mL organic free reagent water.

9.1.4 Anhydrous sodium sulfate (Na_2SO_4), muffled at 400˚ C for 4 hours.

9.1.5 Acidified sodium sulfate: Cover anhydrous (Na_2SO_4) with ether. Carefully add 0.1mL concentrated sulfuric acid (H_2SO_4) per 100 g Na_2SO_4. Use a Buchner funnel to dry. Test pH of Na_2SO_4 by adding 1g to 5 mL deionized H_2O. pH of Na_2SO_4 must be < 4. Store at 130˚ C.

9.1.6 Sodium chloride (NaCl): Muffle salt at 400˚ C for 4 hours

9.1.7 Diazomethane (CH_2N_2): Refer to Appendix 1 for generation of diazomethane using Diazald kit. Reagents for generation include ether, potassium hydroxide, organic free reagent water, 2-(2-ethoxyethoxy) ethanol and Diazald.

9.1.8 Silicic Acid, (H_2SiO_5), 100 mesh powder, store at 130˚ C.

9.2 Solvents – should be screened prior to use. Refer to GL-OA-E-065 for instructions.

9.2.1 Methylene chloride (CH_2Cl_2), pesticide quality or equivalent

9.2.2 Diethyl ether ($C_2H_5OC_2H_5$), pesticide quality or equivalent. Check each bottle of ether for peroxides with peroxide strips. Do not use if peroxides are present.

9.2.3 Hexane (C_6H_{14}), pesticide quality or equivalent

9.2.4 Isooctane ($(CH_3)_3CH_2CHC(CH_3)_2$), pesticide quality or equivalent

9.2.5 Methanol (CH_3OH), pesticide quality or equivalent

9.2.6 LLOQ standard solutions are purchased directly from a certified vendor and may be diluted from the source to make working standards.

10.0 EQUIPMENT MAINTENANCE

Maintenance to these devices are recorded in LIMS in an electronic logbook.

11.0 SAMPLE HANDLING AND PRESERVATION

11.1 Sample Handling and Preservation

11.1.1 Sample containers should be glass or Teflon with a Teflon-lined screw cap. Plastic containers must not be used to prevent phthalate or hydrocarbon contamination.

11.1.2 Sample containers should be protected from light, for some analytes are light sensitive.

11.1.3 Samples are to be maintained at 0° ≤ 6° C until extraction begins.

11.1.4 Samples must be extracted within seven days from collection.

11.1.5 If samples are not in appropriate containers or holding time has expired, notify the Group Leader or Project Manager for further instructions.

12.0 SAMPLE PREPARATION

12.1 All batches (up to 20 samples) will be extracted with a blank, LCS, MS, and MSD. Additional QC may be required by specific clients and/or contracts. If insufficient sample is provided, the MS/MSD will be substituted with a LCS DUP.

12.2 Rinse all glassware with solvent. Label glassware with sample number.

12.3 Check sample pH and record.

NOTE: All pH measurements require the using a clean, 1 mL serological glass pipet to deliver a small aliquot of sample onto a wide-range pH indicator strip.

12.4 Pour sample into a solvent rinsed 2 L separatory funnel. When sufficient sample is provided, use entire 1 L amber bottle and mark water meniscus on sample container. Sample volume may be adjusted due to limited volume. Use 5 mL of sample for TCLP leachates. This sample volume meets TCLP limits.

12.5 Add 50 µL of surrogate to each separatory funnel. Add 1 mL spike to the appropriate separatory funnel (LCS, LCSD, MS, MSD). Surrogate and spike volumes may be adjusted in accordance to sample volume extracted. Peer witnessing is practiced during this process.

12.6 Add approximately 250 g sodium chloride to each sample separatory funnel. Sodium chloride amount may be adjusted in accordance to sample volume extracted. Seal and shake the sample to dissolve the salt.

12.7 Hydrolysis

12.7.1 Adjust sample pH to ≥ 12 with 17 mL 6N sodium hydroxide. Add 17 mL of 6 N sodium hydroxide to the sample, seal, and shake. Check the pH of the sample with pH paper. If the sample does not have a pH greater than or equal to 12, adjust the pH by adding more 6 N sodium hydroxide. Let the sample sit at room temperature until the hydrolysis step is completed (usually 1 to 2 hours), shaking the separatory funnel and contents periodically.

WARNING

METHYLENE CHLORIDE CREATES EXCESSIVE PRESSURE VERY RAPIDLY, POTENTIALLY CAUSING SEPARATORY FUNNEL TO EXPLODE.

12.7.2 Rinse the sample bottle by adding 60 mL of methylene chloride.

12.7.3 Transfer the rinsate to the appropriate 2 L separatory funnel. Seal, invert and vent immediately. Swirl sample to allow pressure release.

12.7.4 Shake vigorously for 2 minutes. Vent often. Allow layers to separate for a minimum of 10 minutes. If emulsion interface between layers is more than 1/3 size of solvent layer, employ one of the following mechanical techniques to break emulsion: stirring, filtration through glass wool, centrifugation, or other physical procedures.

12.7.5 Discard the methylene chloride phase.

12.7.6 Add a second 60 mL volume of methylene chloride to the separatory funnel, and repeat the extraction procedure a second time, discarding the methylene chloride layer.

12.7.7 Repeat step 12.7.6.

13.0 EXTRACTION

13.1 Adjust sample pH to ≤ 2 with 17 mL of cold (0˚ ≤ 6˚ C) 12 N sulfuric acid.

13.2 Add 120 mL of diethyl ether to sample, shake vigorously for 2 minutes.

13.3 Allow layers to separate from the water phase for a minimum of 10 minutes. If the emulsion interface between layers is more than 1/3 the size of the solvent layer, analyst must employ one of the following mechanical techniques to complete separation: swirl, stir with clean glass rod, quantitatively pass filtrate through acid-washed glass wool, or centrifugation.

13.4 Drain the sample (aqueous phase) into a 2 L Erlenmeyer flask.

13.5 Collect the diethyl ether extract in a rinsed 250 mL wide mouth Erlenmeyer flask containing approximately 10 g of acidified sodium sulfate.

13.6 Return the aqueous phase to the separatory funnel.

13.7 Repeat extraction two more times using 60 mL of ether each time combining the three extracts in the same Erlenmeyer.

13.8 If necessary, add more acidified sodium sulfate to the diethyl ether extract until there is no residue of water in the ether.

13.9 Allow extract to dry for at least two hours. Periodically, vigorously swirl the extract and drying agent. If not finishing after the two hours, place this extract in a refrigerator (0˚ ≤ 6˚ C) to keep the diethyl ether from evaporating.

NOTE: The drying step is critical to ensuring complete esterification. Any moisture remaining in the ether will result in low herbicide recoveries. The amount of sodium sulfate is adequate if some free flowing crystals are visible when swirling the flask. If all

the sodium sulfate solidifies in a cake, add a few additional grams of acidified sodium sulfate and again test by swirling. The 2-hour drying time is a minimum, however, the extracts may be held in contact with the sodium sulfate overnight.

13.10 Concentration

13.10.1 Set-up K-D and concentrator tube with a solvent rinsed boiling stone. Use a glass stirring rod to break up the sodium sulfate in the dried extract. If water is present, add more acidified sodium sulfate.

13.10.2 Pour the extract through a funnel containing pre-rinsed acidified glass wool, and collect in a K-D apparatus. Use a glass rod to crush any caked sodium sulfate during the transfer.

13.10.3 Rinse the flask with 20 to 30 mL of diethyl ether, transfer rinse to K-D through drying funnel if used. Add a Snyder column to K-D. Wet column for safety with 1 to 2 mL of ether.

13.10.4 Concentrate the diethyl ether to about 5.0 mL on a water bath with temperature between 60 - 70 °C. Remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes.

13.10.5 Concentrate to approximately 2 mL using the N-EVAP.

13.10.6 Transfer to 15 mL centrifuge tube or sample concentrator tube.

13.11 Esterification

13.11.1 Add 1.0 mL isooctane and 0.5 mL methanol to extract. Dilute to a final volume of 4 mL with diethyl ether.

WARNING

DIAZOMETHANE IS EXTREMELY EXPLOSIVE. USE COLD AND RETURN TO FREEZER WHEN FINISHED USING.

13.11.2 Add 2 mL diazomethane (refer to Appendix 1 for diazomethane generation) and swirl. Extract should stay yellow for a duration of 10-15 minutes. If it does not stay yellow, add more diazomethane.

13.11.3 Allow extract to evaporate in a hood to remove excess diazomethane until colorless (at least 6 hours or overnight), or add 10 mg of silicic acid, which will destroy excess diazomethane. Bring to a final volume of 10 mL with hexane in a 10 mL volumetric flask. The fizzing of the silicic acid should be finished before transferring to sample vials.

13.11.4 The sample is ready for analysis. Cap tightly. Store at $0^{\circ} \leq 6^{\circ}$ C immediately.

14.0 PREPARATION OF STANDARD SOLUTIONS AND QUALITY CONTROL SAMPLES

14.1 Source standards are purchased as certified mixtures. Documentation of the standard's quality and traceability should be provided by the vendor. This documentation entered into the Reference Materials table in LIMS. Standards may be purchased from outside vendors, including o2si smart solutions,

AccuStandard, NSI, Inc., and Supleco. Other vendors on GEL's Approved Vendors list may be also used. Herbicides standards are esterified. Refer to section 13.11. Refer also to GL-OA-E-065 for Reagent/Solvent/Standards Screening for Organic Prep.

14.2 Source standards are assigned a unique code number for the purpose of traceability.

14.3 The standard, along with its code, is recorded in AlphaLIMS. AlphaLIMS can be used to generate a label which is affixed to the standards container, or a handwritten label may be created.

14.4 Stock, intermediate, and working standards are likewise assigned a unique code number and recorded in AlphaLIMS.

15.0 SAFETY AND POLLUTION PREVENTION

Follow all laboratory safety rules for preparation, analysis and handling of the reagents of interest. Reference method SOPs and the GEL safety plan for guidance.

16.0 QUALITY CONTROL REQUIREMENTS

Typically, blank, laboratory control sample (LCS), matrix spike (MS) and matrix spike duplicate (MSD) are extracted and analyzed with each prep batch. However, this may vary depending on such factors as sample availability and client requirements. Other client requirements may include a laboratory control sample duplicate (LCSD). They are carried through all stages of sample preparation, extraction, and analysis.

17.0 DETECTION LIMIT

Method Detection Limit studies are performed annually for each analytical method associated with this SOP. Refer to GL-LB-E-001 for instructions.

18.0 RECORDS MANAGEMENT

18.1 Documentation of Training

18.1.1 Extraction technicians must be properly trained to perform the contents of this SOP. Personnel will extract four laboratory control samples for each analytical SOP referenced within this SOP as training commences. Training records are maintained as quality records.

18.1.2 LCS/LCS DUP will demonstrate on a continuing basis that personnel are properly trained.

18.2 Documentation of Extraction

18.2.1 Complete sample tracker form. Record initial volume of sample, final volume of extract, amount of surrogate and spikes added, and any comments about the extraction process. Record all reagent lot numbers like surrogate, spike and solvent lot numbers. Note any deviations from this standard operating procedure in the comment section. Also, note all discrepancies about sample handling and preservation.

18.2.2 Enter initial volume and final volume of each sample into AlphaLIMS and print a hard copy.

18.2.3 These documents are stored in AlphaLIMS. A copy is also maintained with the analytical data.

19.0 DATA REVIEW, APPROVAL, AND TRANSMITTAL

A review process is used to ensure quality of the data. Extraction logs are peer reviewed by a second technician or group leader. When the reviewer is satisfied that the data have been entered correctly, a data report is generated from AlphaLIMS. The report along with the batch sheets are copied and submitted to the appropriate analytical area for analysis.

20.0 LABORATORY WASTE HANDLING AND DISPOSAL

For the proper disposal of sample and reagent wastes from this procedure, refer to the Laboratory Waste Management Plan, GL-LB-G-001.

21.0 REFERENCES

21.1 Test Methods for Evaluating Solid Waste; Laboratory Manual Physical/ Chemical Methods, Volume 1B, SW-846, 3rd Edition, 1990. Method 8151A, "Chlorinated Herbicides by GC Using Methylation or Pentafluorobenzylation Derivation," Revision 1, December 1996.

21.2 AL-180 Technical Bulletin, "Aldrich, Diazald, MNNG and Diazomethane Generators." Sigma-Aldrich, Milwaukee, WI.

21.3 Dept. of Defense (DOD), Dept. of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories DOD QSM Version 5.1.1, February, 2018.

22.0 HISTORY

Revision 20: Updated QSM version, and clarified use of volumetric glassware in section 13.11

Revision 19: Updated to equipment used in process. Updated the time to process diazomethane. Updated the DOD QSM reference to 5.1.1, February, 2018.

Revision 18: Updated for clarification on process of contacting PM or Client if there is insufficient sample container or if hold time has expired.

Revision 17: Added LLOQ to definitions section. Updated standards section on how LLOQ standards and used in lab. Updated reference with current DOD/DOE QSM. Version 5.1, January, 2017

Revision 16: Changed concentration of diethyl ether from 1 mL to 2 mL.

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APPENDIX 1: GENERATION OF DIAZOMETHANE USING DIAZALD KIT

WARNING

DIAZOMETHANE IS A CARCINOGEN.

DIAZOMETHANE IS EXTREMELY EXPLOSIVE:

AVOID GRINDING SURFACES, GROUND GLASS JOINTS, SLEEVE BEARINGS, GLASS STIRRERS

DO NOT HEAT ABOVE 90˚C

STORE AWAY FROM ALKALI METALS

SOLUTIONS OF DIAZOMETHANE DECOMPOSE RAPIDLY IN THE PRESENCE OF SOLID MATERIALS SUCH AS COPPER POWDER, CALCIUM CHLORIDE AND BOILING CHIPS

1. Wash glassware in Diazald kit from Wheaton. This glassware is specially fitted without using ground glass joints. Do not use brushes. Rinse glassware with ether.

2. In the reaction vessel dissolve 2 g of potassium hydroxide in 4 mL DI water. Add 14 mL 2-(2-ethoxyethoxy) ethanol and 8 mL diethyl ether to the potassium hydroxide solution.

3. Attach condenser to chiller. Check chiller for water level. Add water if needed.

4. Attach receiving vessel to condenser and cool in ice bath.

5. Attach diethyl ether trap containing 1 to 2 mL diethyl ether and cool in ice/salt bath.

6. Fill the separatory funnel with a solution of 5 g Diazald in 45 mL diethyl ether and place over the reaction vessel.

7. Heat the reaction vessel at 65 ˚C ± 5˚ and add the Diazald solution at the rate of distillation.

8. Continue adding Diazald solution. When it is used, slowly add 10 mL of diethyl ether and continue distilling until the distillate is colorless.

9. Diazomethane is ready to use. Place diazomethane in Teflon-lined screw top bottle and store in freezer. Diazomethane may be stored as long as yellow color persists

VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE
FOR
VOLATILE ORGANIC COMPOUNDS (VOC)
BY
GAS CHROMATOGRAPHY/MASS SPECTROMETRY

(GL-OA-E-038 REVISION 28)

APPLICABLE TO METHODS:

EPA SW-846 Method 8000D	EPA SW-846 Method 5030A
EPA SW-846 Method 8260B	EPA SW-846 Method 5030B
EPA SW-846 Method 8260C	EPA SW-846 Method 5035
EPA SW-846 Method 8260D	EPA SW-846 Method 5035A
Standard Methods 6200	EPA SW-846 Method 3585

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1.0 STANDARD OPERATING PROCEDURE FOR VOLATLE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETER

2.0 METHOD OBJECTIVES, PURPOSE, AND CODE

2.1 This procedure is used to determine purgeable organic compounds. The compounds are purged out of a solvent and absorbed onto a trap, separated via the gas chromatograph, and detected using a mass spectrometer. The Mass Selective Detector analyzes minute amounts of material by fragmenting the sample into charged ions, separating them on the basis of molecular weight-to-charge ratio, and then counting the ions as they enter the detector. The counted ions identified by mass can be plotted on a graph with the mass on the x-axis and the counted ions or abundances on the y-axis.

2.2 The procedure can be used to quantify most volatile organic compounds that:

2.2.1 Have a boiling point below 200°C.

2.2.2 Are insoluble or slightly soluble in water.

2.3 Method Codes for this procedure are EPA SW-846 Methods 8000D, 8260B, 8260C, 8260D, 5030A, 5030B, 5035A, 3585 and Standard Methods 6200.

NOTE: For South Carolina samples, only 8260B is applicable.

3.0 APPLICABLE MATRIX AND MATRICES

3.1 Applicable matrices for this method include groundwater, aqueous sludges, caustic liquids, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filtercakes, spent carbons, spent catalysts, soils, tissues, wastewaters, and sediments.

4.0 METHOD SCOPE, DETECTION LIMITS, AND PRECISION/ACCURACY MEASUREMENTS

4.1 Calibration Range: Can be as low as 0.5 ppb up to 100 ppb for most analytes. The range for those analytes with poorer purging efficiencies can be as low as 5.0 ppb up to 5000 ppb.

4.2 Tested Concentration Range: The tested concentration will be the same as the linear calibration range, with no upper limit if dilutions are performed.

4.3 Method Detection Limits (MDL) and Practical Quantitation Limit (PQL):

4.3.1 Method detection limit studies are performed annually. See GL-LB-E-001 for requirements.

4.3.2 The practical quantitation limit is defined as the lowest concentration level used to standardize the instrument.

4.4 Method precision is measured using a Statistical Process Control (SPC). The SPC limits are generated annually for surrogate, laboratory control samples, and matrix spike analyses.

4.5 Method accuracy is measured using spiked samples of known concentration.

5.0 METHOD VARIATIONS

There are no significant variations to the method procedures described in this SOP. It should be noted that method 8260C requires method-specific criteria in addition to the criteria listed

in the body of this SOP. Specific criteria are outlined in Appendix 4 and Appendix 5. Method 6200 requires method-specific criteria in addition to the criteria listed in the body of this SOP. Specific criteria are outlined in Appendix 8. For 8260D method specified criteria is outlined in Appendix 9-11.

6.0 DEFINITIONS

6.1 Calibration Standard (CAL): A solution prepared from the primary dilution, standard solution, or stock standard solutions, and the internal standards and surrogate analytes. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

6.2 Continuing Calibration Verification (CCV) Standard: An aliquot of reagent water or other blank matrix to which known quantities of the method analytes are added in the laboratory. The CCV is analyzed exactly like a sample, at the beginning of the analytical sequence. Its purpose is to determine whether the instrument calibration is in control prior to sample analysis. It may be prepared from the same source as the calibration standards, and is usually of varied concentration. For SC samples the CCV must come from the same source standard that is used to prepare the initial calibration curve.

6.3 Holding Times (Maximum Allowable Holding Time): The maximum times that samples may be held prior to analysis and still be considered valid or not compromised. (SW-846, Table 2-40A)

6.4 Initial Calibration Verification (ICV): A solution of method analytes of known concentrations that is used to fortify an aliquot of blank or sample matrix. The ICV is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to validate an initial calibration curve with externally prepared test materials.

6.5 Initial Standard (ISTD): A known amount of standard added to a portion of the sample extract as a reference for evaluating and controlling the precision and bias of the applied analytical method.

6.6 Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, it must be a separate source standard from the initial calibration): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes of a matrix containing known and verified amounts of analytes. It is generally used to establish intra-laboratory or analyst-specific precision and bias or to assess the performance of all or a portion of the measurement system.

6.7 Limit of Detection (LOD): The lowest concentration level that can be determined by a single analysis and with a defined level of confidence to be statistically different from a blank.

6.8 Limit of Quantitation (LOQ): The lowest level in the calibration curve. With the prep factor applied, the LOQ is referred to as the effective LOQ. The LOQ is equivalent to the PQL and LLOQ.

6.9 Lower Limit of Quantitation (LLOQ): The lowest concentration at which a target analyte can be reliably measured and reported. The LLOQ is the lowest point in the calibration curve and represents a concentration at which both quantitative and qualitative requirements can be consistently demonstrated. The LLOQ is verified quarterly, as the LOQ verification. The verification is performed by extracting and analyzing an LCS spiked at the lowest level of initial calibration curve (see Appendix 3 for concentrations). The LLOQ verification is carried through the same preparation and analytical procedures as environmental samples and QC. The LLOQ is analyzed on every instrument where data are reported and this is the laboratory's normal protocol. Recovery of target analytes in the LLOQ are compared to in-house-statistically-derived limits. Concentrations in samples reported below the LLOQ and above the MDL are qualified as estimated.

6.10 Linear Calibration Range (LCR): The concentration range over which the instrument response is linear.

6.11 Method Detection Limit (MDL): The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined for analysis for a sample in a given matrix containing the analyte. (40 CFR Part 136 Appendix B)

6.12 Practical Quantitation Limit (PQL): The lowest level in the calibration curve. With the prep factor applied, the PQL is referred to as the effective PQL. The PQL is equivalent to the LOQ and the LLOQ.

6.13 Quantitation Limit (DoD clarification): The value at which an instrument can accurately measure an analyte at a specific concentration (i.e., a specific numeric concentration can be quantified). These points are established by the upper and lower limits of the calibration range.

6.14 Relative Percent Difference (RPD): The difference between two duplicate samples, such as a MS/MSD, LCS/LCSD, or sample/sample dup. It is determined by taking the difference between the two results and dividing by the average.

6.15 Statistical Process Control (SPC) Limits: Statistically derived limits, which establish acceptable ranges for recoveries of analytes of interest, including LCS, MS, MSD, PS, PSD and surrogate standards.

6.16 Target Analytes: Identified on a list of project specific analytes of which laboratory analysis is required.

6.17 Refer to GL-QS-B-001 the Quality Assurance Plan for additional lab-wide used definitions.

7.0 INTERFERENCES TO THE METHOD

7.1 Potential contaminants for this procedure include common laboratory solvents, typically ketones, methylene chloride, toluene, freons, and carbon disulfide. These compounds are used frequently in other areas of the laboratory and care must be taken to seclude the volatile organics lab and personnel from the other areas.

7.2 To minimize the contamination, use organic-free deionized water or equivalent for all analyses.

7.3 Cross contamination by carry-over can occur during sequential analyses. If cross-contamination is suspected, the affected sample or samples are reanalyzed.

7.4 The trap and other parts of the system, such as the column, are subject to contamination; therefore frequent bake out and purging of the system are required.

7.5 Sample vials are purchased as EPA Level 2 (pre-cleaned) or Level 3 (certified) and are certified free of organic contaminants. Glassware should be baked in an oven until needed. DO NOT USE HOT GLASSWARE. Wait until the glassware has cooled to room temperature before use.

8.0 SAFETY PRECAUTIONS AND HAZARD WARNINGS

8.1 Treat all chemicals and samples as potential health hazards. Exposure to these chemicals must be reduced to the lowest level possible. GEL maintains a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals as well as a reference file of Material Safety Data Sheets (MSDS). These documents are maintained in the laboratory. Individual sample MSDS forms provided by the clients are kept in Login.

8.2 Personal protective equipment

8.2.1 Approved gloves are required when handling standards and samples in this procedure.

8.2.2 Work under a hood when using concentrated acids, bases, and stock solutions or if samples are suspected of having high VOC contents.

8.2.3 Safety glasses and lab coats are required when handling samples and reagents.

8.3 Prior to handling radioactive samples, analysts must have had radiation safety training and must understand their full responsibilities in radioactive sample handling.

8.4 All samples, chemicals, extracts, and extraction residues must be transferred, delivered, and disposed of safely according to all related SOPs.

8.5 Never leave gas cylinders unchained or untied, including when they are on the moving carts.

8.6 In the event of an accident or medical emergency, seek help immediately. When time and safety permit, file an accident report form regardless of how insignificant the injury may seem.

8.7 Fire escape routes are posted in the lab, and all personnel should be familiar with them. In addition, fire safety equipment such as fire extinguishers is located in the lab. Training is available on the proper operation of this equipment.

8.8 The analyst must use great care when operating and assembling instrumentation and when handling samples and reagents. Check to see that the equipment is properly assembled and connected to gas and power, etc.

8.9 In an effort to maintain instrument integrity, take care to avoid running samples that contain over-range hits of analytes, especially hydrocarbons. Observe samples for foaming. Use caution when analyzing oils, multiphasic samples or samples suspected of containing high concentrations of VOC analytes.

8.10 Refer to SOP GL-LB-N-001 the Safety, Health and Chemical Hygiene Plan for additional general safety and health information pertaining to the laboratory.

9.0 APPARATUS AND MATERIALS; REAGENTS; EQUIPMENT AND INSTRUMENTS

9.1 Apparatus and Equipment

- 9.1.1 5 mL or 10 mL glass syringes
- 9.1.2 Gas Tight syringes: 10, 25, 50, 100, 250, 500, 1000 µL
- 9.1.3 Purge vessels, 40 mL glass vials, EPA certified level 2 or higher.
- 9.1.4 Drying oven
- 9.1.5 Refrigerator and freezer
- 9.1.6 Columns
- 9.1.7 Reactor vials with mininert valves
- 9.1.8 Centrifuge
- 9.1.9 Disposable pipets
- 9.1.10 Balance
- 9.1.11 2 mL vials with screw cap lids for storage of sample extracts (soil)
- 9.1.12 Scintillation vials with screw cap lids for storage of sample aliquots (liquids)

9.2 Instrumentation

- 9.2.1 Purge and trap system for water, soil, and waste samples. This system consists of an autosampler with a heater assembly and a concentrator.
- 9.2.2 The concentrator contains a trap which holds various types of adsorbent materials. Traps are obtained from OI Analytical and are type “10”. Equivalent traps may be used from alternate vendors.
- 9.2.3 The heater assembly is used to maintain samples at 40°C.
  - 9.2.3.1 Recommended Parameters (example only)
  - 9.2.3.2 Standby: 30° C
  - 9.2.3.3 Preheat: 1.0 minute
  - 9.2.3.4 Purge: 11.0 minutes
  - 9.2.3.5 Prepurge: 0.00
  - 9.2.3.6 Sample: 40° C
  - 9.2.3.7 Dry Purge: 0.00
  - 9.2.3.8 Desorb preheat: 150° C (or the recommended temperature per manufacturer for the type of trap installed).
  - 9.2.3.9 Desorb: 0.6 minutes at 180° C
  - 9.2.3.10 Bake: 10.00 minutes at 220° C

9.2.3.11 Purge flow: 25 mL/min to 40 mL/min depending upon each instrument

9.2.3.12 Valve: 120° C, Line: 120° C, Mount: 120° C

9.3 GC/MS System

9.3.1 This device consists of a Gas Chromatograph and Mass Selective Detector, capable of scanning from 35 to 300 amu every two seconds or less. A computerized data system is used to control or assist in instrument tuning, data acquisition, data reduction, report production, and automation.

9.3.2 The laboratory utilizes Agilent 5890, 6890 and 7890 and 7890B Gas Chromatographs and 5972, 5973, 5975 and 5977A Mass Selective Detectors. Agilent Chemstation/Enviroquant Software is used for data processing and evaluations.

9.4 Columns

9.4.1 J & W DB-624 or Restek RTX-624 or equivalent fused silica capillary. column 0.25 mm ID x 60 m, 1.4 µm film thickness.

9.4.2 Temperature Limits: (-20° C to 260° C).

9.4.3 Temperature Program: used to achieve optimal analyte separation.

10.0 REAGENTS AND STANDARDS

10.1 The water used for volatile organic analysis must be high quality organic free water or equivalent.

10.2 Methanol: Spectrum high purity solvent for purge and trap analysis or equivalent. Each lot of methanol is screened for contaminants prior to use. See GL-OA-E-065 Reagent/Solvent/Standard Screening for Organic prep.

10.3 Source Standard Solutions: Most source standard solutions are purchased from approved vendors. Source standards are used to make stock, intermediate, and working standards. The primary source standards used for calibrating the instrument are listed below. The names in parentheses are an alternate description for the compound.

Calibration Short List:

Acrylonitrile	cis-1,4-Dichloro-2-butene
Acrolein	Isopropyl alcohol
Allyl chloride (3-Chloropropene)	tert-Butyl alcohol
Cyclohexanone	Isopropyl ether (Diisopropyl ether)
Ethyl methacrylate	Ethyl-tert-butyl ether (ETBE)
Isobutyl alcohol (2-Methyl-1-propanol)	Methyl-tert-amyl ether (TAME)
Methacrylonitrile	Ethyl acetate
Methyl methacrylate	Benzyl chloride
Trichlorotrifluoroethane (Freon 113)	1-Chlorohexane
Pentachloroethane	2-Nitropropane
Tetrahydrofuran (THF)	bis(2-Chloro-1-methylethyl)ether
Propionitrile (Ethyl cyanide)	1,4-Dioxane
trans-1,4-Dichloro-2-butene	2-Chloro-1,3-Butadiene (chloroprene)

Calibration Long List:

Acetone	Methyl acetate
Acetonitrile	1,2-Dichloropropane
Benzene	2,2-Dichloropropane
Bromobenzene	1,3-Dichloropropane
Bromochloromethane	cis-1,2-Dichloroethene
Bromodichloromethane	cis-1,3-Dichloropropene
Bromoform	trans-1,2-Dichloroethene
Bromomethane (Methyl Bromide)	trans-1,3-Dichloropropene
2-Butanone (Methyl Ethyl Ketone, MEK)	Ethylbenzene
Toluene-d8(surr)	Ethyl ether (Diethyl ether)
n-Butylbenzene	Hexachlorobutadiene
sec-Butylbenzene	2-Hexanone
tert-Butylbenzene	Iodomethane
Carbon disulfide	Isopropylbenzene (Cumene)
Carbon tetrachloride	p-Isopropyltoluene (p-Cymene)
Chlorobenzene	4-Methyl-2-pentanone (MIBK)
Chloroethane	Naphthalene
Chloroform	n-Propylbenzene
Chloromethane (Methyl Chloride)	n-Butyl alcohol
Cyclohexane	Styrene
Cyclohexene	Tetrachloroethylene (Perchloroethylene) (PCE)
2-Chloroethylvinyl ether	Trichlorofluoromethane (Freon II)
2-Chlorotoluene	1,1,1,2-Tetrachloroethane
4-Chlorotoluene	1,1,2,2-Tetrachloroethane
Dibromochloromethane	tert-Butyl methyl ether (MTBE)
Methylene chloride(Dichloromethane)	Toluene
1,2-Dichloroethane-d4 (surr)	Trichloroethene (TCE)
1,2-Dibromoethane (EDB) (Ethylene dibromide)	1,2,3-Trichlorobenzene
Dibromomethane	1,2,4-Trichlorobenzene
1,2-Dichlorobenzene	1,1,2-Trichloroethane
1,3-Dichlorobenzene	Methylcyclohexane
1,4-Dichlorobenzene	1,2,4-Trimethylbenzene
1,1-Dichloroethane	1,3,5-Trimethylbenzene (Mesitylene)
1,1,1-Trichloroethane	o-Xylene
1,1-Dichloroethene	1,2-Dibromo-3-Chloropropane (DBCP)
1,1-Dichloropropene	m,p-Xylene
1,2-Dichloroethane	Vinyl acetate

Dichlorodifluoromethane (Freon 12)	Vinyl chloride
4-Bromofluorobenzene (surr)	1,2,3-Trichloropropane (TCP)

10.4 Surrogates: Toluene-d8, 4-Bromofluorobenzene and 1,2-Dichloroethane-d4. Other compounds may be used as surrogates, depending upon the analysis requirements. Final concentrations of the surrogates should be 50 µg/L. Surrogate analytes are calibrated in the Calibration Long List.

10.5 Internal standards: Fluorobenzene, Chlorobenzene-d5, and 1,4-Dichlorobenzene-d4. Other compounds may be used as internal standards as long as they have retention times similar to the compounds being detected by the GC/MS. Final concentration of internal standards should be 50 µg/L.

10.6 BFB Tune (4-Bromofluorobenzene): Prior to sample analysis, a 5 to 50 ng BFB solution must be analyzed. The BFB solution is 50 µg/L. One microliter of this solution is placed into 5 mL of deionized water. The tune is evaluated against the criteria in Appendix 2.

10.7 Calibration Standards: A minimum of five calibration standards must be used to establish instrument linearity. Each standard is prepared in a 10 mL or 5 mL syringe. The lowest concentration level used to standardize the instrument is considered the practical quantitation limit for the compound of interest. (Refer to Appendix 3 for calibration levels)

10.8 Matrix Spiking/LCS Standards: The spiking solution is a separate source standard from the calibration standards. A set of representative compounds is used to spike sample matrices. Each compound spiked into the sample is reviewed to evaluate the effect of matrix on the spiked compounds. Refer to client specific requirements for spiking list to be reported.

10.9 Volatile organic standards are stored at -10° to -20° C.

10.10 Non-gas standards have an expiration of one month from opening. Standards for the permanent gases must be replaced after one week of opening. Intermediate standards containing gases have a one week expiration date from the day that they are made, or the vendor expiration date if sooner. Volatile working standards are made on a daily basis and are never reused the following day.

10.11 AlphaLIMS software is used to inventory and document standard traceability. For guidance on standard documentation, refer to GL-LB-E-007 for Laboratory Standards Documentation.

11.0 PRESERVATION AND SAMPLE HANDLING REQUIREMENTS

11.1 Aqueous Samples

11.1.1 Aqueous samples must be stored at 0° ≤ 6° C from the time of collection until analysis. The samples should be collected and stored with no headspace, however, pea-size bubbles are acceptable. Large volumes of sediment and any headspace should be noted in the case narrative.

NOTE: Large volumes of headspace indicate that the sample was improperly collected. Notify the Project Manager, who is responsible for contacting the client and requesting that the sample be recollected. If recollection is not possible, flag the report to indicate that the samples were improperly collected. In either case, nonconformance to proper sample collection should be noted in the case narrative.

11.1.2 Water samples should not contain any headspace and should have pH checks with pH paper strips immediately prior to analysis. Aqueous samples are preserved with hydrochloric acid to a pH less than 2 and have a 14-day holding time. Unpreserved waters have a holding time of 7 days from collection. If samples are analyzed after the 7 days with a pH above 2 the nonconformance is documented in the case narrative, the client is notified and the results are flagged accordingly. Samples that request Acrolein and Acrylonitirile must be preserved to a pH of 4-5 and analyzed with 7 days unless the applicable sampling and analysis plan or QAPP specifies otherwise.

11.1.3 Upon obtaining the aliquot for analysis, waters shall be checked for residual chlorine. The residual chlorine check is similar to the pH measurement, using a chlorine test strip. The presence of residual chlorine is documented in the runlog as “Yes” or “No”. If residual chlorine is present in samples, the analyst should document the information on the runlog, in the case narrative, and notify the project manager.

11.2 Soil Samples

11.2.1 Soil samples collected for SW-846 8260 should be unpreserved or preserved in the field with specific collection kits or collected in EnCore samplers. EnCore samplers must be sent to the lab and preserved within 48 hours of collection. Preservation can be client specific however, refer to GL-OA-E-039 (SW-846 5035/SW-846 5035A, collection and preservation) for laboratory standard procedures. Method 5035 list two preservatives-sodium bisulfate and methanol. The laboratory does not analyze samples preserved in sodium bisulfate.

11.2.1.1 Low Level Soil Sample Prep

11.2.1.1.1 Using a sampling device (such as an EnCore), three sets of approximately 5 g of soil are collected per sampling site. Two are prepared for Low Level and one for High Level analysis.

11.2.1.1.2 Transfer the soil from each EnCore device to a tared 40 mL vial. Reweigh the vial (now containing the soil) and record the weight in the Soil Prep Logbook as well as on the sample label. Add 5 mL of organic free water and a magnetic stir bar before sealing the vial. Store unpreserved (water only)

samples in the freezer until analysis. Sample weights are recorded in the soil prep logbook, in AlphaLIMS in the sample prep batch and on the instrument runlog.

11.2.1.2 High Level Soils Collected in EnCore Samplers

11.2.1.2.1 Transfer the soil (approximately 5 g) from the sampling device to a tared 40 mL vial. Record the soil weight in the soil prep logbook. Add 10 mL of purge and trap grade methanol. The volume of methanol added during the extraction process can vary from 5 mL to greater than 10 mL depending on client requirements or matrix. Replace the cap and gently shake for 2 minutes. Allow the soil to settle. Centrifuge if necessary. Transfer an aliquot of the methanol extract to a 2.0 mL vial with minimum headspace, label appropriately, and store in the refrigerator until analysis. Discard the remaining soil/extract in appropriate manner for solvent/solid waste per GL-LB-G-001 for Laboratory Waste Management Plan. Sample weights are recorded in the soil prep logbook, in AlphaLIMS in the sample prep batch and on the instrument runlog.

11.2.1.2.2 A methanol solvent blank (HB) is prepared by placing a portion of the methanol used for sample extractions into a 2.0 mL vial with minimal headspace. The labeled vial is stored with the soil extracts at 0° C ≤ 6° C until the time of analysis. It is analyzed under the same conditions as the associated samples. Methanol used in the laboratory is screened by lot prior to use.

11.3 Solids or liquid waste and oils should not be acid preserved. These types of samples should be stored at 0° ≤ 6° C from the time of collection until analysis.

11.4 Analysts receive the samples directly from the storage cooler. Samples must be tracked into the analyst's custody, then tracked back to the cooler or archive upon completion of the analysis.

11.5 If samples are analyzed out of holding or the correct preservation was not performed, the analyst must immediately notify the Project Manager. Additional information for the nonconformance should be indicated in the batch and/or fractional case narrative.

12.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

12.1 Low Level Soil Sample Preservation (SW-846 5035/SW-846 5035A)

12.1.1 Preserved samples received from the field are wiped to remove any extraneous material from the exterior of the vial. The vial is weighed to the nearest 0.1 g and the weight is recorded in the soil prep logbook. The weights are entered into the AlphaLIMS system in the sample prep batch. The analyst should observe the vial for any indication for leaks or cracks. If any significant observations are detected, the project manager is notified and the appropriate action is taken.

12.1.2 Allow the sample to warm to room temperature. Shake the vial gently to ensure that the contents move freely and that stirring will be effective. Place the vial in the autosampler and ensure the autosampler will add the appropriate volumes of organic-free water, surrogates, and internal standards to all vials. All samples, blanks, and calibration standards are analyzed with the same final volume.

12.1.3 When low level soil analysis produces target analyte results with concentrations higher than the calibration range of the instrument, the sample is reanalyzed using the prepared aliquot from the High Level Soil Sample Prep (11.2.2). Such reanalysis need only address those analytes for which the concentration exceeded the calibration range of the Low Level Method. The maximum volume of methanol analyzed is 100 µL.

12.1.4 Analysts should never open a 5035 preserved low level soil vial from the field or EnCore sampler. All spiking must be performed through the vial septum with a syringe or autosampler needle.

12.1.5 Results are to be reported on a dry weight basis unless otherwise specified by the client.

NOTE: Low level soil samples originating in South Carolina must be collected and analyzed using EPA Methods 5035.

12.2 High Level Soil Sample Preparation (SW-846 5035/SW-846 5035A)

12.2.1 High Level soils collected in EnCore samplers

12.2.1.1 To analyze the methanol extract, transfer 100 µL of the extract to a 5 mL syringe containing 4.9 mL of organic-free water. Transfer the fortified water to an autosampler vial (40 mL vial with septum cap). Place the vial in the autosampler. If concentrations of analytes exceed the calibration range, the sample is reanalyzed using a smaller volume of sample aliquot.

12.2.1.2 For high level soils preserved in the field, this aliquot may or may not be analyzed depending on the concentration of the analytes present in the sample. Prior to analysis, allow the soil/methanol extract to reach room temperature. Withdraw 100 µL or the desired volume from the vial. Transfer the methanol extract to a syringe containing 4.9 mL of organic-free water.

Transfer the fortified water to an autosampler vial (40 mL volatiles vial). Place vial in autosampler.

12.2.1.3 All sample analyses for high level soils using the methanol extract procedure require the preparation and analysis of a methanol blank (HB) if the samples are prepared at the laboratory. Field prepared samples may or may not include a methanol aliquot for analysis. Place 100 µL of the methanol in a syringe containing 4.9 mL of organic-free water. Transfer the water to an autosampler vial (40 mL volatiles vial). Place the vial in the autosampler. The acceptance criteria applied to the HB should be the same as method blank criteria outlined in 17.2.3.

12.3 Low Level Soil analysis: Allow samples to reach room temperature. Weigh approximately 5 g (± 0.1 g) of sample into a 40 mL autosampler vial. Document the weight used in the soil preparation logbook and on the instrument runlog. The weights are also entered into AlphaLIMS system in the sample prep batch. Add 5 mL of organic-free water to the vial and a stir bar. Gently shake the sample vial to ensure the magnetic stir bar moves freely. Place the sample vial in the appropriate position and complete the autosampler sequence as desired to purge each sample.

NOTE: The sample may be diluted by weighing less sample. Do not analyze less than 1.0g of the solid sample. If a higher dilution is required, use the high level soil sample preparation method.

12.4 High Level Soil Preparation (SW-846 5035/SW-846 5030C)

12.4.1 Solid samples that contain concentrations of analytes greater than the highest concentration level in the calibration or samples with high viscosity, are extracted with methanol, and then diluted in water. Approximately 5 g of sample is weighed out and extracted with 10 mL of methanol. The methanol extract is gently shaken for two minutes then allowed to settle until distinct layers form. Centrifuge if necessary. An aliquot of the methanol layer is then transferred to a 2.0 mL vial. The extract is then analyzed immediately or the vial is stored in the sample refrigerator until analysis. The dilution factor is 1:50 if 100 µL of sample is used. The minimum amount of soil that can be used is 1 gram.

12.4.2 Oily waste samples or samples of unknown solid matrix are extracted following SW 846 Method 3585 with some modifications. SW 846 Method 3585 suggests n-Hexadecane or another appropriate solvent be used as the extraction solvent when performing waste dilutions on oily matrices. The laboratory uses purge and trap methanol as the extraction solvent for these types of sample preparations. The samples are prepared by weighing a 1 g aliquot into a VOA vial and adding an appropriate volume of purge and trap methanol to meet the client's detection limit requirements if possible.

This volume is usually 5 to 10 mL but can be as high as 20 mL. The methanol extract is gently shaken for two minutes then allowed to settle until distinct layers form. Centrifuge if necessary. An aliquot of the methanol layer is then transferred to a 2.0 mL vial. The methanol volume may be increased if the matrix absorbs the initial volume of methanol used. The total volume of methanol used is documented in the soil prep logbook and AlphaLIMS. Care should be taken with any oily or unknown matrix to avoid contamination to the analytical system from high concentrations of targets or non-target analytes. Methanol is screened by lot prior to use.

12.5 Aqueous analysis (5030B)

12.5.1 Allow samples to reach room temperature. Invert each sample vial to check for headspace. Gently pour approximately 6 mL of the aqueous sample into a 10 mL syringe. Place the plunger in the syringe and invert, allowing any bubbles to escape through the end fitting. Adjust the sample volume 5.0 mL by depressing the plunger to the mark. The extraneous sample volume may be added back to the original sample vial. Transfer the 5.0 mL of sample to a clean 40 mL autosampler vial and place the vial in the appropriate autosampler position. Complete the autosampler sequence as desired.

12.5.2 After obtaining that aliquot for analysis, the pH of the sample is checked using wide range pH paper. Samples should be preserved to a pH <2. Document pH on instrument run log. Notify project manager of samples with a pH >2 if not analyzed within 7 days from collection. Analysts should always obtain their sample aliquots before taking pH. If sample volumes are limited (one vial per sample) the analyst should transfer a sample aliquot to a smaller container with no headspace (scintillation vial) if additional testing may be required. For limited volume samples, the analyst should obtain the pH of the sample by pipetting a small volume of the sample across the pH strip into a waste container, otherwise the pH strip may be dipped in the sample vial and the vial discarded.
NOTE: Make a note of any sample discrepancies, i.e. unpreserved sample, any headspace, and excessive sediment in the batch case narrative and on the instrument runlog.

12.5.3 After obtaining the aliquot for analysis, waters shall be checked for residual chlorine. Residual chlorine check is similar to the pH measurement, using a chlorine test strip. Additional sample aliquots cannot be taken from the vial once the residual chlorine test has been performed. For limited sample volumes the steps outlined in 12.5.2 may be necessary to preserve sample aliquots for future testing. The presence of residual chlorine is documented in the run log as “Yes” or “No”. If

residual chlorine is present in samples, the analyst should document the information in the case narrative, and notify the project manager.

12.5.4 TCLP extracts and Tumble blanks are diluted prior to analysis for a total 5 mL prep. Transfer the 5 mL of sample to a clean 40 mL autosampler vial and place the vial in the appropriate autosampler position. Complete the autosampler sequence as desired. The batch QC requirements are a tumble blank, method blank, LCS, post spike and post spike duplicate.

13.0 QUALITY CONTROL SAMPLE FOR SOLID AND AQUEOUS SAMPLES

13.1 After preparing and prior to purging, fortify the appropriate samples for matrix spike purposes using an amount of MS/LCS spike solution needed to achieve the desired concentration.

13.2 Preserved soils (5035) should be fortified by inserting the syringe needle into the vial septum and injecting the spike solution into the liquid. Tilt the vial during this process to ensure that the spike solution enters the liquid phase of the sample.

13.3 Aqueous and/or 5035A solid samples can be fortified in the syringe prior to transferring the aqueous sample or organic free water to the autosampler vial.

13.4 Prepare the Laboratory Control Sample (LCS) for solids and liquids by injecting the appropriate amount of spike solution needed to achieve the desired concentration into a gas tight syringe containing 5 mL organic free water. Transfer the syringe contents to an empty 40 mL autosampler vial or one containing 5 g of sand.

13.5 Prepare aqueous method blanks by transferring 5 mL organic free water to an empty 40 mL autosampler vial. Prepare solid method blanks by transferring 5 mL of organic free water to a 40 mL autosampler vial containing a stir bar and 5 g of sand.

13.6 Quality control samples for solids should contain 5 g of sand and a stirbar.

14.0 OVERALL QUALITY CONTROL REQUIREMENTS

14.1 Prior to any quality control or sample analysis, the analyst must perform an instrument check by injecting 5 to 50 ng of Bromofluorobenzene (BFB). The BFB tune check must be performed prior to each analytical sequence. A new BFB check must be performed after 12 hours of analysis.

14.2 Prior to any sample analysis the instrument must be calibrated. Initial calibration standards are analyzed if the calibration verification standards do not meet the acceptance criteria or if instrument maintenance was performed (i.e., source cleaning, new column, replacing source parts).

14.3 The initial calibration must be verified in each 12-hour sequence by purging a mid-level calibration verification standard. For SC samples the calibration verification standard must come from the same source standard that is used to prepare the initial calibration curve.

14.4 A laboratory control sample must be analyzed during each 12-hour sequence by purging a second source mid-level standard.

14.5 A method blank must be analyzed during each 12-hour sequence by purging organic free water.

14.6 Matrix spike and matrix spike duplicate samples are analyzed in each batch of up to 20 samples of the same matrix. Many clients require their sample to be used for matrix spike purposes. Refer to client specifications in AlphaLIMS for this information.

14.7 A laboratory control sample duplicate should be analyzed if matrix volumes are limited.

14.8 Surrogate compounds and internal standards are added to each sample and all quality control samples.

15.0 INSTRUMENT CALIBRATION, STANDARDIZATION AND PERFORMANCE

15.1 Prior to the analysis of calibration standards, inject or introduce 5 to 50 ng of the bromofluorobenzene (BFB) standard into the GC/MS system. The mass spectra of BFB can be obtained using an average of up to three scans and using a background subtraction from no more than 20 scans before the peak starts. However, the analyst may use other documented approaches by the instrument manufacturer.

