Spring 5-2021

SILVER BOW CREEK/BUTTE AREA NPL SITE BUTTE PRIORITY SOILS OPERABLE UNIT

Pioneer Technical Services, Inc.

SILVER BOW CREEK/BUTTE AREA NPL SITE
BUTTE PRIORITY SOILS OPERABLE UNIT

2021

Final

Unreclaimed Sites
Quality Assurance Project Plan (QAPP)

Atlantic Richfield Company

Revision 1. May 2021

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 8, MONTANA OFFICE
FEDERAL BUILDING, 10 West 15TH Street, Suite 3200
Helena, MT 59626-0096
Phone 866-457-2690
www.epa.gov/region8

Ref: 8MO

September 13, 2018

Mr. Josh Bryson
Operations Project Manager
Atlantic Richfield Company
317 Anaconda Road
Butte, Montana 59701

Re: Approval letter for the Butte Priority Soils Operable Unit (BPSOU) Draft Final Unreclaimed Sites Quality Assurance Project Plan (dated 9/6/18)

Dear Josh:

The U. S. Environmental Protection Agency (EPA), in consultation with the Montana Department of Environmental Quality (DEQ), is approving the *Draft Final Unreclaimed Sites Quality Assurance Project Plan (dated 9/6/18),* with the following comments:

- At a minimum, the plan must be reviewed and updated annually, and resubmitted to EPA for review and approval.
- If the content or the technical approach provided in the plan has changed or requires modification between annual reviews, please submit the revised plan to EPA for review and approval.
- In the *Document Modification Summary* table, please revise the first column header to read "modification no.", to be similar to what AR produced for the Butte Reduction Works Phase I QAPP. Please note, the revision tracking number is intended to track changes of an approved QAPP.
- Please review Appendix A.2 (Organizational Chart) and make any necessary revisions.
- Please submit and distribute the Final QAPP with the attached approval page and EPA approved crosswalk.

If you have any questions or concerns, please call me at (406) 457-5019.

Sincerely,

Nikia Greene
Remedial Project Manager

Attachments:
EPA crosswalk
EPA and MDEQ Approval Page

cc: (email only)
Butte File
Jenny Chambers; DEQ
Daryl Reed; DEQ
Jon Morgan; DEQ counsel
Tom Stoops; DEQ
Carolina Balliew; DEQ
Pat Cunneen; State NRD Program
Jim Ford; State NRD Program
Harley Harris; State NRD Program
Mary Capdeville; NRD Program counsel
Dave Palmer; BSBC
Jon Sesso; BSBC
Mollie Maffei; BSBC
David Schultz; BSBC
Eric Hassler; BSBC
Brandon Warner; BSBC
Karen Sullivan; BSBC
Julia Crain; BSBC
Anne Walsh; UP
Robert Bylsma; UP counsel
John Gilmour, UP counsel
Leo Berry; BNSF and UP counsel
Yueh Chuang; BNSF
Brooke Kuhl; BNSF counsel
Jeremie Maehr; Kennedy Jenks for BNSF and UP
Bob Andreoli; Patroit/RARUS
R. Schellig; counsel for Patriot/RARUS
Becky Summerville; counsel for Inland Properties Inc.
Dawn Maack; counsel Inland Properties Inc.
Robert Lowry, BNSF counsel
John Ashworth, BNSF counsel
Cord Harris; AR/BP
Loren Burmeister; AR/BP
Jean Martin; Counsel AR/BP
William Duffy; attorney for AR/BP
Mave Gasaway; attorney for AR/BP

Pat Sampson; Pioneer for AR/BP
Craig Deeney; TREC
Scott Bradshaw; TREC
Mike Borduin; Pioneer for AR/BP
Karen Helfrich; Pioneer for AR/BP
Brad Archibald; Pioneer for AR/BP
Don Booth; AR consultant
Ted Duaime; MBMG
Gary Icopini; MBMG
David Shanight, CDM Smith
Curt Coover, CDM Smith
Chapin Storrar; CDM Smith
Henry Elsen, EPA
Jim Freeman, US DOJ
Joe Vranka; EPA
Chris Wardell; EPA
Jean Belille; EPA
Janice Hogan; CTEC

EPA REGION 8 QA DOCUMENT REVIEW CROSSWALK

QAPP/FSP/SAP for: *(check appropriate box)* ☐ **GRANTEE** ☐ **CONTRACTOR** ☐ **EPA** ☐ **Other**	**Entity** *(grantee, contract, EPA AO, EPA Program, Other)* ATLANTIC RICHFIELD COMPANY	**Regulatory Authority and/or Funding Mechanism**	___ **2 CFR 1500 for Grantee/Cooperative Agreements** ___ **48 CFR 46 for Contracts** ___ **Interagency Agreement** ___ **EPA/Court Order** ___ **EPA Program Funding** ___ **EPA Program Regulation** ___ **EPA CIO 2105**
Document Title *[Note: Title will be repeated in Header]*	Final Butte Priority Soils Operable Unit Unreclaimed Sites Quality Assurance Project Plan		
QAPP/FSP/SAP **Preparer**	Pioneer Technical Services		
Period of Performance *(of QAPP/FSP/SAP)*	**2018**	**Date Submitted for Review**	9/6/18
EPA Project Officer **EPA Project Manager**	Nikia Greene	**PO Phone # PM Phone #**	(406) 457-5019
QA Program Reviewer or **Approving Official**	Nikia Greene	**Date of Review**	

Documents Submitted for QAPP Review (QA Reviewer must complete):

1. QA Document(s) submitted for review:

QA Document	Document Date	Document Stand-alone	Document with QAPP
QAPP	**9/6/18**	**No**	
FSP		No	No
SAP		No	No
SOP(s)			Yes

2. WP/SOW/TO/PP/RP Date _____
WP/SOW/TO/RP Performance Period _____

3. QA document consistent with the:
WP/SOW/PP for grants? Yes / **No**
SOW/TO for contracts? Yes / **No**

4. QARF signed by R8 QAM Yes / No / **NA**
Funding Mechanism IA / contract / grant / **NA**
Amount _____

Notes for Document Submittals:

1. A QAPP written by a Grantee, EPA, or Federal Partner must include for review: Work Plan(WP) / Statement of Work (SOW) / Program Plan (PP) / Research Proposal (RP) and funding mechanism

2. A QAPP written by Contractor must include for review:
- **a)** Copy of Task Order Work Assignment/SOW
- **b)** Reference to a hard or electronic copy of the contractor's approved QMP
- **c)** Copy of Contract SOW if no QMP has been approved
- **d)** Copy of EPA/Court Order, if applicable
- **e)** The QA Review must determine (with the EPA CO or PO) if a QARF was completed for the environmental data activity described in the QAPP.

3. a. Field Sampling Plan (FSP) and/or Sampling & Analyses Plan (SAP) must include the Project QAPP ***or*** must be a stand-alone QA document that contain all QAPP required elements (Project Management, Data Generation/Acquisition, Assessment and Oversight, and Data Validation and Usability).

c. SOPs must be submitted with a QA document that contains all QAPP required elements.

Summary of Comments *(highlight significant concerns/issues)*:

1. The document reviewed is a generic QAPP and therefore needs a site-specific SAP attached to make a complete document. Attach this generic QAPP, an updated version of the document review crosswalk, and the site-specific SAP together to make a SAP/QAPP that is submitted to the agencies for approval.

