



**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 E In Situ Sequestration Treatability Study Work Plan* (the “TSWP”), dated September 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 TSWP, dated September 2020. The TSWP was revised in response to EPA comments dated July 13, 2020, and United States Geological Survey (USGS) comments dated August 2020. The TSWP 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 TSWP and finds that they are acceptable. Therefore, EPA approves the TSWP.

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,

A handwritten signature in black ink, appearing to read "Chris Smith", with a long horizontal line extending to the right.

Christopher Smith
Project Manager

NUCLEAR METALS, INC. SUPERFUND SITE

CONCORD, MASSACHUSETTS

Remedial Design Work Plan - Appendix E

In Situ Sequestration Treatability Study Work Plan

Prepared for:



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September 2020

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LIST OF ACRONYMS

°C	degrees Celsius
µm	micron
µg/L	micrograms per liter
bgs	below ground surface
CD	consent decree
DO	dissolved oxygen
DPT	direct push technology
DU	depleted uranium
FS	feasibility study
FSP	field sampling plan
g	grams
GPS	global positioning system
Hazen	Hazen Research, Inc
HB	holding basin
HCl	hydrochloric acid
HNO ₃	nitric acid
ICP-MS	inductively coupled plasma mass spectrometry
ICP-OES	inductively coupled plasma optical emission spectroscopy
ISS	in situ stabilization
L	liter
M	molar
MCL	maximum contaminant level
mg/kg	milligrams per kilogram
mL	milliliter
mM	millimolar
NMI	Nuclear Metals Inc.
PDI	pre-design investigation
ppb	parts per billion
PVC	polyvinylchloride
PV	pore volume
QA/QC	quality assurance/quality control
QEMSCAN	Quantitative Evaluation of Minerals by SCANning electron microscopy
RA	Remedial Action
RD	Remedial Design
RDWP	remedial design work plan

LIST OF ACRONYMS Cont'd

ROI	radius of influence
RSO	Radiation Safety Officer
SMP	sodium monophosphate
SOP	standard operating procedure
SOW	Statement of Work
SPLP	Synthetic Precipitation Leaching Procedure
STPP	sodium tripolyphosphate
TS	treatability study
U	isotopically natural uranium
Uranium	high-concentration DU
USCS	Unified Soil Classification System
USEPA	United States Environmental Protection Agency
VBW	vertical barrier wall
wt %	weight percent
XRD	X-ray diffraction
ZVI	solid zero valent iron

1. INTRODUCTION

The Consent Decree (CD) and the Statement of Work (SOW) provided as Appendix B to the CD describe the Remedial Design (RD)/Remedial Action (RA) activities to be performed for the Nuclear Metals, Inc. (NMI) Superfund Site (Site). These activities include in situ sequestration (ISS) of depleted uranium in Holding Basin (HB) soils and overburden groundwater and ISS of isotopically natural uranium in bedrock groundwater. Section 3.4(a) of the SOW requires performance of Pre-Design Investigations (PDIs) and Treatability Studies (TS) to support the ISS component of the remedy. The RD work plan (RDWP) to which this TS work plan is an attachment, provides Site background and the vision for ISS being implemented at the Site.

This TS work plan describes laboratory testing that will identify the product(s) and dose(s) for ISS amendments that will be used at the Site. The amendment and dose determined from treatability testing will be field tested as described in Appendix B, and collectively, the results from the TS and PDIs for uranium ISS will inform the RD. As described in this work plan, separate studies are needed to evaluate and select treatment amendment for high-concentration uranium impacted soils within the HB (TS ISS-1), uranium impacted overburden groundwater downgradient of the HB (TS ISS-2), and uranium in bedrock (TS ISS-3). Because of the varying uranium concentration, geochemical conditions and physical limitation (e.g., overburden versus bedrock) within different areas of the Site, a likely outcome of the TS is different ISS amendments and doses for separate areas of the Site.

With respect to geochemistry, treatability testing and remediation of impacted groundwater, there is no difference between depleted uranium in overburden and isotopically natural uranium in bedrock. Thus, the singular term “uranium” is used in this TS Work Plan for both depleted uranium in overburden groundwater and isotopically natural uranium in bedrock groundwater.

The following sections of this TS work plan provide a discussion of the background and objectives for these studies and a scope of work for each component of the TS.

2. BACKGROUND AND OBJECTIVES

Apatite II¹ was successfully demonstrated to sequester high-concentration uranium in overburden groundwater during the Feasibility Study (FS) pilot test program conducted in 2014. The pilot test used columns filled with 100% Apatite II. Results showed >99% uranium removal from the aqueous phase; the predominant sequestering mechanism was precipitation of sparingly soluble calcium uranyl phosphate minerals. Documentation of this pilot test is found in *Field and Laboratory Media Testing, for Depleted Uranium Sequestration in Overburden Groundwater*.² The treatability testing described herein is focused on evaluating the effectiveness of appropriate

¹ Apatite II is the tradename of a meta-stable fish-bone-derived hydroxyapatite product produced by PIMS NW, Inc. (<http://pimsnw.com>).

² Geosyntec, 2014, *Field and Laboratory Media Testing, for Depleted Uranium Sequestration in Overburden Groundwater*, The Nuclear Metals Superfund Site, Concord, Massachusetts. Geosyntec, September, 2014.

concentrations of Apatite II and other amendments suitable for injection (as a solution or an aqueous suspension) into the subsurface. Because Apatite II is being tested at lower concentrations in this phase of testing, amendment performance may deviate from the results of the FS pilot study. Three additional amendments will also be tested during this TS.

Four amendments will be evaluated as part of this TS: solid preformed apatite (Apatite II), solid zero valent iron (ZVI), soluble sodium monophosphate (SMP), and soluble sodium tripolyphosphate (STPP, a phosphate polymer). The TS will identify the most effective amendment for removing aqueous-phase uranium, determine the minimum required dose for each amendment, and evaluate the longevity and stability of the sequestered uranium. Information regarding these amendments, including advantages and disadvantages, is presented in the amendment selection matrix below:

Amendment Selection Matrix

Amendment	Description	Advantages	Disadvantages	Target Media
#1 Apatite II (PIMS NW, Inc.)	Ground fishbone product. Calcium phosphate	Demonstrated to work effectively during FS. Approved by the United States Environmental Protection Agency (USEPA) in Site Record of Decision (ROD)	Preplanning is important to obtain sufficient supply when needed. Particle size results in uncertainty with ability to achieve significant radius of influence. Can increase phosphorous concentration in groundwater. Can contain trace quantities of arsenic. Contains organic matter – potential to induce reducing conditions.	Holding Basin Overburden Bedrock
#2 Soluble phosphate polymer: Sodium tripolyphosphate (STPP)	Tripolyphosphate consists of 3 orthophosphate molecules linked together ($\text{Na}_5\text{P}_3\text{O}_{10}$) In contact with water, this slowly hydrolyzes and releases orthophosphate	In theory, more soluble and slower to react with calcium than SMP (#3), thereby avoiding immediate precipitation at the injection point and allowing better distribution into the aquifer. Inexpensive	In practice, can form an amorphous gel with calcium that may have lower solubility than SMP (#3). Very slow to react and release orthophosphate. Significant reactive uncertainty with calcium and uranium; final solid phases formed may not be ideal (due to higher solubility of these phases than with SMP). Can contain significant quantities of arsenic depending upon purity and source.	Bedrock
#3 Soluble Orthophosphate: Sodium monophosphate (SMP)	Sodium monophosphate salt (Na_3PO_4)	Rapidly reacts with calcium to precipitate calcium phosphate (Apatite) in situ and also reacts with uranium to precipitate uranium phosphates.	Rapid reaction with calcium can limit distribution in aquifer. May not be sufficiently retained in aquifer due to limited sorption to soil. Uncertainty with respect to the final solid phases formed, some amorphous uranium phosphates may dominate solids. Can contain significant arsenic depending upon purity and source.	Overburden

Amendment	Description	Advantages	Disadvantages	Target Media
#4 Zero Valent Iron (ZVI)	Granular (micro- or nanoscale) ZVI from Connolly or Hepure, or other supplier	<p>Reacts with water to form iron corrosion products that will sorb uranium; will also consume dissolved oxygen and drive system reducing, resulting in precipitation of low-solubility U(IV) solids.</p> <p>Micro- or nanoscale forms are injectable.</p> <p>Arsenic may be retained on ZVI.</p> <p>ZVI not likely to introduce arsenic.</p>	<p>Reactions with soluble uranium require that the ZVI surface remains reactive – soluble ions such as calcium, bicarbonate, and sulfate can react on its surface and passivate (reduce reactivity) of ZVI.</p> <p>As corrosion occurs, reactive surfaces are consumed, and treatment capacity becomes limited; potential for desorption of uranium due to displacement by other ions that may sorb to the iron surface.</p> <p>Can generate hydrogen, resulting in accumulation of hydrogen in soil vapor.</p>	Holding Basin Overburden Bedrock

Apatite II has been shown in previous evaluations at NMI to be effective in sequestering uranium from overburden groundwater via formation of low-solubility uranium phosphate mineral phases (e.g., chernikovite, $(\text{H}_3\text{O})_2(\text{UO}_2)_2(\text{PO}_4)_2 \cdot 6\text{H}_2\text{O}$). Additionally, apatite may sequester uranium via substitution within the apatite phase and formation of poorly crystalline calcium-uranium-phosphate precursor phases. Apatite II is a unique form of calcium phosphate because it contains nanocrystalline apatite in a bulk structure that is generally more amorphous (less crystalline) than other phosphate sources. This combination provides high solubility and surface reactivity as well as seed crystals for precipitation of metal-apatite phases such as chernikovite (uranyl phosphate) and autunite (calcium uranyl phosphate). Poor crystallinity makes the solubility of the Apatite II higher than other solid sources of phosphate. Apatite II reacts with uranium through direct sorption and dissolution of the primary apatite mineral to form secondary uranium-bearing phosphate mineral phases.

Our expectation is that Apatite II will continue to show the best results and be the most practical amendment (the amendment with the least uncertainty in terms of treatment effectiveness and permanence) for full-scale application. However, the other amendments may perform better in certain systems and may also be used in combination with Apatite II depending upon their performance. For example, STPP has been used in tailing systems and groundwater for in situ uranium treatment (Gillow et al. 2013³), and ZVI has been shown to effectively sequester uranium

³ Gillow, J., Griffin, A., Christoffersen, L., Divine, C., Hay, M., and DeDycker, P. 2013. Control of tailings seepage through reactive chemical amendments. Proceedings of the International Mine Water Association Conference, 2013.

(Gu et al. 1998⁴, Morrison et al. 2002⁵); however, challenges with both amendments have been identified and are discussed in the sections below.

This TS work plan includes implementation of three separate treatability studies, one for each of the media targets for ISS (**Figure 1**). The USEPA *Guidance for Conducting Treatability Studies under CERCLA Final*,⁶ as supplemented for RD by the *Remedial Design/Remedial Action Handbook*,⁷ was considered in developing this TS work plan. The TS will be conducted according to a schedule that is complimentary with other PDI tasks. For example, certain portions of the overburden injectability pilot testing will not be conducted until sufficient TS testing is completed. Completing the TS before injection testing will eliminate the possibility of pilot injection testing a specific amendment at a specific dose that has not been shown to be effective in a laboratory setting.

Three separate procedures are presented below, one procedure for each of the following:

- TS ISS-1 Holding Basin Soils
- TS ISS-2 Overburden Groundwater
- TS ISS-3 Bedrock Groundwater

3. TS ISS-1: AMENDMENT TESTING FOR SEQUESTRATION OF URANIUM IN HOLDING BASIN SOILS

3.1 Purpose

TS ISS-1 will evaluate the performance of two ISS treatments (Apatite II and ZVI) at sequestering uranium in impacted HB soils under both aerobic and anaerobic geochemical conditions. These conditions are intended to simulate the potential for perturbations to groundwater redox conditions due to planned isolation of impacted HB soils through a vertical barrier wall (VBW) and capping. Anaerobic conditions and stagnant hydraulic gradients are expected to develop in saturated soils contained by the VBW and cap due to a combination of microbial processes and minimal infiltration of aerobic groundwater. Correspondingly, the mobility of uranium in groundwater is expected to decrease following containment of HB soil as the existing highly soluble uranium (VI), present under aerobic conditions, is transformed to sparingly soluble uranium (IV) under reducing conditions, which precipitates as insoluble reduced uranium minerals (e.g., uraninite). However, slight leakage of oxidizing groundwater through the cap and/or VBW is possible and may

⁴ Gu, B., Liang, L., Dickey, M.J., Yin, X., and Dai, S. 1998. Reductive precipitation of uranium (VI) by zero-valent iron. *Environmental Science and Technology* 32(21): 3366-3373.

⁵ Morrison, S.J., Carpenter, C.E., Metzler, D.R., Bartlett, T.R., and Morris, S.A. 2002. Design and performance of a permeable reactive barrier for containment of uranium, arsenic, selenium, vanadium, molybdenum, and nitrate at Monticello, Utah. In D.L. Naftz, S.J. Morrison, J.A. Davis, and C.C. Fuller, Eds., *Handbook of Groundwater Remediation Using Permeable Reactive Barriers: Applications to Radionuclides, Trace Metals, and Nutrient*. San Diego, CA: Academic Press. 371 pp.

⁶ USEPA. 1992. *Guidance for Conducting Treatability Studies under CERCLA Final*. EPA/540/R-92/071a. October.

⁷ USEPA. 1995. *Remediation Design/Remedial Action Handbook*. EPA 540/R-95/059. June.

remobilize uranium via oxidative dissolution of uranium (IV) solid phases. Although unlikely, a cap/containment failure could result in infiltration of a large amount of aerobic groundwater to contact the uranium impacted soils. TS ISS-1 will evaluate whether Apatite II or ZVI is the most effective amendment to sequester leached uranium under both aerobic and anaerobic conditions compared to an unamended control test.

A flow chart of the treatability testing scheme for the HB (TS ISS-1) is shown on **Figure 1A**.

3.2 Scope

3.2.1 Soil Sample Collection

A great deal of historical soil sampling and analysis of uranium-impacted soils beneath the HB have been conducted, and these data were used to develop a three-dimensional (3D) model of uranium-impacted soils. This 3D model was used to select soil sample collection locations and depths for acquiring soils for the TS. The historical data used to create the 3D model were collected during HB characterization studies conducted during or after 1998 (after the HB excavation) included as part of the remedial investigation. These data are described fully in the 2014 FS Report.⁸

Soil sampling locations were selected to target saturated and unsaturated soil samples with the highest uranium concentrations. Consideration was also given to locations that facilitate drill rig access and a broad distribution of uranium mass (i.e., multiple elevated detections of uranium in adjacent vertical profile samples).

A maximum of five unsaturated and five saturated 10-foot soil borings will be advanced and screened for suitability for this TS. Soil borings SB-TS-01001 through SB-TS-01010 target soils with historically elevated uranium concentrations and are shown on **Figure 2**. Coordinates and target sample collection depths for each of these locations are shown on **Figure 2**. Boring locations will be identified using a handheld global positioning system (GPS) unit such as a Trimble GeoExplorer. At each location, a minimum 4-inch-diameter drill casing will be advanced using a track-mounted sonic drill rig to a maximum depth of 65 feet below the elevation of the top of the HB liner by a licensed driller under the oversight of a Geosyntec field engineer or geologist. Each soil boring will be continuously sampled to the target sample interval depth identified on **Figure 2**. All efforts will be made to avoid using water during drilling activities to avoid altering the sample geochemistry.