15.2 Initial Calibration. An unpreserved calibration curve may be used for high level methanol extracts, waters and low level soils. Standards at various concentrations are analyzed and the instrument responses they generate are entered into the analytical method. Traceability of calibration and calibration verification standards is documented per GL-LB-E-007. Individual identification numbers are assigned to each source standard. The calibration curve must be a contiguous subset of the original set.

NOTE: Prior to tuning or running a multi-point calibration, precautions are taken to ensure that the instrument is clean and functioning properly. Standards should be set up from low concentration to high concentration in deionized water (Refer to Appendix 1). Once the instrument methods have been established the mass range and scan time should not be readjusted.

15.2.1 Establish the GC/MS operating conditions using the following guidelines:

15.2.1.1 Mass Range: 35-300

15.2.1.2 Scan time: 0.6-2 sec/scan

15.2.2 Calibration standards are purchased at multiple concentration levels and packaged separately in sealed ampules. Upon cracking the ampule for each standard, transfer each solution into a micro reaction vessel with a mininert cap. Indicate on the vial label the unique standard identification number, date opened, and the expiration date plus the analyst's initials preparing/opening the standards.

15.2.3 Load a 10 mL gas tight syringe with 5mL of organic-free water. Transfer 5 µL of calibration standard into the syringe and then transfer to a 40 mL vial. Repeat this step for each concentration level of standard. The Calibration Long List is separated into gases and non-gas compounds. The analyst must add each set of concentration levels to a syringe in order to analyze for all the compounds in this list. Similarly, the Calibration Short List is prepared by adding 5 µL of the Short List standard plus 5 µL of the

Acrolein/Cyclohexanone standard into a syringe containing 5 mL of deionized water.

15.2.4 Set up the autosampler to analyze each standard from low to high concentration.

15.2.5 Tabulate the area response of the characteristic ions against concentration for each compound and each internal standard. Calculate response factors (RF) for each compound relative to its internal standard. The internal standard selected for the calculation of the RF for a compound should be the internal standard that has a retention time closest to the compound being measured. The RF is calculated as follows:

$$RF = (AxCis) / (AisCx)$$

Where:

Ax = Area of the characteristic ion of the compound being measured.

Ais = Area of the characteristic ion of the specific internal standard.

Cis = Concentration of the specific internal standard.

Cx = Concentration of the compound being measured.

15.2.6 For 8260B the average RF must be calculated for each compound. Five compounds (the System Performance Check Compounds, or SPCCs) are checked for minimum average response factors. These compounds are: Chloromethane, 1,1-Dichloroethane, Bromoform, 1,1,2,2-Tetrachloroethane, and Chlorobenzene. The minimum acceptable average RF for these compounds is: 0.1 for Chloromethane, 1,1-Dichloroethane, and Bromoform, 0.3 for Chlorobenzene and 1,1,2,2-Tetrachloroethane. These compounds typically have RFs of 0.4 to 0.6 and are used to check compound instability and degradation caused by contaminated lines or active sites in the system. Failure to meet the response factor criteria may be attributed to one or more of the following examples:

- Chloromethane: This compound is the most likely compound to be lost if the purge flow is too fast.
- Bromoform: This compound is one of the compounds most likely to be purged very poorly if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response. Response of the quantitation ion (m/z 173) is directly affected by the tuning of BFB at ions m/z 174/176. Increasing the m/z 174/176 ratio may improve Bromoform response.
- 1,1,2,2-Tetrachloroethane and 1,1-Dichloroethane: These compounds are degraded by contaminated transfer lines in purge-and-trap systems and/or active sites in trapping materials.

NOTE: Non-SPCC compounds should have a minimum response factor of 0.01, with the exception of poor purging compounds such as those listed in Appendix 6. Unless otherwise required by client

contract, these compounds may have response factors as low as 0.001. For the analysis of ketones and carbon disulfide at a PQL less than 5 ppb, the relative response factor for these analytes must be equal to or greater than 0.05. Method 8260C has additional requirements for response factors. Refer to Appendix 5. Method 8260D also has additional guidance for response factors however, it is not required.

15.2.7 Calibration curves must be verified using an initial calibration verification standard (ICV). This should be a second source standard from the initial calibration. The laboratory control spike solution can be used for this standard. The response factor or true value (percent difference or drift) is evaluated based on individual client requirements. At minimum the SPCC and CCC's criteria for response and percent drift or difference must be met. For samples from within the state of South Carolina the percent drift or difference should not exceed +/- 30% for requested analytes and +/- 40% for poor purgers (See Appendix 6). The percent drift or difference for non-CCC compounds may be as high as 60% for some clients. For DoD clients the percent drift or percent difference should not exceed ± 20% for requested analytes. If the ICV does not meet the criterion, corrective action should be taken (i.e., inject a different second source, open a new standard mix, perform instrument maintenance and/or reanalyze the initial calibration). If individual client requirements are not met and holding times are expiring, the group leader is notified. Clients will be notified of the nonconformance and analysis of the samples may proceed. Note any nonconformance in the case narrative. See Appendix 4 for 8260C specific criteria. See Appendix 9 for 8260D criteria.

15.2.8 All newly generated calibration curves shall have a secondary review by a Group Leader or Data Validator before being used.

15.2.9 The internal standard responses and retention times in the calibration standards must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds during the calibration, the chromatographic system must be inspected for malfunctions and corrections must be made, as required. If the Extracted Ion Current profile (EICP) area for any of the internal standards changes by a factor of two (-50% to +100%) during the calibration, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made reanalysis of the calibration check standard is necessary.

15.2.10 Using the $\overline{RF}$ s from the initial calibration, calculate the percent relative standard deviation (%RSD) for Calibration Check Compounds (CCCs) and all target analytes:

$$\%\text{RSD} = \frac{\text{SD}}{\bar{x}} \quad 100$$

Where:

RSD = Relative Standard Deviation

$\bar{x}$ = Mean of initial RFs for a compound

SD = Standard deviation of average RFs for a compound

$$SD = \sqrt{\sum_{i=1}^{n} \frac{\left(x_i - \bar{x}\right)^2}{n-1}}$$

15.2.11 If the RSD of any target analyte is 15% or less, then the response factor is assumed to be constant over the calibration range and may be used for quantitation. If the RSD of any target analyte is greater than 15%, then use one of the options in section 15.2.13. A minimum of five calibration standards must be used. The laboratory may use more than five concentration levels. The maximum % RSD for NNSA client is 60% Refer to Appendix 4 for 8260C and Appendix 10 for 8260D

15.2.12 The % RSD for each individual CCC must be less than or equal to 30 percent difference. This criterion must be met in order for the individual calibration to be valid. The CCCs are:

- 1,1-Dichlorethane
- Chloroform
- 1,2-Dichloropropane
- Toluene
- Ethylbenzene
- Vinyl Chloride

15.2.13 Given the potentially large numbers of analytes that may be analyzed by this method, it is likely that some analytes may exceed that 15% acceptance limits for RSD. In those instances, the following steps may be used:
NOTE: Refer to Appendix 4 and 10 for 8260C criteria.

15.2.13.1 Check the instrument operating conditions. Follow the suggested maintenance procedure in this method to make minor adjustments to the system.

15.2.13.2 Review the results of each standard (area counts, calibration or response factors and RSD) to ensure that the problem is not associated with one of the five initial calibration standards. If the problem appears to be associated with a single standard, then the entire calibration must be reanalyzed.

15.2.13.3 High-end standards may be removed on a compound by compound basis thus narrowing the calibration range of the instrument if there are enough remaining calibration points to meet method requirements. The top of the calibration curve may

not be the mid-point spiking concentration. At least one point must be in the calibration curve above this concentration.

15.2.13.4 The analyst may also choose to construct calibration curves of area ratios versus concentration using first order regression fit of the five calibration points. The regression calculation will generate a coefficient of determination (r2) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r2 must be ≥ 0.990. The minimum acceptable correlation coefficient (r) for any compound using linear regression shall be 0.995. The analyst will select only linear regression or average response factor. Higher order curves such as a quadratic curve must never be used. Correlation coefficients may not be rounded up to meet these requirements.

15.2.13.5 Make certain that the instrument response is treated as the dependent variable (y) and the concentration as the independent variable (x). The regression will produce the slope and intercept terms for a linear equation in the form:

$$y = ax + b$$

Where:

y = instrument response

a = slope of the line (also called the "coefficient of x")

x = concentration of the calibration standard

b = the intercept

15.2.13.6 If the analyst chooses to use linear regression, he/she must not force the calibration line through the origin but have the intercept calculated from the data points. In addition, do not include the origin (0,0) as a sixth calibration point.

15.2.13.7 The calculated intercept value needs to be evaluated before reporting sample results. A positive value for the intercept indicates that there is some threshold instrument response that is the limiting factor in establishing linearity. A negative intercept value can be transformed into an x-intercept value that represents a threshold concentration that is the limitation. If the intercept is positive, then as a general rule, results where the instrument response is less than three (3x) the intercept value may be unreliable. This will afford some protection against false positive results. If the intercept is negative, results below the concentration of the lowest concentration calibration standard may be unreliable. The value of the y intercept multiplied by the internal standard concentration must be less than 3 times the

value of the MDL for each matrix for analysis of NNSA client samples. A weighted linear calibration curve may be generated and the intercept re-evaluated for NNSA requirements.

15.2.13.8 In calculating the sample concentrations, the regression equation is rearranged to solve for the concentration (x):

y = ax + b: linear equation

x = (y-b)/a

15.2.14 Exclusion of initial calibration points without technical justification is not allowed. Occasionally, it may be obvious that a condition such as a bad injection or purge had rendered an initial calibration standard unusable.

15.2.14.1 If it is necessary to replace a calibration level in a curve, the entire level must be replaced and not individual analytes.

15.2.14.2 The questionable level may be reanalyzed as long as the reanalysis is performed within the 12 hour window. The second injection must be used in the calibration curve.

15.2.14.2.1 Should the second injection fail, the entire calibration curve must be reanalyzed.

15.2.14.2.2 If the second injection passes, reanalyze the initial calibration verification sample.

15.2.15 Method 8000D outlines two procedures that may be used to determine calibration function acceptability for linear and non-linear curves. These include refitting the calibration data back to the model. % Error and Relative Standard Error (RSE) evaluate the difference between the measured amount and the true amount (or concentration).

15.2.16 % Error is determined as follows:

$$\%Error = \frac{x_i - x_i'}{x_i} \times 100$$

Where:

x′i= Measured amount of analyte at calibration level i, in mass or concentration units

xi= True amount of analyte at calibration level i, in mass or concentration units.

15.2.17 Percent error between the calculated and expected amounts of an analyte should be ≤ 30% for all standards. For some data uses, ≤ 50% may be acceptable for the lowest concentration point.

15.2.18 Relative Standard Error is calculated as follows:

$$RSE = 100 \times \sqrt{\sum_{i=1}^{n} \left[\frac{x_i' - x_i}{x_i} \right]^2 \Big/ (n - p)}$$

Where:

xi= True amount of analyte in calibration level i, in mass or concentration units

x′i= Measured amount of analyte in calibration level i, in mass or concentration units

p= Number of terms in the fitting equation (average =1, linear =2, quadratic =3, cubic =4)

n= Number of calibration points

15.2.19 The RSE acceptance limit criterion for the calibration model is the same as the RSD limit in the determinative method. If the RSD limit is not defined in the determinative method, the limit should be set at ≤ 20% for good performing compounds and ≤ 30% for poor responding compounds.

15.3 Continuing Calibration Verification

15.3.1 Prior to the analysis of calibration verification standards, inject or introduce 5 to 50 ng of the Bromofluorobenzene standard into the GC/MS system. The resulting mass spectra of the BFB must meet all of the criteria given in Appendix 2 before sample analysis begins. The mass spectrum of BFB can be obtained using an average of up to three scans and using a background subtraction of a single scan no more than 20 scans before the peak starts. However, the analyst may use other documented approaches suggested by the instrument manufacturer.

15.3.2 The initial calibration curve for each compound of interest must be checked and verified once every 12 hours of analysis time. This is accomplished by analyzing a calibration standard that is at a concentration near the mid-point concentration for the working range of the GC/MS by checking SPCCs and CCCs. This will determine the validity of the initial calibration on a daily basis. The continuing calibration check standard may be a second source standard. For SC samples the CCV must come from the same source standard that is used to prepare the initial calibration curve. Refer to Appendix 4 for 8260C and Appendix 8 for Standard Method 6200 specific criteria and Appendix 10 for 8260D for specific criteria.

15.3.3 System Performance Check Compounds (SPCCs): A calibration verification check must be made every 12 hours. If the SPCC criteria are met a comparison of response factors is made for all compounds. This is the same check that is applied during the initial calibration. The minimum response factors for volatile SPCCs are listed in section 15.2.6 of this SOP. Some possible problems that may prevent one from meeting the criteria are outlined in that section.

15.3.4 Calibration Check Compounds (CCCs): After the system performance check is met, CCCs are used to check the validity of the initial calibration. Calculate the percent difference using:

$$\%\text{Difference} = \frac{\left|\overline{RF_I} - RF_c\right|}{\overline{RF_I}} \times 100$$

Where:

$\overline{RF_I}$ = Average response factor from initial calibration

RFC = Response factor from current verification check standard

15.3.5 If the percent difference for all CCCs is less than 20%, the initial calibration is assumed to be valid. If the criterion is not met (> 20% difference), corrective action must be taken. Problems similar to those listed under SPCCs could affect this criterion. If no source of the problem can be determined after corrective action has been taken, a new calibration will be analyzed.

15.3.5.1 Passing CCC and SPCC criteria should not be the only factors in evaluating a successful calibration verification. All the analytes in the standard should be evaluated before analysis continues. The percent drift or difference for non-CCC compounds may be as high as 60% for some clients. For DOD-QSM clients the percent drift or percent difference should not exceed 20% for requested analytes. For samples from within the state of South Carolina the percent drift or difference should not exceed ± 30% for requested analytes and ± 40% for poor purging compounds (See Appendix 6). If an analyte is flagged as an outlier on the report the analyst should at least evaluate the significance of the flag. The reason for the outlier may be as simple as the computer missing a quant ion, a poor performance compound, or loss of moisture control. Whatever the reason for the flag, all outliers should be evaluated on how the data will be affected by the analyte list. The 20% criteria for CCCs may be waived if all the required target analytes are less than 20% difference/drift. In addition, if the CCC and SPCC criteria are met despite these compounds not being required, the required target analytes may have differences/drifts greater than 20%. Again, every standard should be evaluated carefully in order to obtain quality data. If individual client requirements are not met and holding times are expiring, the group leader is notified. Clients will be notified of the nonconformance and analysis of the samples may proceed. Note any nonconformance in the case narrative. Refer to Appendix 4 for 8260C and Appendix 8 for Standard Method 6200 specific criteria and Appendix 10 for 8260D specific criteria.

15.3.5.2 Internal Standard Response: Internal standard areas in the daily continuing calibration verification standard (CCV) must be within +100 to -50% and retention times for each internal

standard must be within ±30 seconds of the initial calibration midpoint internal standard. If internal standards do not meet these criteria, reanalyze the CCV to verify the problem was not random. If the areas fail again, corrective action must be taken. Several actions may correct the problem, i.e., refreshing the internal standard solution on the autosampler, using a newly opened solution, checking that instrument conditions have not changed, etc. Document the actions taken to correct the problem in the instrument maintenance log. Refer to Appendix 4 for 8260C specific criteria.

15.3.5.3 If the continuing calibration does not meet the aforementioned criteria, the analyst must determine why and take appropriate corrective action. Samples may not be analyzed until the validity of the calibration has been verified.

16.0 ANALYSIS PROCEDURES AND INSTRUMENTAL OPERATION

16.1 All calibration and verification standards, blanks, quality control samples, and client samples are analyzed using a heated purge. Ultra Pure helium is bubbled through the sample that is contained in a purging chamber at 40° C. The purgables are efficiently transferred from the aqueous phase to the vapor phase that is swept through a sorbent where purgeables are trapped. After purging is complete, the trap is heated and backflushed with the inert gas to desorb the analytes onto a gas chromatograph that is temperature programmed to separate the analytes that are then detected with a mass spectrometer.

16.2 Daily GC/MS Analysis Procedure:

At the beginning of each day or 12-hour tune window, the GC/MS system is checked to see if acceptable performance criteria are achieved. A 5 to 50 ng BFB standard is analyzed and must pass acceptance criteria before any further analyses are performed.

16.3 If the tune passes, a continuing calibration verification sample is analyzed. At a minimum the SPCC and CCC criteria for response factor and percent drift must be met. Additionally for samples within the state of South Carolina, the percent drift or difference should not exceed +/- 30% for the requested target analytes and +/- 40% for poor purgers (See Appendix 6). If the client requested target list does not include SPCC or CCC compounds, all compounds must have a percent difference or drift of less than 20%. For client samples outside of the state of South Carolina, the CCV should be evaluated against client specific requirements if available. The percent drift or difference for non-CCC compounds may be as high as 60%. If individual client requirements are not met and holding times are expiring, the group leader is notified. Clients will be notified of the nonconformance and analysis of the samples may proceed. Note any nonconformance in the case narrative. Refer to Appendices 4 and 5 for 8260C and Appendix 8 for Standard Method 6200 specific criteria and Appendix 10 for 8260D specific criteria.

16.4 Upon passing the calibration verification, a laboratory control sample (LCS) is analyzed. The LCS must pass either the laboratory SPC limits or client specific limits before

continuing analysis. The CCV and LCS may be combined into one analysis as long as the standard is a second source to the initial calibration standards. The standard must pass both sets of criteria for the CCV and LCS to be acceptable. For SC samples the CCV must come from the same source standard that is used to prepare the initial calibration curve and the LCS must come from a second source standard. Some clients may require that the laboratory analyze a CCV and LCS as two separate injections, specific client requirements are indicated on the batch pull sheet. If individual client requirements are not met and holding times are expiring, the group leader is notified. Clients will be notified of the nonconformance and analysis of the samples may proceed. Note any nonconformance in the case narrative.

16.5 Upon analysis of an acceptable LCS(s), the analyst must analyze an acceptable method blank prior to beginning client samples.

16.6 Upon the analysis of an acceptable method blank, client sample analysis may begin. Analysts are encouraged to begin the sequence with the analysis of samples that should be clear of contamination such as trip blanks, field blanks, and methanol blanks.

16.7 If a sample contains target and/or non-target analyte concentrations that exceed the calibration range, the analyst must review subsequent sample analyses for possible carryover contamination of those analytes. Samples containing suspected carryover contamination are reanalyzed. The system is decontaminated either by the analysis of subsequent blanks or subsequent clean samples.

16.8 Sample analysis may continue up to 12 hours from the injection time of the BFB. Any sample analyzed after the 12 hour window will require reanalysis in a valid 12 hour sequence.

16.9 Each instrument has a run log in which each 12 hour sequence is documented. The analyst must document standards used in the analysis, in addition to data files, dates, injection times, sample weights, volumes, pH, residual chlorine, and additional comments.

17.0 QUALITY CONTROL REQUIREMENTS

17.1 Frequency of Quality Control Activities

17.1.1 Instrument Check

Before any initial calibration or continuing calibration checks are performed, the analyst must analyze an acceptable Bromofluorobenzene (BFB) solution (5 to 50ng). Refer to Appendix 2 or 9 for criteria.

17.1.2 Initial and Continuing Calibration

Before the analysis of any blanks, spikes or samples, the instrument must be calibrated. This multilevel calibration is used to establish the linearity of the instrument for the analytes of interest. Once this calibration is in place, the frequency thereafter is determined from the analysis of continuing calibration standards.

17.1.3 The continuing calibration standard is used to verify instrument calibration compared to the last multilevel calibration check and must be performed every 12 hours.

17.1.4 Blank Analysis

17.1.4.1 Method Blanks are used to determine background (laboratory) concentrations of target analytes that have the potential of interfering with sample analysis. A blank is analyzed in each 12-hour analytical sequence.

17.1.4.2 Other types of blanks can be used to determine possible contamination from various points in the process. Trip Blanks can be used for evaluating the bottles in which the samples are collected. Field Blanks are used to discover potential interferences that may originate from the collection process. The frequency at which such blanks are analyzed are determined by the client (internal or external). Refrigerator or Storage Blanks are analyzed on at least a biweekly basis to determine the cleanliness of the storage area.

17.1.5 Spike and Duplicate Analysis

17.1.5.1 Accuracy is monitored through the analysis of sample spikes. Matrix spikes are performed for each batch up to maximum of 20 samples of similar matrix.

17.1.5.2 Precision is monitored through the analysis of duplicates. For method 8260B/C, the matrix spike or sample is duplicated. This ensures the availability of two sets of results for comparison.

17.1.5.3 Laboratory Control Samples (LCS) are used to determine if the process is in control. If a matrix spike fails due to matrix related interference, then the analysis of the LCS demonstrates that the instrument is in control. For non-SC samples, the continuing calibration check may be used as an LCS if it is a second source standard. Analyze the LCS in each 12-hour sequence. The LCS may be analyzed before the method blank as the CCV. For SC samples the LCS must come from a second source standard.

17.1.6 Surrogate Spikes must be added to all standards, blanks, quality control, and client samples.

17.1.7 Internal standards must be added to all standards, blanks, quality control, and client samples.

17.1.8 Samples must be diluted whenever a target analyte concentration exceeds the highest concentration (for that analyte) in the calibration curve. Samples should be diluted in order to put the analyte concentrations near or above the midpoint of the calibration curve. Multiple dilutions may be necessary if multiple analytes at various concentrations require dilutions. Also, if a sample is initially diluted and there are no target or non-target interferences present, the analyst may be required to reanalyze a more concentrated sample. Some factors for consideration when choosing a dilution factor are

pH, nature of the matrix and non-target interferences. Analysts should be aware that diluting samples increases the detection and reporting limits.

17.2 Acceptance Limits for Quality Control Activities

17.2.1 Initial Calibration

17.2.1.1 The initial calibration %RSD and RRF calculated for each compound must meet the criteria in section 15 of this SOP. Refer to Appendix 4 and Appendix 5 for 8260C specific criteria.

17.2.1.2 The internal standard areas and retention times in each calibration standard analysis must meet the criteria in section 15 of this SOP. Refer to Appendix 4 for 8260C and Appendix 10 for 8260D criteria Appendix 8 for 8260D for Standard Method 6200.

17.2.1.3 The initial calibration must be verified by the analysis of an acceptable ICV before client samples can be analyzed. Refer to Appendix 4 for 8260C and Appendix 10 for 8260D criteria.

17.2.2 Continuing Calibration

17.2.2.1 The instrument calibration must be verified every 12 hours by analyzing an acceptable continuing calibration verification standard (CCV). The CCV must meet the criteria indicated in section 15. Refer to Appendix 4 and Appendix 5 for 8260C, and Appendix 8 for Standard Method 6200 specific criteria and Appendix 10 for 8260D criteria.

17.2.3 Method blank criteria require that concentrations of all analytes of interest must be below the client reporting limit or laboratory practical quantitation limit. Ideally, all target analyte concentrations should be below the laboratory method detection limit, however, concentrations below reporting limits are acceptable. Specific client criteria may require that target analyte concentrations in the method blank not exceed one half the reporting limit. The analyst should take corrective action to ensure that target analytes do not exceed the reporting limits. Corrective action may include the analysis of clean up blanks or perhaps, more intense system decontamination. In some cases, concentrations for the common laboratory contaminants (methylene chloride, acetone, 2-butanone, ethyl ether, carbon disulfide) may be present in the method blank above the reporting limit, but the concentration(s) must be less than five times the reporting limit. In this situation if the compounds are detected in the associated samples, the data are flagged and notation is made in the case narrative that detection and quantitation of these analytes in samples would be biased. If the compounds are not detected in the associated samples, no action is required.

17.2.4 Matrix Spikes/Duplicates

17.2.4.1 SW-846 8260C/B/D criteria for both matrix spikes and matrix spike duplicates will be statistically determined after at least 20 to 30 matrix pairs. If data are not available for specific compounds,

a guidance of 70% to 130% recovery for spikes may be used. Some clients may have contract required limits that the laboratory should use.

17.2.4.2 Target analyte concentrations in the parent sample that exceed five times the spike level may cause biased recovery results in the matrix spike samples. Unacceptable recoveries due to high concentrations in the parent sample should be noted in the case narrative.

17.2.4.3 The criteria for sample duplication when analyzing samples using method 8260C/B/D are that any target present over five (5) times the detection limit must be within ± 20% of each other. As a statistical database is built, statistical process control (SPC) limits are generated.

17.2.4.4 Laboratory control sample recoveries must be within the established SPC limits to be considered acceptable. Statistical control limits for each compound should be derived after the analysis of up to 20 to 30 LCSs (per matrix). The SPC limits for most volatile analytes should fall between 70% to 130% recovery; however, due to poor purging efficiencies, water solubilities, and poor chromatography, some analytes may not meet the method recommendation.

17.2.4.5 Surrogates in all quality control and client samples must meet the established SPC limit or client specified limits. Surrogate limits are determined statistically at least semi-annually utilizing a minimum of 20 data points. Samples must be reanalyzed if surrogate recoveries are outside the required limits. If the surrogate recoveries are unacceptable in the reanalysis, the failure is attributed to matrix. One or both analyses may be reported depending on client specifications.

17.2.4.6 Internal standard responses must be within the required criteria in the initial calibration and calibration verification standard (Section 15). In addition to the internal standard area/RT check from the calibration verification standard to the midpoint of the current initial calibration, the laboratory also compares the internal areas and retention times of samples and QC to the associated daily calibration verification standard. If area counts exceed –50% to +100% or retention times are greater than ± 30 seconds, the sample or QC sample must be reanalyzed. If the analyst suspects matrix interference the sample may be diluted to aid in the internal standard recovery. If the response for the internal standards is unacceptable in the reanalysis, the failure is attributed to matrix. One or both analyses may be reported depending on client specifications.

18.0 ANALYST AND METHOD VERIFICATION REQUIREMENTS

18.1 New analysts must complete an Initial Demonstration of Proficiency prior to analyzing client samples. The analyst must be able to demonstrate proficiency by analyzing a passing tune, calibration verification, laboratory control samples, and method blank. The initial demonstration is documented using four acceptable LCS analyses in an analytical sequence.

18.2 Continuing Demonstration of Proficiency results are generated by collecting the data from four LCS analyses per method per analyst. The QA/QC group generates the certificates annually. Refer to GL-QS-E-011 Method Validation and Initial and Continuing Demonstration of Capability.

18.3 Method Detection Limits (MDL) are analyzed annually and verified quarterly. Refer to GL-LB-E-001 for The Determination of Method Detection Limits for additional information.

19.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

19.1 Routine Preventative and Special Operational (Failure)

19.1.1 Preventative maintenance on a GC/MS system involves four basic areas:

19.1.1.1 Vacuum pumps for the analyzer should have an oil change when the system demonstrates that it is needed. This includes the mechanical, turbomolecular, and/or diffusion pumps. Generally, this occurs at least once a year.

19.1.1.2 GC maintenance consists mainly of column and injection port maintenance. These procedures are performed when system sensitivity drops, or air (m/z = 28) is noted present in the system.

19.1.1.3 Analyzer maintenance is most often usage dependent. The type and quantity of samples that have been injected, determine the frequency of ion source and electron multiplier cleaning and/or replacement.

19.1.1.4 Autosampler maintenance is very sample dependent. In general, maintenance consists of changing the trap and line cleaning. Line cleaning is accomplished by back flushing the system with methanol.

19.1.2 Non-routine Maintenance Procedures (Special, Operational or Failure Mode Maintenance)

19.1.2.1 Troubleshooting the autosampler and instrument is a function of analyst experience. In-House service is obtained from GEL's Service Technician. If vendor assistance is needed, then the appropriate vendor is contacted.

19.1.2.2 Electronic maintenance logbooks are kept for each instrument and include entries for both routine as well as non-routine maintenance procedures.

20.0 DATA RECORDING, CALCULATIONS AND REDUCTION METHODS

20.1 Qualitative Analysis:

20.1.1 Samples, standards, and quality control samples are processed using HP Chemstation software. The software performs identification and quantitation based off method information applied to the raw data areas or responses. The program generates electronic reports for review and validation. Raw data are stored electronically on servers and transferred to compact disc media.

20.1.2 Compound Identification

20.1.2.1 A comparison of the sample mass spectrum with the mass spectrum of a standard reference may identify the suspected compound. Mass spectrum for standard reference must be obtained from the NIST library.

20.1.2.2 Elution of sample component at the same GC relative retention time (RRT) as those of the standard component may help identify a compound.

20.1.2.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%).

20.1.2.4 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between the two peaks is less than 25% of the sum of the heights. Otherwise, structural isomers are identified as isomeric pairs.

20.1.2.5 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When GC peaks obviously represent more than one sample, appropriate selection of analyte spectra and background spectra is important.

20.1.2.6 Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra and in the qualitative identification of compounds. When analytes coelute, the identification criteria may be met, but each analyte spectrum will contain extraneous ions contributed by the coeluting compound.

20.1.2.7 Occasionally manual integrations may be required if the sample matrices affect the software's ability to draw appropriate baselines. Manual integrations on calibration levels are permitted although discouraged. In the event that a manual integration is

performed, the software generates a printout of the integration event before the manual integration and after the manual integration. The analyst electronically reviews and initials the data file and includes the reasoning behind the manual integration.

20.1.3 The relative retention time (RRT) of the sample component must be within ± 0.06 RRT units of the RRT of the standard component, analyzed in the same 12 hour period. If coelution of another compound prohibits accurate assignment of the sample component RRT from the total ion chromatogram, the RRT should be assigned by using extracted ion current profiles for ions unique to the components of interest. Analysts should pay close attention to the following compounds for possible coelution and identification 1,1,1-Trichloroethane, and Carbon tetrachloride; m,p-Xylenes and Ethylbenzene; the three Dichlorobenzene isomers; Chloromethane and Dichlorodifluoromethane, cis-1,2-Dichloroethylene and trans-1,2-Dichloroethylene; cis-1,3-Dichloropropylene and trans-1,3-Dichloropropylene; 2-Chlorotoluene and 4-Chlorotoluene; the two Trimethylbenzenes; the three Butylbenzenes; and the two Trichlorobenzenes.

20.1.4 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analysis being conducted. Refer to Appendix 7 for COA and Form 1 TIC reporting.

20.2 Quantitative Analysis:

20.2.1 When a compound has been identified, the quantitation of that compound will be based on the integrated abundance from the EICP of the primary characteristic ion. Quantitation will take place using the internal standard technique. The internal standard used shall be the one nearest the retention time of a given analyte. The concentration of each identified analyte in the sample is calculated as follows:

20.2.2 Water and Water- Miscible Waste using Average RRF:

$$\text{Concentration } (\mu g/L) = [(Ax)(Cis)(Vo)] / [(Ais)(RRF)(Vi)]$$

Where :

Ax = Area of characteristic ion of compound being measured

Cis = Concentration of internal standard purged in µg/L

Ais = Area of Characteristic ion for internal standard

RRF= Relative response factor for compound being measured (see above)

Vo = Total purge volume (mL)

Vi = Volume of sample purged (mL)

20.2.3 Water and Water-Miscible using Linear Regression:

$$\text{Conc. } \mu g/L = \frac{\left[\frac{\left(\frac{Ax}{Ais}\right)}{a} + b\right] * Cis * Vo}{Vi}$$

Where:

Ax = Area of characteristic ion of compound being measured

Cis = Concentration of internal standard purged in µg/L

Ais = Area of characteristic ion for the internal standard

RRF = Relative response factor for compound being measured (see above)

Vo = Total purge volume (mL)

Vi = Volume of sample purged (mL)

a = slope

b = y intercept

20.3 Sediment, Soil, Sludge, and Waste

20.3.1 High level using Average RRF:

Concentration (µg/kg) = [(Ax)(Cis)(Vt)(Vo)] /[(Ais)(RRF)(Ws)(Vi)]

20.3.2 Low Level using Average RRF:

Concentration (µg/kg) = [(Ax)(Cis)(Vt)(Vo)] /[(Ais)(RRF)(Ws)(Vi)]

Where :

Ax, Cis, Ais, RRF = Same as for water

Vt = Volume of total extract (use 10,000 µL or factor of this when dilutions are made.)

Vi = Volume of extract added (µL) for purging

Ws = Weight of sample extracted or purged (g). The wet or dry weight may be used, depending upon the specific application of the data

Vo = Total purge Volume (mL)

20.3.3 Low Level using Linear Regression:

$$\text{Conc. } \mu g/kg = \text{Conc. } \mu g/kg = \frac{\left[\frac{\frac{Ax}{Ais}+b}{a}\right] * Cis * Vo}{W_S}$$

20.3.4 High Level using Linear Regression:

$$\text{Conc. } \mu g/kg = \text{Conc. } \mu g/kg = \frac{\left[\frac{\frac{Ax}{Ais}+b}{a}\right] * Cis * Vo * Vt}{Ws * Vi}$$

20.4 Where applicable, an estimate of concentration for non-calibrated components in the sample is made. The formulas given above shall be used with the following modifications: The areas Ax and Ais shall be from the total ion chromatograms, and the RF for the compound shall be assumed to be 1.

20.4.1 The concentration obtained is an indication that the value is an estimate, and identifies which internal standard was used to determine concentrations. Use the nearest internal standard free of interferences.

20.5 SPC limits are determined by criteria set forth in EPA Method 8000D. For a given matrix, calculate the upper and lower control limit for method performance for each standard of interest as follows:

Upper control limit (UCL) = p + 3 s

Lower control limit (LCL) = p - 3 s

Where:

p = Average percent recovery

s = Standard deviation of percent recovery

20.6 Data are reported in units of ug/L or ppb for waters, and ug/kg or mg/kg for soils and sludges unless otherwise specified by the client.

21.0 POLLUTION/CONTAMINATION CONTROL

21.1 The volatile organics laboratory is physically separated from the other areas of the laboratory. Only authorized personnel have access to this area.

21.2 A separate air handling system and frequent filter changes help keep this area free of common contaminants. The laboratory includes a positive pressure air system.

21.3 Volatile samples are stored separately from other samples. Oil or waste samples for volatile organic analysis are generally not stored in the volatile refrigerator. Samples that may cause contamination in the refrigerator are stored in the main walk-in cooler.

Storage blanks are analyzed at least biweekly from each refrigerator and cooler that stores samples for volatile analysis.

21.4 Soils and waste samples are weighed on a balance within a fume hood in the volatile organics laboratory.

21.5 Radioactive and non-radioactive samples are separated in different refrigerators.

21.6 Organic free water obtained by passing laboratory tap water through a series of carbon filters. This system is located in the volatile organics laboratory.

22.0 DATA REVIEW, APPROVAL AND TRANSMITTAL

22.1 After analysis, each analyst is responsible for reviewing their analytical data for completeness and quality control information.

22.2 The analyst electronically dates and initials the front page of the quantitation report generated by the Chemstation software.

22.3 Occasionally, manual integrations may be required if the sample matrices affected the software's ability to draw appropriate baselines. Manual integrations on calibration levels are permitted although discouraged. In the event that a manual integration is performed, the software generates a printout of the integration event before the manual integration and after the manual integration. The analyst electronically initials and dates the printout and includes the reasoning behind the manual integration.

22.4 After the analyst has completed and reviewed the batch raw data, a second review by a peer or validator is performed before data are given a done status. Data deliverables are client specific, however if a fractional case narrative of CLP-like data package is needed, an additional review of the fractional data package is performed by a validator prior to sending to the client.

22.5 The levels of review responsibility are:

22.5.1 First level review: The analyst must ensure the overall quality of the data. Included in this review are: the BFB tune criteria, samples within tune time, calibration verification standard check, method blank, matrix spikes, laboratory control sample, surrogate recovery, internal standard response, over range concentrations include a dilution, reanalysis as required, manual integrations are appropriate and correct.

22.5.2 Second level review: A peer must review all of the data before it can be released from the lab. The peer performs all the checks that the first analyst did and compares the data with the runlog. Particular attention must be paid to the 12-hour tune window, the check standard, and internal standard area counts. All hits must also be reviewed very carefully. When the second level review is complete, the peer reviewer initials and dates the quantitation report.

22.5.3 A flow chart showing the process by which papers and documents undergo review can be viewed in Figure 2.

22.5.4 Data packages are generated electronically in Portable Document Format or PDF. Data packages are sent directly to the client as a PDF and/or hard copies are generated for submittal.

22.5.5 Data packages are stored on compact disc (CD) by the laboratory computer services group.

23.0 NONCONFORMANCE AND CORRECTIVE ACTION FOR UNACCEPTABLE DATA

23.1 When analyzing a multi-point calibration for many analytes at once, the chances are high that a few may not meet the criteria. Additional standards may be reanalyzed. If they still do not meet the requirements, the instrument may need maintenance before continuing or new standards may be needed.

23.2 If the continuing calibration fails any of the criteria in section 15, the analyst must take action to correct the situation. This may be retuning, reanalyzing the standard, adjusting the purge flow, or any number of maintenance practices in order to meet the daily calibration. If all attempts fail, the analyst must analyze a new series of multi-point calibration standards. If individual client requirements are not met and holding times are expiring, the group leader is notified. Clients will be notified of the nonconformance and analysis of the samples may proceed. Note any nonconformance in the case narrative.

23.3 When the method blank fails the criteria defined in section 17.2.3, the analyst must find and eliminate the source of contamination before proceeding with analyses. Another blank must be analyzed before sample analysis begins. Note any nonconformance in the case narrative.

23.4 If any surrogate recovery is outside the acceptance criteria, the sample must be reanalyzed. If the surrogate recoveries fail a second time, in the same manner, and the blank and LCS recoveries do not indicate a system problem, the failure is attributed to matrix effects. Note any nonconformance in the case narrative.

23.5 All analyte recoveries in the LCS should meet either the SPC limits or client required limits. If the LCS recoveries are outside the acceptance criteria, the analysis should stop and corrective actions should be attempted before continuing with the analysis. Corrective action may be repeating the analysis, opening a new spike mix and reanalyzing, or more complex actions may be attempted. If re-tuning the instrument, the analyst must analyze a tune check (BFB) and calibration verification standard before reanalyzing the LCS. If individual client requirements are not met and holding times are expiring, the group leader is notified. Clients will be notified of the nonconformance and analysis of the samples may proceed. Note any nonconformance in the case narrative.

23.6 The manner in which data will be accepted or rejected is described in the flowchart in figure 1.

24.0 RECORDS MANAGEMENT

24.1 Data generated as a result of this procedure are stored in the laboratory for approximately 1 month.

24.2 After approximately 1 month these records are maintained as quality documents in accordance with GL-QS-E-008 for Quality Records Management and Disposition.

25.0 LABORATORY WASTE HANDLING AND DISPOSAL

25.1 All organic waste solvents, extracts and reagents are stored in a certified hazardous waste can. When the can is full, the GEL hazardous waste disposal specialist is called. Refer to the Laboratory Waste Management Plan (GL-LB-G-001).

26.0 REFERENCES

26.1. Test Methods for Evaluating Solid Waste: Laboratory Manual Physical/ Chemical Methods, Volume 1(Part 2) Section B. SW-846, Third Edition. Method 8260B, "Volatile Organic Compounds by Gas Chromatograph/Mass Spectrometer (GC/MS): Capillary Column Technique," Revision 1, September 1994. USEPA Office of Solid Waste and Emergency Response, Washington, DC 20460.

26.2. Test Methods for Evaluating Solid Waste: Laboratory Manual Physical/Chemical Methods, Volume 1(Part 2) Section B. Sw-846, Third Edition. Method 8000D, "Determinative Chromatographic Separations, "Revision 4, July 2014. USEPA Office of Solid Waste and Emergency Response, Washington, DC 20460.

26.3. Test Methods for Evaluating Solid Waste: Laboratory Manual Physical/ Chemical Methods, Volume 1(Part 2) Section B. SW-846, 3rd Edition. Method 5030B, "Purge-and-Trap for Aqueous Samples," Revision 2, December 1996. USEPA Office Of Solid Waste and Emergency Response, Washington, DC 20460.

26.4. Test Methods for Evaluating Solid Waste: Laboratory Manual Physical/ Chemical Methods, Volume 1(Part 2) Section B. SW-846, Third Edition. Method 5035, "Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples," Revision 0, December 1996. USEPA Office Of Solid Waste and Emergency Response, Washington, DC 20460.

26.5. Dept. of Defense (DOD), Dept. of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories DOD QSM Version 5.3, July 2019.

27.0 HISTORY

Revision 24: Added LOQ and LLOQ definitions. Removed reference to 8000C 5030C. Updated the data reviewed electronically. Updated Equipment section with specific vials used. Updated calibration short and long list tables.

Revision 25: Added clarification on the frequency at which LLOQs are verified and that the LLOQ is verified on every instrument where data is reported.

Revision 26: Updated 26.2 to reference current revision number and revision month.

Revision 27: Updated to include 8260D. Updated reference section for current DoD QSM Version 5.2, December 2018

Revision 28: Updated the preservation requirement for Acrolein and Acrylonitrile to a 4-5 pH and analyzed within 7 days of sampling. Add the requirements for TCLP and tumble blanks. Updated reference for DOD QSM Version 5.3 July 2019.

FIGURE 1: DECISION FLOWCHART

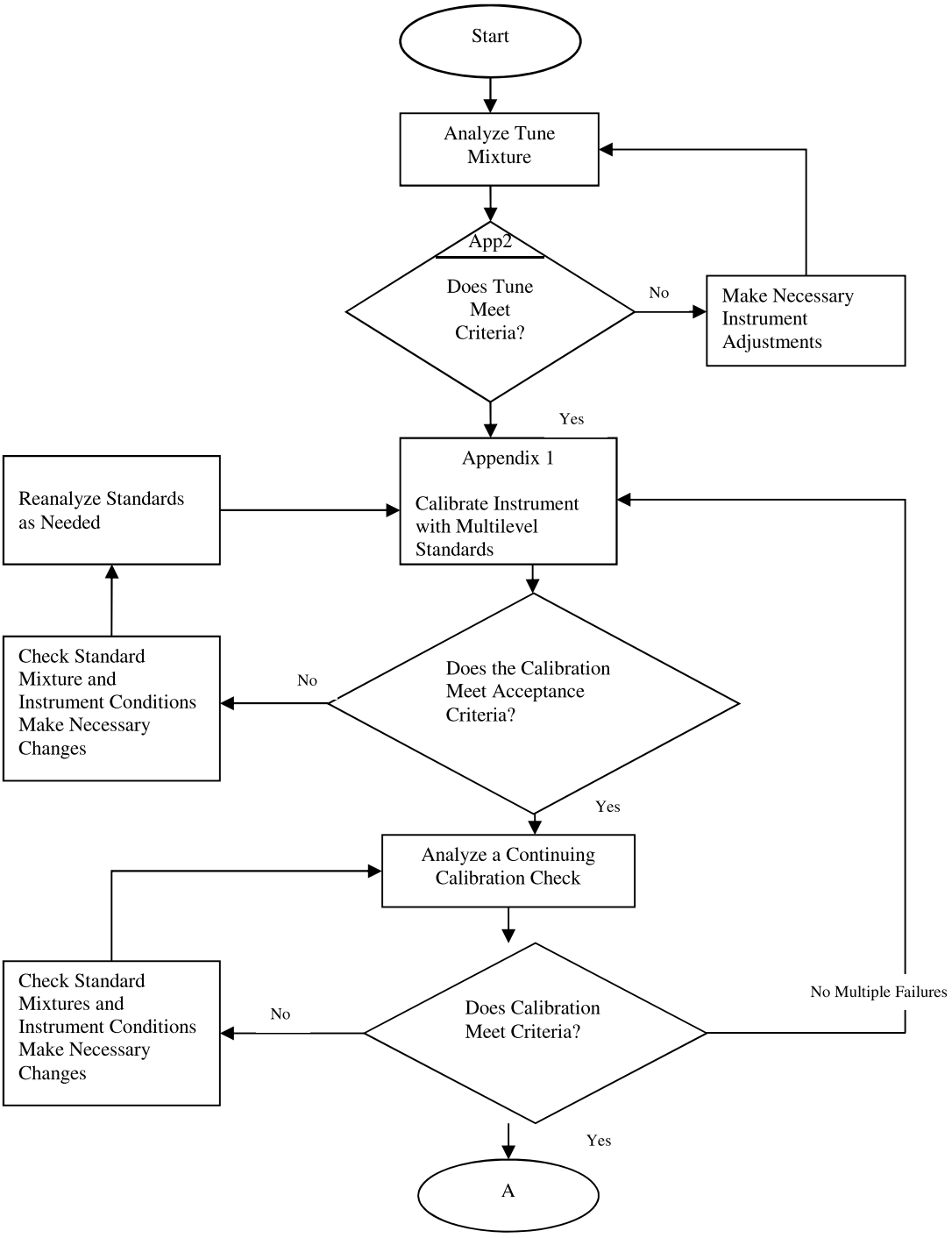


FIGURE 1: DECISION FLOWCHART continued

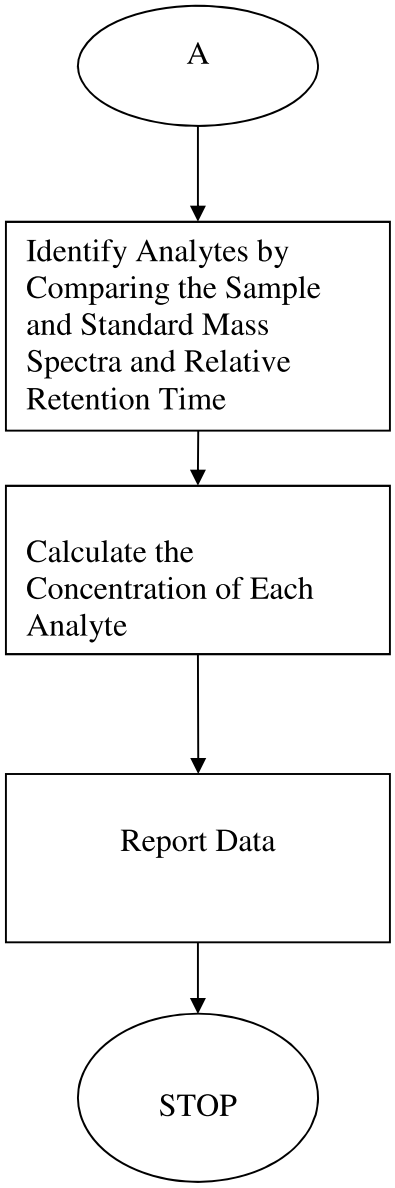
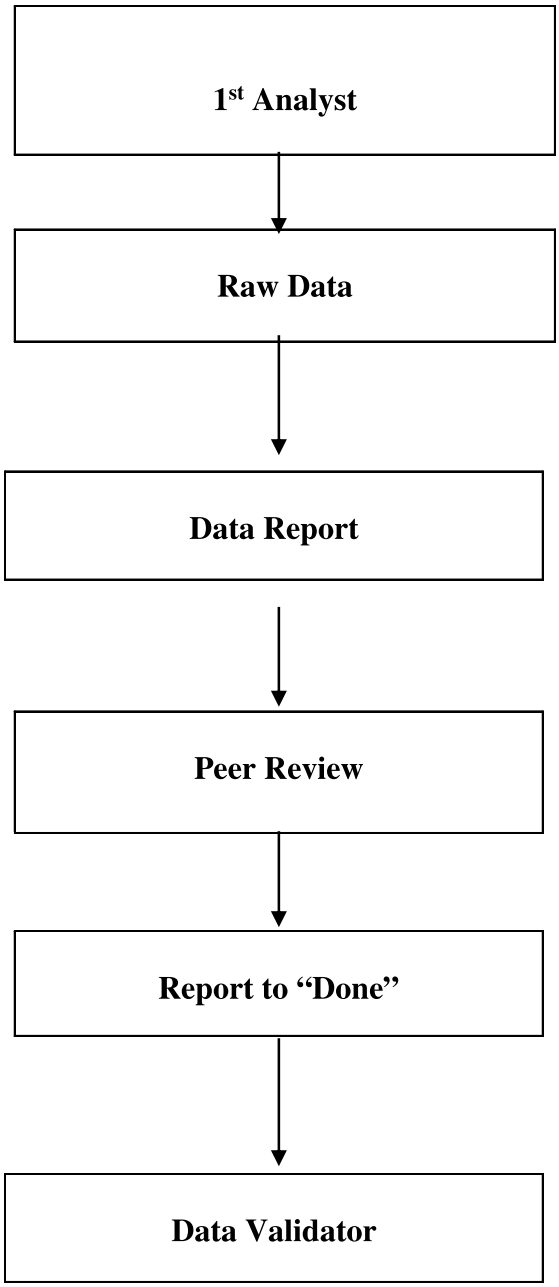


FIGURE 2: LEVELS OF REVIEW



APPENDIX 1: CALIBRATION MIXES

(FOR ILLUSTRATIVE PURPOSES ONLY)

Initial Stock Concentrations

Stock A/B:	0.5ppm	1.0ppm	2.0ppm	5.0ppm	10ppm	20ppm	50ppm	100ppm
Final Conc.								
0.2ppb	2uL							
0.5ppb	5uL							
1.0ppb		5uL						
2.0ppb			5uL					
5.0ppb				5uL				
10ppb					5uL			
20ppb						5uL		
50ppb							5uL	
80ppb								4uL
100ppb								5uL

Initial Stock Concentrations

Stock C:	5ppm	10ppm	25ppm	50ppm	100ppm	250ppm	500ppm
Stock D:	1ppm	2ppm	5ppm	10ppm	20ppm	50ppm	100ppm
Final Conc.							
5.0ppb/1ppb	5uL ea.						
10ppb/2ppb		5uL ea.					
25ppb/5ppb			5uL ea.				
50ppb/10ppb				5uL ea.			
100ppb/20ppb					5uL ea.		
250ppb/50ppb						5uL ea.	
300ppb/ **60ppb**							3uL
500ppb/100ppb							5uL ea.