ATLANTIC RICHFIELD COMPANY **must address the comments in the Summary of Comments, as well as those identified in the Comment section(s) that**

includes a "Response (date)" and Resolved (date)".			
Element	**Acceptable** *Yes/No/NA*	**Page/ Section**	**Comments**
A. Project Management			
A1. Title and Approval Sheet			
a. Contains project title	Yes	1st page	EPA Comments: None
b. Date and revision number line (for when needed)	Yes	vii	EPA Comments: None
c. Indicates organization=s name	Yes	2nd page	EPA Comments: None
d. Date and signature line for organization=s project manager	Yes	i	EPA Comments: None
e. Date and signature line for organization=s QA manager	Yes	i	EPA Comments: None
f. Other date and signatures lines, as needed	Yes	i	EPA Comments: None
A2. Table of Contents			
a. Lists QA Project Plan information sections	Yes	v,vi	EPA Comments: None
b. Document control information indicated	Yes	2.6	EPA Comments: None
A3. Distribution List			
Includes all individuals who are to receive a copy of the QA Project Plan and identifies their organization	Yes	ii - iv	EPA Comments: None
A4. Project/Task Organization			
a. Identifies key individuals involved in all major aspects of the project, including contractors	Yes	2.1	EPA Comments: The corresponding SAP should specifically identify the names of the key individuals that apply to the sampling effort. Atlantic Richfield Response: Comment noted. EPA: Comment addressed (9/11/18).
b. Discusses their responsibilities	Yes	2.1	EPA Comments: None
c. Project QA Manager position indicates independence from unit generating data	Yes	2.1	EPA Comments: Does not specifically indicate independence from unit generating data. Atlantic Richfield Response: Document updated. EPA: Comment addressed (9/11/18).
d. Identifies individual responsible for maintaining the official, approved QA Project Plan	Yes	2.1	EPA Comments: None
e. Organizational chart shows lines of authority and reporting responsibilities	Yes	App. A	EPA Comments: None
A5. Problem Definition/Background			
a. States decision(s) to be made, actions to be taken, or outcomes expected from the information to be obtained	Yes	2.2 – 2.3	EPA Comments: None

b. Clearly explains the reason (site background or historical context) for initiating this project	Yes	1.0, 2.2	EPA Comments: Add text citing ROD actions, goals, and objectives for unreclaimed areas. Atlantic Richfield Response: Document updated. EPA: AR should ensure that the SAP for each sampling effort clearly explains the reason for initiating the sampling effort. Comment addressed (9/11/18).
c. Identifies regulatory information, applicable criteria, action limits, etc. necessary to the project	Yes	2.4 Table 1 & 2	EPA Comments: None
A6. Project/Task Description			
a. Summarizes work to be performed, for example, measurements to be made, data files to be obtained, etc., that support the project's goals	Yes	2.3	EPA Comments: Since the sample preparation proposed is limited and samples will be analyzed through the sample bag, the possibility of analytical error is increased. Therefore in Section 2.3, Analysis, modify the last sentence of this paragraph to read: "...if the field results show the COC levels at 35% above or 35% below established action/screening levels to limit decision errors." The 35% criterion may be adjusted based on statistical analysis on the confirmation sample results. Atlantic Richfield Response: Document updated. EPA: Comment addressed (9/11/18).
b. Provides work schedule indicating critical project points, e.g., start and completion dates for activities such as sampling, analysis, data or file reviews, and assessments	Yes	2.3 Step 4	A general schedule is provided. Since there is currently no specific project, a specific schedule is not applicable. EPA Comments: None
c. Details geographical locations to be studied, including maps where possible	Yes	Figure 1	EPA Comments: None
d. Discusses resource and time constraints, if applicable	Yes	2.3 step 4	EPA Comments: None
A7. Quality Objectives and Criteria			
a. Identifies - performance/measurement criteria for all information to be collected and acceptance criteria for information obtained from previous studies, - including project action limits and laboratory detection limits and - range of anticipated concentrations of each parameter of interest	Yes	2.4 Table 1	Range of anticipated concentrations is unknown. EPA Comments: None
b. Discusses precision	Yes	2.4.1	EPA Comments: None
c. Addresses bias	Yes	2.4.1	EPA Comments: None
d. Discusses representativeness	Yes	2.4.1	EPA Comments: None

e. Identifies the need for completeness	Yes	2.4.1	EPA Comments: None
f. Describes the need for comparability	Yes	2.4.1	EPA Comments: None
g. Discusses desired method sensitivity	Yes	2.4.1 Table 3	EPA Comments: None
A8. Special Training/Certifications			
a. Identifies any project personnel specialized training or certifications	Yes	2.5	EPA Comments: Add HAZWOPER training for field personnel. Delete the second paragraph of Section 2.5. As stated in the crosswalk notes above, the SAP must be combined with the QAPP to make a stand-alone document. Atlantic Richfield Response: HAZWOPER language added and paragraph deleted. EPA: Comment addressed (9/11/18).
b. Discusses how this training will be provided	Yes	2.5	EPA Comments: None
c. Indicates personnel responsible for assuring training/certifications are satisfied	Yes	2.5	EPA Comments: None
d. identifies where this information is documented	Yes	2.5	EPA Comments: None
A9. Documentation and Records			
a. Identifies report format and summarizes all data report package information	Yes	2.6	EPA Comments: None
b. Lists all other project documents, records, and electronic files that will be produced	Yes	2.6	EPA Comments: None
c. Identifies where project information should be kept and for how long	Yes	2.6	EPA Comments: There is no mention in section 2.6 of the time period for which project information shall be kept Atlantic Richfield Response: A data management plan is being completed. The QAPP will be updated as soon as the plan is reviewed and approved. EPA: Comment addressed (9/11/18).
d. Discusses back up plans for records stored electronically	Yes	2.6	EPA Comments: There is no mention in section 2.6 of back up plans for records stored electronically Atlantic Richfield Response: Please see response above. EPA: Comment addressed (9/11/18).
e. States how individuals identified in A3 will receive the most current copy of the approved QA Project Plan, identifying the individual responsible for this	Yes	2.6.8	EPA Comments: In the last paragraph, modify the first sentence as follows: "Any addendums or revisions to the QAPP, *such as annual updates*, will be electronically distributed…" Atlantic Richfield Response: Document updated.
B. Data Generation/Acquisition			
B1. Sampling Process Design (Experimental Design)			

a. Describes and justifies design strategy, indicating size of the area, volume, or time period to be represented by a sample	Yes	3.1	EPA Comments: Size of area that is representative of one composite sample is not described. To enhance the representativeness of the sampling, expanding the number of subsamples collected (e.g., to a minimum of five aliquots) should be considered. Atlantic Richfield Response: Please see updates to section 3.1.1. EPA: Comment addressed (9/11/18).
b. Details the type and total number of sample types/matrix or test runs/trials expected and needed	Yes	3.1	EPA Comments: The total number of samples are not detailed, but this is acceptable at this time Atlantic Richfield Response: Comment noted.
c. Indicates where samples should be taken, how sites will be identified/located	Yes	3.1	Specific sites are not identified since there is no specific project. EPA Comments: None
d. Discusses what to do if sampling sites become inaccessible	Yes	3.1	EPA Comments: None
e. Identifies project activity schedules such as each sampling event, times samples should be sent to the laboratory, etc.	Yes	NA	EPA: This information to be included in the SAP. Comment addressed (9/11/18).
f. Specifies what information is critical and what is for informational purposes only	Yes	NA	Cannot define without a specific project EPA: This information to be included in the SAP. Comment addressed (9/11/18).
g. Identifies sources of variability and how this variability should be reconciled with project information	Yes	3.1	EPA Comments: None
B2. Sampling Methods			
a. Identifies all sampling SOPs by number, date, and regulatory citation, indicating sampling options or modifications to be taken	Yes	3.1, Table 4	EPA Comments: None
b. Indicates how each sample/matrix type should be collected	Yes	3.1	EPA Comments: Please add to the "Collect Samples – Test Pit Method" the procedure to sample test pits in order, from deepest interval to shallowest, to better protect against cross contamination/mixing of soils from different depth intervals. In addition, please change the sampling depth increments to 0-2 inches, 2-6 inches, and 6-12 inches throughout the entire document. Atlantic Richfield Response: Document updated. EPA: The test pit method was clarified. Comment addressed (9/11/18).
c. If in situ monitoring, indicates how instruments should be deployed and operated to avoid contamination and ensure maintenance of proper data	NA	NA	NA