In 2002, the United States Environmental Protection Agency (USEPA) placed approximately 6 feet of clean fill in the bottom of the HB to grade the floor and allow an impermeable liner to gravity drain any rainwater to a culvert at the northern end of the HB. Several unsaturated zone borings identified in **Figure 2** begin at the clean fill/native soil interface; if more than 1 foot of clean fill is observed at the top of these target sample intervals, then the target sample interval will

⁸ de maximis, 2014. Feasibility Study Report, Nuclear Metals, Inc. Superfund Site, Concord Massachusetts. November.

be extended deeper to collect 10-feet of native soil. As shown in **Table 1**, a minimum volume of approximately 17 liters (L) of HB soil will be needed for TS ISS-1.

After setting up the drill rig at a boring location, an exclusion zone will be established around the drill rig, soil staging area, and soil logging areas. The extracted soil cores will be prescreened for radiological activity levels at the drill site and then screened in the on-site laboratory. The following is the sample protocol for the on-site laboratory:

1. The sonic drill core liner will be cut open and soils will initially be screened with a handheld radiation survey unit to ensure radiation levels are safe to handle as determined by a qualified health physics technician and approved by the on-site Radiation Safety Officer (RSO).
2. If the soil core is safe to handle without further controls, the soils in the core will be described using the Unified Soil Classification System (USCS). If the soil is deemed unsafe, protocols will be followed as directed by the health physics technician.
3. The soil core will be screened again using the handheld radiation survey instrument, and the count rate per minute of the core will be logged over the length of the core.
4. If the soil core is collected above the target sample interval for that location (Figure 2), a maximum of one, 1-L subsample per 5 feet may be collected from the portion of the core exhibiting the highest radiological activity and submitted to the on-site laboratory for analysis. The remaining soil will be set aside until the on-site laboratory results are received.
5. While advancing the first boring, the subsample submitted to the on-site laboratory will be evaluated to develop a calibration curve relating readings from the handheld radiation survey instrument to the uranium concentrations measured at the on-site laboratory. The calibration curve will range from a minimum concentration of no greater than 10 milligrams per kilogram (mg/kg) to a maximum concentration of no less than 500 mg/kg. From this curve, the count rate per minute on the handheld radiation survey instrument that correlates to a uranium concentration of 100 mg/kg will be estimated. Additional subsamples beyond what is described may be necessary to construct the full calibration curve. An RSO will be consulted to help select any additional samples for the calibration curve.
6. If the soil core is from the 10-foot target sample interval (identified in Figure 2), those portions of the sample interval with radiation activity levels greater than the predetermined value that equates to greater than 100 mg/kg uranium shall be divided into a maximum of five subsamples with no subsample consisting of less than 1 L of soil. For the first boring, it may be necessary to first collect and analyze a subsample of soil from the target sample interval with the highest screened activity in order to develop the calibration curve described above. If no portion of the target sample interval exhibits radiological activity above the “elevated uranium value,” then two 1-L samples of soil should be collected from the two portions of the target sample interval exhibiting the highest radiological activity

levels and submitted to the on-site laboratory for analysis. The remaining soil will be set aside until the on-site laboratory results are received.

7. Soil samples will be collected in zip top plastic bags. To better preserve the sample's geochemistry during transport to the lab, air will be expelled from the bag to the greatest extent possible prior to sealing the zip top. Using a new, clean pair of nitrile gloves, each zip top plastic bag containing the soil sample will then be placed into a second zip top plastic bag, sealed, labeled appropriately (sample identification number, sample collection date, time of collection, and sampler initials), and placed into a sample cooler. The sample cooler will then be brought to the on-site radiation screening laboratory, where an aliquot of the sample will be analyzed for preliminary total uranium concentration. Following screening and preliminary sampling, soil samples will be sealed in zip top plastic bags to limit unnecessary exposure to the atmosphere and preserve sample geochemistry.
8. Soil borings in the saturated and unsaturated zones shall be advanced following the order identified on **Figure 2**. The drill tooling will be decontaminated between each boring following the procedures outlined in the decontamination standard operating procedure (SOP) (NMI-007, in the Field Sampling Plan [FSP]). After receiving the on-site laboratory results for samples from the three primary borings, the total volume of the subsamples with uranium concentrations greater than 100 mg/kg in each saturation zone shall be measured. If the total volume exceeds 30 L, those samples shall be submitted to the treatability testing laboratory. If less than 30 L of soil with uranium concentration greater than 100 mg/kg have been collected, then additional borings shall be advanced at the secondary locations identified in **Figure 2** or as step-outs from the primary borings with known elevated uranium concentration. This additional sampling shall follow the same procedure described above until a total of 30 L of soil with uranium concentrations greater than 100 mg/kg have been collected from the saturated and unsaturated zones combined.
9. Following collection, the samples will be labeled, placed in coolers with ice, and shipped under standard chain-of-custody procedures (described in NMI-001 of the FSP) to Hazen Research, Inc. (Hazen) of Golden, Colorado, the laboratory conducting the TS. Samples to be stored on-site shall be placed in a refrigerator until packing for shipping is complete. The Hazen laboratory quality assurance/quality control (QA/QC) procedures are included in **Attachment A** of this TS work plan. Soil samples will be shipped to the laboratory under appropriate packing and shipping protocols as determined by the on-site RSO.
10. Soil samples collected from the saturated and unsaturated zones will be homogenized by the laboratory and undergo baseline characterization. Homogenized HB soil was chosen for use in TS ISS-1 because (1) soil texture is similar within the saturated and unsaturated zones and (2) alteration of the redox state of the column influent will overpower any differences in geochemical conditions between the saturated and unsaturated zones. Soils

will be blended via cloth blending, use of a V-blender, or cone-and-quartering techniques.⁹ Representative sub-samples of homogenized soil will be collected based on visual inspection. For baseline characterization, the lab will analyze homogenized HB soil for the following:

- Organic and inorganic carbon (combustion analysis)
 - Environmentally accessible (acid digestible by EPA Method 3050B) uranium, iron, aluminum, calcium, manganese, molybdenum, and arsenic (inductively coupled plasma mass spectrometry [ICP-MS]/inductively coupled plasma optical emission spectroscopy [ICP-OES])
 - Leachable uranium (Synthetic Precipitation Leaching Procedure [SPLP], ICP-MS/ICP-OES) to ensure that the baseline uranium soil content is sufficient to perform testing (i.e., at least 20 mg/kg, with higher concentrations preferable).
 - Fe(II) (colorimetry)
 - Sulfide (turbidimetry)
11. Soil from depth intervals with the highest uranium concentration from the unsaturated and saturated zones will be composited at the lab for use in the flow-through columns during the TS.

3.2.2 Groundwater Collection

Groundwater with low concentrations of uranium representative of the composition of background groundwater will be collected from MW-S30, an overburden monitoring well upgradient of the HB, for use as the flow-through column influent. Groundwater will be collected using a submersible or peristaltic pump fitted with new tubing and then transferred into new containers (e.g., 5-gallon pail, 55-gallon drum). To minimize aeration of the groundwater sample during collection, the discharge tubing will enter the sample container through a rubber seal and will be submerged beneath the water surface of the container. A small vent hole in the container will allow displaced air to escape the container without mixing with the groundwater sample. Sampling methods will follow the low-flow groundwater sampling SOP (NMI-GW-010 in the FSP). During collection, filtered (0.45 µm) and unfiltered samples will be collected, and general groundwater quality parameters will be measured, including pH, dissolved oxygen (DO), oxidation reduction potential (ORP), turbidity, and specific conductance. A minimum volume of approximately 67 L of groundwater will be collected for use in TS ISS-1 (**Table 1**). Extracted groundwater from MW-S30 will be labeled, packed on ice, and shipped to Hazen under standard chain-of-custody procedures (as described in NMI-001 of the FSP). For baseline characterization, the laboratory will analyze filtered and unfiltered groundwater from MW-S30 at the time of collection and prior to initiation of the column tests for the following:

⁹ United States Bureau of Mines, 1995. Laboratory Procedures for Hydrometallurgical-Processing and Waste-Management Experiments. Information Circular 9431.

- pH
- ORP
- Inorganic carbon (coulometry)
- Nitrate (colorimetry)
- Sulfide (turbidimetry)
- Total uranium, calcium, phosphorus, arsenic, manganese, molybdenum, and iron (ICP-MS/ICP-OES)

Comparison of baseline characterization results between the time of collection and the initiation of column tests will be used to identify changes in groundwater composition (e.g., precipitation, biological consumption) during transport and storage.

3.2.2.1 Groundwater Sample Collection Beneath the Holding Basin

As discussed in the sections above, drilling equipment will be mobilized into the Holding Basin to collect soil samples for treatability studies, including from the saturated zone beneath the Holding Basin. A groundwater sample will be collected from the shallow saturated zone (approximately the upper 10-feet) using a temporary well or a Push-Ahead sampler developed by Cascade Drilling. If the Push-Ahead sampler is used, the sampler will be driven at least 5 feet ahead of the override casing and into the native formation without the use of drilling water that could alter the geochemistry. Prior to the sample collection, the temporary well screen or the Push-Ahead sampler will be purged until field parameters (temperature, dissolved oxygen, oxidation-reduction potential, pH, specific conductance and turbidity) stabilize consistent with low-flow groundwater sampling procedures.

The groundwater sample will be analyzed for the following suite of parameters:

- VOCs via Method 8260
- 1,4-dioxane via Method 8270SIM
- SVOCs via Method 8270
- Total and dissolved uranium with U^{235}/U^{238} speciation via Method 6020A ICP-MS
- Total and Dissolved Metals via Method 6020A (ICP-MS)/6010D (ICP-OES) (Al, As, Sb, Ba, Be, Cd, Cr, Co, Cu, Pb, Mn, Mo, Ni, Se, Ag, Tl, Th, V, Zn)
- Nitrate/Nitrite via Method 353.2
- Total Phosphorous via Method 365.1
- Orthophosphate via Method SMP4500P-E
- Dissolved Organic Carbon via Method 9060
- Total and Dissolved Cations (Fe, Mn, Ca, Mg, Na, K) via Method 6020A (ICP-MS)/6010D (ICP-OES)
- Anion (Sulfate, Fluoride, and Chloride) via Method 300
- Carbonate and Bicarbonate Alkalinity via Method 310.1

This suite of analytes is consistent with the November 2019 parameters sampled in monitoring wells MW-S24 and HBPZ-2R to allow for a direct comparison of groundwater beneath the Holding Basin and immediately downgradient.

3.2.3 Column Construction

Flow-through columns will be constructed from clear schedule 40 polyvinylchloride (PVC) with threaded end-caps fitted with tubing to deliver column influent and receive column effluent. Columns will measure 3 inches in diameter and 18 inches in length.

Three columns containing homogenized HB soil will be constructed for testing (**Table 2**):

- One column containing unamended soil (control column)
- One column containing soil amended with 1 weight percent (wt %) ZVI
- One column containing soil amended with 1.5 wt % Apatite II

The mass loading of Apatite II and ZVI amendments in the column tests for Holding Basin soils was selected to be representative of a typical bulk mass loading achieved in the field using direct-push technology (DPT) jet injection. The bulk mass loading of injected amendments is a function of the mass of amendment per fracture, the fracture radius of influence (ROI), the vertical spacing between individual fractures at each injection location, and the overlap of ROIs between adjacent injection locations. As described in **Appendix B**, site-specific injection design parameters will be evaluated based on the results of the treatability studies as well as ISS pilot testing in overburden. Assuming typical values for these parameters based on previous experience implementing DPT direct-push jet injection (e.g., up to 1300 lbs of Apatite II per fracture, 15-ft ROI, 3-ft vertical spacing, and 100% overlap), a dry weight mass loading of 1% to 1.5% was selected for the column tests.

ZVI (Hepure Ferox Flow, 125 microns [μm]¹⁰) and Apatite II (PIMS NW, Inc.) will be amended to HB soil based on dry weight percentage. A subsample of homogenized HB soil will be dried to determine the moisture content, which will be used to calculate the amount of ZVI and Apatite II needed for each column. ZVI and Apatite II will be mixed with undried soil prior to column packing.

Columns will be packed with soil using a plastic scoop. The columns will be shaken/tapped/swirled throughout the packing process to get even compaction of the soil into the column and to avoid creating preferential flow paths and voids in the packed columns. The mass of soil added to each column will be recorded.

3.2.4 Column Test

Site background groundwater amended with a conservative tracer (e.g., bromide) will serve as column influent. Columns will be oriented vertically, and column influent will be pumped at 1

¹⁰<https://hepure.com/products/ferox-flow-zero-valent-iron-powder/>

pore volume (PV) per day in an upflow arrangement using a peristaltic pump. Columns will be operated for 4 weeks. During the column test, redox conditions of the column influent will be altered according to the following schedule:

- Week 1: Column influent will be oxidizing, reflecting the redox conditions of the HB prior to VBW and cap installation.¹¹ Column effluent will be sampled on days 2, 4, and 7.
- Weeks 2-3: Column influent will be amended with organic carbon and nutrients to promote the development of reducing redox conditions within the column. Reducing conditions reflect the expected redox conditions of the HB following installation of the VBW and soil cap. Column effluent will be sampled on days 2, 7, and 14.
- Week 4: Column influent will be oxidizing.¹² A return to oxidizing conditions mimics the infiltration of aerobic groundwater into the anaerobic HB during a potential failure of the VBW or soil cap. Column effluent will be sampled on days 2, 4, and 7.

During the anaerobic phase (weeks 2-3), column influent will be dosed with dissolved organic carbon (glucose) and nutrients (low concentration of ammonium chloride) to stimulate microbial consumption of DO and promote the development of iron-reducing (anaerobic) conditions. Concentrations of DO and nitrate measured in the column influent during baseline characterization will be used to calculate the concentration of glucose required to achieve iron-reducing conditions within the column. This approach is preferred to dosing the column influent with chemical reductants (e.g., bisulfide, dithionite, or ferrous iron) because it produces anaerobic conditions which will more accurately reflect the Site groundwater composition. The column influent will be filtered and sterilized (0.22 µm filtration) to prevent the growth of microbes within the influent container. The expected column effluent during weeks 2-3 will be geochemically reducing; will contain dissolved ferrous iron generated from the Site soil, along with organic acids; and will be a reasonable representation of anaerobic groundwater in the HB after capping and VBW installation. If by week 3 the effluent is not indicative of reducing conditions within the column (e.g., decrease in ORP, increase in dissolved iron), glucose-amended influent will continue to be pumped through the column until reducing conditions develop. Likewise, if by week 4 the effluent is not indicative of a return to oxidizing conditions within the column (return to baseline ORP and dissolved iron concentrations), this phase of the column test will continue until the effluent reflects oxidizing conditions within the column.

Unfiltered samples of column effluent will be collected periodically as described above and analyzed for the following:

¹¹ Although the influent groundwater collected from the Site will be aerobic, this water will be sitting in the lab for periods of time and may lose oxygen due to microbial activity. If necessary, the influent solution will be sparged with air to achieve a DO concentration similar to the concentration measured in the field during sample collection.

¹² If necessary, the influent solution will be sparged with air to achieve a DO concentration similar to the concentration measured in the field during sample collection.