APPENDIX 2: BFB MASS INTENSITY SPECIFICATIONS

Mass	Intensity Required (relative abundance)
50	15-40% of mass 95
75	30-60% of mass 95
95	base peak, 100% relative abundance
96	5-9% of mass 95
173	less than 2% of mass 174
174	greater than 50% of mass 95
175	5-9% of mass 174
176	greater than 95% but less than 101% of mass 174
177	5-9% of mass 176

APPENDIX 3: VOLATILE ORGANIC COMPOUNDS CALIBRATION RANGES

Low level SW846 8260B and Regular level 8260B and EPA 624.1
Calibration Standard Concentration Levels

	Level 1	Level 1a	Level 2	Level 3	Level 4	Level 5	Level 6 #	Level 7 !	Level 7a
Fluorobenzene (IS)	20	20/50	20/50	20/50	20/50	20/50	20/50	20/50	20/50
1,2-Dichloroethane-d4(surr)		20/50	20/50	20/50	20/50	20/50	20/50	20/50	20/50
Dichlorodifluoromethane		0.5	1	2	5	10	20	50	100
Chloromethane		0.5	1	2	5	10	20	50	100
Vinyl chloride		0.5	1	2	5	10	20	50	100
Bromomethane		0.5	1	2	5	10	20	50	100
Chloroethane		0.5	1	2	5	10	20	50	100
Trichlorofluoromethane		0.5	1	2	5	10	20	50	100
1,1-Dichloroethene		0.5	1	2	5	10	20	50	100
Acetone	1	2.5	5	10	25	50	100	250	500
Iodomethane	1	2.5	5	10	25	50	100	250	500
Carbon disulfide	1	2.5	5	10	25	50	100	250	500
Methylene chloride		0.5	1	2	5	10	20	50	100
trans-1,2-Dichloroethene		0.5	1	2	5	10	20	50	100
1,1-Dichloroethane		0.5	1	2	5	10	20	50	100
Ethyl ether		0.5	1	2	5	10	20	50	100
Vinyl acetate	1	2.5	5	10	25	50	100	250	500
cis-1,2-Dichloroethene		0.5	1	2	5	10	20	50	100
1,2-Dichloroethene (total)		1	2	4	10	20	40	100	200
Cyclohexene		0.5	1	2	5	10	20	50	100
2-Chloroethylvinyl ether			5	10	25	50	100	250	500
2,2-Dichloropropane		0.5	1	2	5	10	20	50	100
2-Butanone	1	2.5	5	10	25	50	100	250	500
Bromochloromethane		0.5	1	2	5	10	20	50	100
Chloroform		0.5	1	2	5	10	20	50	100
1,1,1-Trichloroethane		0.5	1	2	5	10	20	50	100
1,1-Dichloropropene		0.5	1	2	5	10	20	50	100
Carbon tetrachloride		0.5	1	2	5	10	20	50	100
Benzene		0.5	1	2	5	10	20	50	100
1,2-Dichloroethane		0.5	1	2	5	10	20	50	100
Trichloroethene		0.5	1	2	5	10	20	50	100
1,2-Dichloropropane		0.5	1	2	5	10	20	50	100
Dibromomethane		0.5	1	2	5	10	20	50	100
Bromodichloromethane		0.5	1	2	5	10	20	50	100
cis-1,3-Dichloropropene		0.5	1	2	5	10	20	50	100
tert-Butylmethylether		0.5	1	2	5	10	20	50	100
Ethyl Ether			1	2	5	10	20	50	100
Acetonitrile			25	50	125	250	500	1250	2500
Methyl acetate			5	10	25	50	100	250	500
Cyclohexane			1	2	5	10	20	50	100
Methylcyclohexane			1	2	5	10	20	50	100
n-Butyl alcohol		50	100	200	500	1000	2000	5000	10000
2-Nitropropane			5	10	25	50	100	250	500
Ethyl acetate			5	10	25	50	100	250	500
Acrolein			5	10	25	50	100	250	500
Trichlorotrifluoroethane			5	10	25	50	100	250	500

APPENDIX 3: VOLATILE ORGANIC COMPOUNDS CALIBRATION RANGES cont'd

Allyl chloride			5	10	25	50	100	250	500
Acrylonitrile			5	10	25	50	100	250	500
1,4-Dioxane			50	100	250	500	1000	2500	5000
Isobutyl alcohol			50	100	250	500	1000	2500	5000
Methacrylonitrile			5	10	25	50	100	250	500
Propionitrile			5	10	25	50	100	250	500
Methyl methacrylate			5	10	25	50	100	250	500
Chlorotrifluoroethylene			5	10	25	50	100	150	200
2-Chloro-1,1,1-trifluoroethane			5	10	25	50	100	150	200
Tetrahydrofuran			5	10	25	50	100	250	500
tert-Butyl alcohol			50	100	250	500	1000	2500	5000
Isopropyl ether			1	2	5	10	20	50	100
Ethyl tert-butyl ether			1	2	5	10	20	50	100
Isopropyl alcohol			50	100	250	500	1000	2500	5000
Methyl tert-amyl ether			1	2	5	10	20	50	100
1-Chlorohexane			1	2	5	10	20	50	100
2-Chloro-1,3-butadiene(chloroprene)			1	2	5	10	20	50	100
Chlorobenzene-d5 (IS)	20	20	20/50	20/50	20/50	20/50	20/50	20/50	20/50
Toluene-d8 (surr)		20	20/50	20/50	20/50	20/50	20/50	20/50	20/50
4-Methyl-2-pentanone	1	2.5	5	10	25	50	100	250	500
Toluene		0.5	1	2	5	10	20	50	100
trans-1,3-Dichloropropene		0.5	1	2	5	10	20	50	100
1,1,2-Trichloroethane		0.5	1	2	5	10	20	50	100
Tetrachloroethene		0.5	1	2	5	10	20	50	100
1,3-Dichloropropane		0.5	1	2	5	10	20	50	100
2-Hexanone	1	2.5	5	10	25	50	20	250	500
Dibromochloromethane		0.5	1	2	5	10	20	50	100
1,2-Dibromoethane		0.5	1	2	5	10	20	50	100
Chlorobenzene		0.5	1	2	5	10	20	50	100
1,1,1,2-Tetrachloroethane		0.5	1	2	5	10	20	50	100
Ethylbenzene		0.5	1	2	5	10	20	50	100
m,p-Xylene		1	2	4	10	20	20	100	200
o-Xylene		0.5	1	2	5	10	20	50	100
Xylenes (total)		1.5	3	6	15	30	60	150	300
Stryene		0.5	1	2	5	10	20	50	100
Ethyl methacrylate			5	10	25	50	100	250	500
1,4-Dichlorobenzene-d4 (IS)	20	20	20/50	20/50	20/50	20/50	20/50	20/50	20/50
Bromofluorobenzene (surr)		20	20/50	20/50	20/50	20/50	20/50	20/50	20/50
Bromoform		0.5	1	2	5	10	20	50	100
Isopropylbenzene		0.5	1	2	5	10	20	50	100
1,1,2,2-Tetrachloroethane		0.5	1	2	5	10	20	50	100
Bromobenzene		0.5	1	2	5	10	20	50	100
1,2,3-Trichloropropane		0.5	1	2	5	10	20	50	100
n-Propylbenzene		0.5	1	2	5	10	20	50	100
2-Chlorotoluene		0.5	1	2	5	10	20	50	100
1,3,5-Trimethylbenzene		0.5	1	2	5	10	20	50	100
4-Chlorotoluene		0.5	1	2	5	10	20	50	100

APPENDIX 3: VOLATILE ORGANIC COMPOUNDS CALIBRATION RANGES CONT'D

1,2,4-Trimethylbenzene		0.5	1	2	5	10	20	50	100
sec-Butylbenzene		0.5	1	2	5	10	20	50	100
1,3-Dichlorobenzene		0.5	1	2	5	10	20	50	100
tert-Butylbenzene		0.5	1	2	5	10	20	50	100
Isopropyltoluene		0.5	1	2	5	10	20	50	100
1,4-Dichlorobenzene		0.5	1	2	5	10	20	50	100
n-Butylbenzene		0.5	1	2	5	10	20	50	100
1,2-Dichlorobenzene		0.5	1	2	5	10	20	50	100
1,2-Dibromo-3-chloropropane		0.5	1	2	5	10	20	50	100
1,2,4-Trichlorobenzene		0.5	1	2	5	10	20	50	100
Hexachlorobutadiene		0.5	1	2	5	10	20	50	100
Naphthalene		0.5	1	2	5	10	20	50	100
1,2,3-Trichlorobenzene		0.5	1	2	5	10	20	50	100
cis-1,4-Dichloro-2-butene			5	10	25	50	100	250	500
trans-1,4-Dichloro-2-butene			5	10	25	50	100	250	500
Pentachloroethane			5	10	25	50	100	250	500
Benzyl chloride			5	10	25	50	100	250	500
Cyclohexanone			25	50	125	250	500	1250	2500
bis(2-Chloro-isopropyl)ether			5	10	25	50	100	250	500

Method	PQL	Concentration range		
SW 846 8260B low level	Level 1&1a	Levels 1-> 7a	IS/SS @ 20 ppb	
SW846 8260B/624.1	Level 2	Levels 1a-> 7a	IS/SS @ 50 ppb	n-butyl alcohol only in 1a

#: Indicates calibration verification concentration level used for low level analysis

!: Indicates calibration verification concentration level used for regular level analysis

APPENDIX 4: Method 8260C Criteria

In addition to the general criteria outlined in the body of this SOP, the following requirements must be met prior to analysis of samples requesting 8260C.

1.0 Initial Calibration

1.1 The calibration curve must be prepared from a minimum of five calibration points. The RSD for all compounds must be less than 20%. If the RSD of any target analyte exceeds 20%, then use one of the options in section 15.2.13.

1.2 If more than 10% of the compounds included in the initial calibration exceed the 20% RSD and do not meet the minimum correlation coefficient of 0.990 for linear regression, system maintenance should be performed and a new calibration curve must be analyzed.

1.3 For compounds utilizing the method of linear regression, a minimum quantitation check should be performed on the lowest calibration point. This involves requantitation of the lowest points of the ICAL as samples (not as calibration levels) and evaluating the recalculated concentrations compared to the true concentrations present. The recalculated concentrations of the low calibration standards should be within $\pm$ 30% of the standard's true concentration. It should be noted that not all compounds are present in the calibration levels at the same concentrations. Documentation is made on the ICAL Validation Check Sheet employed by the data validators.

1.4 It is also recommended that a minimum response factor for the most common target analytes be demonstrated for each individual calibration level. See Appendix 5 for a list of compounds and recommended responses. Due to the large number of compounds analyzed by this method, some compounds may fail to meet the criteria. These compounds may not be critical to the specific project and may be used as qualified data or as estimated values.

1.5 SPCC and CCC compounds are not recognized in method 8260C.

2.0 Initial Calibration Verification

2.1 Calibration curves must be verified using an initial calibration verification standard. This must be a second source standard from the initial calibration. It may be from a different vendor or may be a different lot from the same vendor. The response factor or true value (percent difference or drift) should not exceed $\pm$ 30%. Documentation is made on the ICAL Validation Check Sheet employed by the data validators.

2.2 In the event that a compound falls outside of the $\pm$ 30% acceptance criteria, possible courses of action include reanalysis of the ICV, analysis of a different ICV standard (lot or vendor), maintenance to the analytical system and/or recalibration. If holding times are expiring, sample analysis may proceed. In this case, the Project Manager is notified, documentation is made in the case narrative and the data are qualified.

APPENDIX 4: METHOD 8260C CRITERIA CONTINUED

3.0 Continuing Calibration Verification Analysis

3.1 Each of the most common target analytes in the calibration verification standard should meet the minimum response factors in Appendix 5. If the minimum response factors are not met, check for standard degradation, injection port contamination, column contamination, active sites, or moisture control loss.

3.2 The percent drift or percent difference for each analyte of interest should be +/-20%. The calibration verification standard may be from the same source as the midpoint of the initial calibration or may be a different source. If more than 20% of the total number of requested compounds exceed +/-20% requirement, corrective action should be taken and no samples analyzed. In cases where compounds fail, they may still be reported as non-detects if it can be demonstrated that there was adequate sensitivity to detect the compound at the applicable quantitation limit. For situations when a failed compound is present, the concentrations must be reported as estimated values.

4.0 Internal Standard Retention Time Monitoring

The retention times of the internal standards in the calibration verification standard must be evaluated. If the retention time for any internal standard changes by more than 10 seconds from that in the mid-point standard level of the initial calibration, the system must be investigated for malfunctions and reanalysis of all samples analyzed during this period is required.

5.0 Surrogate Monitoring

The recommended surrogates for this method are Toluene-d8, 4-Bromofluorobenzene, and 1,2-Dichloroethane-d4.

APPENDIX 5: RECOMMENDED RESPONSE FACTORS FOR METHOD 8260C

TABLE 4

RECOMMENDED MINIMUM RELATIVE RESPONSE FACTOR CRITERIA FOR INITIAL AND CONTINUING CALIBRATION VERIFICATION

Volatile Compounds	Minimum Response Factor (RF)[a]	Typical Response Factor (RF)[b]
Dichlorodifluoromethane	0.100	0.327
Chloromethane	0.100	0.537
Vinyl chloride	0.100	0.451
Bromomethane	0.100	0.255
Chloroethane	0.100	0.254
Trichlorofluoromethane	0.100	0.426
1,1-Dichloroethene	0.100	0.313
1,1,2-Trichloro-1,2,2-trifluoroethane	0.100	0.302
Acetone	0.100	0.151
Carbon disulfide	0.100	1.163
Methyl Acetate	0.100	0.302
Methylene chloride	0.100	0.380
trans-1,2-Dichloroethene	0.100	0.351
cis-1,2-Dichloroethene	0.100	0.376
Methyl tert-Butyl Ether	0.100	0.847
1,1-Dichloroethane	0.200	0.655
2-Butanone	0.100	0.216
Chloroform	0.200	0.557
1,1,1-Trichloroethane	0.100	0.442
Cyclohexane	0.100	0.579
Carbon tetrachloride	0.100	0.353
Benzene	0.500	1.368
1,2-Dichloroethane	0.100	0.443
Trichloroethene	0.200	0.338
Methylcyclohexane	0.100	0.501
1,2-Dichloropropane	0.100	0.382

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APPENDIX 5: RECOMMENDED RESPONSE FACTORS FOR 8260C CONT'D

Volatile Compounds	Minimum Response Factor (RF)[a]	Typical Response Factor (RF)[b]
Bromodichloromethane	0.200	0.424
cis-1,3-Dichloropropene	0.200	0.537
trans-1,3-Dichloropropene	0.100	0.515
4-Methyl-2-pentanone	0.100	0.363
Toluene	0.400	1.577
1,1,2-Trichloroethane	0.100	0.518
Tetrachloroethene	0.200	0.606
2-Hexanone	0.100	0.536
Dibromochloromethane	0.100	0.652
1,2-Dibromoethane	0.100	0.634
Chlorobenzene	0.500	1.733
Ethylbenzene	0.100	2.827
meta-/para-Xylene	0.100	1.080
ortho-Xylene	0.300	1.073
Styrene	0.300	1.916
Bromoform	0.100	0.413
Isopropylbenzene	0.100	2.271
1,1,2,2-Tetrachloroethane	0.300	0.782
1,3-Dichlorobenzene	0.600	1.408
1,4-Dichlorobenzene	0.500	1.427
1,2-Dichlorobenzene	0.400	1.332
1,2-Dibromo-3-chloropropane	0.050	0.129
1,2,4-Trichlorobenzene	0.200	0.806

[a] The project-specific response factors obtained may be affected by the quantitation ion selected and when using possible alternate ions the actual response factors may be lower than those listed. In addition, lower than the recommended minimum response factors may be acceptable for those compounds that are not considered critical target analytes and the associated data may be used for screening purposes.
[b] Data provided by EPA Region III laboratory.

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APPENDIX 6: POOR PURGING COMPOUNDS

Dichlorodifluoromethane
Trichlorofluoromethane
Trichlorotrifluoroethane
Acetone
2-Butanone
2-Hexanone
4-Methyl-2-pentanone
Cyclohexanone
Carbon disulfide
n-Butyl alcohol
tert-Butyl alcohol
Isobutyl alcohol
Isopropyl alcohol
Vinyl acetate
Methyl acetate
Ethyl acetate
1,4-Dioxane
Methyl methacrylate
Ethyl methacrylate
Propionitrile
Methacrylonitrile
1,2-Dibromo-3-chloropropane

APPENDIX 7: TENTATIVE IDENTIFICATION PROCEDURES

1. Relative intensities of major ions (> 10%) in the reference spectrum should be present in sample spectrum. This equates to detects of TICs ≥ 5 ppb concentration (10% of 50 ppb internal standard concentration).

2. Relative intensities of the major ions should agree within ± 30% (i.e., for an ion with an abundance of 50% of the standard spectra, the corresponding sample ion abundance must be between 20 and 80 percent.)

3. Molecular ion present in reference spectrum should be present in sample spectrum.

4. Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

5. If no valid identification can be made, the compound should be reported as "unknown." If an additional classification can be given to the unknown (unknown hydrocarbon, aromatic, chlorinated, etc.) it should be listed as such.

6. Peaks that are detected in the sample and analytical blank should be flagged as such on the report.

7. TIC detects with “fit” value < 85 will be identified with J qualifier. Detects with “fit” value > 85 will be identified with NJ qualifier and parameter name.

8. The CAS for calibrated compounds not included on the client requested list of parameters will be the true CASRN, while the CASRN for non-calibrated compounds will be reported with leading zeros (000) in front of the probable CASRN.

9. GEL’s policy for reporting calibrated compounds when requested by the client as TICs only is to follow TIC reporting from National Functional Guidelines and report only ≥ 5ppb for VOA with J or NJ qualification.

APPENDIX 8: STANDARD METHOD 6200

1. Perform continuing calibration with one or more of the concentrations of analytical standards in the initial calibration. Vary actual concentration of continuing calibration standard over calibration range, with a minimum concentration greater than 2 times the reporting limit, the acceptance criteria is 70%- 130%. For gases, the acceptance criteria is 60%-140%. If criteria is not met then re-analyze continuing calibration standard or re-analyze initial calibration.

2. The Continuing Calibration Standard must be the same source (lot number) as the Initial Calibration Standard.

3. Internal Standard responses should be in the range ± 30% compared to the mean calibration curve area response.

4. The LCS/LFB/MS/MSD/LFS are from a different primary mix than that used to develop the initial calibration.

5. A closing continuing calibration standard (same lot number) must be analyzed. The acceptance criteria is 70%-130%. For Gases the acceptance criteria is 60%-140%. The concentration of this standard is varied and not the same as the continuing calibration standard.

APPENDIX 9: 4-BROMOFLUOROBENZENE (BFB) SUGGESTED CRITERIA FOR 8260D

m/z	Intensity (relative abundance)
95	50-200% of mass 174
96	5 to 9% of m/z 95 (5 to 15% when using H_2 carrier)
173	<2% of *m/z* 174
174	50-200% of mass 95
175	5 to 9% of *m/z* 174
176	95 to 105% of *m/z* 174
177	5 to 10% of *m/z* 176

*Criteria based on EPA Method 524.4 (Reference 17), with modified m/z and m/z 174 abundance criteria.

APPENDIX 10: SUMMARY OF QC CRITERIA FOR USE WITH 8260D

SUMMARY OF QC CRITERIA FOR USE WITH 8260D[a]

Quality Control Type	Minimum frequency	Specification	Suggested Acceptance Criteria
Instrument performance check (Secs. 9.3.1, 11.3.1)	Prior to initial calibration	Must be verified prior to initial calibration	Meet ion ratio criteria for reference compound: 4-Bromofluorobenzene Appendix 9, or alternative documented criteria
Initial Calibration (ICAL) (Secs. 9.3.2, 11.3.2-11.3.5)	Prior to analyzing samples, and as needed if continuing performance criteria cannot be met	5 points minimum for RF and linear regressions, 6 points minimum for quadratic regressions; >90% of reported target analytes meet initial calibration criteria	For average response factor (RF) calibration model: ≤20% RSD of RFs; For linear or quadratic regression model: R≥0.995, R^2≥0.99; Independent of calibration model: LLOQ standard recalculation (refit) is within ±50% of true value if it is the low calibration point; All other standards within ±30% of true value; Or, relative standard error (RSE) ≤20% (Refer to Method 8000 and Reference 16 for calculation) See Method 8000 for additional criteria.
ICAL Verification (ICV) (Secs. 9.3.2, 11.3.6)	After each initial calibration, and prior to analyzing samples	Prepared from different source of target analytes than initial calibration standards	Calculated concentrations of target analytes are within ±30% of true value
Continuing Calibration Verification (CCV) (Secs. 9.3.3, 11.4)	Once every 12 hours	>80% of target analytes meet CCV criteria	Targets are ≤20% difference or drift; IS responses are within 50% to 200% of mid-point of ICAL or average of ICAL ISs; and RTs for ISs have not shifted >30 seconds relative to ICAL
Blanks (Secs. 9.5, 9.6.1)	One method blank per preparation batch of 20 or fewer samples; other blanks as needed	NA	Target analyte concentrations in blanks are <1/2 LLOQ, or ≤ 10% of concentration in field samples
Laboratory Control Sample (LCS) (Sec 9.6.2)	One per preparation batch of 20 or fewer samples	NA	Meets recovery criteria (CCV criteria may be used if LCS and CCV are identical)

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APPENDIX 10: SUMMARY OF QC CRITERIA FOR THE USE WITH 8260D (CONTINUED)

Quality Control Type	Minimum frequency	Specification	Suggested Acceptance Criteria
Duplicates and Matrix Spikes (Secs. 9.6.3)	A duplicate and matrix spike, or matrix spike/matrix spike duplicate per preparation batch of 20 or fewer samples, provided adequate material is made available to the laboratory	NA	Meets performance-based or project-defined recovery criteria for matrix spikes; Meets relative % difference between measured concentrations in sample and laboratory duplicate or in matrix spike/matrix spike duplicate;
Surrogates (Secs. 9.7)	Added to each sample	NA	Meets performance-based recovery criteria established by the laboratory or criteria chosen for the project
Internal Standards (Secs. 9.8, 11.5.6)	Added to each sample	NA	IS response is within 50 - 200% of the response of the same IS in the midpoint ICAL standard (or average of ICAL) or most recent CCV
Qualitative Analyte Identification (Sec. 11.6.1)	Each target analyte	NA	RT in sample is within ±10 sec of RT in midpoint ICAL or CCV standard or within ±10 seconds relative to the shift of the associated IS (delta RT of the IS ± 10 seconds) Characteristic ion(s) are within ±30% of expected ion ratio in reference spectrum; or, match to reference library spectra ≥0.8 (only for full mass range acquisition modes)

[a] Default acceptance criteria; alternative criteria may be specified for a given application. Refer to Sec. 9 for more information.

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VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE

FOR

ORGANOCHLORINE PESTICIDES AND CHLORINATED HYDROCARBONS

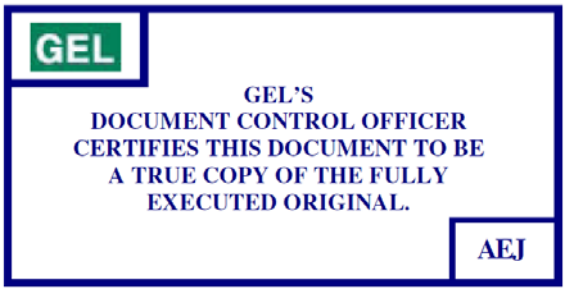
(GL-OA-E-041 REVISION 20)

APPLICABLE TO METHODS:

EPA SW-846 Methods 8000D, 8081A, 8081B and EPA 608.3

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1.0 STANDARD OPERATING PROCEDURE FOR ORGANOCHLORINE PESTICIDES AND CHLORINATED HYDROCARBONS

2.0 METHOD CODES

2.1 EPA SW-846 Methods 8000D, 8081A, 8081B and EPA 608.3

3.0 METHOD OBJECTIVE AND PURPOSE

This is a gas chromatographic procedure for quantitatively determining certain organochlorine pesticides. Separation is performed by gas chromatography (GC) and detection is done by electron capture detector (ECD). The analytical guidance for the detection and quantitation of the applicable organochlorine pesticides has been taken from Methods 8081A, 8081B, 8000D and EPA 608.3 of "Test Methods for Evaluating Solid Waste," EPA Manual SW-846.

4.0 METHOD APPLICABILITY AND METHOD SUMMARY

4.1 The following analytes can be quantitatively determined by this procedure:

Aldrin	Endosulfan I	Aroclor 1016
Alpha-BHC	Endosulfan II	Aroclor 1260
Beta-BHC	Endosulfan Sulfate	Aroclor 1221
Delta-BHC	Endrin	Aroclor 1232
Chlordane (n.o.s)	Endrin Aldehyde	Aroclor 1242
4,4'-DDD	Endrine Ketone	Aroclor 1248
4,4'-DDE	Heptachlor	Aroclor 1254
4,4'-DDT	Heptachlor Epoxide	Aroclor 1262
Dieldrin	Toxaphene	Aroclor 1268
cis-Chlordane	trans-Chlordane	
Lindane	Methoxychlor	
2,4'-DDD	O-Chlordane	
2,4'-DDE	C-Nonachlor	
2,4'-DDT	T-Nonachlor	
Mirex	Hexachlorobenzene	

NOTE: Method 8081B does not include the analysis of PCBs as Aroclors. The analysis of PCBs should be done by method 8082. PCB (Aroclor) analysis is included in method 608.3 as well as the pesticide analytes listed above.

4.2 This method applies to various matrices, including:

4.2.1 Groundwater

4.2.2 Wastewater

4.2.3 Soil

4.2.4 Sludge

4.2.5 TCLP

4.2.6 Miscellaneous matrices

4.3 This method summarizes the procedures necessary to analyze a sample extract for organochlorine pesticides by gas chromatography/electron capture detector.

5.0 METHOD SCOPE AND PERFORMANCE CHARACTERISTICS

5.1 The calibration ranges for pesticides and chlorinated hydrocarbons are compound specific. The lowest concentration level standard is referred to as the lower limit of quantitation (LLOQ), which is equivalent to the practical quantitation limit (PQL).

5.2 The typical calibration ranges follow. These concentrations represent on-column values and do not include the prep factor.

Compound	Concentration Range
4-CMX	8 µg/L – 400 µg/L
Alpha-BHC	4 µg/L – 200 µg/L
Lindane	4 µg/L – 200 µg/L
Beta-BHC	4 µg/L – 200 µg/L
Heptachlor	4 µg/L – 200 µg/L
Delta-BHC	4 µg/L – 200 µg/L
Aldrin	4 µg/L – 200 µg/L
Heptachlor Epoxide	4 µg/L – 200 µg/L
cis-Chlordane	4 µg/L – 200 µg/L
trans-Chlordane	4 µg/L – 200 µg/L
Endosulfan I	4 µg/L – 200 µg/L
4,4'-DDE	8 µg/L – 400 µg/L
Dieldrin	8 µg/L – 400 µg/L
Endrin	8 µg/L – 400 µg/L
2,4'-DDD	4 µg/L – 400 µg/L
2,4'-DDE	4 µg/L – 400 µg/L
2,4'-DDT	4 µg/L – 400 µg/L
Mirex	4 µg/L – 400 µg/L
O-Chlordane	4 µg/L – 400 µg/L
C-Nonachlor	4 µg/L – 400 µg/L
T-Nonachlor	4 µg/L – 400 µg/L
Hexachlorobenzene	4 µg/L – 400 µg/L
4,4'-DDD	8 µg/L – 400 µg/L
Endosulfan II	8 µg/L – 400 µg/L

4,4'-DDT	8 µg/L – 400 µg/L
Endrin Aldehyde	8 µg/L – 400 µg/L
Endrin Ketone	8 µg/L – 400 µg/L
Endosulfan Sulfate	8 µg/L – 400 µg/L
DCB	8 µg/L – 400 µg/L
Methoxychlor	40 µg/L – 2000 µg/L
Toxaphene	100 µg/L – 3000 µg/L
Chlordane	50 µg/L – 3000 µg/L
Chlordane (n.o.s)	50 µg/L – 3000 µg/L

5.3 Precision is determined by the relative percent difference (RPD) between the laboratory control sample (LCS) and the laboratory control sample duplicate (LCS DUP) or the matrix spike (MS) and the matrix spike duplicate (MSD). The RPD acceptance limits are determined through Statistical Process Controls (SPC), which are updated on a semi-annual basis.

5.4 Accuracy is determined by the percent recovery in LCSs and MSs and the control ranges are determined through Statistical Process Controls (SPC), which are updated at least semi-annually. For South Carolina samples, static limits of 70-130% are used as the LCS acceptance range.

5.5 Method Detection Limit (MDL) studies are performed annually for liquid and solid matrices. MDLs are maintained in AlphaLIMS.

6.0 DEFINITIONS

6.1 Definitions specific to this SOP include:

6.2 Limit of Detection (LOD): The lowest concentration level that can be determined by a single analysis and with a defined lever of confidence to be statistically different from a blank. The LOD verification is typically spiked at two times the MDL.

6.3 Limit of Quantitation (LOQ): The lowest level in the calibration curve. With the prep factor applied, the LOQ is referred to as the effective LOQ. The LOQ is equivalent to the PQL and LLOQ.

6.4 Lower Limit of Quantitation (LLOQ): The lowest concentration at which a target analyte can be reliably measures and reported. The LLOQ is lowest point in the calibration curve and represents a concentration at which both quantitative requirements can be consistently demonstrated. The LLOQ is verified quarterly, as the LOQ verification. The verification is performed by extracting and analyzing an LCS spiked at the LOQ (See table above for calibration concentrations). The LLOQ verification is carried through the same preparation and analytical procedures as environmental samples and QC. The LLOQ is analyzed on every instrument where data are reported and this is the laboratories normal protocol. Recovery of target analytes in the LLOQ are compared to in-house statistically-derived limits.

Concentrations in samples reported below the LLOQ and above the MDL are qualified as estimated.

6.5 Relative Standard Error (RSE): Standard Error indicates the extent to which a survey estimate is likely to deviate from the true population and is expressed as a number. The Relative Standard Error (RSE) is the standard error expressed as a fraction of the estimate and is usually displayed as a percentage. The RSE acceptance limit criterion is the same as the RSD limit for average CF or RF in the determinative method.

6.6 Lab-wide used definitions can be found in GL-QS-B-001 the Quality Assurance Plan.

7.0 REFERENCES

7.1 Test Methods for Evaluating Solid Waste: Laboratory Manual Physical/Chemical Methods, Volume 1 (Part 2 and Part 3) Section B. SW-846, 3rd Edition.

7.1.1 Method 8000D, "Determinative Chromatographic Separations," Rev. 4, July 2014.

7.1.2 Method 8081A, "Organochlorine Pesticides by Gas Chromatography," Revision 1, December 1996.

7.1.3 Method 8081B, "Organochlorine Pesticides by Gas Chromatography," Revision 2, February 2007.

7.1.4 EPA Method 608.3

7.2 Dept. of Defense (DOD), Dept. of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories DOD QSM Version 5.2, December, 2018.

8.0 INTERFERENCES TO THE METHOD

8.1 Interference by phthalates can pose a major problem in pesticide determinations. These compounds generally appear in the chromatogram as large late-eluting peaks. Common flexible plastics contain varying amounts of phthalates, which are easily extracted or leached from such materials during laboratory operations. Cross contamination of clean glassware routinely occurs when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled.

8.2 Interference for phthalates can best be minimized by avoiding contact with any plastic materials. Exhaustive cleanup of reagents and glassware may be required to eliminate background phthalate contamination.

8.3 Many of the analytes of interest are susceptible to breakdown from active sites at the front of the column and in the injection port. Care should be taken to ensure that these areas are properly cleaned.

8.4 Additional cleanups are outlined in method 8081B, including silica gel (method 3630), gel permeation chromatography (method 3640), alumina (method 3610), and sulfur clean-up (method 3660).

9.0 SAFETY PRECAUTIONS AND HAZARD WARNINGS

9.1 Treat all chemicals and samples as potential health hazards, and limit exposure to these chemicals to the lowest level possible. GEL maintains a current awareness file

of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals in the laboratory as well as a reference file of Material Safety Data Sheets (MSDS). These documents and client sample MSDS forms are also maintained in the laboratory.

9.2 Personal protective equipment

9.2.1 Gloves are required when handling the samples and chemicals in this procedure.

9.2.2 Safety glasses with side shields and lab coats should also be worn.

9.3 Prior to handling radioactive samples, analysts must have had radiation safety training and must understand their full responsibilities in radioactive sample handling.

9.4 All samples, chemicals, extracts, and extraction residues must be transferred, delivered, and disposed of safely according to all related SOPs.

9.5 Never leave gas cylinders unchained or untied, including when they are on the moving carts.

9.6 In the event of an accident or medical emergency, call for help immediately. When time and safety permit, an accident report form should be completed and turned in to Fire escape routes are posted in the lab and all personnel should be familiar with them. In addition, fire safety equipment such as fire extinguishers is located throughout the lab. Training is available on the proper operation of this equipment.

9.7 Refer to SOP GL-LB-N-001 Safety, Health and Chemical Hygiene Plan for general safety and health information pertaining to the laboratory.

10.0 CAUTION WARNINGS

The helium, nitrogen and hydrogen tanks should be replaced when pressure drops to approximately 250 psi.

11.0 APPARATUS AND EQUIPMENT, REAGENTS, AND INSTRUMENTS

11.1 Apparatus and Equipment:

11.1.1 Volumetric flasks

11.1.2 Pasteur pipettes

11.1.3 2 mL amber auto sampler screw-capped vials with Teflon-lined septa, or 2 mL amber auto sampler crimp top vials with 500 µL inserts.

11.1.4 Microliter syringes (10, 25, 50, 100, 250, 500, and 1000 µL)

11.1.5 Capillary cleaving tool

11.1.6 Supeltex 0.5 and 0.8 mm ferrules (or equivalent)

11.2 Reagents, Chemicals and Standards

11.2.1 Solvents: Acetone, hexane, isooctane, and toluene (pesticide grade or equivalent)

11.2.2 For chemical standards refer to Section 14.0.

11.3 Instrumentation and Columns

11.3.1 Pesticides

11.3.1.1 GC: Agilent 6890,7890 or equivalent.

11.3.1.2 Agilent GC/SFC Auto Sampler (or equivalent)

11.3.1.3 Restek CLP Pesticides 30m X 0.25mm X 0.2µm Column (used for fast GC)

11.3.1.4 Restek CLP Pesticides II 30m X 0.25mm X 0.25µm Column (used for fast GC)

NOTE: The instrumentation and columns listed above are merely the recommended instrumentation and columns for use with this method.

12.0 SAMPLE HANDLING AND PRESERVATION REQUIREMENTS

12.1 Samples must be collected in an amber glass bottle with a Teflon-lined cap. The collection containers are bought pre-cleaned from a certified vender.

12.2 All liquid samples have a holding date of seven days from the time of collection to be extracted while all solid samples have fourteen days from the collection date to be extracted. Method 608.3 stipulates that unpreserved samples need to be extracted within 72 hours. Preserved samples with a pH of 5 > 9 have a hold time of seven days. Samples are protected from light and stored at 0° ≤ 6° C after collection. Once extracted, the extract must be analyzed within forty days. However, during this period of time, the extracts are refrigerated at 0° ≤ 6° C in amber vials with Teflon- sealed screw caps. When holding dates are missed, the data are automatically flagged.

13.0 SAMPLE PREPARATION TECHNIQUES

13.1 Refer to GL-OA-E-070 for Extraction of Semivolatile and Nonvolatile Organic Compounds from Groundwater, Wastewater and Other Aqueous Samples, and GL-OA-E-066 for Extraction of Semivolatile and Nonvolatile Organic Compounds from Soil, Sludge and Other Miscellaneous Solid Samples.

NOTE: It is assumed that materials within the sample container are considered "the sample." Removal of any extraneous material (twigs, leaves, large rocks, etc.) must be documented in the case narrative and bench logbooks.

14.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

14.1 In order to maintain the gas chromatograph's columns and detectors, the gases should be changed when the tank pressure is below 250 psi. As needed, the septum in the injection port should be changed and the injection port cleaned. Column maintenance is performed when baseline rise is present or when the column becomes contaminated.

14.2 Suggested Routine Maintenance

COMPONENT	DAILY	WKLY	MTHLY	AS NEEDED	ANNUALLY
Injector septum				X	
Gases		X			

Column Maintenance	X	
Detector Wipe Test		X
Detector Cleaning	X	

14.3 Non-routine Maintenance

14.3.1 When a check standard fails, the standard is examined for signs of evaporation or degradation. If nothing is found, column maintenance should be performed. Approximately one loop of each column or two loops of guard column should be cleaved. The instrument is then baked out at 320˚ C until an acceptable baseline is obtained. If an acceptable baseline is not obtained, the column is leaking and column maintenance must be performed again.

14.3.2 When contamination occurs, first replace or clean the auto sampler syringe. If contamination is still present, column maintenance should be performed. If contamination is still present after column maintenance has been performed and the system has been cleaned thoroughly, replace the column. The column must then be leak checked and baked. The instrument must then be checked to determine if the problem is solved and if the instrument is stable. If not, the in-house service technician is called.

14.4 When maintenance is done on the instrument, it must be recorded in the maintenance logbook. It must be initialed and dated.

15.0 PREPARATION OF STANDARD SOLUTION AND QUALITY CONTROL SAMPLES

15.1 Source Standard Solutions

15.1.1 Source standard solutions are purchased from certified vendors. These standards are traceable to National Institution of Standards and Technology (NIST) standards. The source standards are received from the company Purchasing Agent. The standard is given a unique identifying number for that day and is entered and traced in AlphaLIMS using the standards and reagents data base.

15.2 For guidance on standard documentation, refer to GL-LB-E-007 for Laboratory Standards Documentation.

15.3 Source and stock standards expire one year after opening or the manufacturer's expiration, whichever is shorter. Working standards expire after six months or the manufacturer's expiration, whichever is shorter.

16.0 INSTRUMENT CALIBRATION

16.1 The instrument should be checked for cleanliness and stability prior to analyzing calibration standards. The standards are loaded onto the auto sampler with the lowest standard being analyzed first to prevent carryover.

16.2 An external standard technique is used to calibrate the instrument. Internal standard techniques are optional but not used. Calibration is achieved by analyzing multiple

standards (minimum of five for 8000 methods and three for 600 methods) at different concentrations. The laboratory uses a minimum of five concentration levels for each individual and multi-component pesticide compound. All target analytes and surrogates are included in each standard. A calibration curve is plotted for each analyte using the total area under the peak versus the mass (in ng) of the analyte injected. For multi-component compounds such as chlordane and toxaphene, a minimum of five major peaks representing the compound should be chosen for calibration and quantitation purposes. Chlordane (n.o.s) peaks should include cis-chlordane, trans-chlordane and heptachlor. The validator selects the most technically sound result for heptachlor so that the concentration of heptachlor is not reported twice. A response factor is obtained for each representative peak chosen in the multi-component compound. The same peaks in the calibration standards must be used to calculate response factors in continuing calibration checks, spike concentrations, and samples results. Multi-component standards are analyzed separately from the individual pesticide compound mix. Typical calibration levels are listed in the following table. Level 1 corresponds to the LLOQ (PQL).

	Level 1 (ug/L)	Level 2 (ug/L)	Level 3 (ug/L)	Level 4 (ug/L)	Level 5 (ug/L)
Hexachlorobenzene	4	20	100	200	400
alpha-BHC	4	10	50	100	200
gamma-BHC (Lindane)	4	10	50	100	200
Heptachlor	4	10	50	100	200
Aldrin	4	10	50	100	200
beta-BHC	4	10	50	100	200
delta-BHC	4	10	50	100	200
Heptachlor epoxide	4	10	50	100	200
Endosulfan I	4	10	50	100	200
gamma-Chlordane	4	10	50	100	200
alpha-Chlordane	4	10	50	100	200
Mirex	4	10	50	100	200
c-Nonachlor	4	10	50	100	200
o-Chlordane	4	10	50	100	200
t-Nonachlor	4	10	50	100	200
2,4-DDD	4	10	50	100	200
2,4-DDE	4	10	50	100	200
2,4-DDT	4	10	50	100	200
4,4'-DDD	8	20	100	200	400
4,4'-DDE	8	20	100	200	400
4,4'-DDT	8	20	100	200	400
Dieldrin	8	20	100	200	400
Endrin	8	20	100	200	400
Endosulfan II	8	20	100	200	400
Endrin aldehyde	8	20	100	200	400
Endosulfan sulfate	8	20	100	200	400

Endrin ketone	8	20	100	200	400
4-CMX (surr)	8	20	100	200	400
DCB (surr)	8	20	100	200	400
1,2-Dichlorobenzene	20	50	100	150	200
Methoxychlor	40	100	500	1000	2000
Chlordane (nos)	50	100	500	1000	3000
Aroclor 1016	100	250	500	1000	4000
Aroclor 1221	100	250	500	1000	4000
Aroclor 1232	100	250	500	1000	4000
Aroclor 1242	100	250	500	1000	4000
Aroclor 1248	100	250	500	1000	4000
Aroclor 1254	100	250	500	1000	4000
Aroclor 1260	100	250	500	1000	4000
Aroclor 1262	100	250	500	1000	4000
Aroclor 1268	100	250	500	1000	4000
Toxaphene	100	500	1000	2000	3000

16.3 The MDL, LOD, and LLOQ (LOQ) are verified quarterly. The MDL verification is spiked at the MDL concentration (approximately one third of the LLOQ). The LOD is spiked at two times the MDL. And the LLOQ is spiked at the lowest calibration level (the PQL). Verification samples are extracted using the same methods and processes used for samples and analyzed on each instrument used for that analysis. Statistical Process Limits (SPC) are calculated for the LLOQ using historical data from the lab and are used to evaluate the LLOQ recoveries.

16.4 The ratio of the response to the amount injected, defined as the calibration factor (CF), can be calculated. Also calculate the mean calibration factor and the relative standard deviation for each analyte at each standard concentration using the formula below.

16.4.1 Calculate the calibration factor for each analyte at each concentration as:

$$CF = \frac{\text{Peak Area of the Compound in the Standard}}{\text{Mass of the Compound Injected (in nanograms)}}$$

The CF can also be calculated using the concentration of the standard rather than the mass in the denominator of the equation above. However, the use of the concentration will require changes to the equations that are used to calculate sample concentration.

16.4.2 Calculate the mean calibration factor for each analyte as:

$$\overline{CF} = \frac{\sum_{i=1}^{n} CF_i}{n}$$

Where n is the number of standards analyzed.

16.4.3 Calculate the standard deviation and the %RSD of the calibration factors for each analyte as:

$$SD = \sqrt{\frac{\sum_{i=1}^{n}(CF_i - \overline{CF})^2}{n-1}} \qquad RSD = \frac{SD}{\overline{CF}} X\ 100$$

If the calculated %RSD for the analyte response factors is ≤ to 20% over the working range, linearity through the origin may be assumed and the mean calibration factor can be used to quantitate sample results. If the %RSD is greater than 20% the only other option the analyst may use to determine linearity is by linear regression using the calibration curve.

16.5 If the analyst chooses to use linear regression, he/she must not force the calibration line through the origin.

16.5.1 Make certain that the instrument response is treated as the dependent variable (y) and the concentration as the independent variable (x). The regression will produce the slope and intercept terms for a linear equation in the form:

$$y = ax + b$$

Where:

y = instrument response

a = slope of line (also called the "coefficient of x")

x = concentration of the calibration standard

b = the intercept

16.5.2 The analyst should not force the line through the origin, but have the intercept calculated from the five data points. Otherwise, the problems noted with the RSD value will occur, i.e., a line through the origin will not meet the QC specifications. In addition, do not include the origin (0, 0) as a sixth calibration point.

16.5.3 The regression calculation will generate a correlation coefficient (r) that can be used to calculate a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be ≥ 0.990. The calculated intercept value needs to be evaluated before reporting sample results. If the system you are using calculates the coefficient of determination (r^2), the value must be ≥ 0.990.

16.5.4 A positive value for the intercept indicates that there is some threshold instrument response that is the limiting factor in establishing linearity. A negative intercept value can be transformed into an x-intercept value that represents a threshold concentration, which is the limitation. If the intercept is positive, then, as a general rule, results where the instrument

response is less than three times (3x) the intercept value may be unreliable. This will afford some protection against false positive results. If the intercept is negative, results below the concentration of the lowest concentration calibration standard may be unreliable.

16.5.5 In calculating the sample concentrations, the regression equation is rearranged to solve for the concentration (x) as shown below:

$$x = \frac{(y - b)}{a}$$

16.6 In some cases, it is likely that certain analytes may not meet the %RSD or linearity criteria. If the calibration curve contains more than the minimum number of concentration levels stated in the method, the analyst may want to narrow the calibration range by eliminating outlying responses. Remember that changing the lowest level in the calibration standard will change the laboratory practical quantitation limit. Consider the project specifications for regulatory limits and action levels to confirm that this option can be used. Individual analyte end points may be eliminated. If the calibration linearity continues to fail despite using the above options, check instrument operating conditions and perform additional maintenance. Changing instrument parameters or columns may be required. Recalibration is required if instrument conditions are changed.

16.7 Method 8000D outlines two procedures that may be used to determine calibration function acceptability for linear and non-linear curves. The calibration data refitted back to the calibration model. % Error and Relative Standard Error (RSE) evaluate the difference between the measured amount and the true amount (or concentration). % Error is determined as follows:

$$\%Error = \frac{x_i - x'_i}{x_i} \times 100$$

Where:

16.8 x_i = True amount of analyte at calibration level i, in mass or concentration units

x'_i = Measured amount of analyte at calibration level i, in mass concentration units

16.9 Percent error between the calculated and expected amount of an analyte should be ≤ 30% for all standards and ≤ 50% for the lowest calibration level.