d. If continuous monitoring, indicates averaging time and how instruments should store and maintain raw data, or data averages	NA	NA	NA
e. Indicates how samples are to be homogenized, composited, split, or filtered, if needed	Yes	3.1.1	EPA Comments: See XRF comments
f. Indicates what sample containers and sample volumes should be used	Yes	3.1.1	EPA Comments: See XRF comments
g. Identifies whether samples should be preserved and indicates methods that should be followed	Yes	3.1.4	EPA Comments: None
h. Indicates whether sampling equipment and samplers should be cleaned and/or decontaminated, identifying how this should be done and by-products disposed of	Yes	3.1.3	EPA Comments: None
i. Identifies any equipment and support facilities needed	Yes	3.1.2	EPA Comments: None
j. Addresses actions to be taken when problems occur, identifying individual(s) responsible for corrective action and how this should be documented	Yes	4.0	Corrective Action Template.pdf EPA Comments: Please include the Corrective Action Template in the QAPP as an Appendix Atlantic Richfield Response: Template included in Appendix C EPA: Comment addressed (9/11/18).
B3. Sample Handling and Custody			
a. States maximum holding times allowed from sample collection to extraction and/or analysis for each sample type and, for in-situ or continuous monitoring, the maximum time before retrieval of information	Yes	3.1.4 Table 5	EPA Comments: None
b. Identifies how samples or information should be physically handled, transported, and then received and held in the laboratory or office (including temperature upon receipt)	Yes	3.1.5, 3.1.6	EPA Comments: None
c. Indicates how sample or information handling and custody information should be documented, such as in field notebooks and forms, identifying individual responsible	Yes	3.1.5	EPA Comments: None
d. Discusses system for identifying samples, for example, numbering system, sample tags and labels, and attaches forms to the plan	Yes	3.1.1, 3.1.5	EPA Comments: None
e. Identifies chain-of-custody procedures and includes form to track custody	Yes	3.1.5	Sample COC EPA Comments: None
B4. Analytical Methods			

a. Identifies all analytical SOPs (field, laboratory and/or office) that should be followed by number, date, and regulatory citation, indicating options or modifications to be taken, such as sub-sampling and extraction procedures	Yes	3.2.1, 3.2.2, Table 3	EPA Comments: In Table 3, Please update the mercury analytical Method 7471B to the most recent version (i.e., 2007). Correct the references accordingly. Atlantic Richfield Response: Document updated EPA: Comment addressed (9/11/18).
b. Identifies equipment or instrumentation needed	Yes	3.1.2, 3.2.1	EPA Comments: None
c. Specifies any specific method performance criteria	Yes	2.4.1, Table 3, 3.5.2, 3.5.3	EPA Comments: None
d. Identifies procedures to follow when failures occur, identifying individual responsible for corrective action and appropriate documentation	Yes	3.1.6, 4.0	EPA Comments: None
e. Identifies sample disposal procedures	Yes	3.4	EPA Comments: None
f. Specifies laboratory turnaround times needed	Yes	3.2.2	EPA Comments: None
g. Provides method validation information and SOPs for nonstandard methods	NA	NA	NA
B5. Quality Control			
a. For each type of sampling, analysis, or measurement technique, identifies QC activities which should be used, for example, blanks, spikes, duplicates, etc., and at what frequency	Yes	3.5	EPA Comments: None
b. Details what should be done when control limits are exceeded, and how effectiveness of control actions will be determined and documented	Yes	4.1, 4.2	EPA Comments: None
c. Identifies procedures and formulas for calculating applicable QC statistics, for example, for precision, bias, outliers and missing data	Yes	2.4.1	EPA Comments: None
B6. Instrument/Equipment Testing, Inspection, and Maintenance			
a. Identifies field and laboratory equipment needing periodic maintenance, and the schedule for this	Yes	3.6.1, 3.6.2	EPA Comments: None
b. Identifies testing criteria	Yes	3.6.1, 3.6.2	EPA Comments: None
c. Notes availability and location of spare parts	Yes	3.6.1	EPA Comments: None
d. Indicates procedures in place for inspecting equipment before usage	Yes	3.6.1, 3.6.2	EPA Comments: None
e. Identifies individual(s) responsible for testing, inspection and maintenance	Yes	3.6.1, 3.6.2	EPA Comments: None

f. Indicates how deficiencies found should be resolved, re-inspections performed, and effectiveness of corrective action determined and documented	Yes	3.6.1, 3.6.2	EPA Comments: None
B7. Instrument/Equipment Calibration and Frequency			
a. Identifies equipment, tools, and instruments that should be calibrated and the frequency for this calibration	Yes	3.5.2.1-3.5.3.5	EPA Comments: None
b. Describes how calibrations should be performed and documented, indicating test criteria and standards or certified equipment	Yes	3.5.2.3	EPA Comments: None
c. Identifies how deficiencies should be resolved and documented	Yes	3.6	EPA Comments: None
B8. Inspection/Acceptance for Supplies and Consumables			
a. Identifies critical supplies and consumables for field and laboratory, noting supply source, acceptance criteria, and procedures for tracking, storing and retrieving these materials	Yes	3.7	EPA Comments: None
b. Identifies the individual(s) responsible for this	Yes	3.7	EPA Comments: None
B9. Use of Existing Data (Non-direct Measurements)			
a. Identifies data sources, for example, computer databases or literature files, or models that should be accessed and used	Yes	2.4 Step 3	EPA Comments: None
b. Describes the intended use of this information and the rationale for their selection, i.e., its relevance to project	Yes	2.4 Step 3	EPA Comments: None
c. Indicates the acceptance criteria for these data sources and/or models	Yes	2.4 Step 3	EPA Comments: None
d. Identifies key resources/support facilities needed	NA	NA	NA
e. Describes how limits to validity and operating conditions should be determined, for example, internal checks of the program and Beta testing	NA	NA	NA
B10. Data Management			
a. Describes data management scheme from field to final use and storage	Yes	3.8	EPA Comments: This section should more closely resemble that same section in the Draft BPSOU 2018 Groundwater Monitoring QAPP (April 2018). Atlantic Richfield Response: A data management plan is being completed. The QAPP will be updated as soon as the plan is reviewed and approved. EPA: Comment addressed (9/11/18).

b. Discusses standard record-keeping and tracking practices, and the document control system or cites other written documentation such as SOPs	Yes	3.8	EPA Comments: Reference the upcoming data management plan. Atlantic Richfield Response: Please see response to B10a above. EPA: Comment addressed (9/11/18).
c. Identifies data handling equipment/procedures that should be used to process, compile, analyze, and transmit data reliably and accurately	Yes	3.8	EPA Comments: Reference the upcoming data management plan. Atlantic Richfield Response: Please see response to B10a above. EPA: Comment addressed (9/11/18).
d. Identifies individual(s) responsible for this	Yes	3.8	EPA Comments: None
e. Describes the process for data archival and retrieval	Yes	3.8	EPA Comments: Specify the process for retrieving data. Atlantic Richfield Response: Please see response to B10a above. EPA: Comment addressed (9/11/18).
f. Describes procedures to demonstrate acceptability of hardware and software configurations	NA	NA	NA
g. Attaches checklists and forms that should be used	NA	NA	NA
C. Assessment and Oversight			
C1. Assessments and Response Actions			
a. Lists the number, frequency, and type of assessment activities that should be conducted, with the approximate dates	Yes	2.6.5, 3.5.2, 4.1	No dates included EPA Comments: More discussion of the XRF and standard laboratory assessment and corrective action activities is needed. Atlantic Richfield Response: The XRF assessment and corrective actions are described in section 3.5.2, Field XRF Quality Control Samples. Standard Laboratory data assessment and corrective action methodology, including deliverables, is described in better detail in section 2.6.5, Analytical Laboratory Records. EPA: Comment addressed (9/11/18).
b. Identifies individual(s) responsible for conducting assessments, indicating their authority to issue stop work orders, and any other possible participants in the assessment process	Yes	4.0, 4.1, 4.2	EPA Comments: None
c. Describes how and to whom assessment information should be reported	Yes	4.0, 4.1, 4.2	EPA Comments: None
d. Identifies how corrective actions should be addressed and by whom, and how they should be verified and documented	Yes	4.1, 4.2, 4.3	EPA Comments: Documentation of corrective actions needs to be better described. Atlantic Richfield Response: Documentation of corrective actions is described in section 4.3, Quality Assurance Reports to Management. EPA: Comment addressed (9/11/18).
C2. Reports to Management			

a. Identifies what project QA status reports are needed and how frequently	Yes	4.3	EPA Comments: None
b. Identifies who should write these reports and who should receive this information	Yes	4.3	EPA Comments: None
D. Data Validation and Usability			
D1. Data Review, Verification, and Validation			
Describes criteria that should be used for accepting, rejecting, or qualifying project data	Yes	5.2	EPA Comments: In the third paragraph, the reference for the National Functional Guidelines for Inorganic Superfund Methods Review should be (EPA, 2017). Atlantic Richfield Response: Document updated. EPA: Comment addressed (9/11/18).
D2. Verification and Validation Methods			
a. Describes process for data verification and validation, providing SOPs and indicating what data validation software should be used, if any	Yes	5.3.2	EPA Comments: None
b. Identifies who is responsible for verifying and validating different components of the project data/information, for example, chain-of-custody forms, receipt logs, calibration information, etc.	Yes	5.1.1, 5.1.2, 5.3.2	EPA Comments: None
c. Identifies issue resolution process, and method and individual responsible for conveying these results to data users	Yes	5.1.1 5.1.2	EPA Comments: None
d. Attaches checklists, forms, and calculations	Yes	Appendix C	EPA Comments: None
D3. Reconciliation with User Requirements			
a. Describes procedures to evaluate the uncertainty of the validated data	Yes	5.3.2	EPA Comments: None
b. Describes how limitations on data use should be reported to the data users	Yes	5.3.1, 5.3.2	EPA Comments: None