- pH
- ORP
- Total uranium, calcium, phosphorus, arsenic, manganese, molybdenum, and iron (ICP-MS/ICP-OES)
- Nitrate (colorimetry)
- Sulfide (turbidimetry)
- Inorganic carbon (coulometry)
- Bromide (ion chromatography)

A summary of samples and analyses that will be conducted during TS ISS-1 is presented in **Table 3**. The Hazen QA/QC procedures are included in **Attachment A** of this TS work plan. Uranium will be monitored to assess the efficacy of soil amendments in sequestering leached uranium from HB soil compared to the unamended column. Calcium and phosphorus will be monitored to assess the dissolution of Apatite II as a source of calcium and phosphate for uranium-phosphate mineral precipitation. Arsenic will be monitored to assess whether trace arsenic present in Apatite II has the potential to impact groundwater quality via dissolution and subsequent release of arsenic from Apatite II solids. Molybdenum will be monitored because it has been detected at elevated levels in the upgradient portion of the uranium plume. In addition to ORP, nitrate, iron (i.e., higher dissolved iron concentrations reflect more reducing conditions), and sulfide will be monitored in select samples as redox indicators. As carbonate enhances uranium mobility in groundwater, inorganic carbon (i.e., total carbonate species) will be monitored to evaluate this effect under the conditions of this study.

Analysis of unfiltered (i.e., “total”) column effluent samples was selected because it provides a more conservative estimate of amendment performance than analysis of filtered (i.e., “dissolved”) column effluent samples. If total uranium results fall above the target for uranium stabilization (total uranium concentrations >0.030 mg/L) in two consecutive sampling events, these samples will be filtered and analyzed for dissolved constituents in order to understand whether colloidal (micro- or nano-particulate) uranium is contributing to the concentration of total (unfiltered) uranium.

3.3 Outcome

Results from TS ISS-1 will be used to determine if Apatite II or ZVI is the most effective soil amendment for sequestering leached uranium in HB soil under both aerobic and anaerobic conditions. Both amended and unamended column tests will be evaluated based on decreases in effluent uranium concentrations measured during periods of reducing conditions (weeks 2-3) and a return to oxidizing conditions (week 4) relative to the initial period of oxidizing conditions (week 1) in the control column.

3.4 Assumptions

The scope of work for this TS includes the following assumptions:

It is assumed that a track-mounted drilling rig is capable of descending into and ascending out of the HB via the southeast sidewall which has a lower grade than the rest of the HB walls. Confirmation of this assumption will be needed during a Site walk with the driller. If the drilling rig cannot descend and ascend the HB sidewalls, it will most likely be necessary to cut and roll back a portion of the existing cover and to regrade a portion of the HB sidewall to allow access. Following the completion of the investigation program, the soil cap and cover would be repaired. Drill rig access information will be provided separately in the implementation plan.

The HB is currently covered by a thick plastic liner. Prior to drilling, the liner shall be cut, peeled away from the boring location, and held down in such a way to prevent tripping hazards or the liner getting caught in the drill tooling. Following completion of drilling and backfill of the borehole, the hole created in the plastic liner will be repaired.

Any soil or groundwater removed from a boring and not collected as a sample will be returned to the boring or appropriately drummed and stored on-site for future disposal. Soil borings will be backfilled with Site soil or engineered sand and sealed with at least 1-foot of hydrated bentonite chips.

The drill tooling will be decontaminated between each boring following the procedures outlined in the decontamination SOP found in the FSP. Additional decontamination and swab sampling will be required prior to free release of any drilling equipment as identified in the decontamination SOP.

de maximis will provide an RSO to evaluate the radioactivity level of soil and groundwater to be shipped off-site. If the activity levels exceed the threshold for more rigorous packaging and shipping methods, *de maximis* will manage the packaging and shipping.

4. TS ISS – 2: AMENDMENT TESTING FOR OVERBURDEN GROUNDWATER

4.1 Purpose

Treatability studies for overburden groundwater will evaluate the performance of three amendments (Apatite II, ZVI, and SMP [Carus Corporation]¹³) for immobilization of uranium. Methods will include (1) a batch reactor study to evaluate the optimum dose rate for each amendment, and (2) a column study to evaluate the efficacy and mechanisms of uranium sequestration under advective flow at the optimal dose rates identified in the batch study. A flow chart of the treatability testing scheme for the overburden groundwater (TS ISS-2) is shown on **Figure 1B**.

¹³ <http://www.caruscorporation.com/remediation/products/phosphates>

4.2 Scope

4.2.1 Soil Collection

Site soil and groundwater will be collected for use in TS ISS-2.

Soil collection will target both low-uranium-content overburden soil (e.g., not immediately downgradient of the HB) and high-uranium-content overburden soil within the overburden uranium groundwater plume with the lowest potential for natural uranium attenuation (e.g., sand). Soil collection locations were selected based on historical overburden soil uranium concentrations. Two soil borings located for soil collection are shown on **Figure 3**, which also shows the current distribution of uranium in overburden groundwater.

The low-uranium-content soil will be collected at the distal end of the uranium plume where there is little or no uranium sorbed to formation soils. The location is designated as TS-SB-01 (**Figure 3**). The high-uranium-content overburden soil will be collected from a boring immediately downgradient of the HB near MW-S24 (TS-SB-02 on **Figure 3**) where previous studies have indicated significant uranium sorbed to formation soils. These soils will be collected from the top 20 feet of saturated overburden (approximately 50 to 70 feet below ground surface [bgs]) using a 4-inch-diameter sonic core barrel. The drill tooling will be decontaminated between each boring following the procedures outlined in the decontamination SOP (NMI-007 in the FSP). The recovered soils will be logged for lithology using the USCS. In total, a minimum of approximately 6 L of high-uranium sandy soil and approximately 45 L of low-uranium sandy soil will be collected for use in TS ISS-2 (**Table 1**). Following collection, the samples will be labeled, placed in coolers with ice, and shipped to Hazen under standard chain-of-custody procedures (described in NMI-001 of the FSP). Appropriate packing and shipping methods will be based on evaluation by the on-site RSO.

Soil samples will be homogenized by the laboratory via cloth blending, use of a V-blender, or cone-and-quartering techniques.¹⁴ Representative sub-samples of homogenized soil will be collected based on visual inspection. For baseline characterization, the lab will analyze homogenized soil for the following:

- Organic and inorganic carbon (combustion analysis)
- Environmentally accessible (acid digestible by EPA Method 3050B) uranium, iron, manganese, arsenic, molybdenum, aluminum, and calcium (ICP-MS/ICP-OES)
- Leachable uranium (SPLP)
- Fe(II) (colorimetry)
- Sulfide (turbidimetry)

¹⁴ United States Bureau of Mines, 1995. Laboratory Procedures for Hydrometallurgical-Processing and Waste-Management Experiments. Information Circular 9431.

If enough uranium is present in the overburden soil (at least 20 mg/kg), a sequential extraction procedure will be performed (described below) to identify the baseline chemical/mineralogical speciation of solid-associated uranium.

4.2.2 Groundwater Collection

Overburden groundwater will be collected from monitoring well MW-S24, which historically and currently has the highest uranium concentration (2,675 micrograms per liter [$\mu\text{g/L}$] in November 2019), for use in the batch reactors and as the flow-through column influent for the first column study. Background (i.e., low-uranium) overburden groundwater from MW-S30 will be collected for use in the SMP sorption capacity test (described in Section 4.2.3) and as the flow-through column influent for the second column study. Sampling methods will follow low-flow protocols as provided in SOP NMI-GW-010 of the FSP. During groundwater collection, filtered (0.45 μm) and unfiltered samples will be collected, and general groundwater quality parameters will be measured, including pH, DO, ORP, turbidity, and specific conductance. Approximately 626 L of high-uranium groundwater will be collected from well MW-S24, and 58 L of low-uranium groundwater will be collected from background well MW-S30 (**Table 1**). Groundwater will be transferred to new drums using a submersible pump fitted with new tubing. To minimize aeration of the groundwater sample during collection, the discharge tubing will enter the drum through a rubber seal and will be submerged beneath the water surface of the drum. A small vent hole in the drum will allow displaced air to escape the drum without mixing with the groundwater sample. Drums will be labeled and shipped under standard chain-of-custody procedures to Hazen. Appropriate packing and shipping methods will be based on evaluation by the on-site RSO. For baseline characterization, the laboratory will analyze filtered and unfiltered overburden groundwater at the time of collection and prior to initiation of the column and batch tests for the following:

- pH
- ORP
- Inorganic carbon (coulometry)
- Total uranium, calcium, phosphorus, arsenic, manganese, molybdenum, and iron (ICP-MS/ICP-OES)
- Nitrate (colorimetry)
- Sulfide (turbidimetry)

Comparison of baseline characterization results between the time of collection and the initiation of column and batch tests will be used to identify changes in groundwater composition (e.g., precipitation, biological consumption) during transport and storage.

4.2.3 Batch Reactor Test

Batch Reactor Construction

The batch reactor study will be performed using 250 milliliter (mL) glass bottles containing undried overburden soil and overburden groundwater. The soil:liquid ratios for the batch reactors will be determined to (1) ensure sequestration by the amendment is detectable in excess of the

control, and (2) prevent dissolution of >10% of the Apatite II amendment in the low-dose condition. Based on the current concentration of uranium in groundwater at well MW-S24 (approximately 2.7 mg/L), a soil:liquid ratio of 1:20 is expected (equivalent to 10 grams [g] of dry soil per 200 mL of liquid).

Three identical reactors containing overburden soil and groundwater will be constructed for each of the following treatments (39 reactors in total) (**Table 2**):

- Unamended control reactor
- Apatite II at 0.5 wt %
- Apatite II at 1.5 wt %
- Apatite II at 3.0 wt %
- Apatite II at 0.5 wt % with guar gum
- Apatite II at 1.5 wt % with guar gum
- Apatite II at 3.0 wt % with guar gum
- ZVI at 0.5 wt %
- ZVI at 1.5 wt %
- ZVI at 3.0 wt %
- SMP at 46 mg/L P
- SMP at 138 mg/L P
- SMP at 277 mg/L P

Guar gum is an injection additive which will likely be required as a carrying fluid during injection of Apatite II; therefore, one set of Apatite II reactors will be amended with guar gum to evaluate the effect of guar gum on remedy performance.

Apatite II (PIMS NW, Inc.) and ZVI (Hepure Ferox Flow [125 µm]) will be dosed based on dry weight percentage. A subsample of homogenized overburden soil will be dried to determine the moisture content, which will be used to calculate the amount of soil and solid treatment amendments (Apatite II and ZVI) on a dry weight percentage needed for each reactor. SMP (Carus Corporation) doses were chosen to give equivalent total phosphorus loading to the Apatite II reactors based on the composition of Apatite II (e.g., ~18 wt.% P).

Once prepared, reactors will be sealed with rubber stoppers. Reactors will be agitated continuously (e.g., placed on a shaker table) for the duration of the study.

Batch Reactor Test

One of the three identical reactors for each condition will be sacrificially sampled at three time points: 3 days, 1 week, and 8 weeks.

At each time point, a subsample of the reactor will be centrifuged, and the supernatant will be analyzed for total U (ICP-MS/ICP-OES). Uranium measured in the supernatant sample consists of dissolved and colloidal uranium, which is equivalent to the fraction of uranium analyzed in the unfiltered column effluent samples in the TS ISS-2 column study (described below). A second subsample will be filtered using a syringe filter (0.45 µm), and the first milliliter of filtrate will be discarded. The remaining filtrate will be analyzed for pH, ORP, inorganic carbon (coulometry), nitrate¹⁵ (colorimetry), sulfide¹⁵ (turbidimetry), and total uranium, calcium, phosphorus, arsenic, manganese, molybdenum, and iron¹³ (ICP-MS/ICP-OES), which represent the dissolved fraction of each constituent. Control tests will be conducted to ensure that any artifacts of filtration on dissolved concentrations (retaining analytes on the filter) are minimal. Sampling and analyses performed during the batch reactor test are summarized in **Table 3**.

Additionally, the sorption capacity of SMP on overburden soil will be determined to evaluate uptake of phosphate by overburden soil in the absence of uranium. Batch reactors containing low-uranium overburden soil and background overburden groundwater from MW-S30 will be amended with increasing concentrations of SMP and allowed to equilibrate for at least 24 hours. Following equilibration, a subsample will be collected, filtered (0.45 µm), and analyzed for total phosphorus (ICP-MS/ICP-OES).

Batch Reactor Outcome

Results from the ISS-2 batch reactor study will be used to determine the best performing dose for each treatment. The best performing dose will be the lowest dose that decreases aqueous uranium concentrations below the maximum contaminant level (MCL) of 30 parts per billion by weight (ppb). If the MCL is not achieved, the dose which results in the largest percent removal of uranium from solution will be deemed the best performing dose. The best performing dose from each treatment will be used in the ISS-2 column study described below.

4.2.4 Column Test

Column Construction

Flow-through columns will be constructed from clear schedule 40 PVC with threaded end-caps fitted with tubing to deliver column influent and receive column effluent. Columns will measure 3 inches in diameter and 18 inches in length, the same column dimensions used in TS ISS-1.

Two column studies will be performed. The first column study will evaluate treatment amendment performance using homogenized, low-uranium overburden soil classified as sand (the same soil as the batch reactor study). Two identical columns containing homogenized overburden soil will be constructed for each of the following conditions (eight columns total) (**Table 2**):

- Unamended soil (control columns)
- Soil amended with Apatite II

¹⁵ Nitrate, iron, and sulfide will be measured in select samples as redox indicators.

- Soil amended with ZVI
- Unamended soil treated with SMP

Apatite II and ZVI will be amended to columns based on the best performing dose of the TS ISS-2 batch reactor study on a dry weight percentage. A subsample of homogenized overburden soil will be dried to determine the moisture content, which will be used to calculate the amount of ZVI and Apatite II needed for each column on a dry weight percentage. ZVI and Apatite II will be mixed with overburden soil prior to column packing.

The SMP treatment columns will be packed with unamended soil, and SMP will be loaded on the column via the influent solution prior to starting the column test. Background overburden groundwater from monitoring well MW-S30 will be dosed with SMP based on the best performing dose of the batch reactor study. The SMP-amended groundwater will be pumped through the column for approximately 1 week to load the column with the amendment. Once phosphorus breakthrough is observed (indicated by detection of phosphorus in the column effluent as described below), the column influent will be switched to uranium-rich groundwater from MW-S24 and the column study will commence.

The second column study will be performed to evaluate (1) phosphate transport in overburden soil and (2) the potential for mobilization of uranium from overburden soil due to changing geochemical conditions associated with the phosphorus-based treatments (e.g., changes in ionic strength, pH). Characterization of phosphate mobility in overburden soil will be necessary to design an injection program for the phosphorus-based amendments under consideration (Apatite II and SMP). This column will be constructed with high-uranium content overburden soil (**Table 2**).

Columns will be packed with soil using a plastic scoop. The columns will be shaken/tapped/swirled throughout the packing process to get even compaction of the soil into the column and to avoid creating preferential flow paths and voids in the packed columns. The mass of soil added to each column will be recorded.