16.10 Relative Standard Error is calculated as follows:

$$RSE = 100 \times \sqrt{\sum_{i=1}^{n} \left[\frac{x'_i - x_i}{x_i} \right]^2 \Big/ (n - p)}$$

Where:

x_i = True amount of analyte in calibration level I, in mass of concentration units

x'_i = Measured amount of analyte in calibration level I, in mass or concentration units

16.11 p = Number of terms in the fitting equation (average = 1, linear =2, quadratic = 3, cubic = 4)

n = Number of calibration points

16.12 The RSE acceptance limit criterion is the same as the RSD limit for CF or RF in the determinative method. If the RSD limit is not defined in the determinative method, the limit should be set at ≤ 20% for well performing compounds and ≤ 30% for poor responders. Please see the appendix for a list of demonstrated poor responders.

16.13 Continuing Calibration

16.14 The initial calibration curve must be verified for each compound of interest prior to sample analysis. Verification is accomplished by analyzing second source standards at or near the midpoint standards in the calibration curve. It is analyzed after every 10 samples. The laboratory performs the initial calibration verification prior to sample analysis and inject continuing calibration verification standards (minimum Pest A/B mix) after every ten samples. The sequence must be concluded with a final calibration verification standard (Pest A/B mix). The ICV and CCV standards should be followed by an instrument blank (IB) as the next injection in the analytical sequence. A second source standard should be provided by a second vendor whenever available. Otherwise, a second lot will be used. Using a second source verification standard will verify the initial calibration on a daily basis.

16.14.1 The initial calibration is verified if each analyte calibration factor in the calibration verification standard is ≤ 15% difference or drift (≤ 20% for 8081B) of the mean calibration factor in the initial calibration. Use the following equation to calculate the % difference.

$$\% \text{ Difference} = \frac{\overline{CF} - CF_v}{\overline{CF}} \text{ X } 100$$

Where:

CF_V = response factor from current verification check standard.

If the linearity of the analyte calibration curve uses linear regression, the % drift must not exceed ± 15% (± 20% for 8081B). Calculate the % drift using the calculated value for the analyte and the true value as follows:

$$\% \text{ drift} = \frac{|\text{true value} - \text{calculated value}|}{\text{true value}} \text{ X } 100$$

16.14.2 The analyst should evaluate the retention time of each analyte in the initial continuing calibration standards with the absolute retention time windows established over a 72-hour time period. Apply the retention

time window to the absolute retention times of each analyte in the calibration curve midpoint standards and ensure that the initial continuing calibration standard analytes are within the established windows. If retention times have drifted outside the windows, the analyst should stop and investigate the cause for the drift. As the calibration curve ages and column maintenance is performed, retention time shifts should be expected. However, if the calibration verification standards are analyzed after the initial calibration curve, retention time shifts should not occur. If analyte retention times are outside the established windows the system is considered out of control and the analyst must attempt to correct the problem before continuing the analysis.

16.14.3 Update the retention time windows for each analyte on both columns using the initial continuing calibration standards absolute retention times prior to sample analysis whenever a new analytical sequence is started. If continuing verification standard analyte retention times are outside the established windows the instrument is considered out of control and the analysis must stop. Samples analyzed before the unacceptable standard must be reanalyzed when the instrument is returned to control. Common problems associated with retention time shifts include inlet leaks, low carrier gal flow, and broken or cracked columns.

17.0 INSTRUMENT PERFORMANCE REQUIREMENTS

17.1 Before samples can be analyzed, the baseline must be stable with little or no back ground noise. If using dual column/detector both sides must be stable. Analysis of an acceptable degradation standard is required prior to analyzing the initial calibration curve and/or initial calibration verification standards and daily every twelve hours. If breakdown for DDT or Endrin exceeds 15% the analyst must stop and perform instrument maintenance before continuing with the analysis. Refer to Section 20 to calculate percent breakdown.

17.2 Each instrument must have a valid initial calibration curve for each analyte of interest on both columns. Hard copies of the continuing calibration standards are kept with the quality control samples and client samples analyzed in the sequence.

17.2.1 Continuing calibration verification standards (CCV) must meet the criteria in Section 16. When a calibration verification standard fails to meet the criteria, all samples that were injected after the last standard that met the criteria must be evaluated to prevent misquantitations and possible false negative results. Reanalysis of sample extracts may be required. More frequent analysis of standards will minimize the number of sample extracts that will have to be reanalyzed if the standard verification criteria are not met.

17.3 Sample injections may continue for as long as the calibration verification standards and standards interspersed with the samples meet the quality control criteria in

Section 16. The sequence ends when the set of samples has been injected or when standards exceed the acceptance criteria.

17.3.1 If during the analysis of a sample group from a single client, the bracketing continuing verification standards do not meet the acceptance criteria, reanalysis of this group of samples is required. The reanalyses are required to confirm that the sample extracts (matrices) are causing the instrument response to fluctuate and standards to fail. The reanalysis must begin with an acceptable CCV. If continuing verification standards fail during the analysis sequence, the failure is attributed to matrix. If the standards meet the acceptance criteria, then matrix interference is not confirmed and the acceptable standards and samples in between should be reported.

17.3.2 In addition, if multiple client sample extracts are analyzed in the analytical sequence where continuing verification standard failures occur, each client's samples must be reanalyzed after an acceptable initial calibration, initial continuing calibration or continuing verification standard.

18.0 ANALYST AND METHOD VERIFICATION REQUIREMENTS

18.1 To establish that the analyst can perform the procedures in an acceptable manner and that the method generates data of acceptable bias and precision, the following operations are performed.

18.1.1 A quality control (QC) check standard must be prepared containing each analyte of interest. It must be prepared from pure standard material or purchased as a certified solution. It must be made from a source independent of that used for calibration. AB, toxaphene (TOX), and chlordane (CHL) IDOCs must be each prepped separately. An IDOC is required for each matrix as well.

18.1.2 Four samples must be prepared and analyzed by the same procedures used to prepare and analyze actual samples.

18.1.3 Calculate the average recovery (X) in µg/L, and the standard deviation of the recovery (S) in µg/L, for each analyte of interest using the four results.

18.1.4 For each analyte compare S and X with the corresponding acceptance criteria for precision and accuracy, respectively, given in the quality control table at the end of the method. If the S and X for all analytes of interest meet the acceptance criteria, the system's performance is acceptable and analysis of actual samples can begin. If any individual S and X exceeds the precision limits or falls out of the range for accuracy, the system's performance is unacceptable for that analyte and a check standard for that analyte must be prepared and reanalyzed.

18.2 Precision and accuracy are matrix dependent and are documented by means of a laboratory control sample (LCS) and a matrix spike (MS) and matrix spike duplicate

(MSD). (Refer to Section 5.4 and GL-LB-E-001 for the Determination of Method Detection Limits.)

18.3 Details of how Initial (IDOC) and Continuing (CDOC) Demonstrations of Capability are performed are in GL-QS-E-011.

19.0 ANALYSIS PROCEDURES AND INSTRUMENTAL OPERATION

19.1 Standards, samples, blanks, and quality control sample extracts are introduced into the instrument via direct injection (autosampler). Retention time windows must be updated for each analyte after the initial verification standard analysis. The absolute retention time windows are determined during a 72-hour period when standards are analyzed. The retention time window is equal to three times the standard deviation of the absolute retention times for each analyte (or peak). Refer to GL-OA-E-001 for Establishing Retention Time Windows for GC and HPLC Analysis.

19.2 Samples are analyzed in a set referred to as an analysis sequence or run sequence. Prior to analyzing a pesticide calibration curve (Pest A/B mixes) or initial check standards, inject a degradation check standard (PEM) to evaluate the potential breakdown of Endrin and DDT. Refer to Section 20 to calculate the percent breakdown. Each sequence begins with a solvent blank and the analysis of the PEM, initial calibration curve or the initial calibration verification standards followed by samples. A mid-level calibration check standard must be analyzed after twenty samples (the lab uses ten) and at the end of the analytical sequence. The sequence ends when all the extracts have been injected and analyzed.

19.3 The data are entered into the computer and a data report is generated. The sequence file is created on the computer by the analyst. It lists every injection made by the instrument, in order, for a given date. The raw data, a copy of the run sequence, batch sheet, case narrative, and a copy of the retention time window are filed together.

19.4 Before the instrument is used, a highly concentrated standard may be analyzed to prime the columns.

19.5 0.5 to 2.0 µL of the extract is injected into the instrument.

19.6 All HP6890 Gas Chromatographs are equipped with the HP 7683 autosampler. It has 100 positions and can be programmed to start from any given position. The digital integrator converts the analog from the instrument to digital information that is processed into a graphic format showing concentration based on peak area.

19.7 A run sequence is entered into the sequence table in the ChemStation software. The samples are entered in the order in which they are to be injected. Additional information, such as vial number, method name, injection vial, and injection volume is also entered. The sequence may be automatically started by selecting “run sequence.”

19.8 Samples containing target analyte concentrations that exceed the linear range of the analyte calibration curve must be diluted. The dilution level should be performed to place the highest analyte concentration between the middle and high points of the calibration curve (on column). If a sample is initially diluted and target analytes are

not detected and non-target analytes are not interfering with the analysis, the sample must be reanalyzed at a lower dilution. Analysts should be aware that diluting samples will increase the detection limits for undetected analytes. Random dilutions without due cause are not acceptable. Samples should undergo appropriate clean up methods prior to diluting for observed matrix problems.

19.9 Samples are analyzed using a dual column procedure. The initial calibration and continuing calibration standards must meet the acceptance criteria on both columns before attempting sample analysis. The analyst may choose one or the other column as the primary (for reporting sample results) and the other as the confirmation column. However, it is the lab's standard practice to report the lower result as per method 8000D. If any analyte in the continuing verification standard does not meet the acceptance criteria on the confirmation column and that target analyte is not detected or confirmed above the analytes practical quantitation limit, then the associated samples do not require reanalysis.

20.0 CALCULATIONS AND DATA REDUCTION METHODS

20.1 The concentration of each analyte in an extract can be determined by comparing the response obtained from analyzing the extract to the calibration curve. The concentration of a specific analyte is matrix specific and is calculated as follows:

AQUEOUS SAMPLE

$$\textit{Concentration } (\mu g/L) = \frac{(C)(D)(V_t)}{1000\ V_i}$$

Where:

C = Concentration (µg/L) calculated by data system from total area substituted into the linear equation derived from the multilevel calibration

D = Dilution factor, if made prior to analysis. If not, D = 1.

V_t = Total volume of the extract in mL

V_i = Initial volume of sample in L

NONAQUEOUS SAMPLES

$$\text{Concentration}(\mu g/kg) = \frac{(C)(D)(V_t)}{W}$$

Where:

C = Concentration (µg/L) calculated by data system from total area substituted into the linear equation derived from the multilevel calibration

D = Dilution factor, if made prior to analysis. If not, D = 1.

V_t = Total volume of the extract in mL

W = Weight of the sample extracted in g

DEGRADATION

$$\% \ Breakdown = [(A_1 + A_2)/(A_1 + A_2 + A_3)](100)$$

Where:

A_1 = Total area of 4,4'-DDE or Endrin Aldehyde

A_2 = Total area of 4,4'-DDD or Endrin Ketone

A_3 = Total area of 4,4'-DDT or Endrin

20.2 Results are reported to the nearest one tenth for all matrices.

20.3 The reporting units are micrograms of analyte per liter of sample for aqueous samples, micrograms of analyte per kilogram of sample for soils, and milligrams per liter for TCLPs.

21.0 DATA RECORDING

Data are recorded and calculated by the Chemstation data acquisition system. They are stored on a remote server. Data are also entered into AlphaLIMS.

22.0 QUALITY CONTROL REQUIREMENTS

22.1 Before sample analysis begins the instrument must have acceptable calibration curves on both columns. Each curve is verified by the analysis of second source continuing calibration standards. Each standard must meet the acceptance criteria for percent difference or drift. Continuing calibration verification standards must be analyzed after every twenty samples. The lab's standard practice is to bracket every ten samples.

22.2 A method blank is used to determine background concentrations of analytes of interest that have the potential to interfere with sample analysis. These blanks are analyzed with every analytical batch that has a maximum number of twenty samples. The criterion for acceptance is that there are no target analytes of interest present above the practical quantitation limit. For DoD QSM, no targets may be present at concentrations > ½ reporting limit (RL). For common lab contaminants, no targets may be detected > RL.

22.3 If the analyte of interest is present at a concentration between the MDL and LLOQ, all data are qualified with a "B" flag and reported. If the analyte of interest is present at a concentration above the LLOQ and the samples contain the analyte of interest at a concentration of greater than 10 times the concentration found in the blank, the data are qualified with a "B" flag and reported. If the concentration found in the sample is less than 10 times that found in the blank and greater than the LLOQ, the samples must be re-extracted.

22.4 The laboratory evaluates the effect of matrix on method performance by performing a matrix spike (MS) and matrix spike duplicate (MSD) for each matrix with up to 20 samples in the same batch. For method 608.3, a matrix spike must be performed for

10% of the samples. Matrix spike recovery limits are generated statistically using at least 20 result pairs of the same matrix. In addition, a laboratory control sample (LCS) and its duplicate (when requested) are analyzed with each sample batch (up to 20 samples of the same matrix). The LCS consists of an aliquot of clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes at the same concentration as the matrix spike. When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. LCS recoveries are statistically generated using at least 20 results of the same matrix type. SPC limits are updated semi-annually.

NOTE: For SCDHEC clients, liquid LCS limits of 70-130% are used.

22.5 Tetrachloro-m-xylene (4CMX) and Decachlorobiphenyl (DCB) surrogates are added to all samples (prior to extraction) and standards. Surrogate recovery limits are statistically determined using at least 20 sample results of the same matrix. SPC limits are updated semi-annually.

22.6 The retention time (RT) window must be established using the initial calibration check standard. Refer to OA-E-001 for Establishing Retention Time Windows for GC and HPLC Analysis. In order to report a concentration from the external standard table, the retention time of that particular analyte must be within its established window. If not, the concentration is not reported.

22.7 Nonconformance

22.7.1 When running a calibration curve for more than one analyte at a time, some of the analytes may not meet the acceptance criteria. Additional standards containing the compounds that were not acceptable may be analyzed. If the curve still does not meet the acceptance criteria, maintenance should be performed or a new standard may be needed. Refer to Section 16.5 for other options addressing the initial calibration.

22.7.2 If the continuing check standard fails any criteria in Section 22.1, the analyst must take action to correct the situation. This may be performing any of the maintenance steps described in Section 14 to get the instrument to meet its daily calibration. If all attempts fail, the analyst must analyze a new series of calibration standards, thus obtaining a new calibration curve.

22.7.2.1 If the standard analyzed after a group of samples exhibits a response for an analyte that is above the acceptance limit (i.e. >15% or > 20%), and the analyte was not detected in the specific samples analyzed during the analytical shift, then the extracts for those samples do not need to be reanalyzed. If an analyte above the QC limits was detected in a sample extract, reinjection of the extract is necessary.

22.7.3 If the percent recovery in the matrix or matrix spike duplicate falls outside the established SPC limits for recovery, the analyst should evaluate the LCS recoveries and method blank analyses. If the LCS and method blank analyses do not indicate a problem with the preparation procedures, the matrix spike recoveries may be attributed to matrix effect. Surrogate recovery data should also be used to evaluate the data. Recoveries of both matrix spike compounds and surrogates that are outside the acceptance limits suggests more pervasive analytical problems then problems with the recoveries of either matrix spikes or surrogates alone. Analysts are not required to reanalyze the matrix spikes for failing recoveries; however they should seek additional technical support before deciding not to reanalyze matrix spikes samples.

22.7.4 If a surrogate in a sample falls outside the acceptable fixed range, the sample must be re-extracted. If the surrogate fails the second time, the failure is attributed to matrix interference, and the data from the first extraction are reported. If the surrogate failures are not confirmed, a nonconformance report is filed. (Refer to GL-QS-E-004 for Documentation of Nonconformance Reporting and Dispositioning and Control of Nonconforming Items.)

22.8 Any positive identification and quantitation of an analyte of interest must be confirmed on a separate column and/or GC/MS. The confirmation column or GC/MS must meet all quality control acceptance described in the method (calibration, retention times, etc.). Calculate the relative percent difference between the two results using the formula:

$$RPD = \frac{|R_1 - R_2|}{\frac{(R_1 + R_2)}{2}} \times 100$$

22.8.1 Since the same method criteria specified in Sections 22.1 through 22.5, and instrument calibration specified in Sections 16.1 through 16.6 are applied uniformly to both columns, either column can be selected to serve as the primary or confirmatory column.

22.8.2 If one result is significantly higher (> 40%), check the chromatograms to see if an obviously overlapping peak is causing an erroneously high result.

22.9 If no anomalies are noted, review the chromatographic conditions. If there is no evidence of chromatographic problems, report the result lower column.

23.0 DATA REVIEW, VALIDATION AND APPROVAL PROCEDURES

Refer to SOP GL-OA-E-044 Organics Data Validation.

24.0 DATA TRANSMITTAL

After the review process is complete, Data Management receives the data.

25.0 RECORDS MANAGEMENT AND DOCUMENT CONTROL

All logbooks and data generated as a result of this procedure are maintained as quality records in accordance with GL-QS-E-008 for Quality Records Management and Disposition.

26.0 LABORATORY WASTE HANDLING AND DISPOSAL: SAMPLES, EXTRACTS, DIGESTATES AND REAGENTS

For the proper disposal of sample and reagent wastes from this procedure, refer to the Laboratory Waste Management Plan, GL-LB-G-001.

27.0 HISTORY

Revision 16: Added clarification to LLOQ definition. Added current reference to DOD/DOE QSM version 5.1 and 3.1 January, 2017.

Revision 17: Added current ECD and FID Method Comparison table in Appendix 1. Update to include DOD/DOE 5.1 requirements.

Revision 18; Updated definitions for variables. Updated missing part of equation in RPD.

Revision 19: MUR update to new 600 methods.

Revision 20: Revised to new method updates 608.3 and QSM 5.2.

APPENDIX 1: ECD, DOD QSM COMPARISON TABLE

ECD Method Comparison Table

Last Updated: March 2019

	8082, 8081A, 8151, 8015B	8082A, 8081B, 8015D	DODQSM 5.2 (QSAS)	608.3
IDOC	Analyze four LCS replicates. Evaluate average and standard deviation of replicates with method limits. Generate QC Summary and training certificate.	Analyze four LCS replicates. Evaluate average and standard deviation of replicates with method limits. Generate QC Summary and training certificate.	Analyze four LCS replicates. Evaluate average and standard deviation of replicates with method limits. Generate QC Summary and training certificate.	Analyze four LCS replicates. Evaluate average and standard deviation of replicates with method limits. Generate QC Summary and training certificate.
MDL & LOD/LOQ	Following initial set-up, MDLs are determined annually using points from each quarter. Q1: 1/2x, 1x, LOD, LOQ, MDL. Q2, Q3, Q4: LOD, LOQ, MDL. Analyzed on every instrument. LOD = 2x MDL LOQ = LLOQ=PQL	Following initial set-up, MDLs are determined annually using points from each quarter. Q1: 1/2x, 1x, LOD, LOQ, MDL. Q2, Q3, Q4: LOD, LOQ, MDL. Analyzed on every instrument. LOD = 2x MDL LOQ = LLOQ=PQL	Following initial set-up, MDLs are determined annually using points from each quarter. Q1: 1/2x, 1x, LOD, LOQ, MDL. Q2, Q3, Q4: LOD, LOQ, MDL. Analyzed on every instrument. LOD = 2x MDL LOQ = LLOQ=PQL .	Following initial set-up, MDLs are determined annually using points from each quarter. Q1: 1/2x, 1x, LOD, LOQ, MDL. Q2, Q3, Q4: LOD, LOQ, MDL. Analyzed on every instrument. LOD = 2x MDL LOQ = LLOQ=PQL
RT Windows	RT study performed over 72 hours at time of method set-up and when significant maintenance is done (column change).	RT study performed over 72 hours at time of method set-up and when significant maintenance is done (column change).	RT study performed over 72 hours at time of method set-up and when significant maintenance is done (column change).	RT study performed over 72 hours at time of method set-up and when significant maintenance is done (column change).
ICAL	Minimum of five levels. RSD for each target analyte ≤ 20% or linear regression for each target analyte R^2 ≥ 0.99. %Error: ± 50%	Minimum of five levels. RSD for each target analyte ≤ 20% or linear regression for each target analyte R^2 ≥ 0.99. %Error: ± 50%	Minimum of five levels. RSD ≤ 20% for all targets or linear regression R^2 ≥ 0.99. Analytes that do not meet either option may not be analyzed per	Minimum of three levels. RSD for each target analyte ≤ 20%.

	for low level and ± 30% for all other levels. RSE: criteria same as RSD for the method. If %Error or RSE fail, consult validator or GL. If adequate sensitivity has been established, ICAL may be acceptable for qualitative analysis.	for low level and ± 30% for all other levels. RSE: criteria same as RSD for the method. If %Error or RSE fail, consult validator or GL. If adequate sensitivity has been established, ICAL may be acceptable for qualitative analysis.	QSAS. If criteria are not met, analyze sample(s) and QC on passing instrument for those compound(s).	
ICV	Second source. Same acceptance criteria as CCV.	Second source. All targets should recover within ± 20%.	Second source. All targets should recover within ± 20%. DOD QSM 5.2 does not allow for exceptions for the ICV. Failing compounds should be re-analyzed on a passing instrument. If re-analysis is not possible, then failures must be documented and corresponding data Q qualified and narrated	Second source ± 20%.
CCV	Analyze at beginning of sequence and after every 10 samples. ± 15% If bracketing CVS fails high and no detects, data may be reported. If CVS fails low on one column with no detects, data may be reported. If CVS fails low on both columns and/or there are detects, bracket	Analyze at beginning of sequence and after every 10 samples. ± 20% If bracketing CVS fails high and no detects, data may be reported. If CVS fails low on one column with no detects, data may be reported. If CVS fails low on both columns and/or there are detects, bracket	Analyze at beginning of sequence and after every 10 samples All targets within ± 20%. QSM 5.2 does not provide exception criteria for failures. Re-analyze failing target compound(s) on a passing instrument. If re-analysis is not possible, then	Analyze every 12 hours. All targets within ± 20%.

	must be reanalyzed to confirm	must be reanalyzed to confirm	failures must be documented and the corresponding data Q qualified and narrated.	
DDT and Endrin Breakdown	≤ 15% for Endrin and DDT.	≤ 15% for Endrin and DDT.	≤ 15% for Endrin and DDT.	≤ 20% for Endrin and DDT.
MB	Detects of target analytes in MB acceptable if not detected in samples or if detects in samples are 10x higher than in MB. Apply B qualifier.	Detects of target analytes in MB acceptable if not detected in samples or if detects in samples are 10x higher than in MB. Apply B qualifier.	No target analytes detected >1/2 LOQ or >1/10 the amount measured in any sample or >1/10 the regulatory limit, whichever is greater. Common contaminants must not be detected > LOQ.	Detects of target analytes in MB acceptable if not detected in samples or if detects in samples are 10x higher than in MB. Apply B qualifier.
LCS	Evaluate with SPC limits. Re-extract samples if LCS fails (can fail high if there are no detects of those analytes in samples). Over-range analytes should be diluted to bring them within the calibration range. When samples are out of holding: consult with validator or GL for industrial clients (re-extraction may not be required); for others, re-extract if within 2x holding. Narrate and DER any failures.	Evaluate with SPC limits. Re-extract samples if LCS fails (can fail high if there are no detects of those analytes in samples). Over-range analytes should be diluted to bring them within the calibration range. When samples are out of holding: consult with validator or GL for industrial clients (re-extraction may not be required); for others, re-extract if within 2x holding. Narrate and DER any failures.	Evaluate with QSM 5.2 limits in Appendix C. If analyte is not listed in the table, use SPC limits. If LCS fails, re-extract samples (can fail high if there are no detects of these analytes). Spike with ALL target analytes. If there are failures and samples cannot be re-extracted, narrate and DER. Identify compounds and validator will apply Q flag.	Evaluate with SPC limits or 608.3 limits. Re-extract samples if LCS fails (can fail high if there are no detects of those analytes in samples). Over-range analytes should be diluted to bring them within the calibration range. When samples are out of holding: consult with validator or GL for industrial clients (re-extraction may not be required); for others, re-extract if within 2x holding. Narrate and DER any failures.
MS/MSD	Evaluate with SPC limits for recovery and RPD. Confirmed failures may be attributed to matrix interference.	Evaluate with SPC limits for recovery and RPD. Confirmed failures may be attributed to matrix interference.	Evaluate with QSM 5.2 limits in Appendix C for recovery and RPD (same limits used for LCS). Confirmed failures may be	Evaluate with SPC limits or 608.3 limits for recovery and RPD. Confirmed failures may be attributed to matrix

			attributed to matrix interference.	interference.
Surrogates	Evaluate with SPC limits. If surrogates fail low, re-extract the sample for confirmation. If there is obvious matrix interference, dilution and re-analysis may be appropriate and re-extraction may not be required. If surrogates fail high with no detects, data may be reported.	Evaluate with SPC limits. If surrogates fail low, re-extract the sample for confirmation. If there is obvious matrix interference, dilution and re-analysis may be appropriate and re-extraction may not be required. If surrogates fail high with no detects, data may be reported.	Evaluate with QSM 5.2 limits. If surrogates fail low, re-extract the sample for confirmation. If there is obvious matrix interference, re-extraction may not be required. Failures are Q qualified.	Evaluate with SPC limits. If surrogates fail low, re-extract the sample for confirmation. If there is obvious matrix interference, dilution and re-analysis may be appropriate and re-extraction may not be required. If surrogates fail high with no detects, data may be reported.

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STANDARD OPERATING PROCEDURE

FOR

DEFINITIVE LOW LEVEL PERCHLORATE ANALYSIS UTILIZING LIQUID CHROMATOGRAPHY/MASS SPECTROMETRY/MASS SPECTROMETRY (LC/MS/MS) BY EPA METHOD 6850 MODIFIED (6850M)

(GL-OA-E-067 REVISON 15)

APPLICABLE TO METHODS:
EPA SW-846 Methods 8000D and 6850 Modified

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1.0 STANDARD OPERATING PROCEDURE FOR DEFINITIVE LOW LEVEL PERCHLORATE ANALYSIS UTILIZING LIQUID CHROMATOGRAPHY/MASS SPECTROMETRY/MASS SPECTROMETRY (LC/MS/MS) BY EPA METHOD 6850 MODIFIED (6850M)

2.0 METHOD CODES

EPA SW-846 Methods 8000D and 6850 Modified.

3.0 METHOD OBJECTIVE AND PURPOSE

3.1 Sample extracts are analyzed using electrospray ionization liquid chromatography/ mass spectrometry/mass spectrometry (ESI/LC/MS/MS). The analyses are performed using positive or negative ion modes and incorporate internal or external quantitation. Chromatography is performed using normal phase, reverse phase, ion exclusion, or ion exchange columns. An ionization interface is chosen based on the functionality and performance of the analyte groups. Once ionized, the molecular ions are analyzed in the first quadrapole and then passed on to the second quadrapole, which is more often called a collision cell. Here the molecular ions are fragmented into daughter ions, which are then passed into the third quadrapole. Finally, the third quadrapole allows specific daughter ions to pass through to the detector.

3.2 This method has been modified from EPA Method 6850 Perchlorate in Water, Soils and Solid Wastes Using High Performance Liquid Chromatography/ Electrospray Ionization/Mass Spectrometry (HPLC/ESI/MS or HPLC/ESI/MS/MS).

NOTE: The following are modifications that GEL Laboratories, LLC has instituted to EPA Method 6850: 1) Sonication is not employed for the extraction of Perchlorate in solid matrices.

3.3 This method has been modified on the basis of GEL's Performance Based Measurement System (PBMS).

4.0 METHOD APPLICABILITY AND METHOD SUMMARY

4.1 This method covers the determination of perchlorate in water and solids using liquid chromatography mass spectrometry mass spectrometry. Water samples are analyzed for perchlorate by a direct inject method. Solid samples undergo a water leach preparation. Before analysis, all samples are filtered through desalting cartridges to eliminate interference from other ions. The sample is then analyzed using electrospray ionization liquid chromatography/mass spectrometry/mass spectrometry (ESI/LC/MS/MS) in the negative ion mode. An anion exchange column is used with an ammonium hydroxide in water mobile phase. The analytes are listed in Table A-3.

4.2 This method applies to the following matrices:

4.2.1 Groundwater

4.2.2 Wastewater

4.2.3 Soil

4.2.4 Sludge

4.2.5 Miscellaneous matrices

5.0 METHOD SCOPE AND PERFORMANCE CHARACTERISTICS

Refer to Appendix A for Determination of Perchlorate in Water and Solid Samples Using ESI/LC/MS/MS.

6.0 DEFINITIONS

6.1 Calibration Standard (CAL): A solution prepared from the primary dilution standard solution or stock standard solutions and the internal standards and surrogate analytes. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

6.2 Continuing Calibration Blank (CCB): An aliquot of reagent water or other blank matrix that is analyzed after each CCV. The CCB is used to determine whether the analytical sequence is in control during sample analysis.

6.3 Continuing Calibration Verification (CCV) Standard: An aliquot of reagent water or other blank matrix to which known quantities of the method analytes are added in the laboratory. The CCV is analyzed exactly like a sample, periodically throughout the run sequence. Its purpose is to determine whether the analytical sequence is in control during sample analysis. It may be prepared from the same source as the calibration standards, and is usually of varied concentration.

6.4 Detection Limit Verification Standard (CRI): An aliquot of reagent water or other blank matrix to which known quantities of the method analytes are added in the laboratory. The CRI is analyzed exactly like a sample, periodically throughout the run sequence. Its purpose is to determine whether the analytical sequence is in control at the low end of the calibration range during sample analysis. It may be prepared from the same source as the calibration standards. This standard is at the concentration of the MDL for the perchlorate analysis.

6.5 Independent Calibration Blank (ICB): An aliquot of reagent water or other blank matrix that is analyzed after each ICV. The ICB is used to determine whether there is carryover contamination after injection of the mid-level ICV.

6.6 Initial Calibration Verification (ICV): A solution of method analytes of known concentrations that is used to fortify an aliquot of Blank or sample matrix. The ICV is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check laboratory performance with externally prepared test materials.

6.7 Instrument Performance Check Solution (IPC): A solution of one or more method analytes, surrogates, internal standards, or other test substances used to evaluate the performance of the instrument system with respect to a defined set of criteria.

6.8 Interference Check Sample (ICS): An aliquot of a standard containing chloride, sulfate, bicarbonate, and carbonate each at 500 ppm that has been spiked with

perchlorate to yield a final concentration of perchlorate of 0.200 ppb. An ICS is extracted with each batch of 20 samples. This sample is used to ensure that in any sample containing high levels of bicarbonate, carbonate, sulfate, and/or chloride, the perchlorate concentration in the sample is not suppressed by these ions.

6.9 Linear Calibration Range (LCR): The concentration range over which the instrument response is linear.

6.10 Limit of Quantitation (LOQ): The lowest concentration level of the initial calibration curve used to quantitate an analyte. The LOQ verification is typically spiked at the PQL.

6.11 Lower Limit of Quantitation (LLOQ): The lowest concentration at which a target analyte can be reliably measured and reported. The LLOQ is ≥ the lowest point in the calibration curve and represents a concentration at which both quantitative and qualitative requirements can be consistently demonstrated. The LLOQ is verified at least annually, but typically quarterly, as the LOQ verification. The verifications performed by extracting and analyzing an LCS spiked at 0.5 to 2 times the LOQ. The LLOQ verification is carried through the same preparation and analytical procedures as environmental samples and QC. It is recommended to analyze the LLOQ on every instrument where data are reported and this is the laboratory's normal protocol. Recovery of target analytes in the LLOQ are compared to in-house-statistically-derived limits. Concentrations in samples reported below the LLOQ and above the MDL are qualified as estimated.

6.12 Method Detection Limit (MDL): The minimum concentration of an analyte that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero. Typically, the MDL is two to three times less than the PQL.

6.13 Practical Quantitation Limit (PQL): Typically the lowest level of the calibration curve. With the prep factor applied, the PQL is referred to as the effective PQL.

6.14 Reporting Limit (RL): The level at which a target analyte would meet the data quality objectives of the laboratory and/or a project, which may include establishing compliance with a regulatory and/or action limit. The reporting limit may be equal the laboratory's practical quantitation limit (PQL).

6.15 Refer to GL-QS-B-001 the Quality Assurance Plan for additional lab-wide used definitions.

7.0 INTERFERENCES TO THE METHOD

7.1 LC/MS/MS is a highly selective analysis that utilizes four means of compound discrimination: chromatographic separation, negative ion generations (where applicable), mass selection, and daughter fragmentation. It is theoretically possible that two different compounds could have the same retention time and generate the same ion. But, it is highly unlikely that these two compounds would fragment to the same daughter ion.

7.2 Method interferences may be caused by contaminants in solvent, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Raw LC/MS/MS data from all blanks, samples, and spikes must be evaluated for interferences. If an interference is detected, it is necessary to determine if the source of interference is in the preparation and/or cleanup of the samples and then take corrective action to eliminate the problem.

7.3 The use of high purity reagents, solvents, and gases help to minimize interference problems.

7.4 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe must be rinsed thoroughly between samples with the strongest organic solvent used in the method. The analyst must monitor Method Blanks to determine if the syringe rinsing is sufficient.

8.0 SAFETY PRECAUTIONS AND HAZARD WARNINGS

8.1 Treat all chemicals and samples as potential health hazards, and limit exposure to these chemicals to the lowest level possible. GEL maintains a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of chemicals. A reference file of Material Safety Data Sheets (MSDS) and individual client MSDS forms are also maintained.

8.2 Personal protective equipment

8.2.1 Gloves are required when handling the chemicals in this procedure. Latex gloves are approved for this procedure.

8.2.2 Laboratory coats should also be worn.

8.3 Prior to handling radioactive samples analysts must have had radiation safety training and understand their full responsibilities in radioactive sample handling.

8.4 All samples, chemicals, extracts, and extraction residues must be transferred, delivered, and disposed of safely according to all related SOPs.

8.4.1 Segregate solid wastes from liquid wastes in the satellite area containers.

8.4.2 Segregate oil wastes from water-soluble wastes in satellite area containers.

8.5 Never leave gas cylinders unchained or untied, even when they are on moving carts.

8.6 In the event of an accident or medical emergency, call for help immediately. When time and safety permit, an accident report form should be completed and turned in to the GEL Safety Officer.

8.7 Fire escape routes are posted in the lab, and all personnel should be familiar with them. In addition, fire safety equipment such as fire extinguishers is located

throughout the lab. Training is available on the proper operation of this equipment.

9.0 CAUTION WARNINGS

Acetonitrile and methanol are flammable solvents. Safety precautions for these flammable solvents must be taken into consideration when dealing with this method.

10.0 APPARATUS AND MATERIALS, REAGENTS, EQUIPMENT, AND INSTRUMENTS

10.1 Liquid Chromatography/Mass Spectrometry/Mass Spectrometry System

An analytical system complete with a ternary gradient, programmable high-pressure liquid chromatography, and a column heater capable of maintaining a temperature of 40° ± 1°C. The liquid chromatography unit must be capable of accurate and repeatable flow rates of 0.2 mL/min to 1.0 mL/min. The mass spectrometer system must possess an electrospray ionization (ESI) interface capable of operating at flow rates stated above. The mass spectrometer system must be capable of negative ion analysis and consist of three quadrapoles for daughter fragmentation.

10.2 Data System

A computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that can search any LC/MS/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as the Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specific time or scan-number limits.

10.3 Autosampler: An autosampler that is capable of injecting 10 to 250 µL with a precision of 5% RSD and that can accommodate at least 24 samples.

10.4 Analytical balance: Used to accurately weigh target analyte for stock standard preparation (± 0.1 mg sensitivity).

10.5 Top loading balance: Used to accurately weigh samples in the sample preparation (± 10 mg sensitivity).

10.6 Pipettes: Pasteur, plastic or glass, disposable, graduated, 5 mL and 10 mL.

10.7 Micropipettes: 100 µL, 1 mL, and 10 mL.

10.8 Vials: High-density polyethylene (HDPE) or glass, amber, 12 mL. For sampling and storage of calibration solutions.

10.9 Weigh boats: Plastic, disposable, for weighing eluent reagents.

10.10 Particulate filters: 0.45 micron syringe filters (Gelman IC Acrodisc, P/N 4485, or equivalent). These cartridges are used to remove particulates from the sample matrix.

10.11 Matrix pretreatment cartridges in the barium form (Dionex On-Guard-Ba cartridges, P/N 046072, or equivalent). These cartridges are conditioned according to the manufacturer's directions and are used to reduce the matrix levels of sulfate.

10.12 Matrix pretreatment cartridges in the hydrogen form (Dionex On-Guard-H cartridges P/N 039596, or equivalent). These cartridges are conditioned according to the manufacturer's directions and are used to reduce the matrix levels of carbonate.

10.13 Custom matrix pretreatment cartridge containing the equivalent of two Barium forms, one Silver form, and one Hydrogen form (Dionex On-Guard II Ba/Ag/H P/N 063955). Cartridge conditioned to the manufacturer's direction.

10.14 Centrifuge capable of holding at least six 25 mL VOA vials.

10.15 LC-MS/MS #2
Computer: IBM ThinkCentre
Software: MassLynx V4.0 SP4

10.16 LC-MS/MS #3
Computer: DELL Precision 360
Software: Analyst V1.4.2

10.17 LC-MS/MS #4
Computer: DELL Precision 370
Software: Analyst V1.4.2

11.0 REAGENT, CHEMICALS AND STANDARDS

11.1 Reagents

11.1.1 Reagent water: Distilled or Deionized (DI) water 17.8 Mohm or better, free of the anions of interest. Water should contain particles no larger than 0.20 microns.

11.1.2 Acetonitrile, reagent grade

11.1.3 Methanol, reagent grade

11.1.4 Ammonium hydroxide, reagent grade

11.2 Standards

11.2.1 Source Standards: Source standards are purchased directly from certified vendors and may be diluted to make stock, intermediate, and/or working standards. Source standards expire per the vendor expiration date, or after one year from the open date, whichever is shorter. Please refer to GL-LB-E-007 for further information regarding standards, their preparation, and control.

11.2.2 Calibration Standards: Working standards are prepared from certified source standard. Calibration standards are prepared at a minimum of five

concentration levels noted in Table A-1. Calibration standards expire after fourteen (14) days.

11.2.3 Second-Source Calibration Standard: A working standard prepared from source standards purchased from a different certified vendor than the calibration standards. Second-Source calibration standards expire after fourteen (14) days.

11.2.4 Laboratory Control Sample (LCS) and Matrix Spike (MS) Standards: The LCS and MS standards contain both Perchlorate and Perchlorate-101. LCS and MS standards may be purchased pre-mixed as source standards, or may be prepared in the laboratory from certified source standards. All LCS and MS standards expire per the source standard vendor expiration date, or after one year from the open date, whichever is shorter.

11.2.5 Retention Time Marker Solution: This standard solution is purchased as a certified source standard, and contains isotopically-labeled Perchlorate. It is added to all samples, QC, and working standards prior to analysis. This standard solution expires per the source standard vendor expiration date, or after one year from the open date, whichever is shorter.

11.2.6 Sodium/Rubidium/Iodide Tuning Solution: A mixture of sodium iodide (NaI) and rubidium iodide (RbI). The tuning solution contains approximately 2 µg/µL of NaI and 0.05 µg/µL of RbI in 1:1 2-Propanol/Water.

11.3 Chemicals

11.3.1 Nitrogen Gas, 99+%

11.3.2 Argon Gas, 99+%.

12.0 SAMPLE HANDLING AND PRESERVATION REQUIREMENTS

12.1 Water samples are collected in clean, 125 mL polyethylene bottles. Whenever possible, water samples should be sterilely filtered in the field at the time of collection using 0.2 µm PTFE membrane filtration in order to remove potentially perchlorate-disgrading microbes.

12.2 Solid samples are collected in clean, 4 oz. amber bottles. Extract solids within 28 days of collection.

12.3 Analyze water samples and the extracts of solid samples within 28 days of collection or preparation, respectively.

12.4 Samples do not need to be shipped cold or stored refrigerated, but every effort should be taken to avoid temperature extremes.

13.0 SAMPLE PREPARATION TECHNIQUES

13.1 Extraction of aqueous samples: Sample is examined for excessive particulate content. If the sample contains a high percentage of particulate, a particulate filter

(0.45 micron syringe filter) is added to the pretreatment process. Take a 10 mL aliquot, and filter 3 mL of sample through the cartridge(s) as an initial sample rinse (particulate filter-if needed, H, Ba, and Ba cartridges or custom cartridge) at a flow rate of 1.0 mL/min or less (approximately 1 drop every 3 to 4 seconds). This flow rate is critical to the pretreatment and must be carefully followed. Discard this fraction and begin collecting the pretreated sample aliquot of collected sample in a disposable 12 mL plastic vial. All data are recorded in the AlphaLIMS-generated preparation logbook.

13.2 Extraction of Solid Samples: Most extractions are performed on solid samples when a known weight of approximately 2.0 g of sample is diluted with DI water to a volume of 20 mL. Shake the extract for approximately 2 minutes. Centrifuge the extract for 30 minutes. Draw off 10 mL of sample and filter and pretreat as described in section 13.1. All data are recorded in the AlphaLIMS-generated preparation logbook.

NOTE: It is assumed that materials within the sample container are considered "the sample." Removal of any extraneous material (twigs, leaves, large rocks, etc.) must be documented in the case narrative and bench logbooks.

13.3 Extraction Quality Control samples: Each batch of samples (20 samples or less) is extracted with at least a Method Blank (MB), an ICS, and a LCS. If there is enough sample, a Matrix Spike (MS) and Matrix Spike Duplicate (MSD) are extracted as well. If there is not enough sample, a LCS duplicate is extracted. All QC samples are filtered and pretreated in exactly the same manner as the samples. HPLC-grade water is used for the MB and LCS, while the ICS is made from the perchlorate suppression solution standard. The ICS, LCS, MS, and MSD are spiked with perchlorate to yield a final concentration of 0.20 µg/L.

NOTE: If a response is observed above the MDL in the pretreated MB, and the samples contain perchlorate at a concentration above the MDL, the sample must be reextacted. If the sample contains perchlorate at a concentration less than the MDL, then the data can be reported without bias.

14.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

14.1 Maintenance for the HPLC

14.1.1 Daily Maintenance

14.1.1.1 Solvents: Adequate volume of solvent needs to be maintained on a daily basis. Failure to do so may result in loss of prime on the LC. This is especially important for the needle wash solvent. Make sure that there is plenty of solvent in each reservoir before using. When a long acquisition run is set up, it is essential to calculate the amount of solvent that will be needed. Add an extra 20% of solvent to the reservoir to make sure the LC does not go dry during the run.

14.1.1.2 Column: The column must be properly equilibrated with the method-specific solvent before batch analysis can occur. At the end of the day or an analysis, the column must be flushed with solvent, usually acetonitrile or methanol, to ensure longevity.

14.1.1.3 Sample Carousel: Make sure the sample carousel is calibrated. (The needle is drawing from the correct vial.)

14.1.1.4 Syringe: Check the operation of the sample syringe on a daily basis to ensure the syringe is drawing the proper amount of sample. Make sure the syringe is free of bubbles and that the needle is injecting properly.

14.1.1.5 Solvent Lines and Pump: Prime the system daily or as needed to ensure proper solvent flow to the column. This is best accomplished by using the system preparation feature from the LC control window. Make sure the seal wash line/syringe wash line is primed and their reservoirs are full.

14.1.2 Weekly Maintenance

14.1.2.1 Waste Container: Check the waste container and dispose if needed. The satellite waste container should never be more than ¾ full. Always label waste properly.

14.1.2.2 Solvent filters: Check the solvent filters for dirt. If filters are dirty then replace with new clean filters.

14.1.2.3 Column filters: May be replaced if an excessive back pressure is observed.

14.1.3 Semi-annual Maintenance

14.1.3.1 Plunger seals: Inspect the plunger and plunger seals. If the pump has been losing prime or if the ripple delta is greater than 30 psi, the seals will need to be replaced.

14.1.3.2 Check valves: Inspect the inlet check valve cartridges. If the pump has been losing prime or if the ripple delta is greater than 30 psi and changing the seals did not fix the problem, the check valves will need to be replaced.

14.1.3.3 Filter: Replace the inline filter. This may need to be replaced sooner if the system is exhibiting high back pressures.

14.1.3.4 Syringe and injector: Inspect the syringe and injector needle. Replace needle if it is bent or damaged. Replace syringe if there are obvious signs of wear. Refer to the Waters Operator's Guide for more details.

14.2 Maintenance for the Mass Spectrometer

14.2.1 Vacuum System Maintenance

14.2.1.1 Daily Maintenance

Vacuum Pressure: Check the analyzer pressure before the start of any analysis. If the reading is greater than 1 x 10^{-4} torr, check for vacuum leaks. The most likely place for the system to develop a leak is around the ion source. An o-ring may not be sealing properly. It may be necessary to disassemble the source to check the conditions of the o-rings. Also, check the oil levels of the rough pumps. Low oil levels in the pumps can cause loss of vacuum.

Rotary Pumps: The E2M28 pump, which backs the quadrupoles, should be ballast for 30 minutes at least once a week. Make sure oil levels in the pumps are sufficient on a daily basis. Check for any leaks.

14.2.1.2 As Needed Maintenance

Oil Odor Element: Change the odor element monthly or whenever the pump emits an oily odor

Mist Element: Change the mist element annually.

Foreline Trap: Change the sorbent (activated alumina) when it becomes discolored (brown). The discolor will reach the region of the trap furthest from the pump (the vacuum side), the sorbent should be replace when the instrument is vented and the pump is changed.

14.2.2 Mass Spectrometer Source Maintenance

14.2.2.1 As Needed Maintenance

14.2.2.1.1 Clean the sample cone, cone gas nozzle, and outer area of the source block with methanol. Before removing the sample cone be sure to close the isolation valve.

14.2.2.1.2 Clean the ion block when needed. A sign that the ion block needs cleaning is gradual loss of sensitivity. Also you may see splits peaks and/or a rough unstable signal.

14.2.2.1.3 The hexapole lenses may need cleaning periodically. It is good to clean these lenses annually but they may need more frequent cleaning depending of the type of samples run. Refer to the Micromass Manual for details on cleaning.

14.3 Electrospray Probe Maintenance

14.3.1 As Needed Maintenance

14.3.1.1 The electrospray probe needs to be properly maintained at all times to ensure maximum sensitivity. Problems that may inform an analyst of needed maintenance are: an unstable ion beam, excessive broadening of chromatographic peaks, and high LC pump backpressure. Refer to the manual for troubleshooting tips.

14.3.1.2 The probe tip may need to be cleaned if dirty matrices are run, especially at low heater temperatures.

14.4 Computer Maintenance

14.4.1 Daily Maintenance

Check Disk Free Space. Check the disk drive free space to determine if there is enough room for the data from the autorun.

14.4.2 Weekly Maintenance

Backup Data system. The data should be copied to a backup directory and stored on the AlphaLIMS system.

15.0 PREPARATION OF STANDARD SOLUTION AND QUALITY CONTROL SAMPLES

15.1 Source Standard Solutions

Source standard solutions are purchased as certified mixtures or as neat chemicals from vendors on the GEL approved vendors list. Documentation of the standard's quality and traceability should be provided by the vendor. This documentation is scanned and linked to the standard in LIMS. Please refer to GL-LB-E-007 for further information regarding standards, their preparation, and control.