SILVER BOW CREEK/BUTTE AREA NPL SITE
BUTTE PRIORITY SOILS OPERABLE UNIT

2021

Final

Unreclaimed Sites
Quality Assurance Project Plan (QAPP)

Prepared for:

Atlantic Richfield Company
317 Anaconda Road
Butte, Montana 59701

Prepared by:

Pioneer Technical Services, Inc.
1101 S. Montana Street
Butte, Montana 59701

Revision 1. May 2021

APPROVAL PAGE

Quality Assurance Project Plan for
Butte Priority Soils Operable Unit
Unreclaimed Sites

Approved: _____ Date: 9-13-18
Nikia Greene, Remedial Project Manager
U.S. Environmental Protection Agency

Approved: _____ Date: 9-5-18
Daryl Reed, State Project Officer
Montana Department of Environmental Quality

Approved: _____ FOR: Date: 9-5-18
Terry Moore, Quality Assurance Manager
Atlantic Richfield Company

Approved: _____ Date: 9-4-18
Josh Bryson, Operations Project Manager
Atlantic Richfield Company

Approved: _____ Date: 4 Sept 2018
Julia Crain, Quality Assurance Manager
Butte-Silver Bow County

Approved: _____ Date: 9/4/18
Eric Hassler, Superfund Operations Manager
Butte-Silver Bow County

Revision 1. 2021
Plan is effective on date of last signature above.

DOCUMENT REVISION SUMMARY

Revision No.	Author	Description	Date
Rev 0	Pioneer Technical Services, Inc.	Cover Sheet – Final	October 2018
Rev 1	Pioneer Technical Services, Inc.	Annual Update	May 2021

DISTRIBUTION LIST

Silver Bow Creek/Butte Area NPL Site Butte Priority Soils Operable Unit Soils
Sampling Quality Assurance Project Plan (QAPP)
Butte, Silver Bow County, Montana

Key Personnel QAPP Recipients	Title	Organization	Telephone Number	E-mail Address
Nikia Greene	Remedial Project Manager	EPA	(406) 457-5019	Nikia.Greene@epa.gov
Erin Agee	Legal Counsel	EPA	(303) 312-6374	Erin.Agee@epa.gov
Daryl Reed	State Project Officer	DEQ	(406) 444-6433	dreed@mt.gov
Jonathan Morgan	Legal Counsel	DEQ	(406) 444-6589	JMorgan3@mt.gov
Josh Bryson	Liability Manager	Atlantic Richfield	(406) 723-1834	josh.bryson@bp.com
Irene Montero	Senior Technologist - RET Lead	Atlantic Richfield	(713) 538-0875	irene.montero@bp.com
David Gratson	Quality Assurance Manager	Atlantic Richfield	(505) 660-8521	dgratson@envstd.com
David Shanight	EPA Contractor	CDM Smith	(406) 441-1400	shanightdt@cdm.com
Eric Hassler	Director, Reclamation and Environmental Services	Butte Silver Bow	(406) 497-5042	ehassler@bsb.mt.gov
Julia Crain	Assistant Director, Reclamation and Environmental Services / Quality Assurance Manager	Butte Silver Bow	(406) 497-6264	jcrain@bsb.mt.gov
Abigail Peltomaa	Superfund Program Data Specialist/Quality Assurance Officer	Butte Silver Bow	(406) 497-5045	apeltomaa@bsb.mt.gov
Chad Anderson	Manager, Human Health/RMAP Division	Butte Silver Bow	(406) 497-6278	canderson@bsb.mt.gov
Brandon Warner	Manager, Environmental Division	Butte Silver Bow	(406) 497-5022	bwarner@bsb.mt.gov
Pat Sampson	Contractor Project Manager	Pioneer Technical Services, Inc.	(406) 490-0706	psampson@pioneer-technical.com
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LIST OF ACRONYMS

Acronym	Definition	Acronym	Definition
BPSOU	Butte Priority Soils Operable Unit	**mm**	millimeter
BSB	Butte-Silver Bow	**NPL**	National Priority List
CD	Consent Decree	**NRDP**	Natural Resource Damage Program
CLP	Contract Laboratory Program	**MS**	Matrix spike
COC	contaminant of concern	**PARCC**	precision, accuracy, representativeness, comparability, and completeness
CoC	chain of custody	**PDF**	Portable Document Format
CPM	Contractor Project Manager	**PPE**	personal protection equipment
DEQ	(Montana) Department of Environmental Quality	**PRR**	Poore, Roth and Robinson
DOJ	Department of Justice	**QA**	Quality assurance
DQA	Data Quality Assessment	**QAM**	Quality Assurance Manager
DQO	Data Quality Objective	**QAO**	Quality Assurance Officer
DSR	Data Summary Report	**QAPP**	Quality Assurance Project Plan
FSP	Field Sampling Plan	**QC**	Quality control
EDD	electronic data deliverable	**RCRA**	Resource Conservation and Recovery Act
EPA	U.S. Environmental Protection Agency	**RL**	reporting limit
GPS	Global Positioning System	**ROD**	Record of Decision
HAZWOPER	Hazardous Waste Operations and Emergency Response	**RPD**	Relative percent difference
HSSE	Health Safety Security and Environment	**RSD**	Relative standard deviation
ICP-AES	Inductively Coupled Plasma Atomic Emission Spectroscopy	**SOP**	Standard operating procedure
IM	Integrity Management	**SRM**	Standard reference material
LCS	laboratory control sample	**SSHASP**	Site-Specific Health and Safety Plan
LCSD	Laboratory control sample duplicate	**USGS**	U.S. Geological Survey
MBMG	Montana Bureau of Mines and Geology	**XRF**	X-ray fluorescence
mg/kg	milligrams per kilogram		

1.0 INTRODUCTION AND PURPOSE

Unreclaimed sites exist within the Butte Priority Soils Operable Unit (BPSOU) that could pose a threat to human health or surface water quality due to the presence of historic mine waste. Although many source areas have been previously reclaimed, areas still exist in which soils have not yet been evaluated; such sites may provide a pathway for human exposure or impact surface water quality via storm water runoff. These unreclaimed sites will be evaluated in accordance with Appendix D, Attachment C, Section 8.0 of the BPSOU Consent Decree (CD) (EPA, 2020).

This Quality Assurance Project Plan (QAPP) describes the activities necessary to conduct soil sampling and characterization activities on unreclaimed sites. It also describes the quality assurance/quality control (QA/QC) policies and procedures to be used during collection and analysis. This QAPP is intended to standardize the sampling process to provide accurate and defensible testing results necessary to make a final site declaration. A Field Sampling Plan (FSP) will be produced to outline the site-specific activities to be performed at each unique site. Supplemental information mentioned throughout the document is included in the appendices below:

Appendix A Figures/Charts
Appendix B Standard Operating Procedures
Appendix C Forms
Appendix D Summary of Revisions and Bibliography of Data Summary Reports

A map in Appendix A shows the BPSOU area. Individual site figures will be provided for site-specific FSPs. Data unique to each site will be provided in a data summary report (DSR), in addition to historic data. Reference to implemented FSPs and completed DSRs will be updated on an annual basis, as provided in Appendix D. A bibliography that includes historic and new site data will be added annually to this document in Appendix D as site sampling is completed. A separate report will be prepared for each site that will include the declaration as to whether reclamation is required (as described further in Section 2.0).

This QAPP was prepared in a manner consistent with the EPA *Requirements for Quality Assurance Project Plans (EPA QA/R-5)* (EPA, 2001) and the BPSOU *Quality Management Plan* (Atlantic Richfield, 2016) and includes the following:

- Project management and objectives.
- Measurement and data acquisition.
- Assessment and oversight.
- Data review.