Column Testing Procedures

Uranium-rich groundwater from MW-S24 amended with a conservative tracer (e.g., bromide) will serve as column influent for the first column study (treatment evaluation columns). Columns will be oriented vertically, and column influent will be pumped at approximately 2 PVs per day in an upflow arrangement using a peristaltic pump. The preferred flow rate will be determined based on measurement of the uranium breakthrough time of the control column, with a target of observing uranium breakthrough in less than 7 days in the control. The primary column for each treatment will be operated for approximately 7 weeks and will be used for effluent analysis. The secondary column for each treatment will be operated for approximately 6 weeks and will be used for solid-phase analysis. During the column test, the column influent will be altered according to the following schedule:

- Weeks 1-6: Influent for both the primary and secondary columns for each treatment will be sparged with air to promote oxidizing geochemical conditions. Oxidizing conditions reflect the redox conditions of the overburden groundwater. Effluent from the primary column of each treatment will be sampled once per week (6 time points total). Effluent from the secondary columns will not be sampled. Following 6 weeks of influent delivery, the secondary column for each treatment will be discontinued and undergo solid-phase analysis (described below).
- Week 7: Influent for the primary column of each treatment will be amended with sodium carbonate at a concentration representative of groundwater in contact with a cement slurry wall or the highest carbonate concentration expected at the Site.¹⁶ Alkaline conditions reflect geochemical conditions following leaching of alkaline fluids from a cement slurry wall, a remedy being considered for the HB VBW. Effluent from the primary columns will be sampled twice during the week of alkaline fluid delivery.

Unfiltered samples of column effluent will be collected periodically from the primary columns as described above and analyzed for the following:

- pH
- ORP
- Total uranium, calcium, phosphorus, arsenic, manganese, molybdenum, and iron (ICP-MS/ICP-OES)
- Inorganic carbon (coulometry)
- Nitrate (colorimetry)
- Sulfide (turbidimetry)
- Bromide (ion chromatography)

A summary of samples and analyses that will be conducted during the column test is presented in **Table 3**. Uranium will be monitored to assess the efficacy of soil amendments in sequestering uranium from overburden groundwater compared to the unamended column. If early breakthrough of uranium is observed from the SMP columns, SMP will be periodically reapplied to the column at a concentration relevant for practical field implementation (e.g., injectable dose). Calcium and phosphorus will be monitored to assess the dissolution of Apatite II as a source of calcium and phosphate and the abundance of SMP for uranium-phosphate mineral precipitation. Arsenic will be monitored to assess whether trace arsenic present in Apatite II has the potential to impact groundwater quality via dissolution and subsequent release of arsenic from Apatite II solids. Molybdenum will be monitored because it has been detected at elevated levels in the upgradient portion of the uranium plume. In addition to ORP, nitrate, iron (i.e., higher dissolved iron concentrations reflect more reducing conditions), and sulfide will be monitored in select samples as redox indicators. As carbonate enhances uranium mobility in groundwater, inorganic carbon

¹⁶ Additional studies will be conducted by the VBW contractor to determine the appropriate carbonate concentration for this part of the study.

(i.e., total carbonate species) will be monitored to evaluate this effect under the conditions of this study.

Analysis of unfiltered column effluent samples was selected because it provides a more conservative estimate of amendment performance than analysis of filtered column effluent samples. Additional analysis of dissolved constituents (i.e., those capable of passing through a 0.45 µm filter) may be performed as needed based on the results of unfiltered samples.

After 6 weeks of operation, the secondary column for each treatment will be disassembled, and the soil will be analyzed to quantify the amount of uranium sequestered, the solid-phase speciation of uranium, and the recalcitrance of the solid-phase uranium to leaching under relevant geochemical conditions. Two subsamples will be collected from each secondary column, one near the column influent and one near the column effluent. The eight samples will be analyzed for the following:

- Environmentally accessible (acid digestible by EPA Method 3050B) uranium, calcium, phosphorus, and iron (ICP-MS/ICP-OES).
- Bicarbonate leaching test: a bicarbonate leaching test will be performed to assess the potential for overburden groundwater with elevated inorganic carbon concentrations to mobilize uranium sequestered by each treatment. A subsample of column soil will be equilibrated with background (i.e., low-uranium) overburden groundwater from MW-S30 amended with 70 mg/L sodium bicarbonate solution and adjusted to pH 7.0. This concentration represents the maximum inorganic carbon concentration expected in overburden groundwater, based on historical groundwater data and effluent concentrations in previously conducted column tests.¹⁷ Following at least 24 hours of equilibration, an aliquot will be collected, filtered (0.45 µm), and analyzed for total uranium (ICP-MS/ICP-OES). The results of the leaching test will indicate the degree of recalcitrance of the uranium solids generated by each treatment.
- Four-step sequential extraction: subsamples of column soil will be sequentially extracted by four different solutions, each of which will target a specific fraction of solid-associated uranium. The sequential extraction procedure is based on the Tessier extraction method for trace metals (Tessier et al. 1979)¹⁸ which has been modified to target solid-associated uranium species based the geochemical behavior of uranium (Salome et al. 2017)¹⁹. Following each extraction step, the soil/extractant mixture will be centrifuged and an aliquot of the supernatant will be collected for analysis. The supernatant will be filtered using a syringe filter (0.45 µm), the first milliliter of filtrate will be discarded, and the

¹⁷ Lammers, L.N., Rasmussen, H., Adilman, D., deLemos, J.L., Zeeb P., Larson, D.G., Quicksall, A.N. 2017. Groundwater uranium stabilization by metastable hydroxyapatite. *Applied Geochemistry* 84, 105-113.

¹⁸ Tessier, A., Campbell, P.G.C., Bisson, M., 1979. Sequential extraction procedure for the speciation of particulate trace metals. *Anal. Chem.* 51, 844e851.

¹⁹ Salome, K.R., Beazley, M.J., Webb, S.M., Sobecky, P.A., and Taillefert, M., 2017. Biomineralization of U(VI) phosphate promoted by microbially-mediated phytate hydrolysis in contaminated soils. *Geochim. et Cosmochim. Acta.* 197, 27-42.

remaining filtrate will be analyzed for total uranium, calcium, phosphorus, and iron (ICP-MS/ICP-OES). The remaining supernatant will be discarded, and the solid residue will be washed once with deionized water before continuing to the next extraction step. The extraction solutions and target uranium species are listed below in order from least to most recalcitrant:

- Step 1: Weakly sorbed/exchangeable uranium. 1.0 molar (M) magnesium chloride in 10 millimolar (mM) nitrilotriacetic acid adjusted to pH 4.5, agitated at room temperature for 1 hour. Nitrilotriacetic acid is included in the extraction solution to prevent the precipitation of uranium desorbed from mineral surfaces by magnesium.
- Step 2: Strongly sorbed/weak acid extractable. 1.0 M sodium acetate adjusted to pH 5.0 with acetic acid, agitated at room temperature for 8 hours. Acetic acid will extract strongly sorbed uranium and uranium associated with carbonate minerals.
- Step 3: Iron- and manganese-associated. 40 mM hydroxylamine in 25% (volume/volume) acetic acid, agitated at 96 degrees Celsius (°C) for 6 hours. Hydroxylamine will target iron- and manganese-oxide associated uranium by reductive dissolution of iron and manganese oxides.
- Step 4: Uranium phosphate minerals and recalcitrant fraction. Reverse aqua regia (3 parts nitric acid [HNO₃] to 1 part hydrochloric acid [HCl]). Acid boiled off at 90 °C, and more acid added until digestion is complete. Dissolve salts in 5% HNO₃ for analysis. This extraction step has previously been shown to target uranium-phosphate solids.²⁰

The sequential extraction procedure will be calibrated to confirm the target uranium phase for each extraction step. Soils predominantly composed of one solid-associated uranium species (adsorbed uranium, uranium-phosphate minerals, or iron- and manganese-associated uranium) will be prepared as follows:

- Adsorbed uranium: high-uranium groundwater from MW-S24 will be mixed with background (low-uranium) overburden Site soil from the new soil boring at the distal end of the plume (TB SB-01).
- Uranium-phosphate minerals: high-uranium groundwater from MW-S24 will be mixed with Apatite II solids.
- Iron- and manganese-associated uranium: high-uranium groundwater from MW-S24 will be mixed with ZVI solids.

After 8 weeks of reaction, the calibration solids will be analyzed. Low-uranium soil from TS-SB-01 will also be analyzed to serve as a control blank to account for any background uranium present

²⁰ Lammers, L.N., Rasmussen, H., Adilman, D., deLemos, J.L., Zeeb P., Larson, D.G., Quicksall, A.N., 2017. Groundwater uranium stabilization by metastable hydroxyapatite. *Applied Geochemistry* 84, 105-113.

in the soil. A subsample of the calibration matrix will be centrifuged, and the supernatant will be discarded. The remaining solids will be washed once in deionized water, and each step of the sequential extraction procedure will be performed in parallel (i.e., not sequentially) on subsamples of the washed solids, following the procedures described above. Total uranium will be measured (ICP-MS/ICP-OES) in the filtered extractant from each extraction step to identify the extraction step that targets the majority of each solid-associated uranium species.

The mass of uranium sequestered by each treatment will be determined by 1) solid phase analysis and 2) mass balance. The mass of environmentally accessible uranium in the column solid after the completion of the column test will be measured and compared to the mass of environmentally available uranium measured during baseline characterization to determine the mass of uranium sequestered by each amendment. Additionally, mass balance calculations using the uranium concentration of the column influent, the uranium concentration of the column effluent, and the total volume flowed through the column will be used to calculate the mass of uranium sequestered on column solids.

Solids for which uranium concentrations are determined to be between 0.1 and 0.5 wt % uranium will be analyzed by X-ray diffraction (XRD) analysis. Up to four solid samples will be dried and analyzed using an automated X-ray diffractometer with copper K-alpha (Cu K- α) X-rays. The diffraction pattern and d-spacings will be matched against a published database (International Center for Diffraction Data Powder Diffraction File-2 [PDF-2]) to determine mineral identity. The goal of this analysis will be to identify the specific predominate uranium mineral in each of the columns that have concentrations of uranium suitable for XRD analysis. If solids contain less than 0.1 wt % uranium or if crystalline uranium phases cannot be resolved by XRD then an alternative approach will be used to identify the uranium minerals formed. The soil samples with the highest concentrations of uranium (up to two samples) will be prepared for Quantitative Evaluation of Minerals by SCANning electron microscopy (QEMSCAN) analysis. Soil will be embedded in epoxy and polished to prepare a smooth surface for the analysis. QEMSCAN combines imaging and high-resolution energy-dispersive X-ray spectroscopy analysis to identify minerals based upon their elemental composition. Although this analytical procedure is significantly more expensive than XRD, if none of the solids contain more than 0.1% uranium, then this procedure is warranted.

For the second column study (evaluation of phosphate transport and uranium leaching), column influent will consist of background (i.e., low-uranium content) overburden groundwater from MW-S30 equilibrated with Apatite II and amended with a conservative tracer (e.g., bromide). Apatite II will be added to the influent container in excess of the solubility (approximately 27 mg/L Apatite II, based on measured concentration of phosphorus in site groundwater equilibrated with Apatite II, Lammers et al., 2017)²¹ to maintain a constant influent phosphorus concentration of approximately 5 mg/L. Columns will be oriented vertically, and column influent will be pumped at approximately 1 PV/day in an upflow arrangement using a peristaltic pump.

²¹ Lammers, L.N., Rasmussen, H., Adilman, D., deLemos, J.L., Zeeb P., Larson, D.G., Quicksall, A.N., 2017. Groundwater uranium stabilization by metastable hydroxyapatite. *Applied Geochemistry* 84, 105-113.

The column will be operated for up to 6 weeks. Unfiltered samples of column effluent will be collected once per week and analyzed for the following:

- pH
- ORP
- Total uranium, calcium, phosphorus, arsenic, manganese, molybdenum, and iron (ICP-MS/ICP-OES)
- Inorganic carbon (coulometry)
- Nitrate (colorimetry)
- Sulfide (turbidimetry)
- Bromide (ion chromatography)

Column Test Outcome

Results from the first TS ISS-2 column study will be used to determine the most effective treatment for sequestering uranium in overburden groundwater. Treatments will be evaluated based on the percent decrease in uranium concentration in the column effluent compared to the unamended control column and the recalcitrance of the solid-associated uranium generated by each treatment. Results of the second TS ISS-2 column study will provide information on the mobility of phosphorus in overburden soil, which will inform the design of the injection program if a phosphorus-based amendment is selected. The information gained from the TS will be used to guide design and implementation of PDI ISS-3.

4.3 Assumptions

The scope of work for this TS PDI includes the following assumptions:

- Overburden soil will be available for sampling and accessible using a sonic drilling rig.
- The required volume of groundwater can be collected that contains elevated concentrations of uranium (> 2.5 mg/L).
- de maximis will provide an RSO to evaluate the activity level of soil and groundwater to be shipped off-site. If the activity levels exceed the threshold for more rigorous packaging and shipping methods, de maximis will manage the packaging and shipping.

There will be adequate time for the TS to be finished prior to the requirement to submit the PDI Report.

5. TS ISS – 3: AMENDMENT SELECTION FOR BEDROCK GROUNDWATER

5.1 Purpose

Prior to initiation of the bedrock groundwater treatability testing (TS ISS-3), a bedrock drilling and groundwater pumping program will be conducted as presented in PDI ISS-2 - Bedrock Pumping and Rebound Testing. The purpose of this pumping is to evaluate if enough uranium mass can be removed to lower uranium concentrations in bedrock groundwater, thereby

remediating via pumping alone, without the need to add amendments to bedrock. If results of PDI ISS-2 indicate that amendments are needed in bedrock, then TS ISS-3 will be initiated according to the plan presented below and illustrated on Figure 1C.

TS ISS-3 will evaluate the performance of three treatment amendments (Apatite II, STPP, and ZVI) at immobilizing uranium in bedrock groundwater via precipitation of uranium solids. The success of treating uranium in bedrock groundwater will rely on identifying an amendment that is both effective under bedrock groundwater conditions and is able to be injected into the target treatment area. For instance, although solid amendments such as Apatite II and ZVI may be more effective at sequestering uranium in bedrock groundwater due to the longevity of the Apatite II treatment and the performance of ZVI under more reducing conditions, the unknown nature of fracture aperture, density, and overall connectivity, combined with low porosity, will make delivery of solid amendments challenging. In turn, although a soluble amendment, such as STPP, has shorter longevity in bedrock fractures, it would be more easily delivered to the target treatment area. Thus, as a first step in identifying the most suitable treatment amendment for bedrock groundwater, Apatite II, STPP, and ZVI will be evaluated in TS ISS-3 as amendments to treat uranium in bedrock groundwater. All testing will be completed as batch reactor studies as shown on **Figure 1C**.

5.2 Scope

5.2.1 Rock Matrix Collection

Crushed bedrock and groundwater will be collected for use in TS ISS-3.