16.0 INSTRUMENT CALIBRATION

16.1 A mass calibration is performed as needed using a sodium iodide/rubidium iodide solution. Refer to Table 1 for the calibration masses. The solution is introduced directly into the mass spectrometer using the ESI interference in positive mode. A mass range of 20 to 1100 amu, with at least six scans across each peak is utilized.

16.2 To verify the mass calibration, a mass calibration verification (tune) is performed daily by infusing a solution containing the analytes of interest. Four masses spanning the high, middle, and low mass range of the daughter ions are visually inspected. If the mass calibration has drifted, peaks in the observed mass range will not be present. The analyst must then perform a mass calibration as stated in section 16.1 above.

16.2.1 Upon performing a mass calibration, the analyst must recalibrate the instrument before proceeding with calibration verification standards and client samples.

16.2.2 Hard copy reports of daily tune data are stored in a logbook near each instrument in order that the analyst may evaluate daughter ion peaks for

broadening and abundance characteristics when compared to previous observations.

16.2.3 These observations may indicate the need for maintenance or cleaning. To ensure that the current calibration is valid, the analyst must analyze a calibration verification standard which meets the method acceptance criteria.

16.3 Initial Calibration Curve

16.3.1 For this analysis, an internal standard method is used.

16.3.2 Typical calibration levels are listed in Table A-1.

16.3.3 A minimum of 6 calibration standards must be used to establish linearity with the correlation coefficient (r) greater than or equal to 0.995 or with an RSD for each analyte (including MRL) of less than or equal to 20%. The calibration is linear and shall not be forced through the origin. The concentration corresponding to the absolute value of the calibration curve's Y intercept must be less than or equal to LOD.

16.4 Calibration Verification

16.4.1 Second-Source Initial Calibration Verification (ICV): A standard of different origin than the calibration standards is used to verify the curve. If a second source calibration is not available a second standard is made to verify the concentration of the stock standard. Refer to related appendices for ICV limits.

16.4.2 Continuing Calibration Verification (CCV): A standard of the same or different origin as the calibration standard is analyzed after every 10 injections. Refer to related appendices for CCV limits.

NOTE: If verification is not achieved the operating conditions of the instrument must be checked and the instrument must be recalibrated.

16.4.3 Calibration Blanks (ICB and CCB)

A solvent blank is analyzed after the ICV and after the CCV. The result must be less than one half the reporting limit concentration.

NOTE: If target analytes are above the RL, check for carryover from a possible high-level sample. Check the LC system for contamination and perform the necessary step to achieve a successful blank.

16.5 For perchlorate analysis, the recovery of perchlorate O-18 standard must be within 50-150% of the average response established in the initial calibration. If the response is outside this range, the samples must be reanalyzed. If the recovery is still outside the limits, a comment about sample matrix interference is included in the case narrative.

16.6 Detection Limit Verification (CRI)

A standard of the same origin as the calibration standard is analyzed after the calibration curve. This standard is at the concentration of the MDL for perchlorate analysis. Perchlorate recovery must be within 70-130%. All samples must be bracketed by passing CRI standards.

NOTE: If verification is not achieved, the operating conditions of the instrument must be checked and the instrument must be recalibrated.

17.0 QUALITY CONTROL

17.1 Preparation Batch: The preparation batch consists of a group of no more than 20 samples of the same matrix processed on the same day. All samples in a batch must be initiated on the same day. Each batch must contain a Method Blank (MB), a Laboratory Control Sample (LCS), an Interference Check Sample (ICS) and an MS/MSD or sample/sample duplicate pair. If insufficient sample is available to prepare an MS/MSD, then a duplicate LCS (LCSD) or sample duplicate must be substituted. Additionally, each sample (including the QC samples) will be spiked with the appropriate surrogate compound(s) when applicable. Refer to GEL Quality Control Policy for additional information regarding data quality assessments and batch processing requirements.

17.2 Method Blank (MB)

17.2.1 MB data are used to assess contamination from the laboratory environment and to characterize spectral background from the reagents used in sample processing.

17.2.2 When samples that are extracted together are analyzed on separate instruments or days, the method blank associated with those samples must be analyzed on at least one of those instruments and/or days. A solvent blank should be analyzed on all other instruments/days on which any of the samples is analyzed.

17.2.3 The MB must not exhibit values that exceed one half the reporting limit (RL) for any target analyte. MB values > ½ RL indicate that a laboratory or reagent contamination should be suspected. If a MB exhibits values greater than ½ RL, the following corrective actions are recommended:

17.2.3.1 The MB may be reanalyzed once to determine if the contamination is instrument related (carryover, etc.). If the reanalysis confirms an instrument-related problem and that the MB has not been contaminated, analysis may proceed.

17.2.3.2 MB values > ½ RL that are associated with samples that do not exhibit concentrations > ½ RL for the analyte observed in the MB may be reported.

17.2.3.3 Samples exhibiting positively detected analytes (i.e., greater than 1/2 RL) that were also detected in the associated MB may

be reported if the sample results are at least 10 times the concentration detected in the MB.

17.2.3.4 Samples exhibiting positively detected analytes (i.e., greater than ½ RL) that were also detected in the associated MB, but are less than 10 times the concentration detected in the MB must be re-extracted and re-analyzed.

NOTE: If there is insufficient sample volume remaining, or if the holding time has expired, the client must be consulted prior to beginning re-extraction. The deficiency must be documented.

17.3 Laboratory Control Sample (LCS)

17.3.1 The LCS consists of an aliquot of blank matrix (deionized water, Ottawa sand, sodium sulfate, etc.) that is spiked with the same analytes at the same concentration as the matrix spike sample. The LCS is used to assess the ability of the analytical system to produce acceptable results and undergoes all sample treatments that the associated samples undergo.

17.3.2 Calculate the percent recovery (%R) for each target analyte using the following equation:

$$\%R = \frac{\text{Found Value}}{\text{True Value}} \times 100$$

17.3.3 If the recovery of any analyte falls outside the required control limits, that analyte is judged out of control, and the source of the problem should be identified and resolved before continuing the analyses. The following corrective actions are recommended:

17.3.3.1 The LCS may be reanalyzed once to determine if the out-of-control result is instrument related. If upon reanalysis, the out-of-control analytes are in control, analysis and reporting of the associated samples may proceed.

17.3.3.2 If the %R for any analyte in the LCS is below the lower control limit, or if the %R is above the upper control and the analyte was detected in the associated samples above the RL, all associated samples must be re-extracted and re-analyzed for that analyte.

NOTE: If there is insufficient sample volume remaining, or if the holding time has expired for any sample associated with the LCS, the client must be consulted prior to beginning re-extraction. The deficiency must be fully documented and it must be noted in the final report.

17.3.3.3 If the %R for any analyte in the LCS is above the upper control limit, and the associated samples do not exhibit concentrations above the RL for that analyte, the sample results may be reported. This indicates a high bias may exist for the batch for

that analyte, but since the samples were free of detections, the potential high bias does not affect the sample result. The deficiency must be documented and noted in the final report.

NOTE: Certain client programs require re-extraction and re-analysis for all LCS failures. The Project Manager must be consulted to ensure that the client will accept data in this instance.

17.4 Interference Check Sample (ICS)

17.4.1 In the perchlorate analysis, an interference check sample is extracted with each batch. This sample is made with a standard containing bicarbonate, carbonate, sulfate and chloride each at a concentration of 500 ppm. This sample is spiked with perchlorate for a final concentration of 0.20 ppb and filtered in the same manner as all other samples in the batch. The acceptance limits for perchlorate recovery are 70 – 130%. This sample is used to ensure that in any sample containing high levels of bicarbonate, carbonate, sulfate, and/or chloride, the perchlorate concentration in the sample is not suppressed by these ions.

17.4.2 Calculate the percent recovery (%R) for each target analyte using the following equation:

$$\%R = \frac{\text{Found Value}}{\text{True Value}} \times 100$$

17.4.3 The ICS may be reanalyzed once to determine if the out-of-control result is instrument-related. If upon reanalysis, the out-of-control result is in control, analysis and reporting of the associated samples may proceed. If the result is still outside of the acceptance limits, all associated samples must be re-extracted.

17.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

17.5.1 A matrix spike is a sample that is spiked with the target analytes and is used to assess the effect of the matrix on the target analytes. If there is insufficient sample volume to prepare an MSD or a sample duplicate, an LCSD must be prepared and analyzed. The accuracy of the MS and MSD is reported as %R and is calculated as follows:

$$\%R = \frac{C_s - C}{S} * 100$$

Where

%R	=	percent recovery
C_s	=	spiked sample concentration
C	=	unspiked sample concentration

S = concentration of the spike added to the sample

17.5.2 The MSD, sample duplicate or LCSD is used to measure the precision of the analytical system. The precision is expressed as relative percent difference (RPD), and is calculated using the following equation:

$$RPD = \frac{|a-b|}{[(a+b)/2]} \times 100$$

Where:

RPD = Relative Percent Difference

A = Result of MS, sample or LCS

B = Result of MSD, sample duplicate or LCSD

17.5.3 The %R for the MS and MSD and the RPD for the MS/MSD should be within control limits. If they are not look for objective evidence of matrix interference such as sample heterogeneity, chromatographic behavior, required dilutions or behavior of the sample during extraction. MS, MSD or MS/MSD RPD failures and sample matrix observations must be documented.

17.5.4 Recovery limits for Quality Control Samples are presented in Table A-2.

17.6 Additional QC Measures.

17.6.1 Instrument Blanks: Solvent blanks (IB) may also be used to check for contamination by carryover from a high-concentration sample into subsequent samples. The IB shall not contain concentrations greater than ½ RL. Reanalyze IB samples until no carryover is observed and reanalyze all samples processed since the contaminated blank.

17.6.2 Carry-over concerns: The analyst should review the results for at least the next sample after a high-concentration sample. If the high-concentration analytes are not present in the subsequent samples, then the lack of carryover has been demonstrated. If there is evidence that carryover may have occurred, then the samples must be reanalyzed.

18.0 CALCULATIONS

18.1 Percent Relative Standard Deviation for Initial Calibration

$$\%RSD = \frac{SD}{RF} x100$$

RF = Mean of RFs from initial calibration for a compound.

SD = Standard deviation of RFs from initial calibration for a compound,

$$= \sqrt{\sum_{i=1}^{N} \frac{(RFi - \overline{RF})^2}{N-1}}$$

RFi = RF for each of the calibration levels

N = Number of RF values

18.2 Continuing calibration percent drift

$$\%Drift = \frac{C_{actual} - C_{found}}{C_{actual}} x100\%$$

C_{actual} = Known concentration in standard

C_{found} = Measured concentration using selected quantitation method

18.3 Concentration in the extract

The concentration of each identified analyte and surrogate in the extract is calculated by average response factor or a regression fit.

18.3.1 Linear Fit

$$C_{ex} = A + B\frac{(R_X C_{IS})}{R_{IS}} \qquad C_{ex} = \frac{(R_X - A)}{B}$$

C_{ex} = Concentration in extract

R_X = Response for Analyte

R_{IS} = Response for internal standard

C_{IS} = Concentration of internal standard

A = Intercept

B = Slope

18.3.2 Average response factor

$$C_{ex} = \frac{(A_X C_{IS})}{(A_{IS}\overline{RF})}$$

C_{ex} = Concentration in extract

C_{IS} = Concentration of internal standard

R_F = Average RF

A_X = Area of the extract

A_{IS} = Area of the internal standard

18.3.3 Method 8000D outlines two procedures that may be used to determine calibration function acceptability for linear and non-linear curves. The calibration data are refitted back to the calibration model. % Error and

Relative Standard Error (RSE) evaluate the difference between the measured amount and the true amount (or concentration). % Error is determined as follows:

$$\%Error = \frac{x_i - x'_i}{x_i} \times 100$$

Where:

x'_i = Measured amount of analyte at calibration level *i*, in mass or concentration units

x_i = True amount of analyte at calibration level *i*, in mass or concentration units.

18.3.4 Percent error between the calculated and expected amounts of an analyte should be ≤30% for all standards and ≤ 50% for the lowest calibration level.

18.3.5 Relative Standard Error is calculated as follows:

$$RSE = 100 \times \sqrt{\sum_{i=1}^{n} \left[\frac{x'_i - x_i}{x_i} \right]^2 \Big/ (n - p)}$$

Where:

x_i = True amount of analyte in calibration level i, in mass or concentration units.

x'_i = Measured amount of analyte in calibration level i, in mass or concentration units.

p = Number of terms in the fitting equation

(average = 1, linear 2, quadratic =3, cubic =4)

n = Number of calibration points

18.3.6 The RSE acceptance limit criterion is the same as the RSD Limit for CF or RF in the determinative method. If the RSD limit is not defined in the determinative method, the limit should be set at ≤20% for well performing compounds and ≤30% for poor responders.

18.4 Concentration Determination of an Aqueous Sample

$$\mu g / L = \frac{C_{ex} V_t}{V_o}$$

V_t = Volume of the total extract in µL taking into account dilutions

V_o = volume of water extracted in µL

18.5 Concentration Determination of a Solid Sample

$$\mu g / kg = \frac{C_{ex} V_t}{W_o}$$

V_t = Volume of the total extract in µL taking into account dilutions

W_o = Weight of the solid extracted in kg

18.6 LCS Percent Recovery

$$LCS\ \mathrm{Recovery} = \frac{SSR}{SA} X100$$

SSR = Spike Sample Result

SA = Spike added

18.7 MS/MSD Spike Recovery calculations

$$MatrixSpike\ \mathrm{Recovery} = \frac{SSR - SR}{SA} X100$$

SSR = Spike sample Result

SA = Spike added

SR = Sample Result

18.8 Relative Percent Difference calculation for the MS/MSD or sample/sample duplicate.

$$RPD = \frac{MS_R - MSD_R}{\frac{1}{2}(MS_R + MSD_R)} X100$$

RPD = Relative Percent Difference

MS_R = Matrix Spike Result or Sample Spike Result

MSD_R = Matrix Spike Duplicate Result or Sample Spike Duplicate Result.

18.9 ICS Percent Recovery

$$ICS\ \mathrm{Recovery} = \frac{SSR}{SA} X100$$

SSR = Spike Sample Result

SA = Spike added

19.0 INSTRUMENT PERFORMANCE REQUIREMENTS

19.1 Refer to Appendix A for instrument set up criteria for compound specific parameters.

19.2 Each instrument must have a valid initial calibration curve for each analyte of interest. Hard copies of the continuing calibration standards are kept with the quality control samples and client samples analyzed in the sequence.

19.2.1 Continuing calibration verification standards must meet the criteria in section 16. When a calibration verification standard fails to meet the criteria, all samples that were injected after the last standard that met the criteria must be evaluated to prevent misquantitations and possible false negative results. Sample reanalysis of sample extracts may be required. More frequent analysis of standards will minimize the number of sample extracts that will have to be reanalyzed if the standard verification criteria are not met.

19.2.2 If the standard analyzed after a group of samples exhibits a response for an analyte that is above the acceptance criteria and the analyte was not detected in a specific sample analyzed during the analytical shift, then the sample extracts do not require reanalysis. The verification standard has demonstrated that the analyte would have been detected were it present due to the elevated response. In contrast, if the analyte with the high response in the verification standard is also detected in samples, then reanalysis of the sample is required since the sample results may be biased high and quantitation unreliable. Reanalyze the sample within acceptable bracketing verification standards.

19.3 Sample injections may continue for as long as the calibration verification standards and standards interspersed with the samples meet the quality control criteria in section 16. The sequence ends when the set of samples has been injected or when standards exceed the acceptance criteria.

19.3.1 If during the analysis of a sample group from a single client, the bracketing continuing verification standards do not meet the acceptance criteria, reanalysis of this group of samples is required. The reanalysis is required to confirm that the sample extracts (matrices) are causing the instrument response to fluctuate and standards to fail. The reanalysis must begin with an acceptable initial continuing calibration set of standards. If continuing verification standards fail during the analysis sequence, the failure is attributed to matrix. If the standards meet the acceptance criteria, then matrix interference is not confirmed and the acceptable standards and samples in between should be reported.

19.3.2 In addition, if multiple client sample extracts are analyzed in the analytical sequence where continuing verification standard failures occur, each client's samples must be reanalyzed after an acceptable initial calibration, initial continuing calibration or continuing verification standard.

20.0 ANALYST AND METHOD VERIFICATION REQUIREMENTS

20.1 To establish that the analyst can perform the procedures in an acceptable manner and that the method generates data of acceptable bias and precision, the following operations are performed:

20.1.1 A Quality Control (QC) check standard must be prepared containing each analyte of interest. It must be prepared from pure standard material or purchased as a certified solution. It must be made from a source independent of that used for calibration.

20.1.2 Four samples must be prepared and analyzed by the same procedures used to prepare and analyze actual samples.

20.1.3 Calculate the average recovery (X) in µg/L, and the standard deviation of the recovery (S) in µg/L, for each analyte of interest using the four results.

20.1.4 For each analyte compare S and X with the corresponding acceptance criteria for precision and accuracy, respectively, given in the quality control table at the end of the method. If the S and X for all analytes of interest meet the acceptance criteria, the system's performance is acceptable and analysis of actual samples can begin. If any individual S and X exceeds the precision limits or falls out of the range for accuracy, the system's performance is unacceptable for that analyte, and a check standard for that analyte must be prepared and reanalyzed.

20.2 Method detection limits are determined and documented annually. Precision and accuracy are matrix dependent and are documented by means of a laboratory control sample (LCS) and a matrix spike and matrix spike duplicate. Refer to GL-LB-E-001 for The Determination of Method Detection Limits.

20.3 Analysts are given continual performance evaluation samples as an ongoing assessment of their ability to perform this procedure.

21.0 ANALYTICAL PROCEDURES AND INSTRUMENT OPERATION

Standards, samples, blanks, and quality control sample extracts are introduced into the instrument via direct injection (autosampler). Samples containing target analyte concentrations that exceed the linear range of the analyte calibration curve must be diluted. The dilution level should be performed to place the highest analyte concentration between the middle and high points of the calibration curve (on column). If a sample is initially diluted and target analytes are not detected and non-target analytes are not interfering with the analysis, the sample must be reanalyzed at a lower dilution. Analysts should be aware that diluting samples will increase the detection limits for undetected analytes. Random dilutions without due cause are not acceptable. Samples should undergo appropriate clean up methods prior to diluting for observed matrix problems.

22.0 CALCULATIONS AND DATA REDUCTION METHODS

22.1 The concentration of each analyte in an extract can be determined by comparing the response obtained from analyzing the extract to the calibration curve. The concentration of a specific analyte is matrix specific and is calculated as follows:

Aqueous Sample

$$\text{Concentration } (\mu g/L) = \frac{(C)(D)(V_t)}{1000V_i}$$

Where:

C = Concentration (µg/L) calculated by data system from total area substituted into the linear equation derived from the multilevel calibration

D = Dilution factor, if made prior to analysis. If not, D = 1.

V_t = Total volume of the extract in mL

V_i =Initial volume of sample in L

Nonaqueous Samples

$$\text{Concentration } (\mu g/kg) = \frac{(C)(D)(V_t)}{1000\ W}$$

Where:

C = Concentration (µg/mL) calculated by data system from total area substituted into the linear equation derived from the multilevel calibration

D = Dilution factor, if made prior to analysis. If not, D = 1.

V_t = Total volume of the extract

W = Weight of the sample extracted in kg

22.2 The isotope ratio for perchlorate can be calculated as follows:

$$\frac{\text{Peak area of Perchlorate } (99 > 83)}{\text{Peak area of Perchlorate } (101 > 85)}$$

The ratio of ^{35}Cl area counts to ^{37}Cl area counts is used as a tool to confirm correct analyte identification. The theoretical ratio is 3.06 and sampled ratios must fall within 2.31-3.85.

Samples having isotope ratios outside the control limits are reanalyzed. If the reanalysis result falls outside the control limits, the result with the best isotope ratio is reported, the variance is noted in the case narrative, and an "X" qualifier is added to the Form I for that sample. For DoD QSM clients, the "Q" qualifier will be used and explained in the case narrative.

23.0 DATA RECORDING

Data are recorded and calculated by the data acquisition system. They are stored on a remote server. The data are also entered into the Laboratory Information Management System (AlphaLIMS).

24.0 QUALITY CONTROL REQUIREMENTS

24.1 Before sample analysis begins, the instrument must have acceptable calibration curves. Each curve is verified by the analysis of second source continuing calibration standards. Each standard must meet the acceptance criteria for percent difference or drift. Continuing calibration verification standards must be analyzed at least after every twenty samples. It is recommended to analyze these verifications after every ten samples.

24.2 A Method Blank (MB) is used to determine background concentrations of analytes of interest that have the potential to interfere with sample analysis. These blanks are analyzed with every analytical batch that has a maximum number of twenty samples. The criterion for acceptance is that there are no target analytes of interest present above the practical quantitation limit.

24.3 If the analyte of interest is present at a concentration between the MDL and PQL, all data are qualified with a "J" flag and reported. If the analyte of interest is present at a concentration above the PQL and the samples contain the analyte of interest at a concentration of greater than 10 times the concentration found in the blank, the data are qualified with a "B" flag and reported. If the concentration found in the sample is less than 10 times that found in the blank and greater than the PQL, the samples must be re-extracted. If the samples do not contain the analytes of interest, the data are reported.

24.4 The laboratory evaluates the effect of matrix on method performance by performing a matrix spike (MS) and matrix spike duplicate (MSD) for each matrix up to 20 samples in the same batch. Matrix spike (MS) and LCS limits are static. In addition, a laboratory control sample (LCS) and its duplicate (DUP) (when requested) are analyzed with each sample batch (up to 20 samples of the same matrix). The LCS consists of an aliquot of clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes at the same concentration as the matrix spike. When the results of the MS analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix. These limits are presented in Table A-2.

24.5 The Retention Time (RT) window for each analyte and surrogate must be established using the mid-point standard of each initial calibration and with the CCV at the beginning of the analytical shift. During the analysis of perchlorate by LC/MS/MS, retention time shifts are commonly observed. These retention time shifts, which are caused by fouling of the column by the sample matrices, are problematic when the retention time is used as one of the criteria of confirmation.

To overcome this problem, a known amount of ^{18}O labeled perchlorate is added to each sample as a retention time standard. The presence of perchlorate is confirmed by the relative retention time (RRT) of the perchlorate peak and the ^{18}O standard. A RRT window of 0.98 to 1.02, as required by Method 332.0, is used. In addition to the isotopic ratio, the presence of perchlorate in the samples associated is confirmed using the relative retention criteria stated above, not the absolute retention time.

24.6 An interference threshold study must be performed at initial setup and when major changes (i.e. addition of cleanup procedures, column changes, and mobile phase changes) occur in the method operating procedures. Measure the threshold of common suppressors (chloride, sulfate, carbonate, bicarbonate) that can be present in the system without affecting the quantitation of perchlorate. The threshold is the concentration of the common suppressors where perchlorate recovery falls outside an 85 -115% recovery.

24.6.1 When running a calibration curve for more than one analyte at a time, some of the analytes may not meet the acceptance criteria. Additional standards containing the compounds that were not acceptable may be analyzed. If the curve still does not meet the acceptance criteria, maintenance should be performed or a new standard may be needed. Refer to section 16 for other options addressing the initial calibration.

24.6.2 If the continuing check standard fails any criteria, the analyst must take action to correct the situation. This may be performing any of the maintenance steps described in section 14 to get the instrument to meet its daily calibration. If all attempts fail, the analyst must analyze a new series of calibration standards, thus obtaining a new calibration curve.

24.6.3 If the standard analyzed after a group of samples exhibits a response for an analyte that is above the acceptance limit, and the analyte was not detected in the specific samples analyzed during the analytical shift, then the extracts for those samples do not need to be reanalyzed. If an analyte above the QC limits was detected in a sample extract, reinjection of the extract is necessary.

24.7 If the percent recovery in the matrix or MSD falls outside the established SPC limits for recovery, the analyst should evaluate the LCS recoveries and MB analyses. If the LCS and MB analyses do not indicate a problem with the preparation procedures, the MS recoveries may be attributed to matrix effect. Analysts are not required to reanalyze the MS for failing recoveries; however they should seek additional technical support before deciding not to reanalyze MS samples.

25.0 DATA REVIEW, VALIDATION AND APPROVAL PROCEDURES

25.1 Upon completion of a batch, the analyst enters the data. A data report is generated and it is placed in a folder along with the batch and all other raw data (chromatograms). This folder is given to the peer review analyst for reviewing.

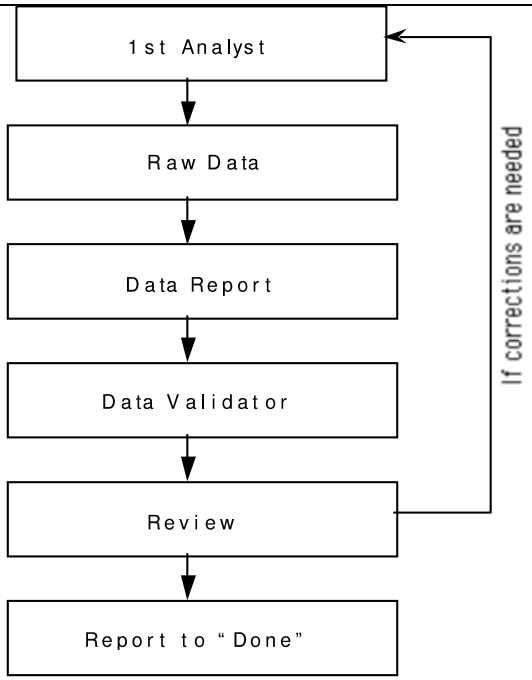
25.2 Levels of review and their responsibilities

25.2.1 First level review - The analyst must check all chromatograms and calculate the percent recoveries for the spikes, and any duplicates. The analyst must verify that the check standard passes for the analytes of interest. If a target compound confirms on the confirmation column, it must be checked to see if the retention time of the analyte is within the daily retention time window. The peak's shape must also be checked to ensure that it is indeed a peak and not noise. If the hit meets both of these requirements, its concentration should be reported. The analyst should also make sure that a review checklist is present.

25.2.2 Second level review - The data validator or other qualified reviewer must ensure that the concentration that appears in the external standards table is indeed what has been entered into the computer. The validator must verify that the calibration check standard is acceptable and if the LCS and its duplicate and the surrogates are all within acceptable ranges. The reviewer must also check the date analyzed, dilution factor, and time of analysis from the raw data against the data report. If everything is acceptable, the reviewer must then initial and date the Target report. The run log must also be dated and initialed. The data report is then issued a status of "Done."

25.2.3 To complete a review process, all chromatograms of the calibration check standard, MB, LCS and its DUP, samples and the spike and/or duplicates of the sample must be present. The batch sheet, sample tracker log, and the data report should all be present

25.3 A flow chart of the review process follows:



26.0 DATA TRANSMITTAL

After the review process is complete, Data Management receives the data.

27.0 RECORDS MANAGEMENT AND DOCUMENT CONTROL

All data associated with the activity described in this procedure, including relevant logbooks, are maintained as quality records in accordance with Quality Records Management and Disposition, GL-QS-E-008.

28.0 LABORATORY WASTE HANDLING AND DISPOSAL: SAMPLES, EXTRACTS, DIGESTATES, AND REAGENTS

For the proper disposal of sample and reagent wastes from this procedure, refer to the Laboratory Waste Management Plan, GL-LB-G-001.

29.0 REFERENCES

29.1 Test Methods for Evaluating Solid Waste: Laboratory Manual Physical/Chemical Methods, Volume 1B, SW846 Update V, Revision 4, Method 8000D, July 2014.

29.2 EPA Method 6850, "Perchlorate in Water, Soils And Solid Wastes Using High Performance Liquid Chromatography/Electrospray Ionization/Mass Spectrometry (HPLC/ESI/MS or HPLC/ESI/MS/MS)," Rev. 0, January 2007.

29.3 Method 332.0, "Determination of Perchlorate in Drinking Water By Ion Chromatograghy With Suppressed Conductivity And Electrospray Ionization Mass Spectrometry," Rev. 1, March 2005.

29.4 Department of Defense, Perchlorate Handbook, Rev. 1, Change 1, August 2007.

30.0 HISTORY

Revision 15: Removed step 11.2.5 and associated NOTE.

Revision 14: Added definitions for LOD and LOQ. Updated the number of calibrations needed.

Revision 13: Update Applicable Methods from 8000B to and 8000D. Updated Reference Section.

Revision 12: Added NOTE to Section 3.2 to clarify GEL modifications to Method 6850.

Revision 11: Removed reference to retired procedure (GL-OA-E-002) and updated standards section to comply with DOECAP audit finding. Calibration standards prepared at a minimum of five concentration levels.

TABLE 1

Calibration Masses for NaI/RbI solution

Amu
22.9898
84.9118
172.8840
322.7782
472.6725
622.5667
772.4610
922.3552
1072.2494

Based on Rb = 85.467, I = 126.9045, Na=22.9897697

APPENDIX A

Determination of Perchlorate in Water and Solid Samples Using ESI/LC/MS/MS

A.1 Analytical Conditions

A.1.1 Chromatography column: A 2 x 50 mm IonPac AG16 Column. Alternate columns are acceptable if they provide acceptable performance.

A.1.2 Typical mass spectrometric conditions are listed in Table A-4.

A.1.3 Typical HPLC conditions are listed in Table A-5.

A.2 Reagents and Standards

A.2.1 Stock Standard: Suggested Concentration = 10 µg/L. A stock standard solution may be purchased as a certified solution or prepared from ACS reagent grade, sodium salt as listed below.

NOTE: Sodium Perchlorate ($NaClO_4$, CAS [7601-89-0]) represents a molar weight fraction of 81.2% perchlorate anion.

A.2.2 Calibration Standards: Six calibration levels are prepared from the 10 µg/L intermediate standard with the lowest level at 0.050 µg/L. Refer to Table A-1. Standards are prepared in reagent water. Additional calibration levels can be prepared at the analyst's discretion.

A.2.3 Second-Source Calibration Verification Standard Solution: A 10 µg/L intermediate solution is prepared from stocks, which is then used to prepare a 0.50 µg/L working standard used to verify the initial calibration.

A.2.4 Spiking Solution: A 10 µg/L solution is made using reagent water. Spikes are prepared at a 0.20 µg/L concentration.

A.2.5 Internal Standard and Retention Time Marker contains perchlorate O-18. This solution should be prepared at a concentration of 25 µg/L. The final extract concentration is 0.5 µg/L. Prior to analysis, a 1 mL aliquot of either a standard, or sample extract is transferred to an analytical vial, and 20 µL of perchlorate O-18 is added. The vials are then mixed.

TABLE A-1

Calibration Levels, ng/mL

Analyte	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
Perchlorate	0.05	0.10	0.25	0.50	1.0	2.0

Calibration solutions are prepared in DI water.

TABLE A-2

Quality Control Sample Recovery Limits

Compound	Recovery	DOD QSM Recovery
Perchlorate-0(18)(RT Marker)	50 – 150 %	50 – 150 %
ICV	85 – 115 %	85 – 115 %
CCV	85 – 115 %	85 – 115 %
LCS (Water)	85 – 115 %	80 – 120 %
LCS (Solid)	70 – 130 %	80 – 120 %
MS/MSD (Water & Solid)	75 – 125 %	80 – 120 %
ICS	70 – 130 %	70 – 130 %
CRI	70 –130 %	70 –130 %
LCS/LCSD and MS/MSD	RPD ≤ 30 %	RPD ≤ 15 %
Blanks	≤ PQL or RL	< ½ (RL)
Isotope Ratio $^{35}Cl/^{37}Cl$	2.31 – 3.85	2.3 – 3.8
Sample/Sample Duplicate	RPD ≤ 20 %	RPD ≤ 20 %

TABLE A-3

Analytes, Characteristic ions and Transitions

Compound	CAS Number	Parent Ion m/z	Daughter Ion m/z
Perchlorate	14797-73-0	99	83
Perchlorate-101	85213-55-4	101	85
Perchlorate-0(18)	N/A	107	89

TABLE A-4

Typical Mass Spectrometer Operating Conditions

Parameter	Setting
Scan Mode	Centroid, Negative Ion
Interface	Electrospray Ionization (ESI)
Capillary Voltage	0.2 kVolts
Multiplier Voltage	750 Volts (This can be adjusted)
Source Temperature	100° C
Desolvation Temperature	500° C
Desolvation Gas Flow	500 L/hr
Cone Gas Flow	0 L/hr
Cone Voltage	45 V
Collision Voltage	Variable
Collision Gas	Argon
Extractor	2 V
RF Lens	Variable

TABLE A-5

Typical HPLC Operating Conditions

Parameter	Setting
Column	250 x 2 mm IonPac AG16 or equivalent
Column Temperature	30° C
Sample Temperature	25° C
Eluent Flow Rate	0.50 mL/min
Eluent Identification	A = 50 mm Ammonium Hydroxide in Water B = Water
Solvent Program	5% A/95%B Isocratic
Injection Volume	50 µL
Run Time	6 Minutes

VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE FOR SOLID PHASE – EXTRACTION

(GL-OA-E-070 REVISION 11)

APPLICABLE TO METHODS:
EPA SW-846 3535A, 3535

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1.0 STANDARD OPERATING PROCEDURE FOR SOLID PHASE-EXTRACTION

2.0 METHOD CODE

EPA SW-846 3535A and 3535.

3.0 PURPOSE

To describe the manner in which aqueous matrices that include groundwaters, wastewaters and Toxicity Characteristic Leaching Procedure (TCLP method 1311) are extracted using Solid Phase Extraction (SPE) media for organic analysis methods, 8081, 8081A, 8081B, 8082A, 8015A, 8015B, 8015C and 8015D and 608. Refer to SOP GL-OA-E-033, appendix 2 for SPE preparation for method 8330A. This method may not be appropriate for aqueous samples with high levels of suspended solids greater than 1%.

4.0 INTERFERENCES

4.1 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks. Specific selection of reagents and purification of solvents by distillation in all glass systems may be necessary.

4.2 The decomposition of some analytes has been demonstrated under basic extraction condition. Organochlorine pesticides may dichlorinate and phthalate esters may hydrolyze. The rate of these reactions increases with increasing pH.

4.3 Phthalate esters contaminate many types of products commonly found in the laboratory. Plastics, in particular, must be avoided because phthalates are commonly used as plasticizers and are easily extracted from materials. Serious phthalate contamination may result at any time if consistent quality control is not practiced.

4.4 Soap residue (e.g., sodium dodecyl sulfate), which results in basic pH on glassware surfaces, may cause degradation of certain analytes. Specifically, aldrin, heptachlor, and most organophosphorous pesticides will degrade in this situation. This problem is especially pronounced with glassware that may be difficult to rinse (e.g., 500 mL Kuderna-Danish flask). These items should be hand rinsed very carefully to avoid this problem.

4.5 Bonded – phase silica (e.g., C18) will hydrolyze on prolonged exposure to aqueous samples with pH levels of less than 2 or greater than 9. Hydrolysis will increase at the extremes of the pH ranger and with longer contact time. Hydrolysis may reduce extraction efficiency or cause baseline irregularities. Styrene divinylbenzene (SDB) extraction disks should be considered when hydrolysis is a problem.

4.6 Sample particulates may clog the solid – phase media and result in extremely slow sample extractions. Aqueous samples with high levels of suspended solids greater

than 1 % as the extraction efficiency may not be sufficient. However, a 5% solution was filtered using this procedure and no problems were encountered.

5.0 METHOD DEVIATION

5.1 Sample pH adjustment to ≤ 2 with 15 mL of 1:1 H_2SO_4.

5.2 No specific wait time for sample/QC after the addition of surrogate/spike but samples are at a stand while analyst is cleaning glassware and conditioning cartridges for the process.

6.0 DEFINITIONS

6.1 Laboratory Duplicate (DUP, LCSD, or MSD): Aliquots of a sample taken from the same container and processed in the same manner under identical laboratory conditions. The aliquot is analyzed independently from the parent sample and the results are compared to measure precision and accuracy.

6.2 Lower Limit of Quantitation (LLOQ): The lowest concentration at which a target analyte can be reliably measured and reported. The LLOQ is the lowest point in the calibration curve and represents a concentration at which both quantitative and qualitative requirements can be consistently demonstrated. The LLOQ is verified quarterly, as the LOQ verification. The verification is performed by extracting and analyzing an LCS spiked at the lowest level of initial calibration curve (see appropriate analytical SOP for calibration concentration). The LLOQ verification is carried through the same preparation and analytical procedures as environmental samples and QC. The LLOQ is analyzed on every instrument where data are reported and this is the laboratory's normal protocol. Recovery of target analytes in the LLOQ are compared to in-house-statistically-derived limits. Concentrations in samples reported below the LLOQ and above the MDL are qualified as estimated.

6.3 Surrogates: Compounds added to every blank, LCS, LCSD, sample and MS and MSD that are used to assess analytical efficiency by measuring recovery. Surrogates are compounds that are not expected to be present in environmental media.

6.4 Refer to GL-QS-B-001 the Quality Assurance Plan for additional lab-wide used definitions.

7.0 APPARATUS AND MATERIALS

7.1 Manifold system

7.2 Collection tube (40 mL vial) - This tube should have an appropriate length so that the drip tip of the standard apparatus can be positioned well into the neck of the tube to prevent splattering.

7.3 Muffle furnace, capable of maintaining 400° C

7.4 Bottle adapter

7.5 Enviro-clean Universal C18 cartridge (#ECUNIC18) and (#ECUNIPAH)

7.6 Cartridge holder
7.7 Kuderna – Danish (KD) apparatus
7.8 Three – Ball macro – synder column
7.9 Two – Ball micro synder column (optional)
7.10 Boiling chips (solvent extracted approximately 10/40 mesh silicon carbide or equivalent.
7.11 Water bath (heated, capable of temperature control to within +/- 5°C. The bath should be in a hood.
7.12 Nitrogen Evaporator apparatus (N – EVAP) Organaomation model 112 or equivalent.
7.13 Glass vials (size 2mL or 10 mL) equipped with polytetrafluoroethylene (PTFE) lined caps for storage.
7.14 Vacuum system
7.15 Graduated cylinders
7.16 Concentrators tubes (10 mL)
7.17 Disposable pipets (1mL)
7.18 Disposable cartridge filter
7.19 Wide range pH indicator paper
7.20 Turbovap II Biotage
7.21 Zymark or Turbovap tubes
7.22 Erlenmeyer flask
7.23 Glass Rods

8.0 REAGENTS AND STANDARDS

8.1 Reagents: Reagents must meet ACS analytical criteria. Reagents will not be stored in plastic containers.

8.1.1 Eluting solvents will be screened prior to use.
8.1.2 Dichloromethane (CH_2CL_2), pesticide quality or equivalent.
8.1.3 Acetone (CH_3COCH_3), pesticide quality or equivalent
8.1.4 Organic – Free reagent water
8.1.5 Sodium Sulfate (granular, anhydrous) NA_2SO4 purify by heating at 400 °C for 4 hours in a shallow tray.
8.1.6 Sulfuric Acid solution (1:1 v/v) - Slowly add 50 mL of concentrated H2SO4 to 50 mL of organic-free reagent water.
8.1.7 Sodium hydroxide solution (10N), NaOH - Dissolve 40 g of NaOH inorganic-free reagent water and dilute to 100 mL.
8.1.8 Methanol (CH_3OH)

8.1.9 Hexane (C_6H_{14})

8.1.10 LLOQ standard solutions are purchased directly from a certified vendor or made by diluting the working standards.

9.0 EQUIPMENT MAINTENANCE

To assure that the manifold is not getting clogged or affected by the acid after samples have completed the extraction process 25 mL of deionized water is flushed through the manifold. Maintenance to these devices are recorded in LIMS in an electronic logbook.

10.0 SAMPLE HANDLING AND PRESERVATION

10.1 Sample containers are glass or Teflon with Teflon-lined screw cap.

10.2 Protect sample containers from light. Some analytes are light sensitive.

10.3 Sample will be maintained at 0° ≤ 6° C until extraction begins.

10.4 Samples for semivolatile organic analysis have a 7 day holding time from the collection date. PCB samples must be extracted within 1 year from collection.

10.5 If samples are not in appropriate containers or holding time has expired, initiate a Data Exception Report (DER). For instructions, refer to GL-QS-E-004.

11.0 SAMPLE PREPARATION

11.1 All batches (up to 20 samples) will be extracted with a blank, LCS, MS, and MSD. If insufficient sample is provided, the MS/MSD will be submitted with a LCS/LCS DUP.

NOTE: It is inappropriate to extract only a portion of a sample from a sample container when using SPE. If < 3 L is received for the QC sample, an LCSD Duplicate will be extracted with the batch also. A matrix spike only will be included if volume is available. The client will also be notified. If this approach is not acceptable to the client, per section 11.1 of the method, an aliquot may be transferred to a graduated cylinder and spiked. However, in such instances, the analyst must take great care to mix the sample well, by shaking, to ensure a homogeneous distribution of the particulate matter and must record the fact that the container was not rinsed.

11.2 Cartridge set-up and conditioning

NOTE: The sample will be inspected visually prior to extraction. If the sample contains > 1 inches of sediment in the bottom, this procedure should not be used. Approximately 1 inch of glass wool is added to each filter to aid in filtration process. If samples contain >5% TSS (>1 inches of sediment in bottom of bottle), the liquid fraction will be decanted and prepped by SPE and the solids will be filtered off and prepped by soxtherm and then physically combined into one extract for analysis.

11.2.1 Arrange the cartridges on the manifold in the closed valve position.

11.2.2 Turn on the vacuum pump and set the vacuum to 10 inches (254 mm) of Hg. Do not exceed the manufacturer's recommendation for manifold

vacuum. Flow rates may be controlled by opening and closing cartridge valves.

11.2.3 Condition the cartridges by adding 10 mL of Dichloromethane to each cartridge and allow soaking on the cartridge for approximately 1.5 min.

11.2.4 Open the valve and pull the Dichloromethane through the cartridge to waste leaving a layer just covering the frit with the valve now close.

NOTE: Cartridge conditioning for Diesel Range Organics (DRO) does not include the use of solvent acetone. Proceed to step 11.2.6.

11.2.5 Add 10 mL of Acetone to the cartridge and let the acetone soak for approximately 1.5 minutes. Open the valve and pull the acetone to the waste and air dry the cartridge with full vacuum for a few seconds and close valve.

11.2.6 Add approximately 10 mL of Methanol to the cartridge and allow soaking for approximately 1.5 minutes. From this point until sample addition the cartridge must not go dry. Open valve and pull some of the methanol through the cartridge leaving a layer just covering the frit and close valve.

11.2.7 Add approximately 20 mL of deionized water to the cartridge and open the valve to pull most of the water through the cartridge to waste but do not allow the sorbent to dry.

12.0 EXTRACTION PROCEDURE

12.1 Sample Preparation

12.1.1 Equilibrate the batch of samples to room temperature.

12.1.2 Sample volume of 1000 mL is used for the extraction or the entire bottle. Mark the level of the sample on the outside of the sample container for later determination of the sample volume used. Check the initial pH of each sample and document. Sample final volume can be determined by using the computer calculation of weight to volume. This information is recorded and generated in AlphaLIMS.

12.1.2.1 If sample bottle(s) is full to the rim, transfer sample to precleaned graduated cylinder.

NOTE: All pH measurements require the use of a disposable pipet to deliver small aliquot of sample to a wide-range pH indicator strip.

12.1.3 Transfer any surrogate and matrix spiking compounds (if applicable) to the samples in the original container or graduated cylinder. A peer must witness and document that the spiking process was correctly performed and the correct spike solutions added.

12.1.4 The pH of the sample needs to be adjusted to a designated value of less the 2 with 15 mL of 1:1 sulfuric acid solution. This pH adjustment should be performed after the addition of the surrogates and spikes. The samples that

are in the graduated cylinder are transferred back to its original container, then recapped and shaken thoroughly for a good mixture.

NOTE: Since C18 cartridges are being used for this method, pH must be < 2; Decachlorobiphenyl surrogate has a partial charge which prevents proper bonding to a C18 functional group. Protonating the DCB has been shown to neutralize the charge allowing for a better bond. This allows acceptable recoveries of the DCB surrogate.

12.1.5 Check the pH to make sure that it is within the required range and record.

12.1.6 Shake the container with the cap tightly sealed, to ensure that any particulate matter is evenly distributed throughout the sample.

12.1.7 Turn on the vacuum. Place the sample containers on the manifold apparatus with a quick flipping of the wrist on the bottle adaptor.

12.1.8 Open the valves of each setup to allow the sample to filter through. The sample should pass through the cartridge in approximately 15 – 20 minutes.

NOTE: The excess sample remaining inside the graduated cylinder after the transfer is to be placed in the original container that has been completely filtered and reapplied back on the manifold for complete filtration of the entire bottle.

12.1.9 Allow the cartridge to dry under full vacuum for 10 minutes and close the valves. The sample waste is collected into a waste carboy that is connected to the apparatus.

12.2 Sample Elution

12.2.1 Place a 40 mL receiving vial under each cartridge.

NOTE: The elution for DRO (Diesel Range Organic) is performed with Dichloromethane only.

12.2.2 Rinse the sample bottle that held the sample with 5 mL of acetone and transfer to the cartridge. Allow the solvent to soak for 1 minute and open the valves to vacuum pulling the acetone into the receiving vial then close valve.

12.2.2.1 First, rinse the graduated cylinder containing the sample(s) with 5 mL of acetone. Transfer the rinse to the sample bottle then transfer onto the cartridge. Allow the solvent to soak for 1 minute and open the valves to vacuum, pulling the acetone into the receiving vial, then close valve. Proceed as instructed in section 12.2.3.

12.2.3 Repeat step 12.2.2 three more times using 10 mL Dichloromethane each time. Allow the solvent to soak for 1 minute, then pull through by vacuum into the receiving vial. The final volume of the elution should not exceed 40 mL.

12.3 K-D Concentrated Technique

12.3.1 Where necessary to meet the sensitivity requirements of the particular

application, sample extract may be concentrated to the final volume necessary for the determinative method and specific application using the K –D technique and nitrogen evaporation or TurboVap evaporator.

12.3.2 Assemble a pre-solvent rinsed Kuderna –Danish (K-D) and 10 mL concentrator tube.

12.3.3 Dry the extract in the collection tube by shaking to mix very well then pour on the thistle funnel containing anhydrous Sodium Sulfate. Collect the dried extract in the K-D concentrator setup. Note: Water is in the extract so while pouring on the sodium sulfate use a glass pipet to mix and break up the clumps of sodium sulfate. Otherwise this will result low recoveries.

12.3.4 Rinse the collection tube and drying column in to the K-D apparatus with an additional 20 mL portion of dichloromethane in order to achieve a quantitative transfer.

12.3.5 Add clean boiling chips to K-D apparatus and attach a pre-wet three ball snyder column. Note: At the proper rate of distillation the balls of the snyder column will actively chatter, but the chambers will not flood.

12.3.6 When the apparent volume of the liquid reaches 2 mL, remove the K-D apparatus from the water bath and allow to drain and cool for 10 minutes.

12.3.7 If a solvent exchange is needed add 10 mL of exchange solvent through the snyder column and mix with a swirling motion. Refer to Appendix 1 for extraction conditions.

12.3.8 Concentrate the extract by placing on the water bath which is at the temperature to maintain proper distillation rate. Allow the sample to remain on the water bath until for an approximate 5 mL final volume or for no more than 30 minutes.

12.3.9 Proceed with clean-up if necessary. Refer to Appendix 1 for appropriate clean-up.

12.3.10 Remove K-D apparatus from the water bath allowing to cool and drain for 10 minutes. Label all concentrator tubes before disassembling the apparatus. If further concentration is needed place extract on a nitrogen evaporator (N-EVAP) at 35°C to reach final volume.

12.3.11 Quantitatively transfer the extract to a 2 ml amber vial with Teflon lined cap.

12.4 Turbovap Concentration Technique

12.4.1 Sample extract may be concentrated to the final volume necessary for the determinative method and specific application using the Turbovap technique.