The sections below provide the basic plan elements and describe the appropriate content required for planning soil sampling and analysis activities at unreclaimed sites within the BPSOU. This QAPP expands or references information from other site-wide documents to comply with the EPA Requirements for QAPPs (EPA, 2001) and to present project-specific requirements.

2.0 PROJECT MANAGEMENT

This section addresses project administrative functions, project concerns, and goals and approaches to be followed during characterization sampling activities on the specific site.

2.1 Project Organization and Responsibilities

An example chart showing the overall organization of the project team is provided in Appendix A. Responsibilities of key individuals comprising a project team are described below.

Liability Manager – Mike Mc Anulty (Atlantic Richfield Company)

The Liability Manager monitors the performance of the contractor(s), consults with the Contractor Project Manager (CPM) and Quality Assurance Officer (QAO) on deficiencies, and helps finalize resolution actions.

Program Director – Eric Hassler (Butte-Silver Bow [BSB])

The Program Director monitors the performance of the contractor(s), consults with the CPM and QAO on deficiencies, and helps finalize resolution actions.

Quality Assurance Manager (QAM) – David Gratson (Atlantic Richfield Company) or Julia Crain (BSB)

The QAM interfaces with the Operations Manager on company policies regarding quality and has the authority and responsibility to approve specific QA documents including this QAPP.

Field Team Supervisor – Brandon Warner (BSB)

The Field Team Supervisor coordinates and oversees BSB-led field evaluation teams and may also oversee specialty contractors. The Field Team Supervisor ensures that the QAPP for each project area has been reviewed by all members of the BSB-led field team and that the QAPP is properly followed during field activities.

Contractor

Atlantic Richfield and/or BSB may assign a Contractor to be responsible for completing individual site investigations.

Contractor Project Manager (CPM)

The CPM is responsible for scheduling all sampling work to be completed and ensuring that the work is performed in accordance with the requirements contained herein. The CPM is also responsible for consulting with the specific project QA personnel regarding any deficiencies and finalizing resolution actions. The CPM for each project will be listed in the supporting documents for each project area under this QAPP.

Field Team Leader

The Field Team Leader ensures that the QAPP for each project area has been reviewed by all members of the field team and that the QAPP is properly followed during field activities. The Field Team Leader will conduct daily safety meetings, assist in field activities, and document activities in the logbook.

The Field Team Leader is responsible for equipment, problem solving and decision making in the field, and for addressing technical aspects of the project. The Field Team Leader will provide "on-the-ground" overviews of project implementation by observing site activities to ensure compliance with technical project requirements, Health Safety Security and Environment (HSSE) requirements, and the Site-Specific Health and Safety Plan (SSHASP). Finally, the Field Team Leader is responsible for identifying potential Integrity Management (IM) issues, as appropriate, and preparing required project documentation.

Contractor Quality Assurance Officer (QAO)

The Contractor QAO is responsible for verifying effective implementation of QAPP requirements and procedures. This includes reviewing field and laboratory data and evaluating data quality. The Contractor QAO for each project will be listed in the supporting documents created for each project area under this QAPP and will be independent from the unit generating the data.

Safety and Health Manager

Where applicable the Safety and Health Manager is responsible for developing the SSHASP and reviewing it with all members of the field team. The Safety and Health Manager will lead applicable Task Risk Assessments and conduct the initial safety meeting prior to starting fieldwork. The Safety and Health Manager will ensure that work crews comply with all site safety and health requirements and will revise the SSHASP, if necessary.

Laboratory

The laboratory selected to analyze the samples will be an approved laboratory within the EPA Contract Laboratory Program (CLP) (a national network of EPA personnel, commercial laboratories, and support contractors whose fundamental mission is to provide data of known and documented quality). The CLP Laboratory will have QA personnel familiar with the approved QAPP. The CLP Laboratory will be responsible for reviewing final analytical reports, scheduling analyses, and supervising in-house custody procedures. Note: Hereafter, the word laboratory (or Laboratory) denotes a CLP Laboratory.

2.2 Problem Definition and Background

As stated previously, unreclaimed sites exist within the BPSOU that could pose a threat to human health or surface water quality due to the presence of historic mine waste. Although many source areas have been previously reclaimed, areas still exist in which soils have not yet been evaluated; such sites may provide a pathway for human exposure or impact surface water quality via storm water runoff. The list of known unreclaimed sites is identified in Appendix D, Attachment C, Section 8.0 of the BPSOU CD (EPA, 2020). Additional unreclaimed sites may be

identified as remedial actions are implemented within BPSOU. If so, the newly identified sites will be evaluated in accordance with this QAPP.

This QAPP will function as a general QA document for all soil sampling activities at unreclaimed sites within the BPSOU. Individual figures and supporting documents will be included in the site-specific FSPs.

2.3 Project/Task Description

Soil sampling will be performed to provide contaminant of concern (COC) concentrations and pH at each site in accordance with this QAPP and site-specific FSPs. These concentrations, as well as other site characteristics, will support making a declaration as to whether site-specific response actions are necessary. The objectives of the QAPP are as follows:

1. Provide consistent results in identifying the specific types and quality of data needed to support decisions regarding each site as a result of the investigation.
2. Describe specific requirements for collecting and analyzing samples.

Below is a summary of project tasks to be completed under this QAPP at each unreclaimed area.

Sampling: Surface soil samples will be collected as described in standard operating procedure (SOP) Surface Soil Sampling General (SOP-S-01) included in Appendix B. The location and number of samples collected will be detailed in the documents specific to each site. The location and number of samples collected will be based on individual site parameters as determined by experienced personnel familiar with the local area.

Analysis: Field samples will consist of 3-point composites. All samples will be analyzed using the Thermo Fisher Scientific Niton Analyzer XL3 X-Ray Fluorescence (XRF) Analyzer (Niton XL3) per Operating XL3 X-Ray Fluorescence Analyzer General SOP (SOP-SFM-02), and for pH per Field Measurement of pH in Soil SOP (SOP-SFM-01) (refer to Appendix B). Confirmation (composite) samples will be analyzed according to laboratory SOP S-MN-I-313 Rev.30 - 6010-200.7 and S-MN-I-359 Rev. 27 in Appendix B). Field personnel will send the confirmation samples to the laboratory at a rate of 1 per 10 samples, with additional samples sent to the laboratory for confirmation if the field results show the COC levels at 35% above and 35% below established action/screening levels to limit decision errors. The 35% criteria may be adjusted based on the statistical analysis of the confirmation sample results.

Quality Control: The QC measures required at each site will be completed as per this QAPP.

Data Management: The Contractor QAO will review and evaluate analytical data for quality (refer to Section 0).

Documentation and Records: The field team will ensure that all samples collected have a corresponding Global Positioning System (GPS) location, XRF measurement, and that each sample is appropriately logged and documented (refer to Section 2.6 and Section 3.0).

Data Summary Report: For each site, the CPM will develop a DSR. The DSR will contain historical data collected from the site (if available), new information about the site, photographs, field notes, and a summary of all results. When finalized, the DSR listing information will be included in Appendix D of this QAPP.

Site Declaration: For each site, the CPM will complete a site declaration as to whether the site is at or above human health action levels or Waste Identification Criteria in Table 1 in Appendix 1 of the BPSOU CD (EPA, 2020), whichever is more stringent, whether the site is contributing metals-impacted sediment to existing or planned wet weather control features, and whether historic mine waste at the site is contributing to the degradation of surface water quality.

2.4 Data Quality Objectives and Criteria

The EPA Data Quality Objective (DQO) process (EPA, 2006a) is used to establish performance or acceptance criteria that serve as the basis for designing a plan to collect data of sufficient quality and quantity to support the goals of a study. Each step of the DQO process defines criteria that will be used to establish the final data collection designs. This QAPP followed the EPA process to develop criteria for each site. The process consists of seven steps as follows:

Step 1: State the Problem.
Step 2: Identify the Goals of the Study.
Step 3: Identify Information Inputs.
Step 4: Define the Boundaries of the Study.
Step 5: Develop the Analytical Approach.
Step 6: Specify Performance and Acceptance Criteria.
Step 7: Develop the Plan for Obtaining Data.

These DQOs (detailed below) will be used to guide the data collection and analysis activities.