As described in PDI ISS-2: Pumping and Rebound Analysis for Uranium in Bedrock Groundwater, four to six new bedrock pumping wells will be installed for evaluating the viability of a short-term bedrock pumping remedy. These wells will be installed using air rotary methods, and rock chips will be collected from the upper 20 feet of bedrock at the new well location. Crushed bedrock cuttings produced during the drilling process will be collected in laboratory-supplied containers. A minimum of approximately 2 L of crushed bedrock will be collected for use in TS ISS-3 (**Table 1**). Following collection, the samples will be labeled, placed in coolers with ice, and shipped to Hazen under standard chain-of-custody procedures. Bedrock samples will be homogenized and sieved to separate the sand-sized fraction to be used in TS ISS-3 batch reactors. Bedrock samples will be homogenized via cloth blending, use of a V-blender, or cone-and-quartering techniques. Representative sub-samples of homogenized bedrock will be collected based on visual inspection. To characterize the baseline uranium content and determine if the uranium content of the bedrock is consistent with historical data, the lab will analyze a subsample of this fraction for the following:

- Organic and inorganic carbon (combustion analysis)
- Environmentally accessible (acid digestible by EPA Method 3050B) uranium, iron, aluminum, manganese, molybdenum, arsenic, and calcium (ICP-MS/ICP-OES)
- Fe(II) (colorimetry)
- Sulfide (turbidimetry)
- Leachable uranium (SPLP)

Based on the results of baseline groundwater monitoring, bedrock groundwater will be collected from either GZW-10-2 or MW-BS03 (these wells have the highest concentrations of uranium [~70 ug/L] as of November 2019). The uranium distribution in bedrock groundwater is shown on Figure 4. Sampling methods will follow low-flow protocols as provided in SOP NMI-GW-010 of the FSP. During collection, filtered (0.45 µm) and unfiltered samples will be collected, and general groundwater quality parameters will be measured, including pH, DO, ORP, turbidity, and specific conductance. A minimum of approximately 13 L of groundwater will be collected and shipped to the laboratory (**Table 1**). Groundwater samples will be labeled and shipped to Hazen under standard chain-of-custody procedures. For baseline characterization, the laboratory will analyze filtered and unfiltered bedrock groundwater at the time of collection and prior to initiation of batch tests for the following:

- pH
- ORP
- Inorganic carbon (coulometry)
- Nitrate (colorimetry)
- Sulfide (turbidimetry)
- Total uranium, calcium, phosphorus, arsenic, manganese, molybdenum, and iron (ICP-MS/ICP-OES) as a baseline characterization.

Comparison of baseline characterization results between the time of collection and the initiation of batch tests will be used to identify changes in groundwater composition (e.g., precipitation, biological consumption) during transport and storage. The Hazen laboratory QA/QC procedures are included in **Attachment A** to this TS work plan.

5.2.2 Batch Reactor Construction

The batch reactor study will be performed using 250-mL glass bottles containing crushed bedrock and bedrock groundwater. The soil:liquid ratios for the batch reactors will be determined to (1) ensure sequestration by the amendment is detectable in excess of the control and (2) prevent dissolution of greater than 10% of the Apatite II amendment in the low-dose condition. Based on the current maximum uranium concentration in bedrock groundwater of 70 µg/L (November 2019), a soil:liquid ratio of 1:200 is initially assumed (equivalent to 1 g of dry bedrock per 200 mL of liquid). Preliminary tests will be conducted to identify the appropriate soil:liquid ratio for the batch reactor test.

Three identical reactors containing crushed bedrock and groundwater will be constructed for each of the following treatments (39 reactors in total) (**Table 2**):

- Unamended control reactor
- Apatite II at 0.5 wt %
- Apatite II at 1.5 wt %
- Apatite II at 3.0 wt %
- Small particle size ZVI at 0.5 wt %

- Small particle size ZVI at 1.5 wt %
- Small particle size ZVI at 3.0 wt %
- Large particle size ZVI at 0.5 wt %
- Large particle size ZVI at 1.5 wt %
- Large particle size ZVI at 3.0 wt %
- STPP at 46 mg/L P
- STPP at 138 mg/L P
- STPP at 277 mg/L P

Apatite II (PIMS NW, Inc.) and ZVI (Hepure Ferox Flow [125 µm]) will be dosed based on dry weight percentage. A subsample of homogenized, crushed bedrock will be dried to determine the moisture content, which will be used to calculate the amount of crushed bedrock and solid treatment amendments (Apatite II and ZVI) needed for each reactor. Doses of STPP (Carus Corporation) were chosen to give equivalent total phosphorus loading to the Apatite II reactors based on the composition of Apatite II (e.g., ~18 wt.% phosphorus).

Once prepared, reactors will be sealed with a rubber stopper. Reactors will be agitated continuously (e.g., placed on a shaker table) for the duration of the study.

5.2.3 Batch Reactor Test

One of the three identical reactors for each condition will be sacrificially sampled at three time points: 3 days, 1 week, and 8 weeks.

At each time point, a subsample of the reactor will be centrifuged and the supernatant will be analyzed for total uranium (ICP-MS/ICP-OES). Uranium measured in the supernatant sample represents the sum of the dissolved and colloidal fractions of uranium. A second subsample will be filtered using a syringe filter (0.45 µm), and the first milliliter of filtrate will be discarded. The remaining filtrate will be analyzed for pH, ORP, inorganic carbon (coulometry), nitrate²² (colorimetry), sulfide²² (turbidimetry) and total uranium, calcium, phosphorus, arsenic, manganese, molybdenum, and iron²² (ICP-MS/ICP-OES), which represent the dissolved fraction of each constituent. Control tests will be conducted to ensure that any artifacts of filtration on dissolved concentrations (retaining analytes on the filter) are minimal.

The best performing amendment dose will be identified as the lowest dose which is able to decrease aqueous uranium concentrations to below the MCL (30 ppb). If an amendment is not able to achieve the MCL, the best performing dose will be identified as the dose which results in the largest percent decrease in uranium from solution compared to the control reactor. For the best performing dose for each amendment (three samples total), the solids generated in the 8-week reactor will be analyzed to quantify the solid-phase speciation of uranium, as follows:

²² Nitrate, iron, and sulfide will be measured in select samples as redox indicators.

Four-step sequential extraction: subsamples of column soil will be sequentially extracted by four different solutions, each of which will target a specific fraction of solid-associated uranium. The sequential extraction procedure is based on the Tessier extraction method for trace metals (Tessier et al. 1979²³) which has been modified to target solid-associated uranium species based the geochemical behavior of uranium (Salome et al. 2017²⁴). Following each extraction step, the soil/extractant mixture will be centrifuged and an aliquot of the supernatant will be collected for analysis. The supernatant will be filtered using a syringe filter (0.45 µm), the first milliliter of filtrate will be discarded, and the remaining filtrate will be analyzed for total uranium, calcium, phosphorus, and iron (ICP-MS/ICP-OES). The remaining supernatant will be discarded, and the solid residue will be washed once with deionized water before continuing to the next extraction step. The extraction solutions and target uranium species are listed below in order from least to most recalcitrant:

- Step 1: Weakly sorbed/exchangeable uranium. 1.0 M magnesium chloride in 10 mM nitrilotriacetic acid adjusted to pH 4.5, agitated at room temperature for 1 hour. Nitrilotriacetic acid is included in the extraction solution to prevent the precipitation of uranium desorbed from mineral surfaces by magnesium.
- Step 2: Strongly sorbed/weak acid extractable. 1.0 M sodium acetate adjusted to pH 5.0 with acetic acid, agitated at room temperature for 8 hours. Acetic acid will extract strongly sorbed uranium and uranium associated with carbonate minerals.
- Step 3: Iron- and manganese-associated. 40 mM hydroxylamine in 25% (volume/volume) acetic acid, agitated at 96 °C for 6 hours. Hydroxylamine will target iron- and manganese-oxide associated uranium by reductive dissolution of iron and manganese oxides.
- Step 4: Uranium phosphate minerals and recalcitrant fraction. Reverse aqua regia (3 parts HNO₃ to 1 part HCl). Acid boiled off at 90 °C, and more acid added until digestion is complete. Dissolve salts in 5% HNO₃ for analysis. This extraction step has previously been shown to target uranium-phosphate solids.²⁵

A summary of samples and analyses that will be conducted during the batch reactor test are summarized in **Table 3**.

²³ Tessier, A., Campbell, P.G.C., Bisson, M., 1979. Sequential extraction procedure for the speciation of particulate trace metals. *Anal. Chem.* 51, 844e851.

²⁴ Salome, K.R., Beazley, M.J., Webb, S.M., Sobecky, P.A., and Taillefert, M., 2017. Biomineralization of U(VI) phosphate promoted by microbially-mediated phytate hydrolysis in contaminated soils. *Geochim. et Cosmochim. Acta.* 197, 27-42.

²⁵ Lammers, L.N., Rasmussen, H., Adilman, D., deLemos, J.L., Zeeb P., Larson, D.G., Quicksall, A.N., 2017. Groundwater uranium stabilization by metastable hydroxyapatite. *Applied Geochemistry* 84, 105-113.

5.3 Outcome

Results from the ISS-3 batch reactor study will be used to determine the best performing dose for each treatment and the best treatment for sequestering uranium in bedrock groundwater. The best performing dose will be the lowest dose that decreases aqueous uranium concentrations below the MCL (30 ppb). If the MCL is not achieved, the dose which results in the largest percent removal of uranium from solution will be deemed the best performing dose.

5.4 Assumptions

The scope of work for this TS includes the following assumptions:

- An adequate volume of groundwater can be obtained from bedrock that contains elevated concentrations of uranium.
- There will be adequate time for the TS to be finished prior to the requirement to submit the PDI Report.
- de maximis will provide an RSO to evaluate the activity level of soil and groundwater to be shipped off-Site. If the activity levels exceed the threshold for more rigorous packaging and shipping methods, de maximis will manage the packaging and shipping.

6. SCHEDULE

A preliminary schedule for the scope of work described above is presented below:

Task	Months after Work Plan Approval									
	1	2	3	4	5	6	7	8	9	10
Collect HB Soil and Groundwater										
Collect Overburden Soil and Groundwater										
Collect Crushed Bedrock and Bedrock Groundwater										
TS ISS-1 - Column Test										
TS ISS-2 - Batch Reactor Test										
TS ISS-2 - Column Test										
TS ISS-3 - Batch Reactor Test										
Prepare Treatability Study Report										

7. REPORTING

Results from TS ISS-1, ISS-2, and ISS-3 will be detailed in final reports prepared by Hazen and submitted to Geosyntec. Geosyntec will evaluate and interpret the results from the TS to identify the most suitable amendment for each target treatment area. The status of this TS, and interim results, will be provided to USEPA during routine project meetings and status reports. Results and the outcome of the TS will be incorporated into the 30% RD report.

TABLES

Table 1
Summary of Soil and Groundwater Collection
 Nuclear Metals, Inc. Superfund Site
 Concord, Massachusetts

Treatability Study ISS-1: Reagent Testing for Sequestration of Uranium in Holding Basin Soils				
Column Test				
Soil				
Sample Location	Number of columns	Mass of soil per column ¹ (kg)	Mass of soil needed ² (kg)	Expected volume of soil needed ³ (L) <i>(assume dry soil = 1.6 g/cm³)</i>
High-uranium Holding Basin soil	3	5.5	26	17
Groundwater				
Sample Location	Number of columns	Pore Volume per column ⁴ (mL)	Duration of study (days)	Expected Volume of groundwater needed ² (L)
Low-uranium Holding Basin groundwater	3	521	28	67

Treatability Study ISS-2: Reagent Testing for Overburden Groundwater				
Batch Reactor Test				
Soil				
Sample Location	Number of reactors	Expected mass of soil per reactor ¹ (kg)	Mass of soil needed ² (kg)	Expected volume of soil needed ³ (L) <i>(assume dry soil = 1.6 g/cm³)</i>
Low-uranium overburden soil	39	0.01	2	2
Groundwater				
Sample Location	Number of reactors	Volume of groundwater per reactor (L)	Expected Volume of groundwater needed ² (L)	
High-uranium overburden groundwater	39	0.2	13	
SMP Sorption Capacity Test				
Soil				
Sample Location	Number of reactors	Expected mass of soil per reactor ¹ (kg)	Mass of soil needed ² (kg)	Expected volume of soil needed ³ (L) <i>(assume dry soil = 1.6 g/cm³)</i>
Low-uranium overburden soil	5	0.01	1	1
Groundwater				
Sample Location	Number of reactors	Volume of groundwater per reactor (L)	Expected Volume of groundwater needed ² (L)	
Low-uranium overburden groundwater	5	0.2	3	
Column Tests				
Soil				
Sample Location	Number of columns	Mass of soil per column ¹ (kg)	Mass of soil needed ² (kg)	Expected volume of soil needed ³ (L) <i>(assume dry soil = 1.6 g/cm³)</i>
Low-uranium overburden soil	8	5.5	67	42
High-uranium overburden soil	1	5.5	9	6
Groundwater				
Sample Location	Number of columns	Pore volume per column ⁴ (mL)	Duration of study (days)	Expected Volume of groundwater needed ² (L)
Low-uranium overburden groundwater (SMP Loading)	2	521	7	22 L for SMP loading
High-uranium overburden groundwater	8	521	49	613
Low-uranium overburden groundwater	1	521	42	33

Table 1
Summary of Soil and Groundwater Collection
 Nuclear Metals, Inc. Superfund Site
 Concord, Massachusetts

Treatability Study ISS-3: Reagent Testing for Bedrock Groundwater				
Batch Reactor Test				
Bedrock				
Sample Location	Number of reactors	Expected Mass of soil per reactor¹ (kg)	Mass of soil needed² (kg)	Expected volume of rock needed³ (L) (assume dry soil = 1.6 g/cm³)
Crushed bedrock	39	0.001	2	2
Groundwater				
Sample Location	Number of reactors	Volume of groundwater per reactor⁴ (L)	Expected Volume of groundwater needed² (L)	
High-uranium bedrock groundwater	39	0.2	13	

Notes:

1. Based on column dimensions (3 inch diameter, 18 inch length) and a bulk soil density of 2.65 grams per cubic centimeter.
2. Includes 50% contingency and additional sample (1 kg soil or 1 L groundwater) for baseline characterization.
3. If soil is wet, add approximately 30% volume.
4. Based on column dimensions (3 inch diameter, 18 inch length) and a bulk soil porosity of 25%.

kg = kilogram

L = liter

mL = milliliter

SMP = sodium monophosphate

Table 2
Summary of Batch Reactor and Column Tests
 Nuclear Metals, Inc. Superfund Site
 Concord, Massachusetts

Treatability Study ISS-1: Reagent Testing for Sequestration of Uranium in Holding Basin Soils				
Column Test				
Column Number	Column Name	Soil	Soil Amendment	Column Influent
1	ISS1-Ap	High-uranium Holding Basin Soil	1.5 wt% Apatite II	Low-uranium Holding Basin groundwater Week 1: aerobic (air-sparged) Weeks 2-3: anaerobic (glucose-amended) Week 4: aerobic (air-sparged)
2	ISS1-ZVI		1.0 wt% ZVI	
3	ISS1-Ctrl		Unamended control	

Treatability Study ISS-2: Reagent Testing for Overburden Groundwater				
Batch Reactor Test				
Reactor Number	Reactor Name	Expected Batch Reactor Composition	Reactor Amendment	Duration
1-3	ISS2-Ap1	10 g Low-uranium overburden soil 200 mL High-uranium overburden groundwater	0.5 wt% Apatite II	8 weeks
4-6	ISS2-Ap2		1.5 wt% Apatite II	
7-9	ISS2-Ap3		3.0 wt% Apatite II	
10-12	ISS2-ApG1		0.5 wt% Apatite II + guar gum	
13-15	ISS2-ApG2		1.5 wt% Apatite II + guar gum	
16-18	ISS2-ApG3		3.0 wt% Apatite II + guar gum	
19-21	ISS2-SMP1		SMP at 46 mg/L P	
22-24	ISS2-SMP2		SMP at 138 mg/L P	
25-27	ISS2-SMP3		SMP at 277 mg/L P	
28-30	ISS2-ZVI1		0.5 wt% ZVI	
31-33	ISS2-ZVI2		1.5 wt% ZVI	
34-36	ISS2-ZVI3		3.0 wt% ZVI	
37-39	ISS2-Ctrl		Unamended Control	