12.4.2 Solvent rinse Turbovap and anhydrous sodium sulfate prior to use.

12.4.3 Transfer the lower layer methylene chloride (top layer is water residue)

from the collection tube to the labeled turbovap tube. Add anhydrous sodium sulfate to the collection tube that contains the water layer. Use a glass rod to break up clumps of sodium sulfate. Not breaking up the clumps may result in lower recoveries. Rinse the collection tube several times with the extraction solvent and add to the turbovap tube.

12.4.4 Check the reagent water level in the turbovap. Make sure to have adequate water covering the bottom rack and not to exceed the level of the top rack.

12.4.5 Open the lid of the Turbovap and gently place the turbovap tube in the water bath rack. Close the cover.

NOTE: During this time, extracts should be monitored periodically, so that extracts do not evaporate completely. Not all extracts evaporate at the same rate.

12.4.6 When the apparent volume of liquid reaches 2-5 mL and if a solvent exchange is needed, add approximately 10-15 mL of the exchange solvent to the turbovap tube. Gently swirl the tube to mix and rinse side of the tube. Return to the water bath for further concentration. Refer to Appendix 1 for extraction conditions.

12.4.7 If clean-up is needed, refer to Appendix 1 for appropriate clean-up.

12.4.8 Quantitatively transfer the final extract to a 2 mL vial with Teflon lined cap.

13.0 PREPARATION OF STANDARD SOLUTIONS AND QUALITY CONTROL SAMPLES

13.1 Source standards are purchased as certified mixtures. Documentation of the standard's quality traceability should be provided from the vendor. This documentation is submitted to the Quality Department. Standards may be purchased from outside vendors, including o2si smart solutions, AccuStandard, Inc., Restek, NSI Solutions, Inc., and Supleco. Other vendors on GEL's vendor list may also be used.

13.2 Source standards are assigned a unique code number for the purpose of traceability. The standard, along with its code, is recorded in AlphaLIMS. AlphaLIMS can be used to generate a label that is affixed to the standards container, or a handwritten label may be created.

13.3 Stock, intermediate, and working standards are likewise assigned a unique code number and recorded in AlphaLIMS.

14.0 SAFETY AND POLLUTION PREVENTION

Follow all laboratory safety rules for preparation, analysis, and handling of the reagents of interest. Refer to method SOPs and the GEL safety plan for guidance.

15.0 SAFETY, HEALTH, AND ENVIRONMENTAL HAZARDS

WARNING

METHYLENE CHLORIDE IS A POSSIBLE CARCINOGEN.

HEXANE IS FLAMMABLE AND TOXIC.

ACETONE IS HIGHLY FLAMMABLE AND TOXIC

SULFURIC ACID IS CORROSIVE, CAUSES SERIOUS BURS AND MAY BE FATAL IF SWALLOWED.

15.1 PREVENT SKIN AND EYE CONTACT BY USING SPECIFIED PERSONAL PROTECTIVE EQUIPMENT WHEN MAKING STOCK REAGENTS.

15.2 WORK UNDER A HOOD TO PREVENT INHALATION WHEN MAKING STOCK REAGENTS.

15.3 Wear eye protection with side shields while working in the laboratory.

15.4 All chemicals and samples should be treated as potential health hazards, and exposure to these chemicals must be reduced to the lowest level possible. GEL maintains a current awareness file of OSHA regulations regarding handling of the chemicals in the laboratory as well as a reference file of Material Safety Data Sheets (MSDS). These documents are maintained in the laboratory. Individual sample MSDS forms provided by the clients are kept in Login.

15.5 Personal protective equipment:

15.5.1 Gloves are required when working with solvents, standards, and samples. Solvents, along with any solute in them, can absorb easily through the skin.

15.5.2 Work under a hood when using concentrated acids and solvents.

15.5.3 To protect clothes and skin from exposure to corrosive material, wear a lab coat.

15.6 Prior to handling radioactive samples, analysts must have had radiation safety training and must understand their full responsibilities in radioactive sample handling. Some general guidelines follow:

15.6.1 Wear a dosimeter at all times to monitor radioactive exposure while working in the lab.

15.6.2 Wear a plastic apron over lab coat when working with radioactive samples.

15.6.3 Protect counter tops with counter paper or work from radioactive sample handling trays.

15.6.4 Prohibit admittance to immediate work area.

15.6.5 Post signs indicating radioactive samples are in the area.

15.6.6 Take swipes of the counter tops upon completion of work. Deliver those swipes to the designated swipe count box.

15.6.7 Segregate radioactive wastes. Radioactive waste containers are obtained from Waste Management.

15.7 All samples, chemicals, extracts, and extraction residues must be transferred, delivered, and disposed of safely according to all related SOPs.

15.7.1 Segregate solid wastes from liquid wastes in the satellite area containers.

15.7.2 Segregate oil wastes from water-soluble wastes in the satellite area containers.

15.8 Snyder columns may shoot off due to pressure inside the K-D.

15.9 In the event of an accident or medical emergency call for help immediately. When time and safety permit, an accident report form should be completed and turned in to the safety committee.

15.10 Fire escape routes are posted in the lab, and all personnel should be familiar with them. In addition, fire safety equipment such as fire extinguishers is located in the lab. Training is available on the proper operation of this equipment.

16.0 QUALITY CONTROL

The analyst must demonstrate that the compounds of interest are being quantitatively recovered before applying this method to actual samples. A recovery check must be performed using standards of the target analytes at known concentrations. Therefore, the LCS, extraction blank and all other quality control samples must be processed with the same procedure as client samples.

17.0 DATA REVIEW, APPROVAL, AND TRANSMITTAL

A review process is used to insure the quality of the data. Extraction logs are peer reviewed by a second technician or group leader. When the reviewer is satisfied that the data have been entered correctly, a data report is generated from AlphaLIMS. The report along with the batch sheets are copied and submitted to the appropriate analytical area for analysis.

18.0 LABORATORY WASTE HANDLING AND DISPOSAL

For the proper disposal of sample and reagent wastes from this procedure, refer to the Laboratory Waste Management Plan, GL-LB-G-001.

19.0 RECORDS MANAGEMENT

19.1 Documentation of Training

19.1.1 Analysts and technicians must be properly trained to perform the contents of this SOP. Personnel will extract 4 laboratory control samples for each analytical SOP referenced within this SOP as training commences. Training records are maintained as quality records. LCS/LCS DUP will demonstrate on a continuing basis that personnel are properly trained.

19.2 Documentation of Extraction

19.2.1 Complete sample tracker form as described in the appropriate extraction

procedure. Record the initial and final volumes of the extract, and any comments about the cleanup process. Also record all reagent lot numbers. Note any deviations from this SOP in the comment section.

19.2.2 Have batch peer reviewed using data review sheet. Note all discrepancies. These documents are stored in AlphaLIMS.

19.3 Documentation of Standards

Refer to GL-LB-E-007 for Laboratory Standards Documentation.

20.0 LABORATORY WASTE HANDLING AND DISPOSAL: SAMPLES, EXTRACTS, DIGESTATES, AND REAGENTS

For the proper handling and disposal of sample and reagent wastes from this procedure, refer to the Laboratory Waste Management Plan, GL-LB-G-001.

21.0 REFERENCES

21.1 Single Laboratory Evaluation of Method 8060-Phthalate Esters EPA/600/4-89/039, V. Lopez-Avila, W .Beckert , et.al.,

21.2 Determination of Eight Organochlorine Pesticides at low Nanogram/Liter Concentration in Groundwater Using Filter Disk Extraction and Gas Chromatography, JAOAC International, 75(6),pp. 1091-1099,1992 3M Data Submission to EPA, C. Markell

21.3 Test Methods for Evaluating Solid Waste EPA manual SW846, 3rd Edition, June 1997, Method 3535A “Solid – Phase Extraction”.

21.4 Dept. of Defense (DOD), Dept. of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories DOD QSM Version 5.3, July 2019.

22.0 HISTORY

Revision 7: Removed HR-E-003 for training reference.

Revision 8: Updated Appendix

Revision 9: Added LLOQ in definitions section. Added reference to new DoD/DoE QSM version 5.1, January 2017.

Revision 10: Updated to include turbovap equipment and process of usage.

Revision 11: Revised to reflect current practices. Updated the DOD QSM Version 5.3, July 2019

APPENDIX 1: EXTRACTION CONDITIONS

Extraction Conditions for Various Water Analyses

Prep	Analysis	Surrogate/Spike[a]	Extraction pH	Solvent Exchange	Clean up Required
Pesticide/ PCB[c]	8081B	Pesticide Surrogate/[d] Pesticide Spike	<2	Hexane	Florisil
PCB	8081B 8082A	Pesticide Surrogate/[e] PCB H_2O Spike	<2	Hexane	$KMnO_4$/ H_2SO_4
TPH/DRO	8015C 8015D	TPH Surrogate/ TPH Spike	<2	None	
Pesticide/ PCB	608	Pesticide Surrogate[d]/ 608 Spike	<2	Hexane	Florisil
PCB	608	Pesticide Surrogate /[e] PCB H_2O Spike	<2	Hexane	$KMnO_4$/ H_2SO_4
Pesticide[b]	TCLP	Pesticide Surrogate / [e] TCLP Pesticide Spike/ Toxaphene/ Chlordane	<2	Hexane	
PCB	TCLP	Pesticide Surrogate/ [e] PCB Spike	<2	Hexane	$KMnO_4$/ H_2SO_4

a: Other spiking compounds may be used if client requests non-regulated compounds.

b: GEL has defined Method 8081/8081A into two classes of compounds: Pesticides and Pesticides/PCBs. When a batch consists of samples requesting Pesticides and/or Pesticides/PCBs, use Pesticide Surrogate and Pesticide Spike.

c: Pesticide surrogate 1000 ug/L

d: Pesticide surrogate 200 ug/L

NOTE: For recipes, concentrations, analytes and diluents generally used, refer to the Create/Edit/View Reference Materials section of AlphaLIMS.

VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE

STANDARD OPERATING PROCEDURE FOR THE EXTRACTION AND ANALYSIS OF PER- AND POLYFLUOROALKYL (PFAS) SUBSTANCES USING LCMSMS

(GL-OA-E-076 REVISION 9)

APPLICABLE TO METHOD:
EPA Method 537.1 and 537.1 Modified
ASTM D7968-17A
Compliant with Table B-15, DOD QSM, Version 5.3 Requirements

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1.0 STANDARD OPERATING PROCEDURE FOR THE ANALYSIS OF PER- AND POLYFLUOROALKYL SUBSTANCES (PFAS) USING SOLID PHASE EXTRACTION AND LIQUID CHROMATOGRAPHY/ TANDEM MASS SPECTROMETRY (LCMSMS)

2.0 METHOD REFERENCE

2.1. EPA 537 Version 1.1
2.2. ASTM D7968-17A
2.3. EPA 537.1 Version 1.0

3.0 METHOD OBJECTIVE AND PURPOSE

This standard operating procedure (SOP) covers the determination of Per- and Polyfluoroalkyl substances (PFAS) in a wide variety of liquid matrices according to USEPA method 537.1 and other analytical protocols such as Department of Defense (DOD) and Department of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental laboratories.

Due to the changing regulatory requirements for drinking water, non-potable water, and other matrices throughout the United States, it is imperative that project objectives be established prior to receipt and analysis of samples. For example, state and federal agencies that have approved drinking water analysis by EPA Method 537.1 do not allow any modifications. Therefore, the analysis of those samples must strictly adhere to all specifications as written in the published method, without any modifications. This includes, but is not limited to, additional compound list, sample collection, preservation, extraction, and analysis.

For all other matrices, an isotopic dilution technique will be used to determine compound concentrations unless otherwise instructed by project data quality objects.

GEL has provided the CAS numbers and additional synonyms for the corresponding analytes in Table 1 so that clients and regulatory agencies can select the appropriate synonym per their requirements.

4.0 METHOD SCOPE AND APPLICABLE MATRIX

4.1. Refer to Table I for compound list of PFAS by LCMSMS.
4.2. This method applies to the following matrices:
- 4.2.1. Groundwater
- 4.2.2. Drinking Water
- 4.2.3. Wastewater
- 4.2.4. Soil
- 4.2.5. Tissue

5.0 DISCUSSION

A 250-mL drinking water sample is fortified with surrogates. A 250 –mL non-drinking water sample is fortified with internal standards. Once fortified, the sample is passed through a solid phase extraction (SPE) cartridge to extract the method analytes and corresponding fortified compounds. The compounds are eluted from the cartridge with 0.1% ammonia/methanol followed by a small amount of methanol. All water samples except drinking waters are passed through an Envicarb SPE cartridge to remove interferences. The extract is concentrated to dryness with nitrogen in a heated water bath, and then adjusted to a final volume of 5-mL with the appropriate solvent.

A 2-gram soil is transferred to a 50 mL tube and fortified extraction internal standards. 5 mL of 0.1% ammonia/Methanol is added to each container. The sample is then shaken on a wrist action shaker for 30 minutes under basic conditions (pH ~ 9-10). The sample is then decanted and the process repeated twice more with 2.5 mL aliquots of 0.1% ammonia/Methanol. The extract is then centrifuged and a portion of the sample passed through an Envicarb SPE cartridge to remove interferences. The sample is then concentrated to 4 mL on an N-EVAP. Add 1 mL of water to each extract. The sample is now ready for analysis.

A 2-gram tissue sample is transferred to a 50 mL tube and fortified extraction internal standards. 10 mL of 0.1% Ammonia/Methanol is added to each container. The sample is then shaken on a wrist action shaker for 30 minutes under basic conditions (pH ~ 9-10). The sample is then decanted and the process repeated twice more with 5 mL aliquots of 0.1% Ammonia/Methanol. The extract is then centrifuged and a portion of the sample passed through an Envicarb SPE cartridge to remove interferences. The sample is then concentrated to 4 mL on an N-EVAP. Add 1 mL of water to each extract. The sample is now ready for analysis.

A 25-µL injection is made into an LC equipped with a C_{18} column that is interfaced to an MS/MS. The analytes are separated and identified by comparing the acquired mass spectra and retention times to reference spectra and retention times for calibration standards acquired under identical LC/MS/MS conditions. The concentration of each analyte is determined by using the internal standard technique for drinking water samples and Isotope Dilution technique for all other matrices. Extraction internal standards or surrogates (drinking water) are added to all Field and QC Samples to monitor the extraction efficiency of the method analytes.

6.0 DEFINITIONS

6.1. Analysis Batch: A set of samples that is analyzed on the same instrument during a 24-hour period, including no more than 20 Field Samples, that begins and ends

with the analysis of appropriate Continuing Calibration Verification (CCV) standards. Additional CCVs may be required depending on the length of the analysis batch and/or the number of Field Samples.

6.2. Calibration Standard (CAL): A solution prepared from the primary dilution standard solution and/or stock standard solution, extraction internal standard(s), and the injection internal standard(s). The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

6.3. Continuing Calibration Check (CCC): This standard will be referred to as a Continuing Calibration Verification (CCV) standard for this SOP. A CCV contains the method analytes, extraction internal standard(s) and injection internal standard(s). The CCV is analyzed periodically to verify the accuracy of the existing calibration for those analytes.

6.4. Method Detection Limit (MDL): The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero. This is a statistical determination of precision, and accurate quantitation is not expected at this level. Typically, the MDL is two to three times less than the lowest concentration used to calibrate the instrument.

6.5. Extraction Batch: A set of up to 20 Field Samples (not including QC samples) extracted together by the same person(s) during a work day using the same lot of SPE devices, solvents, surrogate, internal standard and fortifying solutions. Required QC samples include Laboratory Reagent Blank, Laboratory Fortified Blank, Laboratory Fortified Sample Matrix, and either a Field Duplicate or Laboratory Fortified Sample Matrix Duplicate.

6.6. Field Duplicated (FD1 and FD2): Two separate samples collected at the same time and place under identical circumstances, and treated exactly the same throughout field and laboratory procedures. Analyses of FD1 and FD2 give a measure of the precision associated with sample collection, preservation, and storage, as well as laboratory procedures.

6.7. Field Reagent Blank (FRB): An aliquot of reagent water that is placed in a sample container in the laboratory and treated as a sample in all respects, including shipment to the sampling site, exposure to sampling site conditions, storage, preservation, and all analytical procedures. The purpose of the FRB is to determine if method analytes or other interferences are present in the field environment.

6.8. Laboratory Fortified Blank (LFB): Will be referenced as Laboratory Control Sample (LCS) in this SOP. A volume of reagent water or other blank matrix to which known quantities of the method analytes and all the preservation compounds are added in the laboratory. The LCS is analyzed exactly like a

sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

6.9. Laboratory Fortified Sample Matrix (LFSM): Will be referenced as a Matrix Spike (MS) in this SOP. A field sample to which known quantities of the method analytes are added in the laboratory. The MS is processed and analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate sample extraction and the measured values in the MS corrected for background concentrations.

6.10. Laboratory Fortified Sample Matrix Duplicate (LFSMD): Will be referenced as a Matrix Spike Duplicate (MSD). A duplicate of the Field Sample used to prepare the MS. The MSD is fortified, extracted, and analyzed identically to the MS. The MSD is used instead of the Field Duplicate to assess method precision when the occurrence of method analytes is low.

6.11. Matrix Duplicate (MD): Will be referenced as a Sample Duplicate (DUP). This is a duplicate of the Field Sample. It is extracted and analyzed identically to the sample. The DUP is used to assess method precision when the occurrence of method analytes is low.

6.12. Laboratory Reagent Blank (LRB): Will be referenced as the Method Blank (MB) in this SOP. An aliquot of reagent water or other blank matrix that is treated exactly as a sample including exposure to all glassware, equipment, solvents and reagents, sample preservatives, extraction internal standards, and injection internal standards that are used in the analysis batch. The MB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

6.13. Lowest Concentration Minimum Reporting Level (LCMDL): The single laboratory LCMRL is the lowest true concentration for which a future recovery is expected, with 99% confidence, to be between 50 and 150% recovery.

6.14. Minimum Reporting Level (MRL): The minimum concentration that can be reported as a quantitated value for a method analyte in a sample following analysis. The defined concentration can be no lower than the concentration of the lowest calibration standard for that analyte and can only be used if acceptable QC criteria for this standard are met. A procedure for verifying a laboratory's MRL is provided in Appendix 5. This is also referenced as the LOQ. Typical MRLs for this method 2 ng/L for waters and 500 ng/Kg for solids.

6.15. Quality Control Sample (QCS): Will be referenced as the Initial Calibration Verification (ICV) in this SOP. A solution of method analytes of known concentrations that is obtained from a source external to the laboratory and different from the source of calibration standards. The ICV is analyzed directly

after the calibration curve and is used to check calibration standard integrity. If an alternate source is not available, a different lot from the same source may be used.

6.16. Limit of Detection (LOD): The lowest concentration level that can be determined by a single analysis and with a defined level confidence to be statistically different from a blank. The LOD verification is typically spiked at two times the MDL.

6.17. Limit of Quantitation (LOQ): The lowest concentration level of the initial calibration curve used to quantitate an analyte. The LOQ verification is typically spiked at the PQL. This is also referenced as the MRL (for drinking water only).

6.18. Linear Calibration Range (LCR): The concentration range over which the instrument response is linear.

6.19. Surrogate Standard – A standard added prior to sample extraction used to monitor unusual matrix effects, sample processing errors, and extraction efficiency. These are used for drinking water analysis only. They are: $^{13}C_4$-PFOS, $^{13}C_3$-PFBA, $^{13}C_2$-PFOA, and $^{13}C_2$-PFDA. These surrogates are added at a concentration of 250 ng/L. Surrogates are added to each blank, laboratory control sample (LCS), matrix spike (MS), matrix spike duplicate (MSD) and sample prior to the extraction.

6.20. Refer to GL-QS-B-001 the Quality Assurance Plan for additional lab-wide used definitions.

7.0 INTERFERENCES

7.1. All glassware must be meticulously cleaned. Wash glassware with detergent and tap water, rinse with tap water, followed by a reagent water rinse. Non-volumetric glassware can be heated in a muffle furnace at 400 °C for 2 h or solvent rinsed. Volumetric glassware should be solvent rinsed and not be heated in an oven above 120 °C. Store clean glassware inverted or capped. Do not cover with aluminum foil because PFASs can be potentially transferred from the aluminum foil to the glassware.

NOTE: PFAS standards, extracts and water samples should not come in contact with any glass containers or pipettes as these analytes can potentially adsorb to glass surfaces. PFAS analytes, ES and IS standards commercially purchased in glass ampoules are acceptable; however, all subsequent transfers or dilutions performed by the analyst must be prepared and stored in HDPE containers.

7.2. Method interferences may be caused by contaminants in solvents, reagents (including reagent water), sample bottles and caps, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the chromatograms. The method analytes in this method can also be found in many common laboratory supplies and equipment, such as PTFE (polytetrafluoroethylene) products, LC solvent lines, methanol, aluminum foil, SPE sample transfer lines, etc. All items such as these must be routinely

demonstrated to be free from interferences (less than 1/3 the MRL for each method analyte) under the conditions of the analysis by analyzing laboratory reagent. **Subtracting blank values from sample results is not permitted**.

7.3. Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the water. Humic and/or fulvic material can be co-extracted during SPE and high levels can cause enhancement and/or suppression in the electrospray ionization source or low recoveries on the SPE sorbent. Total organic carbon (TOC) is a good indicator of humic content of the sample.

7.4. Relatively large quantities of the preservative are added to sample bottles for drinking water samples. The potential exists for trace-level organic contaminants in these reagents. Interferences from these sources should be monitored by analysis of laboratory reagent blanks particularly when new lots of reagents are acquired.

7.5. SPE cartridges can be a source of interferences. The analysis of field and laboratory reagent blanks can provide important information regarding the presence or absence of such interferences. Brands and lots of SPE devices should be tested to ensure that contamination does not preclude analyte identification and quantitation.

8.0 SAFETY, HEALTH, ENVIRONMENTAL HAZARDS AND POLLUTION PREVENTION

8.1. Wear eye protection with side shields while working in the laboratory.

8.2. All chemicals and samples should be treated as potential health hazards, and exposure to these chemicals must be reduced to the lowest level possible. GEL maintains a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals. A reference file of Material Safety Data Sheets (MSDS) and individual client sample MSDSs are also maintained.

8.3. Personal protective equipment

8.3.1. Gloves are required when working with solvents, standards and samples. Solvents, along with any solute in them, can absorb easily through the skin.

8.3.2. Work under a hood when using concentrated acids.

8.3.3. To protect clothes and skin from corrosive material, wear a lab coat.

8.4. Prior to handling radioactive samples analysts must have had radiation safety training and understand their full responsibilities in radioactive sample handling. Some general guidelines to follow:

8.4.1. To monitor radioactive exposure, wear a dosimeter at all times while working in the lab.

8.4.2. Wear a plastic apron over lab coat when working with radioactive samples.

8.4.3. Protect counter tops with counter paper or work from radioactive sample handling trays.

8.4.4. Prohibit admittance to immediate work area.

8.4.5. Post signs indicating radioactive samples are in the area.

8.4.6. Take swipes of the counter tops upon completion of work. Deliver those swipes to the designated swipe count box.

8.4.7. Segregate radioactive wastes. Radioactive waste containers are obtained from Waste Management.

8.5. All samples, chemicals, extracts, and extraction residues must be transferred, delivered, and disposed of safely according to all related SOPs.

8.5.1. Segregate solid wastes from liquid wastes in the satellite area containers.

8.5.2. Segregate oil wastes from water-soluble wastes in the satellite area containers.

8.6. In the event of an accident or medical emergency, call for help immediately. When time and safety permit, an accident report form should be completed and turned in to the safety committee.

8.7. Fire escape routes are posted in the lab, and all personnel should be familiar with them. In addition, fire safety equipment such as fire extinguishers is located in the lab. Training is available on the proper operation of this equipment.

8.8. Refer to SOP GL-LB-N-001 Safety, Health and Chemical Hygiene Plan for additional general safety and health information pertaining to the laboratory.

9.0 APPARATUS AND MATERIALS

9.1. Labware

9.1.1. Polypropylene Conical Tubes – 15 mL and 50 mL

9.1.2. pH paper – pH range 1-14

9.1.3. Vials: 4 mL narrow- mouth HDPE bottles with plastic caps for standards storage.

9.1.4. Autosampler Vials – Polypropylene 0.3-mL autosampler vials with polypropylene caps.

9.1.5. Graduated Cylinders – appropriate sizes

9.1.6. Eppendorf Pipet and tips

9.2. Top loading balance

9.3. Vortex mixer

9.4. Wrist Action Shaker

9.5. Solid Phase Extraction (SPE) apparatus for using cartridges.

9.5.1. SPE Cartridges – Strata™ XL-AW 100 µm Polymeric Weak Anion

9.5.2. SPE Cartridges - Strata SDBL 100 um Styrene-divinylbenzene(0.5g) 6 mL – Used for drinking waters only.

9.5.3. SupelClean™ ENVI-Carb™ SPE Cartridge - (0.25g) 6 mL

9.5.4. Vacuum Extraction Manifold – A manual vacuum manifold with large volume sampler for cartridge extractions.

9.5.5. Sample Delivery System – Use of a polypropylene transfer tube system, which transfers the sample directly from the sample container to the SPE cartridge.

9.6. Liquid Chromatography (LC)/Tandem Mass Spectrometer (MS/MS) With Data System

9.6.1. Liquid Chromatography/Mass Spectrometry/Mass Spectrometry System - An analytical system complete with a binary gradient, programmable high-pressure liquid chromatograph, and a column heater capable of maintaining a temperature of 40° ± 1° C. The liquid chromatography unit must be capable of accurate and repeatable flow rates of 0.2 mL/min to 1.0 mL/min. The mass spectrometer system must possess an electrospray ionization (ESI) interface capable of operating at flow rates stated above. The mass spectrometer system must be capable of negative ion analysis and consist of three quadrupoles for daughter fragmentation.

9.6.2. Data System - A computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that can search any LC/MS/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as the Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between specific time or scan-number limits.

9.6.3. Analytical Column – Gemini 3 µm C18 110 Å LC column 50 x 2 mm.

9.6.4. Delay Column - Luna® 5 µm C18 (2) 100 Å 30 x 3 mm

10.0 REAGENTS AND STANDARDS

10.1. Source standards are purchased directly from certified vendors and may be diluted to make stock, intermediate, and/or working standards. Source standards expire

per the vendor expiration date. Please refer to GL-LB-E-007 for further information regarding standards, their preparation, and control.

10.2. Calibration Standards: Calibration standards are prepared from intermediates of certified source standards. Calibration standards expire per the source standard vendor expiration date, or after one year from the open date, whichever is shorter. The calibration standards should be monitored for signs of degradation, and replaced as necessary.

10.3. Second-Source Calibration Standards: A standard purchased from a different certified vendor than the calibration standards or a different lot number from the same vendor as the calibration standards. These standards expire per the vendor's expiration date.

10.4. Laboratory Control Sample (LCS) and Matrix Spike (MS) Standards: The LCS and MS standards contain all of the compounds of interest. LCS and MS standards may be purchased pre-mixed as source standards, or may be prepared in the laboratory from certified source standards. All LCS and MS standards expire per the source standard vendor expiration date, or after one year from the open date, whichever is shorter.

10.5. Extracted Internal Standard (ES): The standard added to all samples and QC prior to extraction to monitor the efficiency of the extraction procedure. The ES standard is prepared as an intermediate source standard and expires per the source standard vendor expiration date, or after one year from the open date of the source, whichever is shorter. This is used as the internal standard for drinking water samples.

10.6. Polypropylene Glycol Tuning Solution (PPG): A positive and negative PPG standard.

10.7. Nitrogen Gas, 99+%

10.8. Reagent water: Purified and does not contain interfering compounds or measurable quantities greater than 1/2 LOQ (1/3 LOQ for drinking water) of each method analyte of interest. (See Appendix 6 for all drinking water criteria). Prior to daily use, at least 3 L of reagent water should be flushed from the purification system to rinse out any build-up of analytes in the system's tubing. Water can also be purchased from a certified vendor.

10.9. White Quartz Sand -50+70 mesh.

10.10. Methanol - High purity, demonstrated to be free of analytes and interferences.

10.11. AMMONIUM ACETATE ($NH_4C_2H_3O_2$, CAS#: 631-61-8) – 97 % mass fraction or greater, demonstrated to be free of analytes and interferences.

10.12. 20M AMMONIUM ACETATE/REAGENT WATER – To prepare 1 L, add 1.54 g ammonium acetate to 1 L of reagent water. This solution is prone to volatility losses and should be replaced at least every 48 hours.

10.13. TRIZMA® PRESET CRYSTALS, pH 7.0 (Sigma cat# T-7193 or equivalent) – Reagent grade. A premixed blend of Tris [Tris(hydroxymethyl)aminomethane] and Tris HCL [Tris(hydroxymethyl)aminomethane hydrochloride]. Alternatively, a mix of the two components with a weight ratio of 15.5/1 Tris HCL/Tris may be used. These blends are targeted to produce a pH near 7.0 at 25 °C in reagent water. Trizma® functions as a buffer, and removes free chlorine in chlorinated finished waters. This will be added to drinking water samples only.

10.14. Acetic acid (CH_3COOH) – 99.9 % mass fraction.

10.15. Ammonium Hydroxide - ~25 % mass fraction.

10.16. Acetate Buffer 0.025 mol/l, pH 4. - Mix 0.5 mL of acetic acid (10.15) with 349.5 ml of water (10.9). Dissolve 0.116 g of ammonium acetate (10.11) in 60 mL of water (10.9). Mix 200 ml of the diluted acetic acid with 50 mL of the ammonium acetate solution.

10.17. Ammonia/methanol solution, 0.1 % mass fraction. - Mix 0.4 mL of 25 % ammonia solution (10.16) with 99.6 mL of methanol (10.11).

10.18. Acetic Acid/Methanol solution – Mix 1.3 mL of Acetic Acid (10.15) with 98.7 mL methanol (10.12).

11.0 SAMPLE HANDLING AND PRESERVATION

11.1. All samples must be collected in a 250 mL HDPE bottle with a polyethylene screw cap.

11.2. Additional containers for field blanks are included with each shipment. One container is filled with reagent water and preservatives (if applicable) in the lab and shipped with field sample containers. A second empty sealed bottle (no preservatives) is included. At the sampling site, the container containing the reagent water is poured into the empty bottle and returned with the field samples.

11.3. 1.25 g Trizma must be added to the 250 mL water sample before sample collection, if applicable. This should be used for drinking water samples only.

11.4. Samples must be shipped on ice. They must be received at the lab at 10 °C or less within 48 hours of collection for waters (6 °C or less for solids). The temperature should be confirmed to be within specification when they are received. All samples stored in the lab must be stored at or below 6 °C until analysis and should not be frozen.

11.5. Upon sample receipt, drinking water samples only are checked for dechlorination. If the free chlorine is > 0.1 mg/L, samples are rejected and a new sample must be requested.

11.6. Water samples must be extracted within 14 days of collection and analyzed within 28 days after extraction. Solid samples must be extracted and analyzed within 28 days of collection for solids. Extracts must be stored at room temperature.

11.7. If samples are not in appropriate containers or holding time has expired, initiate a Nonconformance Report (DER). For instructions, refer to GL-QS-E-004 for Documentation of Nonconformance Reporting and Dispositioning and Control of Nonconforming Item.

12.0 SAMPLE PREPARATION

All batches (up to 20 samples) will be extracted with a method blank (MB), laboratory control sample (LCS), matrix spike (MS), and matrix spike duplicate (MSD). If insufficient sample is provided, the MS/MSD will be substituted with a laboratory control sample duplicate (LCSD).

13.0 EXTRACTION

13.1. See Appendix 2 for the water extraction procedure and Appendix 9 and 10 for extraction check lists

13.2. See Appendix 3 for the solids extraction procedure.

13.3. See Appendix 4 for the AFFF Procedure

14.0 PREPARATION OF STANDARD SOLUTIONS AND QUALITY CONTROL SAMPLES

14.1. Analyte standard solutions – Analyte standards may be purchased commercially as ampoulized solutions or prepared from neat materials. Purchased standards must contain linear and branched isomers, if available. PFHxS and PFOS are not available as the acids but rather as their corresponding salts, such as Na+ and K+. These salts are acceptable starting materials for the stock standards provided the measured mass is corrected for the salt content (adjusted to the anion concentration) according to the equation below. Prepare the Analyte Stock and Primary Dilutions Standards as described below.

$$\text{Mass}_{\text{acid}} = \text{Measured Mass}_{\text{salt}} \times \frac{MW_{acid}}{MW_{salt}}$$

Where:

MW_{Acid} = Molecular weight of the PFAA

MW_{Salt} = Molecular weight of the purchased salt

14.2. Standards

14.2.1. Source standard solutions are purchased from certified vendors. Standards were purchased from Wellington Labs Inc. Once opened, they are stored in HDPE containers. The standard expires on the vendor expiration date. They are stored at 4 °C. Refer to Appendix 7 and 8 of this document for standard calibration concentration levels.

14.2.2. The working and intermediate standard solutions are prepared in 80:20 (vol/vol) MeOH/water (96:4 Vol/vol MeOH/water for drinking water)

from the stock standard. These solutions are stored in HDPE bottles/vials and may be used for up to 6 months from the date of preparation for working standards and up to one year for intermediate standards from the date prepared or the vendor expiration date whichever is shorter.

14.3. For guidance on standard documentation and traceability, refer to GL-LB-E-007 for Laboratory Standards Documentation.

15.0 QUALITY CONTROL REQUIREMENTS FOR PROCESSING AND EXTRACTION

15.1. QC requirements include the Initial Demonstration of Capability (IDC) and ongoing QC requirements that must be met when preparing and analyzing Field Samples. This section describes the QC parameters, their required frequencies, and the performance criteria that must be met in order to meet EPA quality objectives.

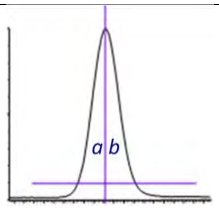
15.2. INITIAL DEMONSTRATION OF CAPABILITY – The IDOC must be successfully performed prior to analyzing any Field Samples. Prior to conducting the IDC, the analyst must first generate an acceptable Initial Calibration following the procedure outlined in Section 17.4.

15.2.1. INITIAL DEMONSTRATION OF LOW SYSTEM BACKGROND – Any time a new lot of SPE cartridges, solvents, centrifuge tubes, disposable pipets, and autosampler vials are used, it must be demonstrated that a method blank is reasonably free of contamination and that the criteria in Section 18.1.4 are met.

15.2.2. INITIAL DEMONSTRATION OF PRECISION (IDP) – Prepare, extract, and analyze four to seven replicate LCSs spiked near the midrange of the initial calibration curve according to the procedure described in Appendix 2 or 3. The relative standard deviation (RSD) of the results of the replicate analyses must be less than 20%.

15.2.3. INITIAL DEMONSTRATION OF ACCURACY (IDA) - Using the same set of replicate data generated for Section 15.2.2, calculate average recovery. The average recovery of the replicate values must be within ± 30% of the true value.

15.2.4. INITIAL DEMONSTRATION OF PEAK ASYMMETRY FACTOR (IDPA) - A peak asymmetry factor must be calculated using the equation below during the IDL and every time a calibration curve is generated. The peak asymmetry factor for the first two eluting peaks in a mid- level CAL standard must fall in the range of 0.8 to 1.5. Modifying the standard or extract composition to more aqueous content to prevent poor shape is not permitted. This measurement is performed by the instrument software.



$$A_s = \frac{B}{A}$$

Where:

A_s = Peak asymmetry factor

B = Width of back half of the peak measured (at 10% peak height) from the trailing edge of the peak to a line dropped perpendicularly from the peak apex

a = The width of the front half of the peak measured (at 10% peak height) from the leading edge of the peak to a line dropped perpendicularly from the apex.

15.2.5. MINIMUM REPORTING LEVEL (MRL) Confirmation - The lowest CAL standard used to establish the Initial Calibration will be at or below the concentration of the MRL. See LOQ.

15.2.6. CALIBRATION CONFIRMATION – Analyze an ICV as described in Section 17.5.1.1 to confirm the accuracy of the standards/calibration curve.

15.2.7. DETECTION LIMIT DETERMINATION – The minimum concentration of an analyte that can be measured, identified and reported with 99% confidence that the concentration is greater than zero. This is a statistical determination of precision, and accurate quantitation is not expected at this level.

15.3. On-going QC Requirements- This section summarizes the ongoing QC criteria that must be followed when processing and analyzing wastewater and solid samples. See Appendix 6 for ongoing QC criteria for drinking water samples.

15.3.1. CONTINUING CALIBRATION VERIFICATION (CCV) - CCV standards are analyzed at the beginning of each analysis batch, after every 10 field samples, and at the end of the analysis batch. See Section 17.5.2 for acceptance criteria solid and water and Appendix 1.

15.3.2. FIELD REAGENT BLANK (FRB) – The purpose of the FRB is to ensure that PFAAs measured in the samples were not inadvertently introduced into the sample during sample collection/handling. The FRB is processed, extracted and analyzed in exactly the same manner as a sample. For drinking waters, if the method analyte(s) found in the sample is present in the FRB at a concentration greater than $^1/_3$ LOQ, then all samples collected with that FRB are invalid and must be recollected and reanalyzed.

15.3.3. INITIAL CALIBRATION VERIFICATION (ICV) – An ICV is a standard from a source different from the source of the CAL standards. If a second vendor is not available, then a different lot of the standard from the same vendor should be used. The ICV should be prepared and analyzed just like a CCV. Acceptance criteria for the ICV are identical to the CCVs; the calculated amount for each analyte must be ± 30% of the expected value. If measured analyte concentrations are not of acceptable accuracy, check the entire analytical procedure to locate and correct the problem. This standard is analyzed whenever a new initial calibration curve is analyzed.

15.3.4. INSTRUMENT SENSITIVITY CHECK (ISC) – A standard spiked at the lowest level of the calibration curve (LOQ). The calculated amount for each analyte must be ± 50% ((± 30% when following DODQSM Table B-15) of the expected value. It should be analyzed prior to sample analysis and at least every 12 hours. The ISC can be used as the initial CCV of the day.

16.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

16.1. Maintenance for the LCMSMS

16.1.1. Daily Maintenance

16.1.1.1. Solvents: Adequate volume of solvent needs to be maintained on a daily basis. Failure to do so may result in loss of prime on the LC. This is especially important for the needle wash solvent. Make sure that there is plenty of solvent in each reservoir before using. When a long acquisition run is set up, it is essential to calculate the amount of solvent that will be needed. Add an extra 20% of solvent to the reservoir to make sure the LC does not go dry during the run.

16.1.1.2. Column: The column must be properly equilibrated with the method specific solvent before batch analysis can occur. At the end of the day or an analysis, the column must be flushed

with solvent, usually acetonitrile or methanol, to ensure longevity.

16.1.1.3. Syringe: Check the operation of the sample syringe on a daily basis to ensure the syringe is drawing the proper amount of sample. Make sure the syringe is free of bubbles and that the needle is injecting properly.

16.1.1.4. Solvent Lines and Pump: Prime the system as needed to ensure proper solvent flow to the column.

16.2. Weekly Maintenance

16.2.1. Waste Container: Check the waste container and dispose if needed. The satellite waste container should never be more than ¾ full. Always label waste properly.

16.3. Semi-Annual Maintenance

16.3.1. Plunger seals: Inspect the plunger and plunger seals. If the pump has been losing prime or if the ripple delta is greater than 30 psi, the seals will need to be replaced.

16.3.2. Syringe and injector. Inspect the syringe and injector needle. Replace needle if it is bent or damaged. Replace syringe if there are obvious signs of wear.

16.4. Maintenance for the Mass Spectrometer

16.4.1. Vacuum System Maintenance

16.4.1.1. Daily Maintenance

16.4.1.1.1. Rotary Pumps: The Agilent MS 40+ pump, which backs the quadrupoles. Make sure oil levels in the pumps are sufficient on a daily basis. Check for any leaks.

16.4.2. Mass Spectrometer Source Maintenance

16.4.2.1. Clean the orifice and skimmer plate when discoloration appears.

16.4.2.2. Refer to the AB SCIEX Manual for other required maintenance

16.5. ESI Probe Maintenance

16.5.1. The ESI probe needs to be maintained at all times to ensure maximum sensitivity. Problems that may inform an analyst of needed maintenance are: an unstable ion beam (spray), peak broadening or tailing, low LC pump back pressure, high LC pump back pressure, or gas flow problems. Refer to the manual for troubleshooting tips.

16.5.2. The probe tip may need to be cleaned if dirty matrices are run, especially at low heater temperatures.

17.0 INSTRUMENT CALIBRATION

17.1. A mass calibration is performed initially prior to use. It should be verified before calibration and updated on an as- needed basis (e.g., QC failures, after major preventative maintenance). Refer to the manufacturer's instructions for instrument tuning and conditions.

17.2. Mass Spectral Acquisition Rate – A minimum of 10 spectra scans are acquired across each chromatographic peak for each analyte and Extracted Internal Standard.

17.3. Ion Transitions (Parent →Product) – The chemical derivation of the ion transitions must be documented. A minimum of two ion transitions (Precursor → quant ion and precursor → confirmation ion) and the ion transitions ratio per analyte are required for confirmation for all samples except drinking waters. Confirmation ions are monitored but not required for drinking water samples. Exception is made for analytes where two transitions do not exist. In-house ion ratios are used for all applicable analytes. The recoveries must fall within +/- 50%. Please see Table 2 for the transitions used for each analyte, ES and surrogate and typical ion ratios.

17.4. Initial Calibration Curve

17.4.1. Isotope Dilution Quantitation must be used for all analyses except drinking water. The isotopically labeled analog of each analyte (Extracted Internal Standard) must be used if commercially available. If a labeled analog is not available, the Extracted Internal standard with the closest retention time to the analyte should be used (Internal Standard Quantitation).

NOTE: Isotope Dilution Quantitation is not be used when analyzing drinking water samples. See Appendix 6 for drinking water calibration criteria.

17.4.2. A minimum of five calibration points is required for this analysis unless a second order regression fit is used. If a second order fit is used, six calibration standards are required. If analyzing drinking waters, the curves must be forced through zero. See Appendix 7 for ground water calibration concentration levels. See Appendix 8 for drinking water calibration concentration levels.

17.4.3. When quantitated using the initial calibration curve, the calibration point for each analyte must calculate to be within 70-130% of its true value for all standards except the lowest level. The lowest level must fall within 50-150% (70-130% when following DODQSM criteria If these criteria

cannot be met, the analyst will have difficulty meeting ongoing QC criteria. It is recommended that corrective action is taken to reanalyze the CAL standards, restrict the range of calibration, or select an alternate method of calibration.

CAUTION: When acquiring MS/MS data, LC operating conditions must be carefully reproduced for each analysis to provide reproducible retention times. If this is not done, the correct ions will not be monitored at the appropriate times. As a precautionary measure, the chromatographic peaks in each window must not elute too close to the edge of the segment time window.

17.4.4. When an internal standard is used, the ratio of the area response of the target to the area response of the nearest internal standard is used. The internal standard area count for each point in the ICAL should fall within -50% to +50% of the average area measured in all ICAL standards. The IS response in any chromatographic run must also fall within 70-140% of the most recent CCV.

17.4.5. When Isotope Dilution technique is used, the ratio of the area response of the target to the area response of the isotopically labeled analog of that analyte is used. If a labeled analog is not commercially available, the nearest internal standard may be used. All area counts must be within 50% to 150% of the ICAL midpoint standard or area measured in the initial CCV on days when an ICAL was not performed.

17.4.6. Calibrations are modeled as average response factor and calibration curves. For average response factor the %RSD must be ≤ 20%. For least-square linear regression fits, unweighted, 1/X or $1/X^2$ weighting can be used. The correlation coefficient (r) must be ≥ 0.995 unless otherwise stated. For second order regression fits, weighting can be used and the coefficient of determination (r^2) must be ≥ 0.990 unless otherwise stated. While second-order regression equations may be used, the intercept and degree of curvature should be examined to be sure that results would be reliable throughout the range of the curve.

17.4.7. Generally, it is not acceptable to remove points from a calibration for the purpose of meeting calibration criteria, unless the points are at the high or low ends of the curve. If a point is removed from the low end, the reporting limit may be adjusted accordingly. If a point is removed from the high end, the linear calibration range must be adjusted accordingly. Whenever a point is removed, it must be clearly documented on the instrument log.

17.4.8. If there is a problem with the calibration that appears to be associated with a single standard, that one standard may be re-analyzed. The

calibration function would then be recalculated against the acceptance criteria. If the criteria still cannot be met, then the entire initial calibration should be performed again. An initial calibration should be considered a single event. Therefore, the re-analysis of a calibration standard should be performed immediately to ensure that the reanalysis is still part of the original initial calibration event, and before any samples are analyzed.

17.4.9. All initial calibration points must be analyzed without any changes to instrument conditions.

17.5. Initial and Continuing Calibration Verification

17.5.1. Initial Calibration Verification (ICV)

17.5.1.1. An ICV standard is analyzed following the analysis of a new initial calibration, and must be created from a second source standard. The acceptance limits are 70-130%. See Appendix 6 for criteria used for drinking water analysis.

17.5.2. Continuing Calibration Verification (CCV)

17.5.2.1. Verify the initial calibration at the beginning and end of each group of analyses, and after every tenth sample during analyses. In this context, a "sample" is considered to be a field sample. MBs, CCVs, LCSs, MSs, FDs, FRBs and MSDs are not counted as samples.

17.5.2.2. The beginning CCV of each analysis batch must be at or below the LOQ in order to verify instrument sensitivity prior to any analyses. If standards have been prepared such that all low ICAL points are not in the same solution, it may be necessary to analyze two standards to meet this requirement. Analyte concentrations in subsequent CCVs must range from the LOQ to the mid-level calibration concentration. These low level standards must be analyzed every 12 hours.

17.5.2.3. Inject an aliquot of the appropriate concentration CAL standard and analyze with the same conditions used during the initial calibration.

17.5.2.4. Determine that the absolute areas of the quantitation ions of the IS(s) meet the acceptance criterion for each analyte. If any of the IS areas do not fall within the acceptance criterion, adjustments must be made to restore system sensitivity. These adjustments may include cleaning of the MS ion source, or other maintenance as indicated in Section 16.4. Major instrument maintenance requires recalibration and verification of sensitivity by analyzing a CCV at or below the LOQ.

17.5.2.5. Calculate the concentration of each analyte in the CCV. The calculated amount for each analyte must be within ±30% of the true value (±50% if foloowing DODQSM criteria). If these conditions do not exist, then all data for the problem analyte must be considered invalid, and one of the following remedial action should be taken.

17.5.2.5.1. Samples analyzed since the last acceptable calibration verification should be reanalyzed after adequate calibration has been restored, with the following exception. If the CCV fails because the calculated concentration is greater than the acceptance criteria for any analyte, and sample extracts show no detection for that analyte, non- detects may be reported without re-analysis.

17.5.2.5.2. Immediately analyze two additional consecutive CCVs. If both CCVs pass, samples may be reported without re-analysis. If either fails, or if two consecutive CCVs cannot be analyzed, perform corrective action(s) and re-analyze the CCV and all associated samples since the last acceptable CCV.

17.6. Instrument Blanks (IB)

17.6.1. A solvent blank is analyzed immediately following the highest standard analyzed and daily prior to sample analysis. The concentration of each target must be ≤ ½ LOQ when analyzing all matrices except drinking waters. For drinking water samples, the acceptance criterion is ≤ 1/3 LOQ

NOTE: Successful analysis following the highest standard analyzed determines the highest concentration that carryover does not occur. The highest standard may be a part of the calibration curve or following the calibration curve. If analyzed following the calibration curve, it can only be used to document the highest concentration at which carryover does not occur and cannot be used to extend out the calibration curve range. If sample concentrations exceed this range and the sample(s) following exceed the acceptance criteria, they must be re-analyzed.