Step 1: State the Problem.

The purpose of this step is to describe the problem to be studied so that the focus of the investigation will not be ambiguous.

Unreclaimed sites are identified as areas that could negatively impact human health and/or materially degrade water quality in downgradient waterways. Site evaluations will determine which, if any, COCs are present within the soil, if concentrations are above action/screening levels listed in Table 1 and Table 2 (on page 7) and support future remedial action efforts within the BPSOU area.

Step 2: Identify the Goals of the Study.

This step identifies the principal question the study will attempt to resolve and what actions may result.

Specific to each unreclaimed site, the key question would be:

- Are contaminants, if present on site, the result of historic mining operations or related activities?
- Are the residual concentrations of arsenic, lead, or mercury present and above the human health action levels shown on Table 1 (on page 7)?
- Are the residual concentrations of cadmium, copper, zinc, arsenic, lead, or mercury present and above the storm water screening criteria shown on Table 2 (on page 8)?

Resulting alternative actions addressing the principal question regarding COC levels include the following:

- Perform additional remedy in the area if COC concentrations exceed action levels.
- Perform additional site-specific analyses if COCs exceed storm water screening criteria.
- If acceptable levels of COCs are met, take no action. (See Unreclaimed Area Decision Logic diagram in Appendix A.)

Step 3: Identify Information Inputs.

The purpose of this step is to identify the informational variables that will be required to resolve the decision statements and determine which variables require environmental measurements.

For each individual site, the following information is required to satisfy or resolve the decision statements:

- Existing data from the individual project area or a similar area to provide preliminary information on variability in sample measurements across the site. This will be important when designing the sampling strategy.
- Arsenic, cadmium, copper, lead, mercury, and zinc results from soil samples that are representative of metals concentrations within the individual project sites.
- BPSOU EPA-developed risk-based action levels for arsenic, mercury, and lead that will dictate the action level, according to land zoning; and will lead to a resolution of the decision statement.
- BPSOU EPA-developed risk-based screening levels for cadmium, copper, and zinc that will dictate the screening level and inform possible remediation efforts.

Step 4: Define the Study Boundaries.

The purpose of this step is to define the spatial and temporal boundaries of the problem.

For each identified unreclaimed area, the site and sample locations will be delineated on a drawing and submitted with supporting documents to the Agencies for review and comment. Samples will be collected at each site to determine if the COC concentrations are above action/screening levels (Table 1 and Table 2 on page 7). Each site is within the BPSOU

boundaries and, generally, the sites are connected by the main drainages at the base of the contributing areas. The work will focus on each individual site and on how any possible contamination will affect the connected drainage.

Potential constraints that could delay fieldwork include adverse weather conditions or the inability to obtain property access. Major project delays resulting from these constraints will be recorded in the field logbooks and reported to the agencies. Individual site sampling efforts are expected to take one to two days to complete. Sampling will be performed as weather conditions permit but most of the effort will be completed from June through October until all collective sites have been characterized.

Step 5: Develop the Analytical Approach.

The purpose of this step is to define the parameters of interest, specify action levels, and integrate any previous DQO inputs into a single statement.

For the BPSOU area, the EPA developed specific risk-based screening levels for human health COCs (arsenic, mercury. and lead) based on land-use exposure scenarios. Current BSB zoning will inform individual site action levels. The screening levels for cadmium, copper, and zinc will inform possible future remediation efforts. Field samples will be tested for pH at a minimal rate of 1 per 200-foot x 200-foot area. The action/screening levels are in Table 1 and Table 2 following.

Table 1. BPSOU Soil Action Levels for Human Health

Analyte	Solid Media	Action Levels
Lead[1]	Non-Residential/ Residential	2,300 mg/kg/1,200 mg/kg
Arsenic[1]	Recreational/Commercial/Residential	1,000 mg/kg/500 mg/kg/250 mg/kg
Mercury[2]	Residential	10 mg/kg

1. From EPA Record of Decision (ROD) BPSOU, Table 12-1 (EPA, 2006b).
2. From Field Screening Criteria and Procedures Phase 7 and 8 Remedial Action, Streamside Tailings Operable Unit removal action levels (Pioneer, 2011).
mg/kg: milligrams per kilogram

Table 2. BPSOU Soil Screening Criteria for Storm Water COCs

Analyte	Action/Screening Levels
Cadmium [1,2]	20 mg/kg
Copper [1,2]	1,000 mg/kg
Zinc [1,2]	1,000 mg/kg
Lead[1,2]	1,000 mg/kg
Arsenic[1,2]	200 mg/kg
Mercury[1,2]	10 mg/kg

1. From Field Screening Criteria and Procedures Phase 7 and 8 Remedial Action, Streamside Tailings Operable Unit removal action levels (Pioneer, 2011).
2. Screening levels to determine possible remediation efforts.

mg/kg: milligrams per kilogram.

Elevated levels of arsenic, cadmium, copper, mercury, lead, and zinc may have negative impacts on human health and surface water quality. If 3 of the 6 contaminant screening level criteria listed in Table 2, are exceeded or if 1 of the contaminant criteria exceeds 5,000 milligrams per kilogram (mg/kg), the site will be further analyzed to determine the materiality of the load to the degradation of surface water.

If results from any of the project site samples are above human health action levels, the site will be addressed in future remediation efforts. If screening criteria are exceeded for surface water analytes, additional analysis will be performed to determine the materiality of the load to the degradation of surface water.

The usability of all analytical data will be evaluated and validated consistent with the procedures described within this document.

Step 6: Specify Performance and Acceptance Criteria

The purpose of this step is to specify the decision maker's tolerable limits on decision errors, which are used to establish performance goals for the data collection design.

There are limitations in evaluating data over a given area and the inherent variability of the matrix being sampled. Measurement error occurs from the inherent variability in the collection, preparation, and analysis of an environmental sample. Individual site FSPs will specify the process to obtain the necessary data to determine the residual COCs within the site while minimizing the matrix, collection, preparation, and analysis variability. Sampling design and measurement errors will be minimized by following the procedures outlined in this QAPP and the SOPs in Appendix B. All FSPs will specify that an adequate quantity of information will be collected to define the residual COCs within the site, and that the data should have confidence and precision factors in fair agreement with previously collected data and QC criteria.

Step 7: Develop the Plan for Obtaining Data.

The purpose of this step is to identify a resource-effective data collection design to generate data that satisfies the DQOs.

The FSP detailed in Section 3.0 is designed to ensure that data will be of sufficient quality and quantity to determine COCs concentrations at each unreclaimed site and help determine if additional remedial action is required. Any site-specific instructions or conditions will be detailed in the supporting documents for each site. The plan will ensure that data from other (related and current) investigations will be comparable due to compatible approaches. Within the sampling design, representatives from the Agencies are encouraged to participate in the field activities and provide input on specific sample locations.

Evaluation of unreclaimed sites will include the following tasks and follow the specific measurement performance criteria listed in Section 2.4.1. This will allow the data gathered to be used in future remediation efforts.

- Complete a site condition inspection and geotechnical analysis of subsidence areas, if necessary.
- Determine any rill depths and adjust sampling depths as needed if rill depths exceed stated sampling depths.
- Conduct the soil sampling activities.
- Capture pertinent data with daily logs and photographs.
- Develop draft and final data summary documents.

2.4.1 Measurement Performance Criteria for Data

Specific data validation processes ensure that analytical results are within acceptable limits. All the information and data gathered according to this QAPP for each unreclaimed site will be checked to ensure they are usable for their intended purposes. The data will be classified as screening data with definitive confirmation and are anticipated to meet data quality requirements for the soil sampling process. An evaluation of analytical control limits and of the precision, accuracy, representativeness, comparability, and completeness (PARCC) parameters will be performed. If significant issues with the data are found, data results will be discussed with the EPA and Montana Department of Environmental Quality (DEQ) project managers. The EPA, in consultation with DEQ, will then decide if the total study error could factor into or cause an incorrect decision. Using this approach, the probability of making an incorrect decision (i.e., either a false negative or positive) based on the information collected is considered small.

The definitions of the PARCC parameters are provided below along with the acceptance criteria for data collected.