SMP Sorption Capacity Test				
Reactor Number	Reactor Name	Expected Batch Reactor Composition	Reactor Amendment	Duration
1	SMP-1	10 g Low-uranium overburden Soil 200 mL Low-uranium overburden groundwater	SMP at 10 mg/L P	At least 24 hours
2	SMP-2		SMP at 50 mg/L P	
3	SMP-3		SMP at 100 mg/L P	
4	SMP-4		SMP at 200 mg/L P	
5	SMP-5		SMP at 300 mg/L P	

Column Test				
Column Number	Column Name	Soil	Soil Amendment	Column Influent
1	ISS2-ApA	Low-uranium overburden soil (same as ISS-2 batch reactor study)	Best performing Apatite II dose from ISS-2 batch reactor test	High-uranium overburden groundwater (same as ISS-2 batch reactor study) Weeks 1-6: aerobic (air-sparged) Week 7: highly alkaline (Na ₂ CO ₃ amended)
2	ISS2-ApB		Best performing SMP dose from ISS-2 batch reactor test	
3	ISS2-SMPA		Best performing ZVI dose from ISS-2 batch reactor test	
4	ISS2-SMPB		No amendment (control)	
5	ISS2-ZVIA			
6	ISS2-ZVIB			
7	ISS2-CtrlA			
8	ISS2-CtrlB			
9	ISS2-Leach	High-uranium overburden soil	No amendment	Low-uranium overburden groundwater equilibrated with Apatite II

Table 2
Summary of Batch Reactor and Column Tests
 Nuclear Metals, Inc. Superfund Site
 Concord, Massachusetts

Treatability Study ISS-3: Reagent Testing for Bedrock Groundwater				
Batch Reactor Test				
Reactor Number	Reactor Name	Expected Batch Reactor Composition	Treatment Amendment	Duration
1-3	ISS3-Ap-1	1 g Crushed bedrock 200 mL High-uranium bedrock groundwater	0.5 wt% Apatite II	8 weeks
4-6	ISS3-Ap-2		1.5 wt% Apatite II	
7-9	ISS3-Ap-3		3.0 wt% Apatite II	
10-12	ISS3-STPP-1		STPP at 46 mg/L P	
13-15	ISS3-STPP-2		STPP at 138 mg/L P	
16-18	ISS3-STPP-3		STPP at 277 mg/L P	
19-21	ISS3-ZVI-A1		0.5 wt% ZVI - small particle size	
22-24	ISS3-ZVI-A2		1.5 wt% ZVI - small particle size	
25-27	ISS3-ZVI-A3		3.0 wt% ZVI - small particle size	
28-30	ISS3-ZVI-B1		0.5 wt% ZVI - large particle size	
31-33	ISS3-ZVI-B2		1.5 wt% ZVI - large particle size	
34-36	ISS3-ZVI-B3		3.0 wt% ZVI - large particle size	
37-39	ISS3-Ctrl		Unamended control	

Notes:

g = grams

mg/L = milligram per liter

mL = milliliter

P = phosphorus

SMP = sodium monophosphate

STPP = soluble triphosphate

wt % = weight percent

Table 3
Summary of Samples and Analyses
 Nuclear Metals, Inc. Superfund Site
 Concord, Massachusetts

Treatability Study ISS-1: Reagent Testing for Sequestration of Uranium in Holding Basin Soils				
Baseline Characterization				
Sample Matrix	Time		Sample Preparation	Analyses
High-uranium Holding Basin Soil	Baseline characterization prior to starting column test		Homogenized	- Organic and inorganic carbon (combustion analysis) - Acid digestion for total U, Fe, Al, Ca, Mn, Mo, As (ICP-MS/ICP-OES) - Leachable U (SPLP, ICP-MS) - Fe(II) (colorimetry) - Sulfide (turbidimetry)
Column influent (Low-uranium Holding Basin groundwater)	Baseline characterization at time of sampling		Unfiltered and filtered (0.45 µm)	- Field parameters: pH, ORP, DO, turbidity, specific conductance - pH - ORP
	Baseline characterization prior to starting column test		Unfiltered and filtered (0.45 µm)	- Inorganic carbon (coulometry) - Nitrate (colorimetry) - Total U, Ca, P, As, Mn, Mo, and Fe (ICP-MS/ICP-OES)
Column Test				
Sample Matrix	Time		Sample Preparation	Analyses
Column effluent	Week 1 (aerobic)	Days 2, 4, and 7	Unfiltered	- pH - ORP
	Weeks 2-3 (anaerobic)	Days 2, 7, and 14	Unfiltered	- Total U, Ca, P, As ¹ , Mo ¹ , Mn, and Fe ² (ICP-MS/ICP-OES) - Nitrate ² (colorimetry) - Sulfide ² (turbidimetry)
	Week 4 (aerobic)	Days 2, 4, and 7	Unfiltered	- Inorganic carbon (coulometry)

Table 3
Summary of Samples and Analyses
 Nuclear Metals, Inc. Superfund Site
 Concord, Massachusetts

Treatability Study ISS-2: Reagent Testing for Overburden Groundwater				
Baseline Characterization				
Sample Matrix	Time		Sample preparation	Analyses
Low-uranium overburden soil (Batch and column treatment tests)	Baseline characterization prior to starting batch and column tests		Homogenized	- Organic and inorganic carbon (combustion analysis) - Acid digestion for total U, Fe, Mn, Mo, As, Al, and Ca (ICP-MS/ICP-OES) - Leachable U (SPLP, ICP-MS)
High-uranium overburden soil (phosphate transport column test)	Baseline characterization prior to starting batch and column tests		Homogenized	- 4-step sequential extraction for U ³ (high-uranium soil only) - Fe(II) (colorimetry) - Sulfide (turbidimetry)
Low-uranium Overburden Groundwater (SMP sorption test, phosphate transport column test)	Baseline characterization at time of sampling		Unfiltered and filtered (0.45 µm)	- Field parameters: pH, ORP, DO, turbidity, specific conductance - pH - ORP
	Baseline characterization prior to starting batch and column tests			
High-uranium overburden groundwater (Batch and column treatment tests)	Baseline characterization at time of sampling		Unfiltered and filtered (0.45 µm)	- Inorganic carbon (coulometry) - Total U, Ca, P, As, Mo, Mn, and Fe (ICP-MS/ICP-OES) - Nitrate (colorimetry) - Sulfide (turbidimetry)
	Baseline characterization prior to starting batch and column tests			
Batch Reactor Test				
Sample Matrix	Time		Sample preparation	Analyses
Batch Reactors			Centrifuge, unfiltered supernatant	- Total U (ICP-MS/ICP-OES)
	Day 3 Day 7 (1 week) Day 56 (8 weeks)		Filtered (<0.45 µm)	- pH - ORP - Inorganic carbon (coulometry) - Total U, Ca, P, As ¹ , Mo ¹ , Mn, and Fe ² (ICP-MS/ICP-OES) - Nitrate ² (colorimetry) - Sulfide ² (turbidimetry)
SMP Sorption Capacity Test				
Sample Matrix	Time		Sample preparation	Analyses
Batch Reactors	After at least 24 hours of equilibration		Filtered (<0.45 µm)	- Total P (ICP-MS/ICP-OES)
Column Test (treatment evaluation)				
Sample Matrix	Time		Sample Preparation	Analyses
Primary column effluent	SMP loading period (SMP treatment column only)	Once per day for approximately 7 days	Unfiltered	- Total P (ICP-MS/ICP-OES)
	Unamended groundwater influent period	Once per week for 6 weeks	Unfiltered	- pH - ORP - Total U, Ca, P, As ¹ , Mo ¹ , Mn, and Fe ² (ICP-MS/ICP-OES) - Inorganic carbon (coulometry)
	Alkaline groundwater influent period	Twice per week for 1 week	Unfiltered	- Nitrate ² (colorimetry) - Sulfide ² (turbidimetry)
Secondary column soil	After completion of 6-week unamended groundwater influent period		Centrifuge, remove supernatant	- Acid digestion for total U, Ca, P, Fe (ICP-MS/ICP-OES) - Bicarbonate leaching test ³ , U (ICP-MS) - 4-step sequential extraction ³ , U, Ca, P, and Fe (ICP-MS/ICP-OES) - XRD or QEMSCAN on select samples
Column Test (phosphate transport and uranium leaching evaluation)				
Sample Matrix	Time		Sample Preparation	Analyses
Column effluent	Once per week for up to 6 weeks		Unfiltered	- pH - ORP - Total U, Ca, P, As ¹ , Mn, and Fe ² (ICP-MS/ICP-OES) - Inorganic carbon (coulometry) - Nitrate ² (colorimetry) - Sulfide ² (turbidimetry)

Table 3
Summary of Samples and Analyses
 Nuclear Metals, Inc. Superfund Site
 Concord, Massachusetts

Treatability Study ISS-3: Reagent Testing for Bedrock Groundwater			
Baseline Characterization			
Sample Matrix	Time	Sample preparation	Analyses
Crushed bedrock	Baseline characterization prior to starting batch reactor test	Homogenized and sieved (target sand fraction)	- Organic and inorganic carbon (combustion analysis) - Acid digestion for total U, Fe, Al, Ca, Mn, Mo, and As (ICP-MS/ICP-OES) - Leachable U (SPLP, ICP-MS) - Fe(II) (colorimetry) - Sulfide (turbidimetry)
Bedrock groundwater	Baseline characterization at time of sampling	Unfiltered and filtered (0.45 µm)	- pH - ORP - Inorganic carbon (coulometry)
	Baseline characterization prior to starting batch tests		- Total U, Ca, P, As, Mn, Mo, and Fe (ICP-MS/ICP-OES) - Nitrate (colorimetry) - Sulfide (turbidimetry)
Batch Reactor Test			
Sample Matrix	Time	Sample preparation	Analyses
Batch reactors (aqueous analyses)	Day 3 Day 7 (1 week) Day 56 (8 weeks)	Centrifuge, unfiltered supernatant	- Total U (ICP-MS/ICP-OES)
		Filtered (<0.45 µm)	- pH - ORP - Inorganic carbon (coulometry) - Total U, Ca, P, As ¹ , Mo ¹ , Mn, and Fe ² (ICP-MS/ICP-OES) - Nitrate ² (colorimetry) - Sulfide ² (turbidimetry)
Batch reactors (solid analyses)	Day 56 (Week 8)	Centrifuge, remove supernatant	- 4-step sequential extraction ³ on best-performing dose for each treatment, U, Ca, P, and Fe (ICP-MS/ICP-OES)

Notes:

1. Arsenic and molybdenum will be analyzed less frequently than the other analytes.
2. Nitrate, dissolved iron, and sulfide will be analyzed in select samples to monitor the redox conditions of the batch reactors and columns.
3. Details of the bicarbonate leaching test and 4-step sequential extraction are provided in the text.

µm = micrometer

As = arsenic

Ca = calcium

DO = dissolved oxygen

EPA = United States Environmental Protection Agency

Fe = iron

ICP-MS = inductively coupled plasma mass spectrometry

ICP-OES = inductively coupled plasma optical emission spectroscopy

ORP = Oxidation reduction potential

P = phosphorus

QEMSCAN = Quantitative Evaluation of Minerals by SCANing electron microscopy

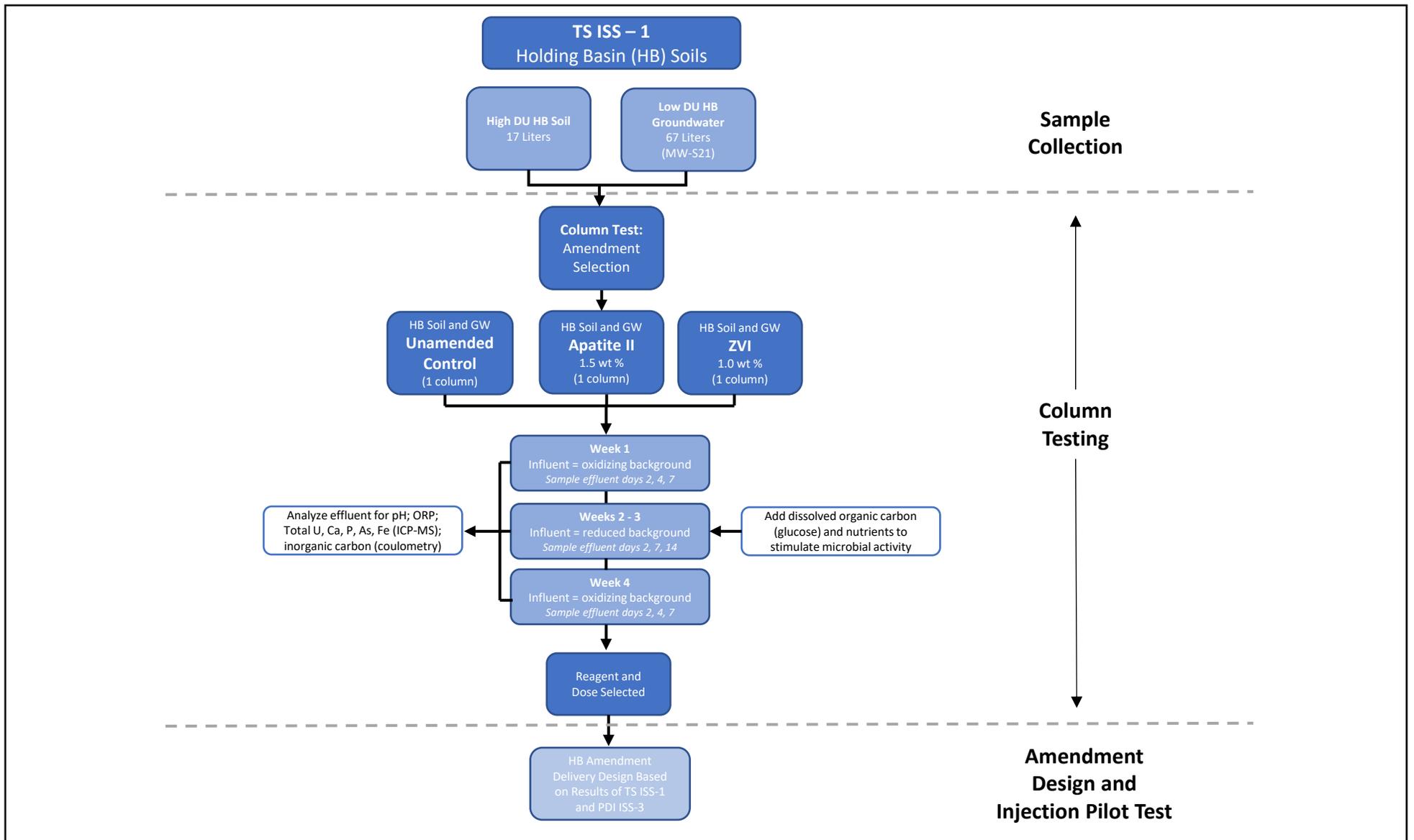
SMP = sodium monophosphate

SPLP = synthetic precipitation leaching procedure

U = uranium

XRD = X-ray diffraction

FIGURES



Notes:
 GW = groundwater
 ISS = in situ stabilization
 mg/L = milligram per liter
 P = phosphorus
 SMP = sodium monophosphate
 STPP = sodium tripolyphosphate
 TS = treatability study
 U = uranium
 Wt % = weight percentage
 ZVI = zero valent iron

Flow Chart for Holding Basin Uranium in Soils Treatability Testing (TS ISS-1)