17.6.1.1. If target analytes are less than the acceptance criteria, calibration must be performed using a lower concentration for the highest standard until the acceptance criterion is met. If acceptance criteria are not met after the highest standard

which is not included in the calibration curve, the standard cannot be used to determine the highest concentration in samples at which carryover does not occur.

17.6.1.2. If acceptance criteria are not met after samples, additional instrument blanks must be analyzed until acceptance criteria are met. Additional samples should not be analyzed until the acceptance criteria are met.

Note: This practice is not used for drinking water samples.

18.0 ANALYTICAL QUALITY CONTROL REQUIREMENTS AND CONTINGENCIES

18.1.1. Method Blank (MB)

18.1.2. MB data are used to assess contamination from the laboratory environment and to characterize spectral background from the reagents used in sample processing.

18.1.3. When samples that are extracted together are analyzed on separate instruments or days, the method blank associated with those samples must be analyzed on at least one of those instruments and/or days. A solvent blank should be analyzed on all other instruments/days on which any of the samples are analyzed.

18.1.4. The MB must not exhibit values that exceed the 1/2 Limit of Quantitation (LOQ) (1/3 LOQ for drinking waters) for any target analyte. MB values > 1/2 LOQ (1/3 LOQ for drinking waters) indicate laboratory or reagent contamination should be suspected. (See Appendix 6 for all drinking water criteria):

18.1.4.1. The MB may be reanalyzed once to determine if the contamination is instrument related (carryover, etc.). If the reanalysis confirms an instrument-related problem and that the MB has not been contaminated, analysis may proceed.

18.1.4.2. Upon confirmation of the contamination, samples exhibiting positively detected analytes (i.e., greater than the LOQ) that were also detected in the associated MB may be reported if the sample results are at least 10 times the concentration detected in the MB.

18.1.4.3. Samples exhibiting positively detected analytes (i.e., greater than the LOQ) that were also detected in the associated MB but are less than 10 times the concentration detected in the MB must be re-extracted and re-analyzed.

18.1.4.4. If there is insufficient sample volume remaining, or if the holding time has expired, the client must be consulted prior to beginning re-extraction. The deficiency must be documented.

18.2. Laboratory Control Sample (LCS)

18.2.1. The LCS consists of an aliquot of blank matrix that is spiked with the same analytes at the same concentration as the matrix spike sample. The LCS is used to assess the ability of the analytical system to produce acceptable results and undergoes all sample treatments that the associated samples undergo

Calculate the percent recovery (%R) for each target analyte using the following equation:

$$\%R = \frac{\text{Found Value} \times 100}{\text{True Value}}$$

18.2.2. An LCS is required with each extraction batch. It will be spiked at, or less than, the midpoint of the ICAL. Recoveries must fall within the DoD/DOE QSM limits. If the LCS results do not meet these criteria for method analytes, then all data for the problem analyte(s) must be considered invalid for all samples in the extraction batch. If the recovery of any analyte falls outside the established control limits, that analyte is judged out of control, and the source of the problem should be identified and resolved before continuing the analyses.

The following corrective actions are recommended:

18.2.2.1. The LCS may be reanalyzed once to determine if the out of control result is instrument related. If upon reanalysis the out of control analytes are in control, analysis and reporting of the associated samples may proceed.

18.2.2.2. If the %R for any analyte in the LCS is below the lower control limit, or if the %R is above the upper control and the analyte was detected in the associated samples above the LOQ, all associated samples must be re-extracted and re-analyzed for that analyte.

NOTE: If there is insufficient sample volume remaining or if the holding time has expired for any sample associated with the LCS, the client must be consulted prior to beginning re-extraction. The deficiency must be fully documented and it must be noted in the final report.

18.2.2.3. If the %R for any analyte in the LCS is above the upper control limit, and the associated samples do not exhibit concentrations above the LOQ for that analyte, the sample results may be reported. This indicates a high bias may exist for the batch for that analyte, but since the samples were free of detections, the potential high bias does not affect the

sample result. The deficiency must be documented and noted in the final report.

18.3. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

18.3.1. A matrix spike is a sample that is spiked with the target analytes and is used to assess the effect of the matrix on the target analytes. If there is insufficient sample volume to prepare a MSD or a sample duplicate, a LCSD must be prepared and analyzed. The accuracy of the MS and MSD is reported as %R and is calculated as follows:

$$\%R = \frac{C_s - C}{S} * 100$$

Where:

%R =	Percent recovery
C_s =	Spiked sample concentration
C =	Unspiked concentration
S =	Concentration of the spike added to the sample

18.3.2. The MSD, sample duplicate or LCSD is used to measure the precision of the analytical system. The precision is expressed as relative percent difference (RPD), and is calculated using the following equation:

$$RPD = \frac{|a - b|}{[(a + b)/2]} \times 100$$

Where:

RPD =	Relative Percent Difference
a =	Result of MS, sample or LCS
b =	Result of MSD, sample duplicate or LCSD

18.3.3. The %R should fall within the DOD QSM limits for all matrices except drinking water. Drinking water recoveries should be between 70%-130%. If the accuracy of any analyte falls outside the designated range, and the laboratory performance for that analyte is shown to be in control in the CCVs, the recovery is judged to be matrix biased. The result for that analyte in the unfortified sample is qualified and a statement made in the case narrative to notify the end user that the results are suspect due to matrix effects. Also, look for objective evidence of matrix interference such as sample heterogeneity, chromatographic behavior, LCS

recoveries, method blank, required dilutions or behavior of the sample during extraction. MS, MSD or MS/MSD RPD failures and sample matrix observations must be documented.

18.3.4. RPDs for MSDs, sample duplicate or LCSD should be ≤30%. Greater variability may be observed when FDs have analyte concentrations that are within a factor of 2 of the LOQ. At these concentrations, FDs should have RPDs that are ≤50%. If the RPD of any analyte falls outside the designated range, and the laboratory performance for that analyte is shown to be in control in the LCS, the recovery is judged to be matrix biased. The result for that analyte in the unfortified sample is appropriately qualified and the issue of the results being suspect due to matrix effects documented in the case.

18.4. Extracted Internal Standards

18.4.1. The ES is the isotopically labeled analog of the analyte being analyzed, when available. It is spiked into all samples (except drinking waters) in the batch, including QC samples prior to extraction. The recovery is used to assess the effectiveness of the extraction procedure and matrix effects. Recoveries must be in the range of 50-150% of the true value.

18.4.2. If the recoveries are not within the control limits for the MB, LCS or LCSD, and/or samples, the following corrective actions are recommended:

18.4.2.1. The extract may be reanalyzed to determine if the out of control result is instrument related. If upon reanalysis the criterion is met, analysis and reporting of the associated samples may proceed.

18.4.2.2. If the %R is outside the control limits, all associated samples may be re-prepped and re-analyzed for that analyte (greater dilution may be needed). If there is insufficient sample volume remaining or if the holding time has expired for any associated sample, the client must be consulted prior to beginning re-extraction.

18.4.2.3. The sample can also be diluted and re-analyzed to remove the matrix interference. If this option is chosen, additional ES standard must be added to the diluted sample.

19.0 CALCULATIONS

19.1. Calculate response factors (RF) for each compound relative to one of the internal standards. The internal standard selected for the calculation of the RF for a compound is the internal standard that has a retention time closest to the compound being measured for drinking waters and the isotopically labeled analog

The RF is calculated as follows:

$$RF = (A_X C_{IS}) / (A_{IS} C_X)$$

Where:

A_X = Area of the characteristic ion for the compound being measured.

A_{IS} = Area of the characteristic ion for the specific internal standard.

C_{IS} = Concentration of the specific internal standard.

C_X = Concentration of the compound being measured.

19.2. The average RF must be calculated for each compound.

19.3. The percent relative standard deviation (%RSD) should be less than 20% for each analyte.

$$\%RSD = \frac{SD}{RF} x100$$

Where:

$\overline{RF}$ = Mean of RFs from initial calibration for a compound

SD = Standard deviation of RFs from initial calibration for a compound

$$SD = \sqrt{\sum_{c=1}^{n} \frac{\left(x_1 - \bar{x}\right)^2}{N-1}}$$

19.4. Continuing Calibration percent drift

$$\%Drift = \frac{C_{actual} - C_{found}}{C_{actual}} x100\%$$

Where:

C_{actual} = Known concentration in standard

C_{found} = Measured concentration using selected quantitation method

19.5. Concentration in the Extract

The concentration of each identified analyte in the extract is calculated by average response factor or a regression fit.

19.5.1. Linear fit

$$C_{ex} = A + B\frac{(R_X C_{IS})}{R_{IS}} \qquad C_{ex} = \frac{(R_X - A)}{B}$$

Where:

C_{ex} = Concentration in the extract
C_{is} = Concentration of the Internal Standard
R_x = Response for the analyte
R_{IS} = Response for the IS
A = Intercept
B = Slope

19.5.2. Quadratic Fit

Instrument data reduction software performs the quadratic equation that has been validated by the Quality department. For more information refer to the owner's manuals or contact the instrument's manufacturer.

19.5.3. Average response factor

$$C_{ex} = \frac{(A_X C_{IS})}{(A_{IS}\overline{RF})}$$

Where:

C_{ex} = Concentration in extract
C_{IS} = Concentration of internal standard
$\overline{RF}$ = Average RF
A_x = Area of the extract
A_{IS} = Area of the internal standard

19.5.3.1. Concentration Determination of an Aqueous Sample

$(ng/L) = (C_{ex})(V_t)(D_f)/(V_o)$

Where:

C_{ex} = Concentration found in the extract in ng/L
V_t = Final Volume of the extract in mL

D_f = Dilution Factor.

V_o = Volume of water extracted (mL).

19.5.3.2. Sediment/Soil Sludge (on a dry-weight basis) and Waste and Tissue (normally on a wet-weight or as received basis):

Concentration

$$(ng/Kg) = (C_{ex})(V_t)(D_f)/(W_s)(D)$$

Where:

C_{ex}, V_t, Ais, D_f = Same as for water matrix

Ws = Weight of sample in grams.

D = (100% - moisture in sample)/100, or 1 for wet-weight basis.

19.6. LCS Percent Recovery

$$LCS\ \text{Recovery} = \frac{SSR}{SA} X100$$

Where:

SSR = Spike Sample Result

SA = Spike added

19.7. MS/MSD Spike Recovery calculations

$$MatrixSpike\ \text{Recovery} = \frac{SSR - SR}{SA} X100$$

Where:

SSR = Spike Sample Result

SA = Spike Added

SR = Sample Result

19.8. Relative Percent Difference calculation for the MS/MSD or sample/sample duplicate.

$$RPD = \frac{MS_R - MSD_R}{\frac{1}{2}(MS_R + MSD_R)} X100$$

Where:

RPD = Relative Percent Difference

MS_R = Matrix Spike Result or Sample Spike Result

MSD_R = Matrix Spike Duplicate Result or Sample Spike Duplicate Result

20.0 INSTRUMENT PERFORMANCE REQUIREMENTS

See Appendix 1 for all criteria.

21.0 ANALYST AND METHOD VALIDATION REQUIREMENTS

21.1. To establish that the analyst can perform the procedures in an acceptable manner and that the method generates data of acceptable bias and precision, the following operations are performed.

21.1.1. A quality control (QC) check standard must be prepared containing each analyte of interest. It must be prepared from pure standard material or purchased as a certified solution. It must be made from a source independent of that used for calibration if commercially available.

21.1.2. Four samples must be prepared and analyzed by the same procedures used to prepare and analyze actual samples.

21.1.3. Calculate the average recovery (X) in ng/L, and the standard deviation of the recovery (S) in ng/L, for each analyte of interest using the four results.

21.1.4. For each analyte compare S and X with the corresponding acceptance criteria for precision and accuracy, respectively, given in the quality control table at the end of the method. If the S and X for all analytes of interest meet the acceptance criteria, the system's performance is acceptable and analysis of actual samples can begin. If any individual S and X exceeds the precision limits or falls out of the range for accuracy, the system's performance is unacceptable for that analyte and a check standard for that analyte must be prepared and reanalyzed.

21.2. Method detection limits are also determined and documented annually. Aqueous method detection limits and verifications are performed as stated in the GL-LB-E-001 for the Determination of Method Detection Limits. Typical MDLs for this method are 0.67 ng/L for waters and 166 ng/Kg for solids.

21.3. Minimum Reporting Limit (MRL) confirmation is determined for drinking water samples. A minimum of seven LRBs are fortified at the proposed MRL and extracted an analyzed per the EPA 537.1 method. See Appendix 5 for all calculations and acceptance criteria.

21.4. Precision and accuracy are matrix dependent and are documented by means of a laboratory control sample (LCS) and a matrix spike and matrix spike duplicate.

22.0 INSTRUMENT PROCEDURES AND QUALITY CONTROL

22.1. Before sample analysis begins, the instrument must have acceptable calibration curves. Each Curve is verified by the analysis of second source continuing calibration standards. Each standard must meet the acceptance criteria for percent difference or drift. Continuing calibration verification standards must be analyzed after every ten samples.

22.2. An instrument blank is analyzed daily prior to sample analysis. The concentration of each target must be ≤ ½ LOQ when analyzing all matrices except drinking waters. For drinking water samples, the acceptance criterion is ≤ 1/3 LOQ. Samples are not analyzed until the blank has met all acceptance criteria. .

22.3. Nonconformance

22.3.1. When analyzing a calibration curve for more than one analyte at a time, some of the analytes may not meet the acceptance criteria. Additional standards containing the compounds that were not acceptable may be analyzed. If the curve still does not meet the acceptance criteria, maintenance should be performed or a new standard may be needed.

22.3.2. If the continuing check standard fails any of the criteria, the analyst must take action to correct the situation. This may be performing any of the maintenance steps described in the appropriate SOP to get the instrument to meet its daily calibration. If all attempts fail, the analyst must analyze a new series of calibration standards, thus obtaining a new calibration curve.

22.3.3. If the standard analyzed after a group of samples exhibits a response for an analyte that is above the acceptance limit, and the analyte was not detected in the specific samples analyzed during the analytical shift, then the extracts for those samples do not need to be reanalyzed. If an analyte above the QC limits was detected in a sample extract, reinjection of the extract is necessary.

23.0 DATA RECORDING

Data are recorded and calculated by the instrument data acquisition system. They are stored on a remote server. The data are also entered into the Laboratory Information Management System (AlphaLIMS).

24.0 RECORDS MANAGEMENT

24.1. Documentation of Training

Extraction technicians and analysts must be properly trained to perform the contents of this SOP. Personnel will extract or analyze four laboratory control samples for this SOP as training commences. Training is documented per GL-HR-E-003 for Maintaining Technical Training Records.

24.2. Documentation of Extraction

24.2.1. In AlphaLIMS, complete the Sample Tracker Form. Record initial weight of the sample, final volume of the extract, amount of ES and spikes added, and any comments about the extraction process. Also, record all reagent lot numbers and note any deviation from this standard operating procedure.

24.2.2. Print a hard copy to submit with the extracts

24.2.3. Have the batch peer reviewed.

24.2.4. All documents are stored in AlphaLIMS.

24.3. Documentation of Standards

Refer to GL-LB-E-007 for Laboratory Standards Documentation.

25.0 DATA REVIEW, APPROVAL AND TRANSMITTAL FOR EXTRACTION AND ANALYTICAL GROUPS

25.1. Extraction Group: A review process is used to ensure the quality of the data. Extraction logs are peer reviewed by a second technician or Group Leader. When the reviewer is satisfied that the data have been entered correctly, a data report is generated from AlphaLIMS. The report along with the batch sheets are copied and submitted to the appropriate analytical area for analysis.

25.2. Analytical Group

25.2.1. Levels of review and their responsibilities

25.2.1.1. First level review - The analyst must check all chromatograms. They will also ensure that the percent recoveries for the spike, duplicate, and all IS recoveries are acceptable. They must check to see if the check standard passes for the analytes of interest. The peak's shape must also be checked to ensure that it is indeed a peak and not noise. If the detect meets all requirements (see Appendix 1 for DOD QSM criteria), its concentration should be reported. Upon completion of a batch, the analyst enters the data. A data report is generated and it is placed in a folder along with the batch and all other raw data (chromatograms). This folder is given to the peer review analyst for reviewing.

25.2.1.2. Second level review - The data validator or other qualified reviewer must ensure that the concentration that appears in the external standards table is indeed what has been entered into the computer. They must check to see if the calibration check standard is acceptable and if the LCS and its duplicate and the ISs are all within acceptable ranges. The reviewer must also

check the date analyzed, dilution factor, and time of analysis from the raw data against the data report. If everything is acceptable, the reviewer must then initial and date the data report. The data report is then sent to a status of "Done."

25.2.1.3. To complete a review process, all chromatograms of the calibration check standard, blank, LCS and its duplicate, samples and the spike and/or duplicates of the sample must be present. The batch sheet, sample tracker log, and the data report should all be present.

25.2.1.4. Refer to SOP GL-OA-E-044, Organics Data Validation for additional information on validation.

26.0 METHOD VERIFICATION AND VARIATION

26.1. This procedure deviates from EPA 537.1 for waste water and solid analysis only by using Isotope Dilution to calculate analyte concentrations in place of internal standards. We refer to these isotopes as Extraction Internal Standards. The Extracted Internal Standards are added to the samples and QC prior to extraction in place of surrogates.

26.2. This procedure deviates from EPA 537.1 for waste water and solid analysis only by using 80:20 methanol/water (vol/vol) as the final solvent for standards and samples instead of the method prescribed 96:4 methanol/water solvent when extracting samples other than drinking waters.

26.3. The Method Detection Limit studies were performed in accordance with GL-LB-E-001for the Determination of Method Detection Limits.

26.4. Per EPA 537.1, the IS must deviate no more than ± 50% of the initial calibration and must fall within 70% - 140% of the most recent CCV. This criterion is used for drinking water s

27.0 REFERENCES

27.1. EPA Method 537.1 Determination of Selected Perfluorinated alkyl acids in drinking water by Solid Phase Extraction and liquid chromatography/tandem mass spectrometry (LC/MC/MS) Version 1.1, September 200.9 National Exposure Research Laboratory Office of Research and Development U. S. Environmental Protection agency Cincinnati, Ohio 45268.

27.2. ISO (2009)ISO 25101:2009(E)Water Quality- Determination of perfluorooctanesulfonate (PFOS) and perfluorooctonate (PFOA)-Method for unfiltered sample using solid phase extraction and liquid chromatography/mass spectrometry. First Edition 2009-03-01.

27.3. ASTM D7979-17 Standard Test Method for Determination of Per- and Polyfluoroalkyl Substances in Water, Sludge, Influent, Effluent and Wastewater by Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS).

27.4. ASTM D7968-17a Standard Test Method for Determination of Perfluorinated Compounds in Soil by Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS).

27.5. Dept. of Defense (DOD), Dept. of Energy (DOE) Consolidated Quality Systems Manual (QSM) for Environmental Laboratories DOD QSM Version 5.3, May, 2019.

27.6. Dept. of Defense (DOD) Bottle Selection and other Sampling Considerations When Sampling for Per-and Poly-Fluoroalkyl Substances (PFASs), Rev. 1.1, Oct 2016.

27.7. EPA Method 537.1 Version 1.0. Determination of Selected Perfluorinated alkyl acids in drinking water by Solid Phase Extraction and liquid chromatography/tandem mass spectrometry (LC/MC/MS), November 2018 National Exposure Research Laboratory Office of Research and Development U. S. Environmental Protection agency Cincinnati, Ohio 45268.

28.0 WASTE MANAGEMENT

Laboratory waste is disposed in accordance with the Laboratory Waste Management Plan, GL-LB-G-001

29.0 HISTORY

Revision 9: Updated to use TNI nomenclature for analytes of interest. Added Instrument Synonym column in Table 1.

Revision 8: Updated for new compounds and clarifications for state agencies.

Revision 7: Updated for DoD QSM Version 5.3 changes and added Appendix 9 and 10.

Revision 6: Revised to appropriately reference Appendices 7 and 8. Update Appendix header for consistency. Revised standards expiration date usage.

Revision 5: Added Calibration Concentration Chart (Appendix 7) and added new compounds and Appendix 8. Added tissue as additional matrix for analysis.

APPENDIX 1: DOD/DOE QSM 5.3 QC REQUIREMENTS

Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Aqueous Sample Preparation	Each sample and associated batch QC samples.	Solid Phase Extraction (SPE) must be used unless samples are known to contain high PFAS concentrations (e.g., AFFF formulations). Inline SPE is acceptable. Entire sample plus bottle rinsate must be extracted using SPE. Known high PFAS concentration samples require serial dilutions be performed in duplicate. Samples of known high PFAS concentrations can be prepared by serial dilution instead of SPE.	NA.	NA.	Samples with >1% solids may require centrifugation prior to SPE extraction. Pre-screening of separate aliquots of aqueous samples is recommended.
Solid Sample Preparation	Each sample and associated batch QC samples.	Entire sample received by the laboratory must be homogenized prior to subsampling	NA.	NA.	NA.
Biota Sample Preparation	Each sample and associated batch QC samples.	Sample prepared as defined by the project (e.g., whole fish versus filleted fish).	NA.	NA.	NA.
AFFF and AFFF Mixture Sample Preparation	Each sample and associated batch QC samples.	Each field sample must be prepared in duplicate (equivalent to matrix duplicate).	NA.	NA.	Adsorption onto bottle is negligible compared to sample concentration so subsampling is allowed. Mulitple dilutions will most likely have to be reported in order to achieve the lowest LOQ possible for each analyte.
Sample Cleanup Procedure	Each sample and associated batch QC samples. Not applicable to AFFF formulation samples.	ENVI-Carb™ or equivalent must be used on each sample and batch QC sample.	NA.	Flagging is not appropriate.	Cleanup should reduce bias from matrix background.

Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Mass Calibration	Instrument must have a valid mass calibration prior to any sample analysis. Mass calibration is verified after each mass calibration, prior to initial calibration (ICAL).	Calibrate the mass scale of the MS with calibration compounds and procedures described by the manufacturer. Mass calibration range must bracket the ion masses of interest. The most recent mass calibration must be used for every acquisition in an analytical run. Mass calibration must be verified to be ±0.5 amu of the true value, by acquiring a full scan continuum mass spectrum of a PFAS stock standard.	If the mass calibration fails, then recalibrate. If it fails again, consult manufacturer on corrective maintenance	Flagging is not appropriate.	Problem must be corrected. No samples may be analyzed under failing mass calibration. The Mass calibration is updated on a as-needed basis (e.g., QC failures, ion masses fall outside of the ±0.5 amu of the true value, major instrument maintenance is performed, or the instrument is removed).
Mass Spectral Acquisition Rate	Each analyte, Extracted Internal Standard (EIS) Analyte	A minimum of 10 spectra scans are acquired across each chromatographic peak.	NA.	Flagging is not appropriate.	NA.
Calibration, Calibration Verification, and Spiking Standards	All analytes.	Standards containing both branched and linear isomers must be used when commercially available. PFAS method analytes may consist of both branched and linear isomers, by quantitative standards that contain the linear and branched isomers do not exist for all method analytes. For PFAS that do not have a quantitative branched and linear standard, identify the branched isomers by analyzing a qualitative standard that includes both linear and branched isomers and determine retention times, transitions and transition ion ratios. Quantitate samples by integrating the total response (i.e. accounting for peaks that	NA.	Flagging is not appropriate.	Standards containing both branched and linear isomers are to be used during method validation and when reestablishing retention times, to ensure the total response is quantitated for that analyte. Technical grade standards cannot be used for quantitative analysis.

Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
		are identified as linear and branched isomers) and relying on the initial calibration that uses the linear isomer quantitative standard.			
Sample PFAS Identification	All analytes detected in a sample.	The chemical derivation of the ion transitions must be documented. A minimum of two ion transitions (Precursor → quant ion and precursor → confirmation ion) and the ion transitions ratio per analyte are required for confirmation. Exception is made for analytes where two transitions do not exist (PFBA and PFPeA). Documentation of the primary and confirmation transitions and the ion ratio is required. In-house acceptance criteria for evaluation of ion rations must be used and must not exceed 50-150%. Signal to Noise Ratio (S/N) must be ≥ 3 for all ions used for confirmation. Quant ion and confirmation ion must be present and must maximize simultaneously (± 2 seconds).	NA.	PFAS identified with ion ratios that fail acceptance criteria must be flagged. Any quantitation ion peak that does not meet the maximization criteria shall be included in the summed integration and the resulting data flagged as "estimated, biased high".	For example: Ion Ratio = (quant ion abundance/confirm ion abundance) Calculate the average ratio (A) and standard deviation (SD) suing the ICAL standards. An acceptance range of ratio could be within A ±3SD for confirmation of detection.

Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Ion Transitions (Parent-> Product)	Every field sample, standard, blank, and QC sample.	In order to avoid biasing results high due to known interferences for some transitions, the following transitions must be used for the quantification of the following analytes: PFOA: 413 —› 369 PFOS: 499 —› 80 PFHxS: 399 —› 80 PFBS: 299 —› 80 4:2 FTS: 327 —› 307 6:2 FTS: 427 —› 407 8:2 FTS: 527 —› 507 NEtFOSAA: 584 —› 419 NMeFOSAA: 570 —› 419 If these transitions are not used, the reason must be technically justified and documented (e.g., alternate transition was used due to observed interferences).	NA.	Flagging is not appropriate.	NA.

Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration (ICAL)	At instrument set-up and after ICV or CCV failure, prior to sample analysis.	The isotopically labeled analog of an analyte (Extracted Internal Standard Analyte) must be used for quantitation if commercially available (Isotope Dilution Quantitation). Commercial PFAS standards available as salts are acceptable providing the measured mass is corrected to the neutral acid concentration. Results shall be reported as the neutral acid with appropriate CAS number. If a labeled analog is not commercially available, the Extracted Internal Standard Analyte with the closest retention time or chemical similarity to the analyte must be used for quantitation. (Internal Standard Quantitation) Analytes must be within 70-130% of their true value for each calibration standard. ICAL must meet one of the two options below: Option 1: The RSD of the RFs for all analytes must be ≤ 20%. Option 2: Linear or non-linear calibrations must have r^2 ≥0.99 for each analyte.	Correct problem, then repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until ICAL has passed. External Calibration is not allowed for any analyte. Calibration can be linear (minimum of 5 standards) or quadratic (minimum of 6 standards); weighting is allowed.
Retention Time window position establishment	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	Calculated for each analyte and EIS.

Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Retention Time (RT) window width	Every field sample, standard, blank, and QC sample	RT of each analyte and EIS analyte must fall within 0.4 minutes of the predicted retention times from the daily calibration verification or on days when ICAL is performed, from the midpoint standard of the ICAL. Anaytes must elute within 0.1 minutes of the associated EIS. This criterion applies only to analyte and labeled analog pairs	Correct problem and reanalyze samples	NA.	Calculated for each analyte and EIS.
Instrument Sensitivity Check (ISC)	Prior to analysis and at least once every 12 hours.	Analyte concentrations must be at LOQ; concentrations must be within ±30% of their true values.	Correct problem, rerun ISC. If problem persists, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until ISC has met acceptance criteria. ISC can serve as the initial daily CCV.

Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	Analyte concentrations must be within ±30% of their true value.	Correct problem, rerun ICV. If problem persists, repeat ICAL.	Flagging is not appropriate.	No samples shall be analyzed until calibration has been verified.
Continuing Calibration Verification (CCV)	Prior to sample analysis, after every 10 field samples, and at the end of the analytical sequence.	Concentration of analytes must range from the LOQ to the mid-level calibration concentration. Analyte concentrations must be within ±30% of their true value.	Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, or if two consecutive CCVs cannot be run, perform corrective action(s) and repeat CCV and all associated samples since last successful CCV. Alternately, recalibrate if necessary; then reanalyze all associated samples since the last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Results may not be reported without valid CCVs. Instrument Sensitivity Check (ISC) can serve as a bracketing CCV.
Instrument Blanks	Immediately following the highest standard analyzed and daily prior to sample analysis.	Concentration of each analyte must be ≤ ½ the LOQ. Instrument Blank must contain EIS to enable quantitation of contamination.	If acceptance criteria are not met after the highest calibration standard, calibration must be performed using a lower concentration for the highest standard until acceptance criteria is met. If sample concentrations exceed the highest allowed standard and the sample(s) following exceed this acceptance criteria (> ½ LOQ), they must be reanalyzed.	Flagging is only appropriate in cases when the sample cannot be reanalyzed and when there is no more sample left.	Note: sample shall be analyzed until instrument blank has met acceptance criteria. Note: Successful analysis following the highest standard analyzed determines the highest concentration that carryover does not occur. When the highest standard analyzed is not part of the calibration curve, it cannot be used to extend out the calibration range, it

Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
					is used only to document a higher concentration at which carryover still does not occur.

GEL Laboratories LLC
2040 Savage Road Charleston, SC 29407
P.O. Box 30712 Charleston, SC 29417
Main: 843.556.8171 Fax: 843.766.1178
www.gel.com

Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Extracted Internal Standard Analytes	Every field sample, standard, blank, and QC sample.	Added to solid sample prior to extraction. Added to aqueous samples, into the original container prior to extraction. For aqueous samples prepared by serial dilution instead of SPE, added to samples prior to analysis. Extracted Internal Standard Analyte recoveries must be within 50% to150% of the ICAL midpoint standard area or area measured in the initial CCV on days when an ICAL is not performed.	Correct problem. If required, re-extract and reanalyze associated field and QC samples. If recoveries are acceptable for QC samples, but not field samples, the field samples must be re-extracted and reanalyzed (greater dilution may be needed). Samples may be re-extracted and reanalyzed outside of hold times, as necessary for corrective action associated with QC failure.	Apply Q-flag and discuss in the Case Narrative only if reanalysis confirms failures in exactly the same manner.	Failing analytes shall be thoroughly documented in the Case Narrative. EIS should be 96% (or greater) purity. When the impurity consists of the unlabeled analyte, the EIS can result in a background artifact in every sample, standard and blank, if the EIS is fortified at excessive concentrations.
Method Blank (MB)	One per preparatory batch.	No analytes detected > ½ LOQ or > 1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater.	Correct problem. If required, re-extract and reanalyze MB and all QC samples and field samples processed with the contaminated blank. Samples may be re-extracted and reanalyzed outside of the hold times, as necessary for corrective action associated with QC failure. Examine the project-specific requirements. Contact the client as to additional measures to be taken.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid MB. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Laboratory Control Sample (LCS)	One per preparatory batch.	Blank spiked with all analytes at a concentration ≥ LOQ and ≤ the mid-level calibration concentration. A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Correct problem, then re-prep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes if sufficient sample material is available. Samples may be re-extracted and analyzed outside of hold times, as necessary for corrective action associated with QC failure. Examine the project-specific requirements. Contact the client as to additional measures to be taken.	If reanalysis cannot be performed, data must be qualified and explained in the Case Narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike	One per preparatory batch. Not required for aqueous samples prepared by serial dilution instead of SPE.	Sample spiked with all analytes at a concentration ≥ LOQ and ≤ the mid-level calibration concentration. A laboratory must use the DoD/DOE QSM Appendix C Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified. If the analyte(s) are not listed, use in-house LCS limits if project limits are not specified.	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	For matrix evaluation only. If MS results are outside the limits, the data shall be evaluated to determine the source(s) of difference (i.e., matrix effect or analytical error).

Table B-15. Per- and Polyfluoroalkyl Substances (PFAS) Using Liquid Chromatography Tandem Mass Spectrometry (LC/MS/MS) With Isotope Dilution or Internal Standard Quantification in Matrices Other Than Drinking Water

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	For MSD: One per preparatory batch. For MD: Each aqueous sample prepared by serial dilution instead of SPE.	For MSD: Sample spiked with all analytes at a concentration ≥ LOQ and ≤ the mid-level calibration concentration. A laboratory must use the DoD/DOE QSM Limits for batch control if project limits are not specified. If the analyte(s) are not listed, use in0house LCS limits if project limits are not specified. RPD ≤ 30 % (between MS and MSD or sample and MD).	Examine the project-specific requirements. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met and explain in the Case Narrative.	The data shall be evaluated to determine the source of difference. For Sample/MD: RPD criteria only apply to analytes whose concentration in the sample is greater than or equal to the LOQ. The MD is a second aliquot of the field sample that has been prepared by serial dilution.
Post Spike Sample	Only applies to aqueous samples prepared by serial dilution instead of SPE that have reported value of "<LOQ" for analyte(s).	Spike all analytes reported as <LOQ in to the dilution that the result for that analyte is reported from. The spike must be at the LOQ concentration to be reported for this sample a <LOQ. When analyte concentrations are calculated as < LOQ, the post spike for that analyte must recover within 70-130% of its true value.	When analyte concentrations are calculated as < LOQ, and the spike recovery does not meet the acceptance criteria, the sample, sample duplicate, and post spike sample must be reanalyzed at consecutively higher dilutions until the criteria is met.	Flagging is not appropriate.	When analyte concentrations are calculated as <LOQ, results may not be reported without acceptable post spike recoveries.

APPENDIX 2: WATER EXTRACTION

1.0 Setup:

1.1. Weigh the sample container to the nearest 1 g.

1.2. Shake the container for several minutes, with the cap tightly sealed, to ensure that any particulate matter is evenly distributed.

1.3. Prepare a Method Blank (MB) and a Laboratory Control Sample (LCS) from a 250 mL volume of organic-free reagent water, or a volume of reagent water similar to that being used for the samples in HDPE containers.

1.4. Add 125 uL of a 10 ug/L ES Standard to the samples in their original containers, the MB, LCS, and any Matrix Spikes associated to the extraction batch.

Note: If these are drinking water samples, use 125 uL of the 10 ug/L IS standard.

1.5. Add 250 uL of a 20 ug/L Spike intermediate standard to the LCS, MS and MSD in their original containers.

1.6. Shake the samples then allow to stand for several minutes. This allows the surrogate and spikes time to disperse in the samples, and will allow any particulate matter to settle.

2.0 Cartridge Conditioning:

2.1. Attach the SPE cartridge to the top of the manifold.

2.2. Fill the cartridge with 4 mL of ammonia/methanol solution. Pull the solvent through the sorbent bed. Do **not** allow the cartridge to go dry.

2.3. With a thin layer of solvent above the sorbent bed, add 4 mL of methanol. Allow the methanol to flow through the sorbent bed.

2.4. 4 mL of water is then added. Allow the water to flow through the sorbent bed. Stop the flow just before the cartridge goes dry. Close the manifold valve.

2.5. Dispose the discarded rinse solvents in the appropriate waste drum.

3.0 Sample Loading onto SPE Cartridge:

3.1. Attach a tube adapter with a polyethylene sample reservoir line to the top of the cartridge. Insert weighted end into sample container.

3.2. Engage the vacuum system to draw the sample through the cartridge a rate of approximately 10mL/min, until all of the sample has passed through the cartridge. Do **not** allow the cartridge to go dry until all of the sample has passed through it. If a cartridge runs dry, the preparation technician will notify the Group leader, and place a notation in the extraction log.

APPENDIX 2: WATER EXTRACTION - CONTINUED

3.3. Once all of the sample has loaded, rinse the bottle with 4 mL of reagent water. Allow the reagent water to pass through the transfer tubes onto the cartridge. Allow the cartridge to go dry at the end of the transfer. Discard this eluate.

3.4. Rinse the bottle with 4 mL of acetate buffer and transfer to the dried cartridge via the transfer lines. Apply a vacuum to completely remove the residual solution from the cartridge allowing the cartridge to go dry at the end of the transfer. This eluate is also discarded.

4.0 Sample Elution from the SPE Cartridge:

4.1. Place a collection tube under the cartridge in the vacuum manifold in which to collect the sample extract.

4.2. Connect a methanol rinsed ENVI-Carb cartridge to each sample cartridge prior to elution.

4.3. Rinse the bottle with 4 ml of 0.1 % ammonia/methanol. Elute the analytes from the cartridge by pulling this solution through the transfer lines onto the cartridge at a rate of one drop/second.

4.4. Rinse the bottle with 4 ml of Acetic Acid/methanol. Elute the cartridge by pulling this solution through the transfer lines onto the cartridge at a rate of one drop/second.

4.5. Elute the cartridge with 2 mL of Methanol.

4.6. Allow the cartridge to go dry.

4.7. Reweigh the empty sample bottle with its original cap and calculate the net mass of sample, to the nearest 1 g, from the difference in weight. Assuming a density of 1 g/ml, the value of the net mass (in grams) is equivalent to the volume (in milliliters) of the water used in the extraction.

5.0 Concentration

5.1. Evaporate the eluate with a gentle stream of nitrogen gas at 50 ± 5 ºC to a final volume of 4 mL.

5.2. Adjust the final volume to 5 mL with 1 mL of reagent water for all liquids except drinking water. Adjust the final volume to 5 mL with 0.8 mL of methanol and 0.2 mL of water for drinking water.

6.0 Extract Preparation

6.1. Label an autosampler vial with each sample number including batch QC.

6.2. Add 200 uL of each extract to the vial.

6.3. Add 20 uL of Injection Internal standard to each vial.

APPENDIX 2: WATER EXTRACTION -CONTINUED

Note: For Drinking water samples, use 20 uL of the Extraction Internal Standard.

6.4. Cap the vial and vortex. The extract is now ready for analysis.

APPENDIX 3: SOLIDS EXTRACTION

1.0 Setup:

1.1. Perform daily calibration verification of the open top balance.
1.2. Homogenize the sample following procedure GL-LB-E-029.
1.3. Weigh 2.0 +/- 0.5g of sample in a 50 mL tube. Ottawa sand or prep analyzed fish tissue is used for the MB and LCS QC sample.
1.4. Add 250 µL of the ES standard to each sample and QC.
1.5. Add 500 µL of the spike intermediate standard to LCS, LCS DUP, matrix spike, and matrix spike DUP.
1.6. Add 5 mL (10 mL for tissue samples) of 0.1% Ammonia/Methanol to each vial.
1.7. Cap and gently shake to mix contents.
1.8. Shake the samples on a wrist action shaker for 30 minutes.
1.9. Remove the samples from the shaker and decant the liquid portion into a clean 50 mL tube.
1.10. Add 2.5 mL (5 mL for tissue samples) of 0.1% Ammonia/Methanol and return the sample to the shaker. Shake an additional 15 minutes.
1.11. Decant the sample into the 50 mL tube.
1.12. Repeat step 1.10 -1.11 once more.
1.13. Centrifuge the extract for 5 minutes.

Please note: The final volume for soil samples is 10 mL. The final volume for tissue is 20 mL.

2.0 ENVI-Carb Cartridge Conditioning:

2.1. Attach the ENVI-Carb SPE cartridge to the top of the manifold.
2.2. Fill the cartridge with 4 mL of methanol. Pull the solvent through the sorbent bed.
2.3. Dispose the discarded rinse solvents in the appropriate waste drum.

3.0 Sample Loading onto SPE Cartridge:

3.1. Place a collection tube under the cartridge in the vacuum manifold in which to collect the sample extract.
3.2. Add 5 mL of the centrifuged extract to the pre-rinsed cartridge.
3.3. Allow the sample to pass through the cartridge under the flow of gravity.
3.4. Rinse the cartridge with 4 mL of Acetic acid/methanol.
3.5. The sample is now ready for concentration.

APPENDIX 3: SOLIDS EXTRACTION-CONTINUED

4.0 Concentration

4.1. Evaporate the eluate with a gentle stream of nitrogen gas at 50 ± 5 ºC to a final volume of 4 mL.

4.2. Adjust the final volume to 5 mL with water.. The extract is now ready for analysis.

5.0 Extract Preparation

5.1. Label an autosampler vial with each sample number including batch QC.

5.2. Add 200 uL of each extract to the vial.

5.3. Add 20 uL of Injection Internal standard to each vial.

5.4. Cap the vial and vortex. The extract is now ready for analysis

APPENDIX 4: Aqueous Film Forming Foam (AFFF) Samples

1.0 Setup:

1.1. Weigh the sample container to the nearest 1 g.

1.2. Shake the container for several minutes, with the cap tightly sealed, to ensure that any particulate matter is evenly distributed.

1.3. Weigh out three separate aliquots of each sample (usually 0.5 - 1 mL) in HDPE containers. **Note: A sample duplicate (DUP) and matrix spike (MS) must be analyzed with each sample.**

1.4. Prepare a Method Blank (MB) and a Laboratory Control Sample (LCS) from a 1 mL volume of organic-free reagent water, or a volume of reagent water similar to that being used for the samples in HDPE containers.

1.5. Add 125 uL of a 10 ug/L ES Standard to the samples, the MB, LCS, and any Matrix Spikes associated to the extraction batch.

1.6. Add 250 uL of a 20 ug/L Spike intermediate standard to the LCS only.

1.7. Add 25 uL of a 20 ug/L Spike intermediate standard to the MS only. **Note: The MS must be spiked at the LOQ for all non-detected analytes.**

1.8. Add 3.875 mL of Methanol to the samples and method blank.

1.9. Add 3.625 mL of Methanol to the LCS and 3.850 mL of Methanol to the MS and MSD.

1.10. Cap the vial and vortex. The extract is now ready for analysis

2.0 Extract Preparation

2.1. Label an autosampler vial with each sample number including batch QC.

2.2. Add 200 uL of each extract to the vial.

2.3. Add 20 uL of Injection Internal standard to each vial.

2.4. Cap the vial and vortex. The extract is now ready for analysis.

APPENDIX 5: INITIAL DEMONSTRATION OF CAPABILITY QUALITY CONTROL REQUIREMENTS FOR DRINKING WATERS

Requirement	Specification and Frequency	Acceptance Criteria
Initial Demonstration of Low System Background	Analyze LRB prior to any other IDC steps.	All method analytes must be below 1/3 the . Must also ensure that possible interferences from extraction
Initial Demonstration of Precision (IDP)	Analyze four to seven replicate LFBs fortified near the midrange calibration concentration.	%RSD must be <20%
Initial Demonstration of Accuracy (IDA)	Calculate average recovery for replicates used in IDP.	Mean recovery ± 30% of true value
Initial Demonstration of Peak Asymmetry Factor	Calculate the peak asymmetry factor using the equation in Section 15.2.4 for the first two eluting chromatographic peaks in a mid-level CAL standard.	Peak asymmetry factor of 0.8 - 1.5
Minimum Reporting Limit (MRL) Confirmation	Fortify, extract and analyze seven replicate LFBs at the proposed MRL concentration. Calculate the Mean and the Half Range (HR). Confirm that the upper and lower limits for the Prediction Interval of Result (Upper PIR, and Lower PIR) meet the recovery criteria.	Upper PIR ≤150% Lower PIR ≥ 50%

APPENDIX 5: INITIAL DEMONSTRATION OF CAPABILITY QUALITY CONTROL REQUIREMENTS FOR DRINKING WATERS -CONTINUTED

Requirement	Specification and Frequency	Acceptance Criteria
Quality Control Sample (QCS)	Analyze a standard from a second source, as part of IDC.	Results must be within 70-130% of true value.
Detection Limit (DL) Determination (optional)	Over a period of three days, prepare a minimum of seven replicate LFBs fortified at a concentration estimated to be near the DL. Analyze the replicates through all steps of the analysis. Calculate the DL using the equation in Sect. 15.2.7	Data from DL replicates are not required to meet method precision and accuracy criteria. If the DL replicates are fortified at a low enough concentration, it is likely that they will not meet precision and accuracy criteria.

APPENDIX 6: ONGOING QUALITY CONTROL REQUIREMENTS (SUMMARY) FOR DRINKING WATERS

Requirement	Specification and Frequency	Acceptance Criteria
Sample Holding Time	14 days with appropriate preservation and storage as described in Section 11.0.	Sample results are valid only if samples are extracted within the sample holding time.
Extract Holding Time	28 days when stored at room temperature in centrifuge tubes.	Extract results are valid only if extracts are analyzed within the extract holding time.
Laboratory Reagent Blank (LRB)	With each extraction batch of up to 20 samples, whichever is more frequent.	Demonstrate that all method analytes are below 1/3 the MRL, and confirm that possible interferences do not prevent quantification of method analytes. If targets exceed 1/3 the MRL or if interferences are present, results for these analytes in the extraction batch are invalid.
Laboratory Fortified Blank (LFB)	Analyze at least one LFB daily or one for each extraction batch of up to 20 Field Samples. Rotate the fortified concentrations between low, medium and high amounts.	Results of LFB analyses must be 70-130% of the true value for each method analyte for all fortified concentrations except the lowest CAL point. Results of the LFBs corresponding to the lowest CAL point for each method analyte must be 50-150% of the true value.

APPENDIX 6: ONGOING QUALITY CONTROL REQUIREMENTS (SUMMARY) FOR DRINKING WATERS-CONTINUED

Requirement	Specification and Frequency	Acceptance Criteria
Internal Standard (IS)	Internal standards, $^{13}C_4$-PFBA, $^{13}C_5$-PFPeA, $^{13}C_3$-PFBS, $^{13}C_5$-PFHxA, $^{13}C_2$-4:2 FTS, $^{13}C_4$-PFHpA, $^{13}C_3$-PFHxS, $^{13}C_8$-PFOA, $^{13}C_2$-6:2 FTS, $^{13}C_9$-PFNA, $^{13}C_8$-PFOS, $^{13}C_8$-PFOSA, $^{13}C_6$-PFDA, $^{13}C_2$-8:2 FTS, $^{13}C_7$-PFUdA, d_3-NMeFOSAA, d_5-NEtFOSAA, $^{13}C_2$-PFDoA,$^{13}C_2$-PFTeDA, MFHEA, MFOEA and MFDEA are added to all standards and sample extracts, including QC samples just prior to analysis. Compare IS areas to the average IS area in the initial calibration and to the most recent continuing calibration standard.	Peak area counts for all ISs in all injections must be within ± 50% of the average peak area calculated during the initial calibration and 70-140% from the most recent CCV If ISs do not meet this criterion, corresponding target results are invalid.
Surrogate Standards	Surrogate standards, $^{13}C_2$-PFOA, $^{13}C_2$-PFDA, $^{13}C_4$-PFOS and $^{13}C_3$-PFBA, are added to all CAL standards and samples, including QC samples. Calculate the surrogate recoveries.	Surrogate recoveries must be 70-130% of the true value. If a SUR fails this criteria, report all results for sample as suspect/surrogate recovery.

APPENDIX 6: ONGOING QUALITY CONTROL REQUIREMENTS (SUMMARY) FOR DRINKING WATERS-CONTINUED

Requirement	Specification and Frequency	Acceptance Criteria
Laboratory Fortified Sample Matrix (LFSM)	Analyze one LFSM per extraction batch (20 samples or less) fortified with method analytes at a concentration close to but greater than the native concentration, if known. Calculate LFSM recoveries.	Recoveries at mid and high levels should be within 70-130% and within 50-150% at the low-level fortified amount (near the MRL). If these criteria are not met, results are labeled suspect due to matrix effects.
Laboratory Fortified Sample Matrix Duplicate (LFSMD) or Field Duplicates (FD)	Extract and analyze at least one FD or LFSMD with each extraction batch (20 samples or less). A LFSMD may be substituted for a FD when the frequency of detects are low. Calculate RPDs.	Method analyte RPDs for the LFMD or FD should be ≤30% at mid and high levels of fortification and ≤50% near the MRL. If these criteria are not met, results are labeled suspect due to matrix effects.
Field Reagent Blank (FRB)	Analysis of the FRB is required only if a Field Sample contains a method analyte or analytes at or above the MRL. The FRB is processed, extracted and analyzed in exactly the same manner as a Field Sample.	If the method analyte(s) found in the Field Sample is present in the FRB at a concentration greater than 1/3 the MRL, then all samples collected with that FRB are invalid and must be recollected and reanalyzed.
Peak Asymmetry Factor	Calculate the peak asymmetry factor for the first two eluting chromatographic peaks in a mid-level CAL standard every time a calibration curve is generated.	Peak asymmetry factor of 0.8 - 1.5
Quality Control Sample (QCS)	Analyze at least quarterly or when preparing new standards, as well as during the IDC.	Results must be within 70-130% of true value.
Initial Calibration	Use IS calibration technique to generate a first or second order calibration curve forced through zero. Use at least five standard concentrations. Check the calibration curve as described in Sect. 17.4.	When each CAL standard is calculated as an unknown using the calibration curve, the analyte results should be 70-130% of the true value for all except the lowest standard, which should be 50-150% of the true value. Recalibration is recommended if these criteria are not met.