Precision
Data precision is assessed by determining the agreement between replicate measurements of the same sample and/or measurements of duplicate samples. The overall random error component of

precision is a function of sampling. The analytical precision is determined by the analyses of field duplicates and by replicate analyses of the same sample. An analytical duplicate is the preferred measure of analytical method precision. When analytes are present in samples at concentrations below or near the quantitation limit, precision may be evaluated using duplicate analyses of laboratory-prepared samples such as laboratory control sample (LCS) duplicates (LCSD) and laboratory matrix spike (MS) duplicate samples. Precision can be measured as relative percent difference (RPD) or as relative standard deviation (RSD, also known as a coefficient of variation). See Precision Calculations in Appendix A.

For this QAPP, precision will be determined by the analyses of field duplicates, field replicates, laboratory (analytical) duplicates, confirmation samples, and the evaluation of the RPD or RSD for these various paired measurements. The RPD goals for measures of laboratory (analytical) precision are provided in example SOPs in Appendix B. Information related to specific sites will be included in the individual site FSP or remedial action work plan. The RPD field precision goal for soil field duplicates will be 35% for sample pairs with both sample results being greater than 5 times the reporting limit (RL). For soil field duplicate pairs with 1 or both sample results being less than 5 times the RL, a difference of less than or equal to 2 times the RL (difference ≤ 2xRL) will be used as the precision goal.

Accuracy/Bias

Accuracy of sample analysis is controlled primarily by the laboratory and is reported as bias. Accuracy is the degree of difference between the measured or calculated value and the true value. It is a measure of the bias or systematic error of the entire data collection process. Potential sources of systematic errors include the following:

- Sample collection methods.
- Physical or chemical instability of the samples.
- Interference effects during sample analysis.
- Calibration of the measurement system.
- Contamination.

Field and laboratory field blanks will be analyzed to assess artifacts introduced during sampling, transport, and/or analyses that may affect the accuracy of the data. The XRF field check sample data will be completed and included in the summary reports. Laboratory accuracy will be determined by LCS results. Proposed minimum detection limits and reporting limits for the specific analytes are listed in Table 3. Accuracy in the field is assessed through the adherence to all sample handling, preservation, and holding times.

Table 3. Proposed Minimum Detection Limits and Reporting Limits for Specific Analytes

Analyte	Proposed Minimum Detection Limits (mg/kg)	Reporting Limit (mg/kg)
Arsenic[1]	0.200	1.00
Cadmium[1]	0.0095	0.15
Copper[1]	0.0400	0.50
Lead[1]	0.100	0.50
Zinc[1]	0.278	1.00
Mercury[2]	0.00931	0.02

1. EPA Method 6010 (EPA, 2014).
2. EPA Method 7471B (EPA, 2007).
mg/kg: milligrams per kilogram.

Representativeness
Data representativeness is defined as the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, or environmental conditions. Representativeness is a qualitative parameter that is most concerned with the proper design of the sampling program. Representativeness will be achieved through judicious selection of sampling locations and methods. This QAPP has been designed to ensure that the sample locations selected are representative of the medium being sampled and that there are a sufficient number of samples to meet the project DQOs and to satisfy the project remedial action design elements. Sample representativeness may also be evaluated using the RPD values for field duplicate results.

Comparability
Data comparability is defined as the measure of the confidence with which one data set can be compared to another. Comparability is a qualitative parameter but must be considered in the design of the sampling plan and selection of analytical methods, QC protocols and data reporting requirements. Comparability will be ensured by analyzing samples obtained in accordance with this QAPP as well as the appropriate SOPs, which are comparable to the sampling methods used during previous investigations at similar sites. All data will be reported in units consistent with standard reporting procedures so that the results of the analyses can be compared with results from previous investigations. Soil will be reported in units of milligrams per kilogram (mg/kg).

Completeness
Completeness refers to the amount of usable data produced during a sampling and analysis program. The procedures established in this QAPP are designed to ensure, to the extent possible, that data will be valid and usable. To achieve this objective, every effort will be made to collect each required sample and to avoid sample loss.

2.5 Special Training/Certification

All field personnel conducting site investigations will be trained to collect samples and will review the requirements of this QAPP in a project meeting held prior to fieldwork. Hazardous Waste Operations and Emergency Response (HAZWOPER) training will be required for field sampling personnel. All field personnel will read the QAPP document prior to the start of

fieldwork and will acknowledge that they have read and understand the document at the time of the project meeting. Field personnel will be trained on how to use field equipment and in decontamination procedures and custody procedures in accordance with field data collection SOPs used for the sampling event (Section 3.2.5). This training will be documented within the appropriate section of each SOP. The CPM and Safety and Health Manager will be responsible for ensuring that training requirements are fulfilled.

Depending on individual company or agency safety policies, a review of the associated SSHASPs will be conducted with all field personnel prior to fieldwork to assess the particular hazards at the specific site and the control measurements that have been put in place to mitigate these hazards. The SSHASP review will cover all other safety aspects of working at the site including personnel responsibilities and contact information, additional site-specific safety requirements and procedures, and the emergency response plan.

Laboratories providing analytical services will have a documented QC program that complies with EPA Requirements for QAPPs (EPA, 2001). The laboratory QA personnel will be responsible for ensuring that all laboratory personnel have been properly trained and are qualified to perform assigned tasks.

2.6 Documentation and Records

This section describes procedures for documentation management and record keeping related to this QAPP and the individual site investigation reports from initial record generation through final data formatting and storage.

2.6.1 Property Access Agreements

Atlantic Richfield or BSB will request that property owners grant access to their properties for all remedial action-related activities including sampling. The CPM will manage access requests, track their status, and maintain copies of completed agreements received from property owners. Completed agreements will be photocopied and scanned with the electronic version stored on a server. Photocopied access agreements will also be copied to the project record files. Fieldwork will not proceed until access agreements have been finalized.

2.6.2 Field Logbook

All field sampling activities and field data collection will be recorded in a bound field logbook dedicated to the project or on field data sheets (XRF results) that are referenced in the logbook. All documents will follow SOP-SA-05 Project Documentation General (Appendix B). The CPM or Field Team Leader will be responsible for recording information including the sample collection date and time, weather conditions, field crew members, site visitors, samples collected, procedures used, field data collected, and deviations from the site FSP. Sufficient information should be recorded to allow the sampling event to be reconstructed without having to rely on the sampler's memory. Individual field team members may be responsible for required documentation based on specific tasks assigned by the CPM or the Field Team Leader.

Completed field data sheets and logbooks will be photocopied and scanned with the electronic version stored in the project file. Photocopied field records will also be copied to the project record files (refer to Section 3.9). No bound field logbooks will be destroyed or thrown away even if they are illegible or contain inaccuracies that require a replacement document.

2.6.3 Field Photographs

Field personnel will also document field-sampling activities using a digital camera. Documentation of all photographs taken during sampling activities will be recorded in the bound field logbook or appropriate field data sheets (refer to field SOPs for the individual site), and will specifically include the following for each photograph taken:

- The photographer's name, date, time, and the general direction faced.
- A brief description of the subject and the fieldwork portrayed in the picture.
- Sequential number of the photograph.

The digital files will be placed in project files with copies of supporting documentation from the bound field logbooks.

2.6.4 Chain of Custody Records

After samples have been collected, they will be maintained under strict chain of custody (CoC) protocols in accordance with SOP-SA-04 Chain of Custody Form for Environmental Samples General (Appendix B). The field sampling personnel will complete a CoC form (Appendix C) for each shipping container of samples to be delivered to the laboratory for analysis. A copy of each as-transmitted CoC form will be scanned and stored in the project file. The CoC records will also be copied to the project record files (refer to Section 3.9). For complete custody protocols refer to Section 3.2.5.

2.6.5 Analytical Laboratory Records

Results received from the laboratory will be documented both in report form and in an electronic format. Laboratory documentation will include copies of the signed CoC forms, laboratory confirmation reports that include information on how samples were batched and the analyses requested, sample data packages that include the laboratory report and the electronic data deliverable (EDD), and any change requests or corrective action requests. Section 5.1.3 lists the laboratory reporting requirements in detail. The deliverable ("data package" or "report") issued by the laboratory will include data necessary to complete level 2 validation of laboratory results in accordance with specifications included in Section 5.2. Original hard copy deliverables and electronic files received from laboratory will be maintained with the project QA/QC records.

2.6.6 Project Data Reports

A summary report for each site will be prepared following data collection, evaluation, and interpretation. The report will include figures displaying sample locations, analytical results, required declarations about the results (Section 2.3), and program records as detailed in Section

2.6.8. The summary report will be submitted to the Agencies for comment and approval. The approved summary report will be included as an appendix to this QAPP.