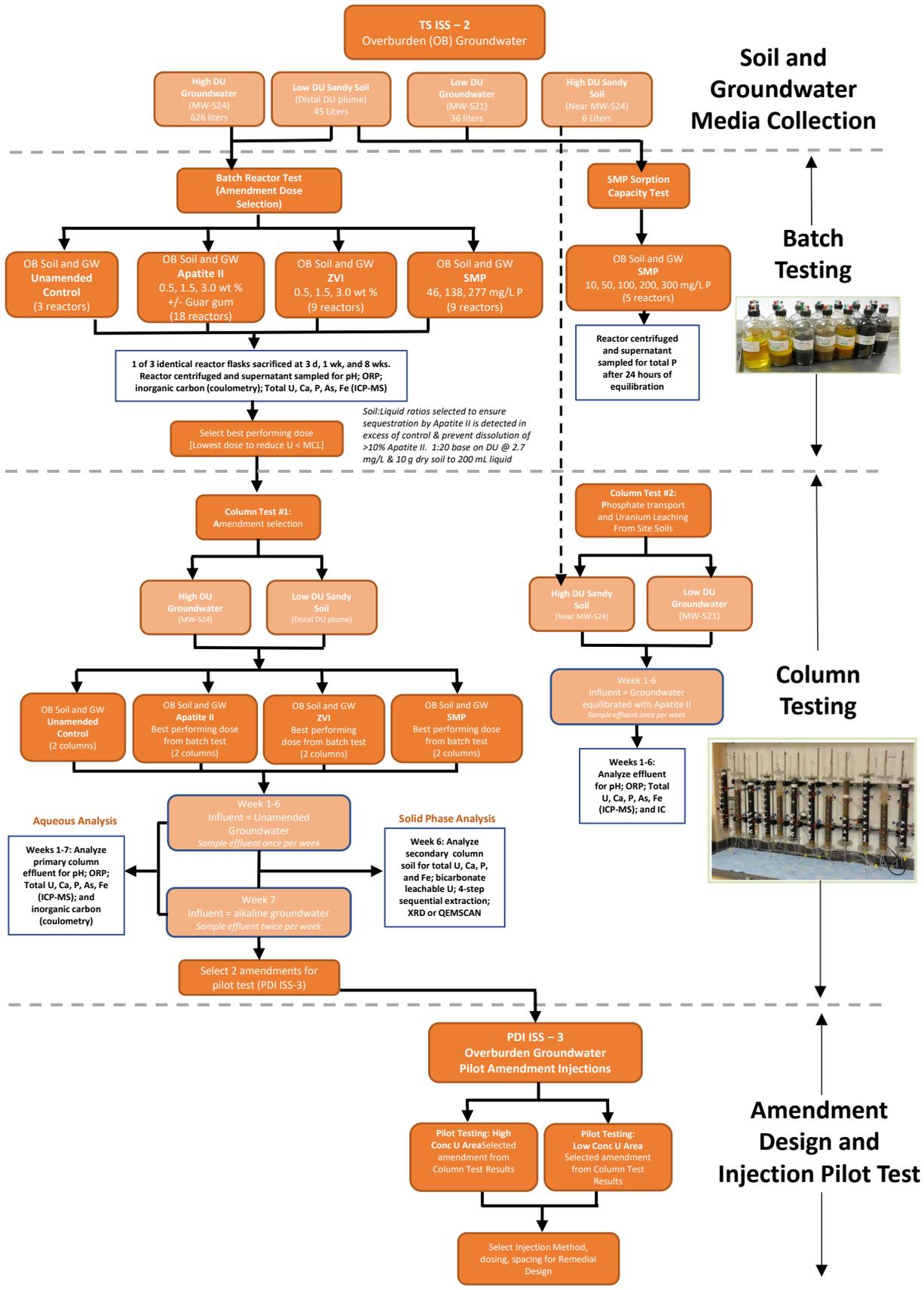
Nuclear Metals, Inc. Superfund Site
 Concord, Massachusetts

Geosyntec
 consultants

BR0090

September 2020

**Figure
 1A**



Notes:
 GW = groundwater
 ISS = in situ stabilization
 mg/L = milligram per liter
 P = phosphorus
 SMP = sodium monophosphate
 STPP = sodium triphosphate
 TS = treatability study
 U = uranium
 Wt % = weight percentage
 ZVI = zero valent iron

Flow Chart for Overburden Uranium in Groundwater Treatability Testing (TS ISS-2)

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Figure
1B

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PDI ISS-2
Bedrock Pumping & Rebound Testing for Uranium Removal

Results indicate Pumping/Treating **Is Not a Viable** Short-term Remedy

Results indicate Pumping/Treating **is a viable** Short-term Remedy

Perform Treatability Testing for Uranium in Bedrock

Design Pumping Remedy

Bedrock Pumping and Rebound Testing

TS ISS – 3
Bedrock Groundwater (GW)

Crushed Bedrock
2 Liters

Uranium Impacted
Bedrock Groundwater
13 Liters

Treatability Testing
Bedrock and Groundwater
Media Collection

Batch Reactor Test:
Amendment Selection

Crushed rock and GW
Unamended Control
(3 reactors)

Crushed rock and GW
Apatite II
0.5, 1.5, 3.0 wt %
(9 reactors)

Crushed rock and GW
ZVI
Small and large particle size
0.5, 1.5, 3.0 wt %
(18 reactors)

Crushed rock and GW
STPP
46, 138, 277 mg/L P
(9 reactors)

Batch Testing

1 of 3 identical reactor flasks sacrificed at 3 d, 1 wk, and 8 wks.
Reactor centrifuged and supernatant sampled for pH; ORP;
inorganic carbon (coulometry); Total U, Ca, P, As, Fe (ICP-MS)

Select best performing dose
[Lowest dose to reduce U < MCL]

Solids from best performing dose analyzed for 4-step sequential
extraction (solid phase uranium speciation)

Reagent and
Dose Selected

Design Amendment
Injection Remedy for
Bedrock

Amendment Design and Injection Pilot Test

Notes:
GW = groundwater
ISS = in situ stabilization
mg/L = milligram per liter
P = phosphorus
SMP = sodium monophosphate
STPP = sodium triphosphate
TS = treatability study
U = uranium
Wt % = weight percentage
ZVI = zero valent iron

**Flow Chart for Bedrock Uranium in Groundwater
Treatability Testing (TS ISS-3)**

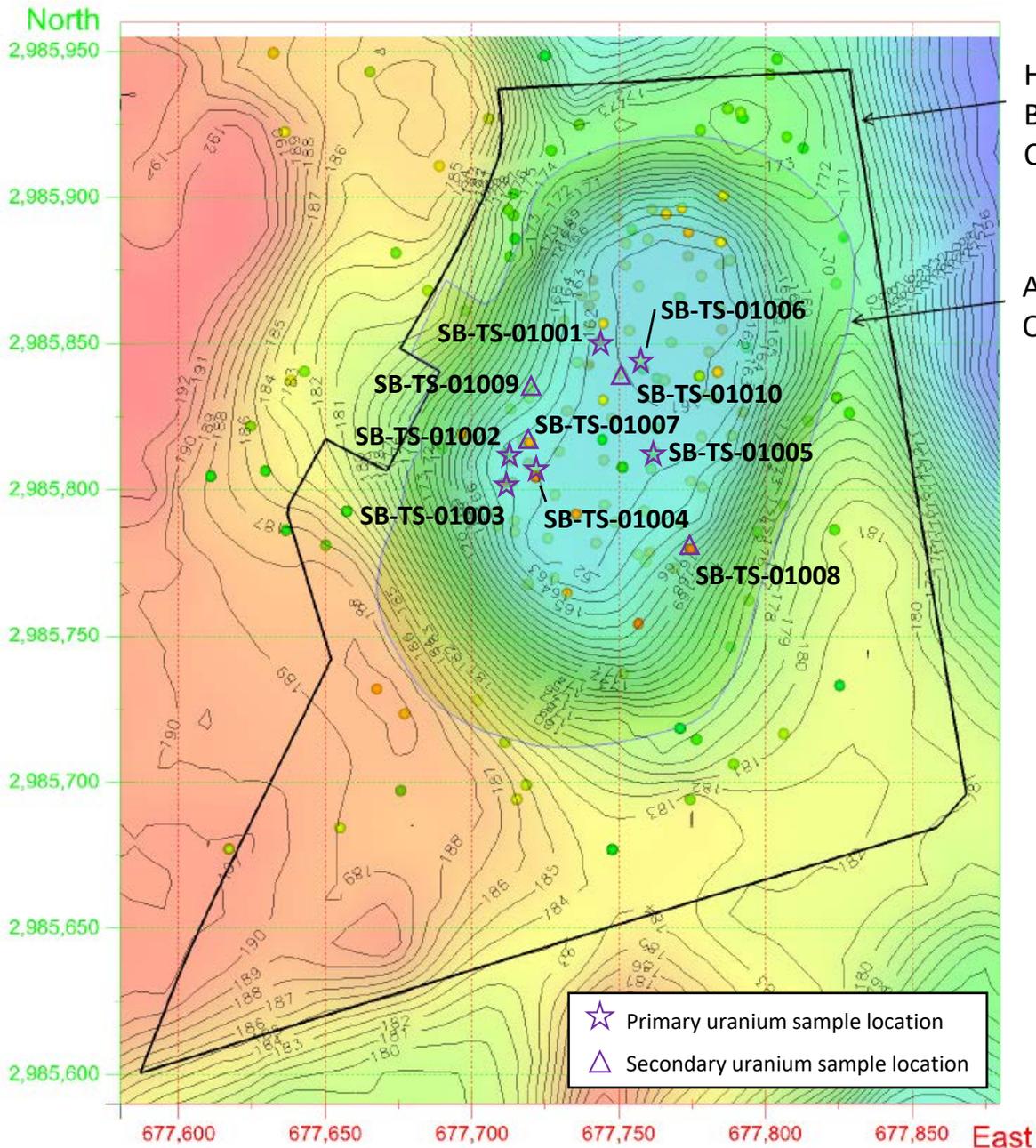
Nuclear Metals, Inc. Superfund Site
Concord, Massachusetts



**Figure
1C**

BR0090

September 2020



Holding Basin Outline

AOI 1 Outline

Boring ID	Primary Boring	Easting	Northing	Sample Depth Interval (ft bgs)	Closest Historical Sample	
					Sample ID	Uranium Conc. Range (mg/kg)
<i>Unsaturated</i>						
SB-TS-01001	Y	677,743	2,985,851	8 – 18	SB-8	3,868 – 12,023
SB-TS-01002	Y	677,712	2,985,812	7 – 17	HB-308	1,150 – 2,740
SB-TS-01003	Y	677,714	2,985,803	6 – 16	SB-3	939 – 1,977
SB-TS-01007	N	677,717	2,985,817	13 – 23	HB-437	686 – 1,188
SB-TS-01008	N	677,775	2,985,780	4 – 14	SB-5	579 – 1,309
<i>Saturated</i>						
SB-TS-01004	Y	677,720	2,985,808	45 – 55	HB-439	464 – 1,317
SB-TS-01005	Y	677,762	2,985,812	47 – 57	HB-440	462 – 545
SB-TS-01006	Y	677,715	2,985,886	46 – 56	HB-441	106 – 391
SB-TS-01009	N	677,720	2,985,835	50 – 60	HB-503	213 – 388
SB-TS-01010	N	677,751	2,985,840	26 – 36	HB-423	272 – 1,096

1) Abbreviations: AOI = area of investigation; conc. = concentration; ft bgs = feet below ground surface; mg/kg = milligram per kilogram