APPENDIX 6: ONGOING QUALITY CONTROL REQUIREMENTS (SUMMARY) FOR DRINKING WATERS-CONTINUED

Requirement	Specification and Frequency	Acceptance Criteria
Continuing Calibration Check (CCV)	Verify initial calibration by analyzing a low level (at the MRL or below) CCV prior to analyzing samples. CCVs are then injected after every 10 samples and after the last sample, rotating concentrations to cover the calibrated range of the instrument.	Recovery for each analyte and SUR must be within 70-130% of the true value for all but the lowest level of calibration. Recovery for each analyte in the lowest CAL level CCV must be within 50-150% of the true value and the surrogate must be within 70-130% of the true value.

APPENDIX 7: PFCS CALIBRATION CONCENTRATIONS FOR GROUNDWATER

Groundwater Calibration Chart	Level 1 (ng/L)	Level 2 (ng/L)	Level 3 (ng/L)	Level 4 (ng/L)	Level 5 (ng/L)	Level 6 (ng/L)	Level 7 (ng/L)	Level 8 (ng/L)	Level 9 (ng/L)
N-Ethyl perfluorooctanesulfonamidoacetic acid (NEtFOSAA)	100	200	500	750	1000	1500	2000	5000	10000
N-Methyl Perfluorooctanesulfonamidoacetic acid (NMeFOSAA)	100	200	500	750	1000	1500	2000	5000	10000
Perfluorobutanoic acid (PFBA)	100	200	500	750	1000	1500	2000	5000	10000
Perfluorodecanoic acid (PFDA)	100	200	500	750	1000	1500	2000	5000	10000
Perfluorododecanoic acid (PFDoA)	100	200	500	750	1000	1500	2000	5000	10000
Perfluoroheptanoic acid (PFHpA)	100	200	500	750	1000	1500	2000	5000	10000
Perfluorohexanoic acid (PFHxA)	100	200	500	750	1000	1500	2000	5000	10000
Perfluoropentanoic acid (PFPeA)	100	200	500	750	1000	1500	2000	5000	10000
Perfluorononanoic acid (PFNA)	100	200	500	750	1000	1500	2000	5000	10000
Perfluorooctanesulfonamide (PFOSAm)	100	200	500	750	1000	1500	2000	5000	10000
Perfluorooctane sulfonic acid (PFOS)	100	200	500	750	1000	1500	2000	5000	10000
Perfluorooctanoic acid (PFOA)	100	200	500	750	1000	1500	2000	5000	10000
Perfluorotetradecanoic acid (PFTDA)	100	200	500	750	1000	1500	2000	5000	10000
Perfluorotridecanoic acid (PFTrDA)	100	200	500	750	1000	1500	2000	5000	10000
Perfluoroundecanoic acid (PFUnDA)	100	200	500	750	1000	1500	2000	5000	10000
1H, 1H, 2H, 2H-perfluorohexane sulfonic acid (4:2 FTS)	93.5	187.0	467.5	701.3	935.0	1402.5	1870.0	4675	9350
1H, 1H, 2H, 2H-Perfluorooctane sulfonic acid (6:2 FTS)	95.0	190.0	475.0	712.5	950.0	1425.0	1900.0	4750	9500
1H, 1H, 2H, 2H-Perfluorodecane sulfonic acid (8:2 FTS)	96.0	192.0	480.0	720.0	960.0	1440.0	1920.0	4800	9600
Perfluorobutane sulfonic acid (PFBS)	88.5	177.0	442.5	663.8	885.0	1327.5	1770.0	4425	8850
Perfluorodecanesulfonate (PFDS)	96.5	193.0	482.5	723.8	965.0	1447.5	1930.0	4825	9650
Perfluoroheptane sulfonic acid (PFHpS)	95.0	190.0	475.0	712.5	950.0	1425.0	1900.0	4750	9500
Perfluorohexane sulfonic acid (PFHxS)	91.2	182.4	456.0	684.0	912.0	1368.0	1824.0	4560	9120
Perfluorononane sulfonic acid (PFNS)	96.0	192.0	480.0	720.0	960.0	1440.0	1920.0	4800	9600
Perfluoropentane sulfonic acid (PFPeS)	94.0	188.0	470.0	705.0	940.0	1410	1880.0	4700	9400
Hexafluoropropyleneoxide dimer acid (GenX) (HPFO-DA)	100	200	500	750	1000	1500	2000	5000	10000
Perfluoro-2-methoxyacetic acid (PFMOAA)	100	200	500	750	1000	1500	2000	5000	10000
Perfluoro-3-methoxypropanoic acid (PFMPA)	100	200	500	750	1000	1500	2000	5000	10000
Perfluoro-4-methoxybutanoic acid (PFMBA)	100	200	500	750	1000	1500	2000	5000	10000
Perfluoro(3,5-dioxahexanoic) acid (PFO2HxA)	100	200	500	750	1000	1500	2000	5000	10000
Perfluoro(3,5,7-trioxaoctanoic) acid (PFO3OA)	100	200	500	750	1000	1500	2000	5000	10000

Perfluoro(3,5,7,9-tetraoxadecanoic) acid (PFO4DA)	100	200	500	750	1000	1500	2000	5000	10000
Nafion Byproduct 1	100	200	500	750	1000	1500	2000	5000	10000
Nafion Byproduct 2	100	200	500	750	1000	1500	2000	5000	10000
Perfluorooctadecanoic acid (PFODA)	100	200	500	750	1000	1500	2000	5000	10000
Perfluorohexadecanoic acid (PFHxDA)	100	200	500	750	1000	1500	2000	5000	10000
4,8-Dioxa-3H-perfluorononanoic acid (DONA)	100	200	500	750	1000	1500	2000	5000	10000
N-Ethylperfluorooctane sulfonamidoethanol (EtFOSE)	100	200	500	750	1000	1500	2000	5000	10000
N-Ethylperfluorooctane sulfonamide (EtFOSAm)	100	200	500	750	1000	1500	2000	5000	10000
Perfluroethoxypropyl carboxylic acid (PEPA)	100	200	500	750	1000	1500	2000	5000	10000
N-Methylperfluorooctane sulfonamido ethanol (NMeFOSE)	100	200	500	750	1000	1500	2000	5000	10000
N-Methylperfluorooctane sulfonamide (N-MeFOSA)	100	200	500	750	1000	1500	2000	5000	10000
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9-Cl-PF3ONS)	93.2	186.4	466	699	932	1398	1864	4660	9320
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11-Cl-PF3OUdS)	94.2	188.4	471	706.5	942	1413	1884	4710	9342
Perfluoro-3,5,7,9,11-pentaoxadodecanoic acid (TAF) (PFO5DOA)	100	200	500	750	1000	1500	2000	5000	10000
4-(Heptafluoroisopropoxy)hexafluorobutanoic acid (PFECA-G)	100	200	500	750	1000	1500	2000	5000	10000
1H, 1H, 2H, 2H-Perfluorododecan sulfonic acid (10:2 FTS)	96.0	192.0	480.0	720.0	960.0	1440.0	1920.0	4800	9600
Perfluoro-1-dodecane sulfonic acid (PFDoS)	96.8	193.6	484	726	968	1452	1936	4840	9680
2-Perfluorohexyl Ethanoic Acid (FHEA)(6:2 FTA)	100	200	500	750	1000	1500	2000	5000	10000
2-Perfluorooctyl Ethanoic Acid (FOEA) (8:2 FTA)	100	200	500	750	1000	1500	2000	5000	10000
2-Perfluorodecyl Ethanoic Acid (FDEA)(10:2 FTA)	100	200	500	750	1000	1500	2000	5000	10000
4,4,5,5,6,6-Heptafluorohexanoic acid (3:3 FTCA)	100	200	500	750	1000	1500	2000	5000	10000
2H, 2H, 3H, 3H,-Perfluorooctanoic acid(5:3 FTCA)	100	200	500	750	1000	1500	2000	5000	10000

2H, 2H, 3H, 3H-Perfluorodecanoic acid (7:3 FTCA)	100	200	500	750	1000	1500	2000	5000	10000
MFHEA (EXTRACTED INTERNAL STANDARD)	250	250	250	250	250	250	250	250	250
MFOEA (EXTRACTED INTERNAL STANDARD)	250	250	250	250	250	250	250	250	250
MFDEA (EXTRACTED INTERNAL STANDARD)	250	250	250	250	250	250	250	250	250
$^{13}C_2$PFHxDA (EXTRACTED INTERNAL STANDARD)	250	250	250	250	250	250	250	250	250
d-N-EtFOSA (EXTRACTED INTERNAL STANDARD)	250	250	250	250	250	250	250	250	250
d_9-N-EtFOSE (EXTRACTED INTERNAL	250	250	250	250	250	250	250	250	250
d_7-N-MeFOSE (EXTRACTED INTERNAL	250	250	250	250	250	250	250	250	250
d-N-MeFOSA (EXTRACTED INTERNAL STANDARD)	250	250	250	250	250	250	250	250	250
$^{13}C_3$-PFPrOPrA (EXTRACTED INTERNAL STANDARD)	250	250	250	250	250	250	250	250	250
$^{13}C_4$-PFHpA (EXTRACTED INTERNAL STANDARD)	250	250	250	250	250	250	250	250	250
$^{13}C_8$-PFOSA (EXTRACTED INTERNAL STANDARD)	250	250	250	250	250	250	250	250	250
d_3-MeFOSAA (EXTRACTED INTERNAL STANDARD)	250	250	250	250	250	250	250	250	250
$_5$-EtFOSAA (EXTRACTED INTERNAL STANDARD)	250	250	250	250	250	250	250	250	250
$^{13}C_2$-PFDoA (EXTRACTED INTERNAL STANDARD)	250	250	250	250	250	250	250	250	250
$^{13}C_8$-PFOS (EXTRACTED INTERNAL STANDARD)	250	250	250	250	250	250	250	250	250
$^{13}C_3$-PFBS (EXTRACTED INTERNAL STANDARD)	250	250	250	250	250	250	250	250	250
$^{13}C_2$-4:2 FTS (EXTRACTED INTERNAL STANDARD)	250	250	250	250	250	250	250	250	250
$^{13}C_5$-PFHxA (EXTRACTED INTERNAL STANDARD)	250	250	250	250	250	250	250	250	250
$^{13}C_3$-PFHxS (EXTRACTED INTERNAL STANDARD)	250	250	250	250	250	250	250	250	250
$^{13}C_2$-6:2 FTS (EXTRACTED INTERNAL STANDARD)	250	250	250	250	250	250	250	250	250
$^{13}C_8$-PFOA (EXTRACTED INTERNAL STANDARD)	250	250	250	250	250	250	250	250	250
$^{13}C_9$-PFNA (EXTRACTED INTERNAL STANDARD)	250	250	250	250	250	250	250	250	250

APPENDIX 7: PFCS CALIBRATION CONCENTRATIONS FOR GROUNDWATER- CONTINUED

$^{13}C_2$-8:2 FTS (EXTRACTED INTERNAL STANDARD)	250	250	250	250	250	250	250	250	250
$^{13}C_6$-PFDA (EXTRACTED INTERNAL STANDARD)	250	250	250	250	250	250	250	250	250
$^{13}C_7$-PFUdA (EXTRACTED INTERNAL STANDARD)	250	250	250	250	250	250	250	250	250
$^{13}C_2$-PFTeDA (EXTRACTED INTERNAL STANDARD)	250	250	250	250	250	250	250	250	250
$^{13}C_4$-PFBA (EXTRACTED INTERNAL STANDARD)	250	250	250	250	250	250	250	250	250
$^{13}C_5$-PFPeA (EXTRACTED INTERNAL STANDARD)	250	250	250	250	250	250	250	250	250
$^{13}C_2$-PFDoA (EXTRACTED INTERNAL STANDARD)	250	250	250	250	250	250	250	250	250
$^{13}C_8$-PFOS (EXTRACTED INTERNAL STANDARD)	250	250	250	250	250	250	250	250	250
$^{13}C_3$-PFBS (EXTRACTED INTERNAL STANDARD)	250	250	250	250	250	250	250	250	250
$^{13}C_2$-4:2 FTS (EXTRACTED INTERNAL STANDARD)	250	250	250	250	250	250	250	250	250
$^{13}C_5$-PFHxA (EXTRACTED INTERNAL STANDARD)	250	250	250	250	250	250	250	250	250

APPENDIX 8: PFCS CALIBRATION CONCENTRATIONS FOR DRINKING WATER

Drinking Water Calibration Chart	Level 1 (ng/L)	Level 2 (ng/L)	Level 3 (ng/L)	Level 4 (ng/L)	Level 5 (ng/L)	Level 6 (ng/L)	Level 7 (ng/L)	Level 8 (ng/L)	Level 9 (ng/L)
N-Ethyl perfluorooctanesulfonamidoacetic acid (NEtFOSAA)	100	200	500	750	1000	1500	2000	5000	10000
N-Methyl perfluorooctanesulfonamidoacetic acid (NMeFOSAA)	100	200	500	750	1000	1500	2000	5000	10000
Perfluorobutanoic acid (PFBA)	100	200	500	750	1000	1500	2000	5000	10000
Perfluorodecanoic acid (PFDA)	100	200	500	750	1000	1500	2000	5000	10000
Perfluorododecanoic acid (PFDoA)	100	200	500	750	1000	1500	2000	5000	10000
Perfluoroheptanoic acid (PFHpA)	100	200	500	750	1000	1500	2000	5000	10000
Perfluorohexanoic acid (PFHxA)	100	200	500	750	1000	1500	2000	5000	10000
Perfluoropentanoic acid (PFPeA)	100	200	500	750	1000	1500	2000	5000	10000
Perfluorononanoic acid (PFNA)	100	200	500	750	1000	1500	2000	5000	10000
Perfluorooctanesulfonamide (PFOSAm)	100	200	500	750	1000	1500	2000	5000	10000
Perfluorooctane sulfonic acid (PFOS)	100	200	500	750	1000	1500	2000	5000	10000
Perfluorooctanoic acid (PFOA)	100	200	500	750	1000	1500	2000	5000	10000
Perfluorotetradecanoic acid (PFTDA)	100	200	500	750	1000	1500	2000	5000	10000
Perfluorotridecanoic acid (PFTrDA)	100	200	500	750	1000	1500	2000	5000	10000
Perfluoroundecanoic acid (PFUnDA)	100	200	500	750	1000	1500	2000	5000	10000
1H, 1H, 2H, 2H-Perfluorohexane sulfonic acid (4:2 FTS)	93.5	187.0	467.5	701.3	935.0	1402.5	1870	4675	9350
1H, 1H, 2H, 2H-Perfluorooctane sulfonic acid(6:2 FTS)	95.0	190.0	475.0	712.5	950.0	1425.0	1900	4750	9500
1H, 1H, 2H, 2H-Perfluorododecane sulfonic acid (8:2 FTS)	96.0	192.0	480.0	720.0	960.0	1440.0	1920	4800	9600
Perfluorobutane sulfonic acid (PFBS)	88.5	177.0	442.5	663.8	885.0	1327.5	1770	4425	8850
Perfluorodecane sulfonic acid (PFDS)	96.5	193.0	482.5	723.8	965.0	1447.5	1930	4825	9650
Perfluoroheptane sulfonic acid (PFHpS)	95.0	190.0	475.0	712.5	950.0	1425.0	1900	4750	9500
Perfluorohexane sulfonic acid (PFHxS)	91.2	182.4	456.0	684.0	912.0	1368.0	1824	4560	9120
Perfluorononane sulfonic acid (PFNS)	96.0	192.0	480.0	720.0	960.0	1440.0	1920	4800	9600
Perfluoropentane sulfonic acid (PFPeS)	94.0	188.0	470.0	705.0	940.0	1410.0	1880	4700	9400
Hexafluoropropyleneoxide dimer acid (HFPO-DA) (Gen-X)0HDOI(GenX) (PFPrOPrA)	100	200	500	750	1000	1500	2000	5000	10000

Perfluoro-2-methoxyacetic acid (PFMOAA)	100	200	500	750	1000	1500	2000	5000	10000
Perfluoro-3-methoxypropanoic acid (PFMPA)	100	200	500	750	1000	1500	2000	5000	10000
Perfluoro-4-methoxybutanoic acid (PFMBA)	100	200	500	750	1000	1500	2000	5000	10000
Perfluoro(3,5-dioxahexanoic) acid (PFO2HxA)	100	200	500	750	1000	1500	2000	5000	10000
Perfluoro(3,5,7-trioxaoctanoic) acid (PFO3OA)	100	200	500	750	1000	1500	2000	5000	10000
Perfluoro(3,5,7,9-tetraoxadecanoic) acid (PFO4DA)	100	200	500	750	1000	1500	2000	5000	10000
Nafion Byproduct 1	100	200	500	750	1000	1500	2000	5000	10000
Nafion Byproduct 2	100	200	500	750	1000	1500	2000	5000	10000
Perfluorooctadecanoic acid (PFODA)	100	200	500	750	1000	1500	2000	5000	10000
Perfluorohexadecanoic acid (PFHxDA)	100	200	500	750	1000	1500	2000	5000	10000
4,8-Dioxa-3H-perfluorononanoic acid (DONA)	100	200	500	750	1000	1500	2000	5000	10000
N-Ethylperfluorooctane sulfonamido ethanol (EtFOSE)	100	200	500	750	1000	1500	2000	5000	10000
N-Ethylperfluorooctane sulfonamide (EtFOSAm)	100	200	500	750	1000	1500	2000	5000	10000
Perfluoroethoxypropyl carboxylic acid (PEPA)	100	200	500	750	1000	1500	2000	5000	10000

APPENDIX 8: PFCS CALIBRATION CONCENTRATIONS FOR DRINKING WATER-CONTINUED

Drinking Water Calibration Chart	Level 1 (ng/L)	Level 2 (ng/L)	Level 3 (ng/L)	Level 4 (ng/L)	Level 5 (ng/L)	Level 6 (ng/L)	Level 7 (ng/L)	Level 8 (ng/L)	Level 9 (ng/L)
N-Methylperfluorooctane sulfonamideethanol (N-MeFOSE)	100	200	500	750	1000	1500	2000	5000	10000
N-methylperfluorooctane sulfonamide (N-MeFOSA)	100	200	500	750	1000	1500	2000	5000	10000
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9-Cl-PF3ONS)	93.2	186.4	466	699	932	1398	1864	4660	9320
11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11-Cl-PF3OUdS)	94.2	188.4	471	706.5	942	1413	1884	4710	9342
Perfluoro-3,5-7,9-11-pentaoxadodecanoic acid (PFO5ODA) (TAF)	100	200	500	750	1000	1500	2000	5000	10000
4-(Heptafluoroisopropoxy)hexafluorobutanoic acid (PFECA-G)	100	200	500	750	1000	1500	2000	5000	10000
1H, 1H, 2H, 2H-Perflourododecane sulfonic acid (10:2 FTS)	96.0	192.0	480.0	720.0	960.0	1440.0	1920.0	4800	9600
Perfluorododecanesulfonate (PFDoS)	96.8	193.6	484	726	968	1452	1936	4840	9680
2-Perfluorohexyl ethanoic acid (FHEA)(6:2 FTA)	100	200	500	750	1000	1500	2000	5000	10000
2-Perfluorooctyl ethanoic acid (FOEA)(8:2 FTS)	100	200	500	750	1000	1500	2000	5000	10000
2-Perfluorodecyl ethanoic acid (FDEA)(10:2 FTA)	100	200	500	750	1000	1500	2000	5000	10000
4,4,5,5,6,6-Heptafluorohexanoic acid (3:3 FTCA)	100	200	500	750	1000	1500	2000	5000	10000
2H, 2H, 3H, 3H-Perfluorooctanoic acid (5:3 FTCA)	100	200	500	750	1000	1500	2000	5000	10000
2H, 2H, 3H, 3H-Perfluorodecanoic acid (7:3 FTCA)	100	200	500	750	1000	1500	2000	5000	10000
MFHEA (INTERNAL STANDARD)	1000	1000	1000	1000	1000	1000	1000	1000	1000
MFOEA (INTERNAL STANDARD)	1000	1000	1000	1000	1000	1000	1000	1000	1000
MFDEA (INTERNAL STANDARD)	1000	1000	1000	1000	1000	1000	1000	1000	1000

$^{13}C_2$PFHxDA (INTERNAL STANDARD)	1000	1000	1000	1000	1000	1000	1000	1000	1000
d-N-EtFOSA (INTERNAL STANDARD)	1000	1000	1000	1000	1000	1000	1000	1000	1000
d_9-N-EtFOSE (INTERNAL STANDARD)	1000	1000	1000	1000	1000	1000	1000	1000	1000
d_7-N-MeFOSE (INTERNAL STANDARD)	1000	1000	1000	1000	1000	1000	1000	1000	1000
d-N-MeFOSA (INTERNAL STANDARD)	1000	1000	1000	1000	1000	1000	1000	1000	1000
$^{13}C_3$-PFPrOPrA (INTERNAL STANDARD)	1000	1000	1000	1000	1000	1000	1000	1000	1000
$^{13}C_4$-PFHpA (INTERNAL STANDARD)	1000	1000	1000	1000	1000	1000	1000	1000	1000
$^{13}C_8$-PFOSA (INTERNAL STANDARD)	1000	1000	1000	1000	1000	1000	1000	1000	1000
d_3-MeFOSAA (INTERNAL STANDARD)	1000	1000	1000	1000	1000	1000	1000	1000	1000
d_5-EtFOSAA (INTERNAL STANDARD)	1000	1000	1000	1000	1000	1000	1000	1000	1000
$^{13}C_2$-PFDoA (INTERNAL STANDARD)	1000	1000	1000	1000	1000	1000	1000	1000	1000
$^{13}C_8$-PFOS (INTERNAL STANDARD)	1000	1000	1000	1000	1000	1000	1000	1000	1000
$^{13}C_3$-PFBS (INTERNAL STANDARD)	1000	1000	1000	1000	1000	1000	1000	1000	1000
$^{13}C_2$-4:2 FTS (INTERNAL STANDARD)	1000	1000	1000	1000	1000	1000	1000	1000	1000
$^{13}C_5$-PFHxA (INTERNAL STANDARD)	1000	1000	1000	1000	1000	1000	1000	1000	1000
$^{13}C_3$-PFHxS (INTERNAL STANDARD)	1000	1000	1000	1000	1000	1000	1000	1000	1000
$^{13}C_2$-6:2 FTS (INTERNAL STANDARD)	1000	1000	1000	1000	1000	1000	1000	1000	1000

APPENDIX 8: PFCS CALIBRATION CONCENTRATIONS FOR DRINKING WATER-CONTINUED

Drinking Water Calibration Chart	Level 1 (ng/L)	Level 2 (ng/L)	Level 3 (ng/L)	Level 4 (ng/L)	Level 5 (ng/L)	Level 6 (ng/L)	Level 7 (ng/L)	Level 8 (ng/L)	
$^{13}C_8$-PFOA (INTERNAL STANDARD)	1000	1000	1000	1000	1000	1000	1000	1000	1000
$^{13}C_9$-PFNA (INTERNAL STANDARD)	1000	1000	1000	1000	1000	1000	1000	1000	1000
$^{13}C_2$-8:2 FTS (INTERNAL STANDARD)	1000	1000	1000	1000	1000	1000	1000	1000	1000
$^{13}C_6$-PFDA (INTERNAL STANDARD)	1000	1000	1000	1000	1000	1000	1000	1000	1000
$^{13}C_7$-PFUdA (INTERNAL STANDARD)	1000	1000	1000	1000	1000	1000	1000	1000	1000
$^{13}C_2$-PFTeDA (INTERNAL STANDARD)	1000	1000	1000	1000	1000	1000	1000	1000	1000
$^{13}C_4$-PFBA (INTERNAL STANDARD)	1000	1000	1000	1000	1000	1000	1000	1000	1000
$^{13}C_5$-PFPeA (INTERNAL STANDARD)	1000	1000	1000	1000	1000	1000	1000	1000	1000
$^{13}C_4$-PFOS (SURROGATE)	250	250	250	250	250	250	250	250	250
$^{13}C_3$-PFBA (SURROGATE)	250	250	250	250	250	250	250	250	250
$^{13}C_2$-PFOA (SURROGATE)	250	250	250	250	250	250	250	250	250
$^{13}C_2$-PFDA (SURROGATE)	250	250	250	250	250	250	250	250	250

APPENDIX 9: WASTER WATER EXTRACTION

Waste Water Extraction

Preservative Type

_____ (4°C) no preservative

- **May contain Trizma**

Condition Cartridge

_____ 4 mL 0.1% ammonia in MeOH
_____ 4 mL methanol
_____ 4 mL water

Surrogate and Spike

_____ All samples (125 uL of **EIS**)

_____ LCS, MS, MSD (250 uL **PFAS** Spike)

Rinse Samples

_____ 4 mL water (wet)
_____ 4 mL acetate buffer (dry)

Elute Samples

_____ Stack preconditioned cartridge (4 mL MeOH)

_____ 4 mL 1% ammonia in MeOH (wet)
_____ 4 mL methanol (dry)

Final Volume

_____ Concentrate sample to Approximately 4 mL
_____ Add 0.2 mL of water
_____ Adjust final volume to 5 mL with methanol

APPENDIX 10: DRINKING WATER EXTRACTION

Drinking Water Extraction

The Extraction of Per and Polyfluroalkyl Substances

Preservative Type

_____ Trizma - 1.25g for MB, LCS, LCSD

Condition Cartridge

_____ 18 mL methanol
_____ 15 mL water

Add Surrogate

_____ All samples (125 uL of **SURROGATE**)

Add Spike

_____ LCS, MS, MSD (250 uL **PFAS** Spike)

Rinse Samples

_____ 7.5 mL water (wet)

_____ 7.5 mL water (dry)

Elute Samples

_____ 4 mL Methanol (wet)
_____ 4 mL Methanol (dry)

Final Volume

_____ Concentrate sample to dryness

_____ Adjust final volume to 5 mL with 96:4 methanol

APPENDIX 11: TISSUE HOMOGENIZATION

1. Place the samples on the counter to thaw.
2. Each sample shall be homogenized using a laboratory hand held blender with a stainless-steel.
3. Prior to homogenizing the tissue samples, the blender must be certified as PFAS free by decontamination using an Alconox (or equivalent PFAS free soap) wash, water rinse using PFAS-free water, solvent rinse using high purity methanol that has been demonstrated to be PFAS-free, and a final water rinse using PFAS-free water.
4. Add the entire contents of each sample container to a clean beaker.
5. Homogenize sample to a paste-like consistency that does not contain recognizable chunks. The sample is now ready to be subsampled.
6. Weigh out 2.0 ± 0.5 g of the tissue into two (2) separate 50 mL Falcon tubes. Six (6) tubes are needed if this is the QC sample for the batch.
7. After the aliquot is removed for extraction, the remaining homogenate is returned to its original container and returned to the freezer.
8. Each batch of 20 or fewer samples shall include a blank consisting of pre-certified PFAS-free tissue and a laboratory control sample prepared using the same tissue as the blank. Blank samples and laboratory control samples shall undergo the same homogenization (blender) and processing procedures as the field samples.
9. All extraction equipment is decontaminated between samples using an Alconox (or equivalent) wash, water rinse using PFAS-free water, solvent rinse using high purity methanol that has been demonstrated to be PFAS-free, and a final water rinse using PFAS-free water.

TABLE 1. ANALYTE LIST

Compound	Instrument Synonym	CAS Number
1H, 1H, 2H, 2H-Perfluorododecane sulfonic acid (10:2 FTS)	10:2 FTS	120226-60-0
Hexafluoropropyleneoxide dimer acid (HFPO-DA) (GenX)	PFPrOPrA	13252-13-6
Perfluorooctadecanoic acid (PFODA)	PFODA	16517-11-6
N-Ethylperfluorooctane sulfonamido ethanol (NEtFOSE)	N-EtFOSE	1691-99-2
Perfluorooctane sulfonic acid (PFOS)	PFOS	1763-23-1
Perfluoroundecanoic acid (PFUnDA)	PFUdA	2058-94-8
N-methyl perfluorooctane sulfonamido acetic acid (NMeFOSAA)	N-MeFOSAA	2355-31-9
N-Methylperfluorooctane sulfonamido ethanol (NMeFOSE)	N-MeFOSE	24448-09-7
Perfluoroethoxypropyl carboxylic acid (PEPA)	PEPA	267239-61-2
Perfluoropentanoic acid (PFPeA)	PFPeA	2706-90-3
Perfluoropentane sulfonic acid (PFPeS)	PFPeS	2706-91-4
1H, 1H, 2H, 2H-Perfluorooctane sulfonic acid (6:2 FTS)	6:2 FTS	27619-97-2
2-Perfluorooctyl ethanoic acid (8:2 FTA) (FOEA)	FOEA	27854-31-5
Nafion Byproduct 1 (NAFION_BP1)	NAFION_BP1	29311-67-9
N-ethyl perfluorooctane sulfonamido acetic acid (NEtFOSAA)	N-EtFOSAA	2991-50-6
Perfluorohexanoic acid (PFHxA)	PFHxA	307-24-4
Perfluorododecanoic acid (PFDOA)	PFDoA	307-55-1
N-Methylperfluorooctane sulfonamide (N-MeFOSA)(MeFOSA)	N-MeFOSA	31506-32-8
Perfluorooctanoic acid (PFOA)	PFOA	335-67-1
Perfluorodecanoic acid (PFDA)	PFDA	335-76-2
Perfluorodecane sulfonic acid (PFDS)	PFDS	335-77-3
Perfluorohexane sulfonic acid (PFHxS)	PFHxS	355-46-4
4,4,5,5,6,6,6-Heptafluorohexanoic acid (3:3 FTCA)	FPrPA	356-02-5
Perfluorobutanoic acid (PFBA)	PFBA	375-22-4
Perfluorobutane sulfonic acid (PFBS)	PFBS	375-73-5
Perfluoroheptanoic acid (PFHpA)	PFHpA	375-85-9

Perfluoroheptane sulfonic acid (PFHpS)	PFHpS	375-92-8

Compound	Instrument Synonym	CAS Number
Perfluorononanoic acid (PFNA)	PFNA	375-95-1
Perfluorotetradecanoic acid (PFTDA)	PFTeDA	376-06-7
Perfluoro-3-methoxypropanoic acid (PFMPA)	PFMOPrA	377-73-1
1H, 1H, 2H, 2H-Perfluorodecane sulfonic acid (8:2 FTS)	8:2 FTS	39108-34-4
Perfluoro(3,5-dioxahexanoic) acid (PFO2HxA)	PFO2HxA	39492-88-1
Perfluoro(3,5,7-trioxaoctanoic) acid (PFO3OA)	PFO3OA	39492-89-2
Perfluoro(3,5,7,9-tetraoxadecanoic) acid (PFO4DA)	PFO4DA	39492-90-5
Perfluoro-3,5,7,9,11-pentaoxadodecanoic acid (PFO5DOA) (TAF)	TAF	39492-91-6
N-Ethylperfluorooctane sulfonamide (EtFOSAm)	N-EtFOSA	4151-50-2
2-Perfluorohexyl ethanoic acid (6:2 FTA) (FHEA)	FHEA	53826-12-3
2-Perfluorodecyl ethanoic acid (10:2 FTA) (FDEA)	FDEA	53826-13-4
Perfluoro-2-methoxyacetic acid (PFMOAA)	PFMOAA	674-13-5
Perfluorohexadecanoic acid (PFHxDA)	PFHxDA	67905-19-5
Perfluorononane sulfonic acid (PFNS)	PFNS	68259-12-1
Perfluorotridecanoic acid (PFTrDA)	PFTrDA	72629-94-8
Nafion Byproduct 2 (NAFION_BP2)	NAFION_BP2	749836-20-2
Perfluorooctane sulfonamide (PFOSAm) (PFOSA)	PFOSA	754-91-6
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9-Cl-PF3ONS)	PF3ONS	756426-58-1
1H, 1H, 2H, 2H-Perfluorohexane sulfonic acid (4:2 FTS)	4:2 FTS	757124-72-4
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11-Cl-PF3OUdS)	PF3OUdS	763051-92-9
Perfluorododecane sulfonic acid (PFDoS)	PFDoS	79780-39-5
4-(Heptafluoroisopropoxy)hexafluorobutanoic acid (PFECA-G)	PFECA_G	801212-59-9

2H, 2H, 3H, 3H-Perfluorodecanoic acid (7:3 FTCA)	FHpPA	812-70-4
Perfluoro-4-methoxybutanoic acid (PFMBA)	PFMOBA	863090-89-5
2H, 2H, 3H, 3H-Perfluorooctanoic acid (5:3 FTCA)	FPePA	914637-49-3
4,8-Dioxa-3H-perfluorononanoic acid (DONA)	ADONA	919005-14-4

TABLE 2. MS/MS METHOD CONDITIONS

	Parent Ion Mass	Quantification Daughter Ion Mass	Confirmation Daughter Ion Mass [1]	Typical Ion Ratio Quant./Conf.[2]	Quantified using
Method Analytes					
PFBA	212.9	169	n.a.	n.a.	$^{13}C_4$-PFBA
PFPeA	262.9	219.0	na	n.a.	$^{13}C_5$-PFPeA
PFHxA	313.0	269.0	119	0.06	$^{13}C_5$-PFHxA
PFHpA	363.0	319.0	169.0	0.22	$^{13}C_4$-PFHpA
PFOA	413.0	369.0	169.0	0.3	$^{13}C_8$-PFOA
PFNA	463.0	419.0	219.0	0.21	$^{13}C_9$-PFNA
PFDA	513.0	469.0	219.0	0.11	$^{13}C_6$-PFDA
PFUnDA	563.0	519.0	269.0	0.11	$^{13}C_7$-PFUnA
PFDoA	613.0	569.0	319.0	0.11	$^{13}C_2$-PFDoA
PFTrDA	663.0	619.0	169.0	0.15	$^{13}C_2$-PFTA
PFTDA	713.0	669.0	169.0	0.15	$^{13}C_2$-PFTA
PFBS	298.7	80.0	99.0	0.36	$^{13}C_3$-PFBS
PFPeS	349.0	80.0	99.0	0.30	$^{13}C_3$-PFHxS
PFHxS	399.0	80.0	99.0	0.25	$^{13}C_3$-PFHxS
PFHpS	449.0	80.0	99	0.17	$^{13}C_8$-PFOS
PFOS	499.0	80.0	99.0	0.10	$^{13}C_8$-PFOS
PFNS	549.0	80.0	99.0	0.14	$^{13}C_8$-PFOS
PFDS	599.0	80.0	99.0	0.25	$^{13}C_8$-PFOS
PFDoS	699.0	80.0	99.0	0.30	$^{13}C_8$-PFOS
4:2FTS	327.0	307.0	81.0	0.55	$^{13}C_2$- 4:2FTS
6:2FTS	427.0	407.0	81.0	0.65	$^{13}C_2$-6:2FTS
8:2FTS	527.0	507.0	81.0	0.77	$^{13}C_2$-8:2FTS
PFOSA	498.0	78.0	169.0	0.015	$^{13}C_8$-PFOSA

	Parent Ion Mass	Quantification Daughter Ion Mass	Confirmation Daughter Ion Mass [1]	Typical Ion Ratio Quant./Conf.[2]	Quantified using
N-MeFOSA	512.0	169.0	65.0	0.30	D_3-NMeFOSA
N-EtFOSAm	526.0	169.0	269.0	0.95	D_5-NEtFOSA
N-MeFOSAA	570.0	419.0	483.0	0.66	D_3-N-MeFOSAA
N-EtFOSAA	584.0	419.0	526.0	0.67	D_5-N-EtFOSAA
N-MeFOSE	616.0	59.0	n.a.	n.a.	D_7-N-MeFOSE
N-EtFOSE	630.0	59.0	n.a.	n.a.	D_9-N-EtFOSE
HFPO-DA Gen-X	284.9	169.0	184.9	0.65	$^{13}C_3$-HFPO-DA
DONA	377.0	251.0	85.0	0.49	$^{13}C_8$-PFOA
9Cl-PF3ONS	531	351.0	n.a.	n.a.	$^{13}C_8$-PFOS
11Cl-PF3OUdS	631	451	n.a.	n.a.	$^{13}C_8$-PFOS
PFODA	913.0	869.0	169.0	0.14	$^{13}C_2$-PFHxDA
PFHxDA	812.8	769.0	169.1	0.12	$^{13}C_2$-PFHxDA
PFMOAA	179	85	n.a.	n.a.	$^{13}C_3$-HFPO-DA
PFMPA	229	185	n.a.	n.a.	$^{13}C_3$-HFPO-DA
PFMBA	279	235	n.a.	n.a.	$^{13}C_3$-HFPO-DA
PFO2HxA	245	85	n.a.	n.a.	$^{13}C_3$-HFPO-DA
PFO3OA	311	85	n.a.	n.a.	$^{13}C_3$-HFPO-DA
PFO4DA	377	85	n.a.	n.a.	$^{13}C_3$-HFPO-DA
NAFION BP_1	442.9	147	n.a.	n.a.	$^{13}C_3$-HFPO-DA
NAFION BP_2	462.9	185	n.a.	n.a.	$^{13}C_3$-HFPO-DA
TAF, PF5DOA	442.9	85	n.a.	n.a.	$^{13}C_3$-HFPO-DA
PFECA_G	378.9	184.9	n.a.	n.a.	$^{13}C_3$-HFPO-DA
10:2 FTS	626.8	606.9	n.a.	n.a.	$^{13}C_2$-8:2FTS
FHEA	377	293	n.a.	n.a.	MFHEA
FOEA	477	393	n.a.	n.a.	MFOEA

	Parent Ion Mass	Quantification Daughter Ion Mass	Confirmation Daughter Ion Mass [1]	Typical Ion Ratio Quant./Conf.[2]	Quantified using
FDEA	577	493	n.a.	n.a.	MFDEA
FPePA	341	237	n.a.	n.a.	$^{13}C_8$-PFOA
FHpPA	441	337	n.a.	n.a.	$^{13}C_8$-PFOA
FPrPA	241	177	n.a.	n.a.	$^{13}C_8$-PFOA
MFHEA	379	294	n.a.	n.a.	n.a.
MFOEA	479	394	n.a.	n.a.	n.a.
MFDEA	579	494	n.a.	n.a.	n.a.
$^{13}C_4$-PFBA	216.8	171.9	n.a.	n.a.	n.a.
$^{13}C_4$-PFOS	503	80	n.a.	n.a.	n.a.
$^{13}C_8$-PFOSA	506	78	120.3	n.a.	n.a.
$^{13}C_2$-PFDA	515	470	n.a.	n.a.	n.a.
d3-MeFOSAA	573	419	n.a.	n.a.	n.a.
d5-EtFOSAA	589	419	n.a.	n.a.	n.a.
$^{13}C_2$-PFDoA	615	570	n.a.	n.a.	n.a.
$^{13}C_3$-PFBA	216	172	n.a.	n.a.	n.a.
$^{13}C_2$-PFOA	415	370	n.a.	n.a.	n.a.
$^{13}C_8$-PFOS	507	99	n.a.	n.a.	n.a.
$^{13}C_3$-PFBS	302	99	98.9	n.a.	n.a.
$^{13}C_2$-4:2 FTS	329	81	98.8	n.a.	n.a.
$^{13}C_5$-PFHxA	318	273	79.9	n.a.	n.a.
$^{13}C_3$-PFHxS	402	99	309.0	n.a.	n.a.
$^{13}C_2$-6:2 FTS	429	81	409.0	n.a.	n.a.
$^{13}C_8$-PFOA	421	376	509.0	n.a.	n.a.

	Parent Ion Mass	Quantification Daughter Ion Mass	Confirmation Daughter Ion Mass [1]	Typical Ion Ratio Quant./Conf.[2]	Quantified using
$^{13}C_9$-PFNA	472	427	n.a.	n.a.	n.a.
$^{13}C_2$-8:2 FTS	529	81	n.a.	n.a.	n.a.
$^{13}C_6$-PFDA	519	474	n.a.	n.a.	n.a.
$^{13}C_4$-PFOS	503	80	n.a.	n.a.	n.a.
$^{13}C_8$-PFOSA	506	78	n.a.	n.a.	n.a.
$^{13}C_2$-PFDA	515	470	n.a.	n.a.	n.a.
$^{13}C_7$-PFUnDA	570	525	n.a.	n.a.	n.a.
$^{13}C_2$-PFTDA	715	670	n.a.	n.a.	n.a.
$^{13}C_4$-PFBA	217	172	n.a.	n.a.	n.a.
$^{13}C_5$-PFPeA	267.9	223	n.a.	n.a.	n.a.
$^{13}C_2$PFHxDA	815	770	n.a.	n.a.	n.a.
d-N-EtFOSA	531	169	n.a.	n.a.	n.a.
d_9-N-EtFOSE	639	59	n.a.	n.a.	n.a.
d_7-N-MeFOSE	623	59	n.a.	n.a.	n.a.
d-N-MeFOSA	515	169	n.a.	n.a.	n.a.
$^{13}C_3$-PFPrOPrA	332	169	n.a.	n.a.	n.a.

Page: _____ of _____
Project # _____
GEL Quote #: _____
COC Number (1): _____
PO Number:

GEL Laboratories LLC
gel.com
Chemistry | Radiochemistry | Radiobioassay | Specialty Analytics

Chain of Custody and Analytical Request

GEL Work Order Number: ***GEL Project Manager:***

GEL Laboratories, LLC
2040 Savage Road
Charleston, SC 29407
Phone: (843) 556-8171
Fax: (843) 766-1178

Client Name: Phone #
Project/Site Name: Fax #
Address:
Collected By: Send Results To:

Sample Analysis Requested (5) (Fill in the number of containers for each test)

Sample ID ** For composites - indicate start and stop date/time*	*Date Collected (mm-dd-yy)	*Time Collected (Military) (hhmm)	QC Code (2)	Field Filtered (3)	Sample Matrix (4)	Should this sample be considered: Radioactive (If yes, please supply isotopic info.)	Should this sample be considered: (7) Known or possible Hazards	Total number of containers										<-- Preservative Type (6)
																		Comments Note: extra sample is required for sample specific QC

Chain of Custody Signatures

Relinquished By (Signed)	Date	Time	Received by (signed)	Date	Time
			1		
2			2		
3			3		

TAT Requested: Normal: _____ Rush: _____ Specify: _____

Fax Results: [ ] Yes [ ] No

Select Deliverable: [ ] C of A [ ] QC Summary [ ] level 1 [ ] Level 2 [ ] Level 3 [ ] Level 4

Additional Remarks:

For Lab Receiving Use Only: *Custody Seal Intact?* [ ] *Yes* [ ] *No* ***Cooler Temp: _____ °C***

> For sample shipping and delivery details, see Sample Receipt & Review form (SRR.)

Sample Collection Time Zone : [ ] Eastern [ ] Pacific [ ] Central [ ] Mountain [ ] Other:

1.) Chain of Custody Number = Client Determined

2.) QC Codes: **N** = Normal Sample, **TB** = Trip Blank, **FD** = Field Duplicate, **EB** = Equipment Blank, **MS** = Matrix Spike Sample, **MSD** = Matrix Spike Duplicate Sample, **G** = Grab, **C** = Composite

3.) Field Filtered: For liquid matrices, indicate with a **- Y -** for yes the sample was field filtered or **- N -** for sample was not field filtered.

4.) Matrix Codes: **DW**=Drinking Water, **GW**=Groundwater, **SW**=Surface Water, **WW**=Waste Water, **W**=Water, **ML**=Misc Liquid, **SO**=Soil, **SD**=Sediment, **SL**=Sludge, **SS**=Solid Waste, **O**=Oil, **F**=Filter, **P**=Wipe, **U**=Urine, **F**=Fecal, **N**=Nasal

5.) Sample Analysis Requested: Analytical method requested (i.e. **8260B**, **6010B/7470A**) and number of containers provided for each (i.e. *8260B* - **3**, *6010B/7470A* - **1**).

6.) Preservative Type: **HA** = Hydrochloric Acid, **NI** = Nitric Acid, **SH** = Sodium Hydroxide, **SA** = Sulfuric Acid, **AA** = Ascorbic Acid, **HX** = Hexane, **ST** = Sodium Thiosulfate, If no preservative is added = leave field blank

7.) **KNOWN OR POSSIBLE HAZARDS**

RCRA Metals
As = Arsenic **Hg**= Mercury
Ba = Barium **Se**= Selenium
Cd = Cadmium **Ag**= Silver
Cr = Chromium **MR**= Misc. RCRA metals
Pb = Lead

Characteristic Hazards
FL = Flammable/Ignitable
CO = Corrosive
RE = Reactive

TSCA Regulated
PCB = Polychlorinated biphenyls

Listed Waste
LW= Listed Waste
(F,K,P and U-listed wastes.)
Waste code(s):

Other
OT= Other / Unknown
(i.e.: High/low pH, asbestos, beryllium, irritants, other misc. health hazards, etc.)
Description:

Please provide any additional details below regarding handling and/or disposal concerns. *(i.e.: Origin of sample(s), type of site collected from, odd matrices, etc.)*

CHAIN OF CUSTODY

PAGE_____ OF _____

ALPHA ANALYTICAL

WESTBORO, MA
TEL: 508-898-9220
FAX: 508-898-9193

MANSFIELD, MA
TEL: 508-822-9300
FAX: 508-822-3288

Date Rec'd in Lab:

ALPHA Job #:

Client Information

Client:

Address:

Phone:

Fax:

Email:

❑ These samples have been previously analyzed by Alpha

Project Information

Project Name:

Project Location:

Project #:

Project Manager:

ALPHA Quote #:

Turn-Around Time

❑ Standard ❑ RUSH *(only confirmed if pre-approved!)*

Date Due: **Time:**

Report Information - Data Deliverables

❑ FAX ❑ EMAIL

❑ ADEx ❑ Add'l Deliverables

Billing Information

❑ Same as Client info PO #:

Regulatory Requirements/Report Limits

State /Fed Program | *Criteria*

MA MCP PRESUMPTIVE CERTAINTY --- CT REASONABLE CONFIDENCE PROTO

❑ Yes ❑ No Are MCP Analytical Methods Required?

❑ Yes ❑ No Is Matrix Spike (MS) Required on this SDG? (If yes see note in Comments)

❑ Yes ❑ No Are CT RCP (Reasonable Confidence Protocols) Required?

Other Project Specific Requirements/Comments/Detection Limits:

If MS is required , indicate in Sample Specific Comments which samples and what tests MS to be performed.
(Note: All ***CAM*** methods for inorganic analyses require MS every 20 soil samples)

ANALYSIS

SAMPLE HANDLING

*Filtration*_____
❑ Done
❑ Not needed
❑ Lab to do
Preservation
❑ Lab to do
(Please specify below)

TOTAL # BOTTLES

ALPHA Lab ID (Lab Use Only)	Sample ID	Collection Date	Collection Time	Sample Matrix	Sampler's Initials	Sample Specific Comments

Container Type	
Preservative	

PLEASE ANSWER QUESTIONS ABOVE!

IS YOUR PROJECT MA MCP *or* CT RCP?

Relinquished By:	Date/Time	Received By:	Date/Time

Please print clearly, legibly and completely. Samples can not be logged in and turnaround time clock will not start until any ambiguities are resolved. All samples submitted are subject to Alpha's Terms and Conditions.
See reverse side.

FORM NO: 01-01 (rev. 18-Jan-2010)