2) Coordinates are in state plane

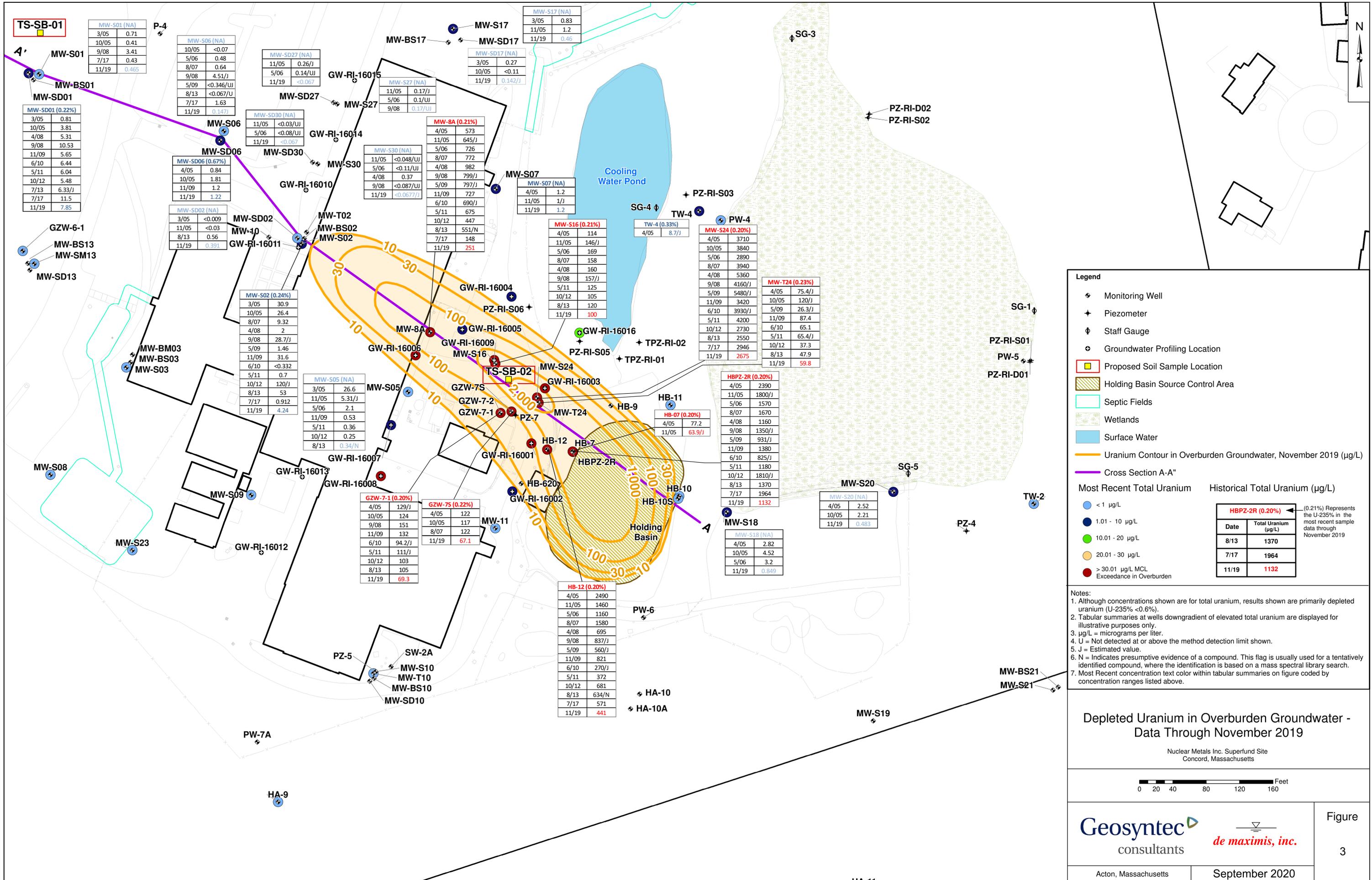
Geosyntec
consultants

Acton, Massachusetts

Proposed Holding Basin Soil Boring Locations
ACTON, MASSACHUSETTS

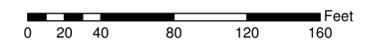
PROJECT: BR0090

FIGURE 2

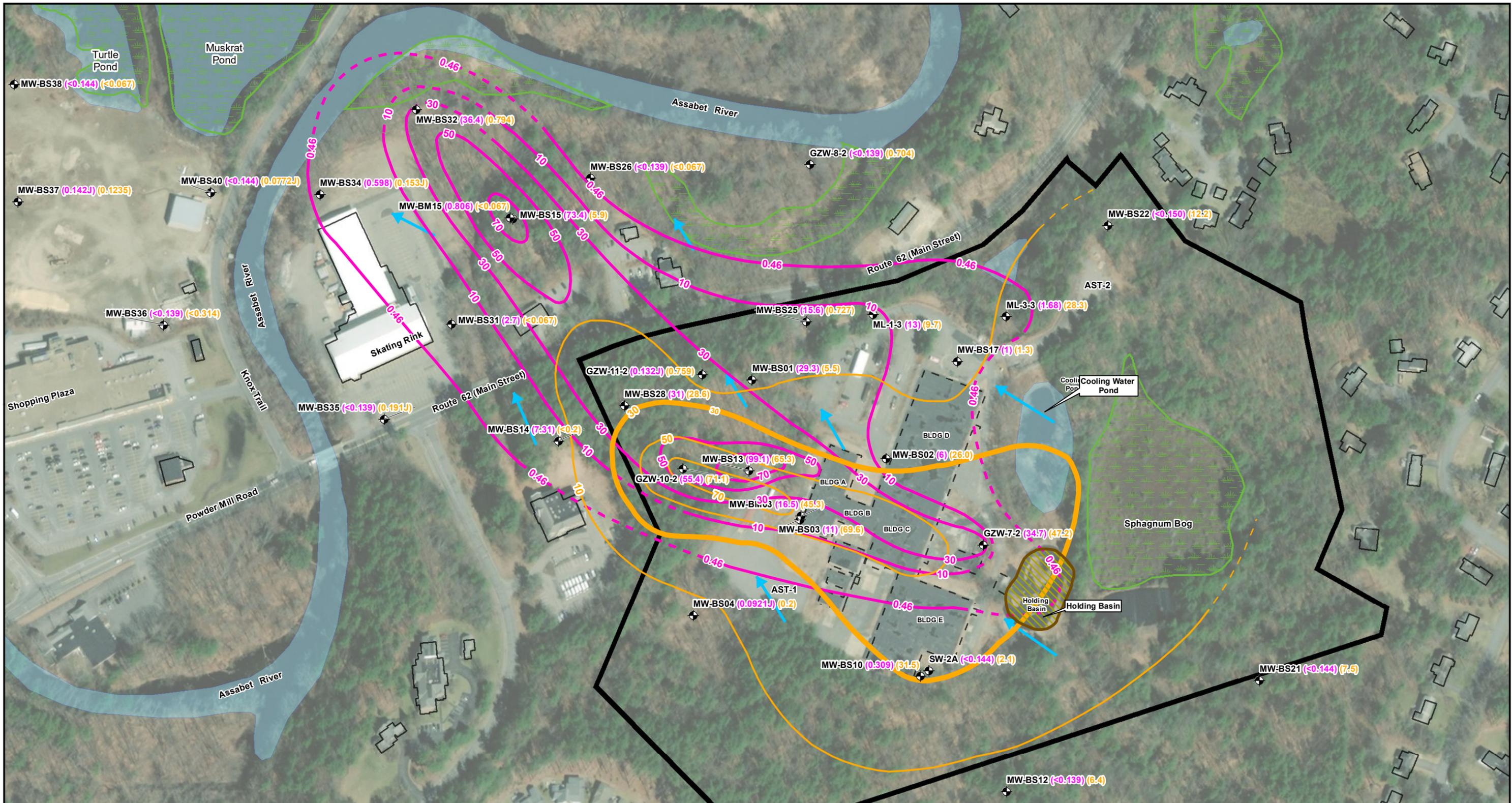


Depleted Uranium in Overburden Groundwater - Data Through November 2019

Nuclear Metals Inc. Superfund Site
Concord, Massachusetts



Q:\GISProjects\BR0090-NMISite\Projects\Updates_2020\Figure 2-5 Depleted Uranium in OB GW_v2.mxd



Legend

- Bedrock Monitoring Well
- Site Boundary
- Building Outline
- Former Building Concrete Foundation
- Bedrock Groundwater Flow Direction Inferred from November 2019 Groundwater Elevations
- Surface Water
- Wetlands
- Uranium ISO Contour in Bedrock November 2019 (µg/L)
- Estimated Uranium ISO Concentration Contour in Bedrock November 2019 ug/L
- 1,4-Dioxane ISO Concentration Contour in Bedrock November 2019 (µg/L)
- Estimated 1,4-Dioxane ISO Concentration Contour in Bedrock November 2019 (µg/L)

- 0.57 1,4-Dioxane Concentrations November 2019 (µg/L)
- 76.9 Uranium Concentrations November 2019 (µg/L)

Note:
 1. The uranium concentrations shown represent total uranium. Uranium in bedrock groundwater is characterized as isotopically natural (U-235% > 0.6%).
 2. < = less than laboratory method detection limit.
 3. J = estimated detection below method quantitation limit.

DRAFT



Uranium and 1,4-Dioxane Concentrations in Bedrock Groundwater - November 2019

Nuclear Metals, Inc. Superfund Site
 Concord, Massachusetts

Geosyntec consultants **de maximis, inc.**

Acton, Massachusetts

July 2020

Figure

4

ATTACHMENT A

**THE HUFFMAN HAZEN LABORATORIES'
QA/QC PLAN**



TITLE: **THE HUFFMAN HAZEN LABORATORIES' QA/QC PLAN**

I. PURPOSE:

This describes Huffman Hazen Laboratories' Quality Assurance/Quality Control (QA/QC) plan.

II. SCOPE:

This procedure applies to analyses performed at Huffman Hazen Laboratories, Inc., 4630 Indiana St., Golden, CO 80403 (Huffman Hazen Labs). Special analytical requirements, client's special needs, or client-supplied procedures may be considered in view of this procedure -- in the case of conflicts, an agreement between the parties may be reached, as deemed appropriate by a suitable representative of each party.

III. PROCEDURE:

A. Quality Control Data

At a minimum, the following Quality Control (QC) analyses shall be performed for all samples, unless otherwise deemed appropriate by a suitable representative of Huffman Labs. It is acceptable to exceed the minimum QC requirements outlined herein. Deviations shall be noted and/or reported appropriately (Refer to Appendices I and II for definitions).

1. Daily:

- a) Initial Calibration Standard(s) (ICS)
- b) Instrument Blanks (IB)

2. At least one per sample batch:

- a) Method Blank (MB)
- b) Laboratory Control Sample (LCS)

3. For every ten samples, at least one per batch:

- a) Duplicate samples (DUP)
- b) Continuing Calibration Standard (CCS) - may also be run at the end of the batch.

4. At least once every 12 months

- a) Instrument detection limits (IDL) shall be confirmed (if applicable).

5. Raw data sufficient to recalculate results shall be maintained for an appropriate period of time, but for at least 30 days. This data shall be appropriately identified.

6. Acceptable values for standards and duplicates shall be as specified in the Standard Operating Procedure (SOP) for individual analytical procedures and/or methods. If not otherwise specified, standards shall be within 10% of predicted values (e.g. calculated values, theoretical values, etc.). Duplicates shall be within 10% of their mean values if quantifiable.

7. Blanks shall be as specified in the SOP for the specific analysis -- if not otherwise specified, variation in the blanks shall be less than half of the reporting limit for the subject analysis.

8. In any case deemed appropriate by a suitable representative of Huffman Hazen Labs, additional measures (e.g. spikes, duplicates, dilutions, etc.) may be added.

B. Corrective Action

- 1. Samples analyzed while QC values are out of the specified range shall be reanalyzed after the system is brought back into control.



2. In cases where client data has been reported, and wherein it is subsequently determined by a suitable representative of Huffman Hazen Labs that there is a discrepancy in the reported data, the client shall be notified of the discrepancy.

C. Exceptions

1. Additional and/or modified QC requirements may be specified in analytical method SOP's, and/or may be specified by clients.
2. If results are used for an analysis in which QC data is not within designated control limits (for any reason), the QC data shall be summarized and included in the raw data associated with the samples. Based upon client requests and/or requirements, a suitable representative of Huffman Hazen Labs shall determine if the QC data and/or an appropriate disclaimer shall be reported to the client.
3. In some cases, calibration blanks and instrument blanks may be the same.
4. In some cases (e.g. BTU determinations) blanks may not be required. In these cases, a note shall be included in the individual SOP for the relevant analysis.
5. In the cases where a standard reference material cannot be obtained, this shall be noted in the raw data package. A suitable representative of Huffman Hazen Labs may approve the use of a single reference material, if he/she deems it appropriate.

D. Quality Assurance

1. Analysts shall monitor QC data and, if possible, make appropriate corrections to any out of control situation.
2. Managers and/or supervisors (i.e. suitable representatives of Huffman Hazen Labs) are responsible for checking to see that QC data has been obtained for all analyses, and that values are acceptable as outlined in SOP DOC-04.
3. The QA/QC Officer or designee shall on an annual basis:
 - a) Review a random report for each method to confirm that proper procedures outlined in the SOPs have been followed.
 - b) Report non-conforming QA/QC data to the Lab Director of Huffman Hazen Labs, or his/her designee using the CAR/CUC form.
4. The Lab Director of Huffman Hazen Labs, or his/her designee shall make the final decision as to whether QA/QC data are acceptable.
 - a) If the data are deemed unacceptable, a representative of Huffman Hazen Labs or his/her designee shall further determine whether analyses and/or QA/QC shall be rerun.
5. Blind samples shall be inserted into routine analyses periodically, as deemed necessary by a suitable representative of Huffman Hazen Labs.
 - a) Lab Coordinator and/or Analysts shall not be made aware that these blind samples are standards.
 - b) Results of these blind samples shall be reported to a suitable representative of Huffman Hazen Labs, who may then make results available to the supervisors, as he/she deems appropriate.
6. Any individual failing to follow this QA/QC plan shall be subject to disciplinary action, up to and including termination.



E. Labeling

1. All sample containers, including client samples and laboratory QC samples such as blanks, standards, etc., from point of preparation to point-of-use, shall be clearly labeled with appropriate information to provide unambiguous traceability to the source of the contents of the container.



APPENDIX I

GLOSSARY

BATCH:

A “batch” shall mean a group of similar samples that are run together for a particular test/analysis. In the case where a client submits a single sample, it may be considered a batch by itself, or may alternatively be combined with similar samples to comprise a batch. A batch does not indicate or imply any particular number of samples, as the term is utilized, herein.

BLANKS:

- IB (Instrument Blank) - Blank for instrument calibration reagents.
MB (Method Blank) - Blank carried through entire analytical method.

CALIBRATION STANDARDS:

- CCS (Calibration Check Standard) - May be same as ICS or LCS. Run periodically during run to ensure continuing calibration.
- ICS (Initial Calibration Standards) – Calibration standards that are traceable to NIST, when possible.
- IDLs (Instrument Detection Limit Standard) - Calibration standard at 3 to 5 times the detection limit to ensure that the instrument performs at the specified detection limit. The IDL shall be set to three times the standard deviation of 10 non-consecutive runs. Blanks may be used to determine IDL, where appropriate.
- LCS (Laboratory Control Sample) A standard reference material carried through an entire analysis. The LCS matrix shall be as similar to the sample matrix as possible. The LCS shall not be the same as the ICS.

LIMITS:

- MDL (Method Detection Limit) – the minimum amount of a given analyte that can be detected with a sufficient level of confidence.
- PQL (Practical Quantitation Limit) – the minimum amount of a given analyte that can accurately be quantified.
- MRL (Reporting Limit) – the minimum level of a given analyte that can be reported (e.g. to a client).

NOTE: Standards used for calibration are typically NIST traceable, they are purchased from third parties, and they are typically provided with an expiration date. While it is understood that these expiration dates are somewhat arbitrary, they shall be acknowledged and dealt with in the following manner:

Standards that are beyond their expiration date may be revalidated on an as-needed basis, in order to keep them in service. Expired standards shall be analyzed alongside current (i.e. valid, unexpired) standards for comparison. If their values are within acceptable levels, based on analytical precision and/or instrumentation limits, a “revalidation sticker” may be placed on the expired standard. The revalidation sticker shall indicate the new expiration date, which shall be the same duration as that indicated by the original expiration date. This revalidation process may be repeated for any chemical standard, as needed.



SAMPLE CONTROLS

DUP (Duplicate Sample) - Carried through the entire analytical procedure.

SPIKE (Spike) - A known quantity of calibration standard (e.g. an ICS) added to a known quantity of sample.

SUITABLE REPRESENTATIVE OF HUFFMAN LABS:

A “suitable representative of Huffman Hazen Labs”, as used in this document and in other related Standard Operating Procedures of Huffman Hazen Labs, shall be any employee (full-time, part-time, or semi-retired) who, in a given situation or circumstance, has the background, training, wherewithal, and/or understanding of a situation to render a well-reasoned decision that produces a viable and sound outcome in the situation at hand. Examples of a “suitable representative of Huffman Hazen Labs” include, but are not limited to the Lab Director, a staff member with an advanced degree in chemistry or related field, the Lab Coordinator, and/or a lab supervisor.



APPENDIX II

QA EXAMPLES

Carbonate Carbon - Although this is an absolute method, coulometer calibration is checked daily by running standards. An acceptable initial instrument blank (IB) shall be obtained followed by an ICS. An LCS shall be run, then every 10 samples a duplicate and a CCS shall be run. Note: The CCS may be the same as the ICS and/or the LCS.

Exemplary analytical sequence:

Calibration: IB, ICS

Calibration Check: LCS

Analysis: 10 samples , DUP, IB, CCS, 10 samples, DUP, IB, CCS...

End of run: CCS

Metals in Solid Material by ICP - A method blank (MB) and a standard reference material (LCS) shall be carried through the entire procedure. The ICP shall be calibrated using the instrument blank (IB) and one or more calibration standards (ICS) and shall be checked with initial calibration check standard (LPC - laboratory performance check) that has all the elements of interest present but made from a second source traceable to NIST (if possible).

Next, the MB and LCS shall be analyzed. If values are satisfactory then 10 samples and 1 duplicate (DUP) shall be analyzed. Next, a continuing calibration standard (CCS or LPC(laboratory performance check)) shall be run. The CCS may be one or more standards similar to the concentrations observed in the samples.

Suggested analytical sequence:

Calibration: IB, ICS

Calibration Check: LPC

Analysis: MB, LPC, 10 samples , DUP, IB, LPC, 10 samples, DUP, IB, LPC...

End of run: LPC

Oxygen - The instrument blank (IB) is analyzed until a low reproducible value is obtained (very close to zero). Then a method blank (MB) that is an empty tin capsule or indium capillary is determined (this depends on the sample matrix being analyzed). After a satisfactory IB is obtained and a MB calibration is performed then an initial calibration standard (ICS) will be run. This will be followed by a laboratory control standard (LCS – this is a second source standard that is different than the calibration standard). If values are satisfactory then 10 samples and 1 duplicate (DUP) are analyzed. Next a CCS would be run. Note: The CCS could be the same as the ICS the LCS.

Typical analytical sequence:

Calibration: IB, ICS

Calibration Check: LCS

Analysis: 10 samples (usually 5 samples in duplicate if sufficient provided), CCS, IB, 10 samples, DUP, CCS, IB...

End of run: CCS

APPROVED _____

DATE _____