2.6.7 Site Declaration

A Site Declaration as to whether a specific site is at or above human health action levels, whether the site is contributing significant metals-impacted sediment to existing or planned wet weather control features, and whether the site is materially contributing to the degradation of surface water quality will be submitted to the Agencies for comment and approval. The approved site declaration will be included as an appendix to this QAPP.

2.6.8 Program Quality Records

Program quality records are documents that furnish objective evidence of the quality of items or services, activities affecting quality, or the completeness of data. These records will be organized and managed by the remedial action entity and will include the following, at a minimum:

- This QAPP and any approved revisions or addenda.
- Site-specific figures and supporting documentation.
- SSHASP and any addenda.
- Copies of SOPs for field data collection, with any updates or revisions or addenda to those SOPs.
- Incoming and outgoing project correspondence.
- Copies of completed access agreements for the individual properties sampled.
- Individual property maps including any field drawings and field photographs.
- Field documentation forms.
- Copies of all bound field logbooks.
- Copies of all field data sheets.
- Electronic field forms.
- Electronic copies of completed sample CoC forms.
- Copies of all laboratory agreements and amendments.
- As-received laboratory data packages (hard copy and electronic).
- Documentation of field and/or laboratory audit findings and any corrective actions.
- Draft and final delivered versions of all reports and supporting documents.

Any addendums or revisions to this QAPP, such as annual updates, will be electronically distributed to all parties identified on the distribution list by the Atlantic Richfield Liability Manager. All records will be maintained and archived electronically for future reference.

3.0 DATA ACQUISITION

This section describes the requirements to complete sampling events at a site to ensure the collection methods and handling procedures result in reliable data that can inform possible future efforts at the site.

3.1 Site Evaluation Objectives

The primary objective of preliminary site evaluations is to characterize the site to determine if sampling and testing are required due to historic mining operations. Site evaluations include visual examination of the site area to determine historic mining activity, identify presence of erosion such as gullies and/or rills, and the potential contribution to downstream contaminated sediment accumulations.

3.2 Soil Sampling Objectives

The primary objective of sampling the unreclaimed sites is to comprehensively characterize COC concentrations in the soils. Samples will be collected from multiple, hand dug test holes from possible waste sources as identified by trained professionals and outlined in the specific supporting documents for each individual site. If no potential source areas are identified, general samples will be collected to characterize soil types and usage areas.

For a specific site, the site layout figure and supporting documents will identify the number of potential samples to be collected, show the locations of each sample, and list any specific sample labeling requirements. Sampling will be conducted by professionals familiar with the sampling processes and the local area. If, during field activities, additional samples need to be collected to evaluate a potential source, the reason and sample collection method will be recorded in the field logbook. Field personnel and representatives from the Agencies (if present) will make the decisions regarding collection of additional "opportunistic" samples to characterize site conditions accurately.

If a site becomes inaccessible due to weather conditions, the sampling date will be adjusted as required. If access to the site is not granted (access agreement not signed by private property owner), the site will remain uncharacterized and be removed from further consideration, barring Agency intervention on the behalf of the sampling team.

To mitigate variability within soil samples, field personnel will use field XRF analysis, which provide instantaneous data that allows the field team to adjust the location and number of samples while at the site. Field XRF confirmation samples will be submitted to the laboratory for arsenic, cadmium, copper, lead, mercury, and zinc analysis.

All sampling will be conducted as per SOPs listed in the Table 4 below. All applicable SOPs are provided in Appendix B.

Table 4. List of Applicable SOPs for Sampling

Reference Number	Title and Revision Date	Originating Organization
SOP-S-01	Surface Soil Sampling General 1/4/2018	Pioneer
SOP-SA-01	Soil and Water Sample Packaging General 1/4/2018	Pioneer
SOP-SA-04	Chain of Custody Forms for Environmental Samples General 1/4/2018	Pioneer
SOP-SA-05	Project Documentation General 1/4/2018	Pioneer
SOP-SFM-01	Field Measurement of pH in Soil 1/4/2018	Pioneer
SOP-SFM-02	Operating XL3-X-Ray Fluorescence Analyzer General 1/4/2018	Pioneer
SOP-DE-01	Personal Decontamination Procedures General 1/4/2018	Pioneer
SOP-DE-02	Equipment Decontamination General 1/4/2018	Pioneer
S-MN-I-313	6010-200.7 Rev. 30 4/14/2017	Pace
S-MN-I-359	7471B Rev. 27 3/1/2018	Pace
S-MN-I-460	Preparation of Solid Samples Rev 19 7/17/2017	Pace

3.2.1 General Sampling Procedure

All unreclaimed site areas will be sampled according to the general procedures in this QAPP and the more detailed procedures listed in the specific site layout figure and supporting documents. Prior to soil sampling activities, a site condition inspection and geotechnical analysis of subsidence areas, if necessary, will be completed. Sample locations identified in the site layout figure will be checked to ensure they meet the sampling objectives. Potential source areas will be sampled preferentially. Depending on real time XRF readings, additional samples can be obtained to define the extent of any contaminants found. If no visually identifiable source areas are present, samples will be collected from general locations to characterize soil types and usage areas. A minimum of 5 combination samples (15 subsamples) will be collected at smaller sites (1 acre or less), and a minimum of 3 combination samples will be collected per acre at larger sites (greater than 1 acre). Subsamples will be collected in a 3-point (triangular) pattern. At each point, a subsample of predetermined depth will be collected. As a rule, the diagonal distance between the points will be 10 feet, depending on the area of soil homogeneity. The diagonal distance can be adjusted in the field to account for soil differences.

Three discrete aliquots of equal amounts of soil from each designated subsample location will be composited into 1 sample. Materials such as plant matter, debris, and large rocks will be removed, to a reasonable extent, prior to placing the sample in the sample container. Samples will be collected from the 0 to 12-inch depth at 0-2 inch, 2-6 inch, and 6-12 inch intervals. Samplers will collect samples using the following protocol:

Collect Samples – Test Pit Method

1. Don a new pair of disposable nitrile gloves.
2. Use a new disposable plastic scoop for each sample.
3. Remove vegetation and debris from the surface prior to digging. If a vegetative mat is present, separate it from the soil surface with the plastic scoop. Shake and scrape the

removed vegetative mat over the sample collection bag to dislodge any soil particles. Include all the dislodged soil particles in the composite sample.

4. Excavate the hole to 0-2 inches, 2-6 inches, and 6-12 inches below ground surface and collect a sample from each interval separately (see step 5-10). Excessive vegetation, tree roots, hard rock areas, and other sampling obstacles may cause problems with planned sample locations. If obstacles are encountered during sampling, choose a new subsample location within 10 feet of the original location.
5. Using a tape measure, mark the sample interval.
6. Use the disposable plastic scoop to scrape the wall of the pit to expose a fresh surface for sampling.
7. Collect the samples from the bottom to the top to avoid cross contamination.
8. Collect a sample from the freshly cleaned interval with the plastic scoop by scraping from the base of the interval to the top of the interval removing material evenly from all around the pit in accordance with SOP-S-01, Surface Soil Sampling-General (Appendix B).
    a. Screen the soils with a stainless steel #10 (2-millimeter [mm]) screen into a new disposable foil pan.
    b. Collect and screen at least one-half to a full plastic scoop of soil from each subsample hole.
9. Place the sieved sample into an appropriately labeled resealable plastic bag.
10. If debris is identified in the screen, remove the debris and make a note in the field logbook.
11. Record the debris information along with a count in the field logbook or on the field data sheet.

Collect Samples – Stainless Steel Probe

1. Define the composite sampling interval and test locations.
2. Insert probe to the sampling depth.
3. Remove and composite proper depth profile (i.e., 0-2 inches, 2-4 inches, etc.)
4. Sieve the sample if gravelly as described in step 7a under **Collect Samples – Test Pit Method** (listed previously).
5. Place the sample into an appropriately sized resealable plastic bag
6. Record appropriate data in the field logbook.

Field personnel will analyze samples in the field using a Niton XL3 XRF. This will allow the field team to adjust the location and number of samples to characterize each site accurately. Prior to field XRF analysis, the sampler will follow the general procedures below. Specific details are included in SOP-SFM-02 (Appendix B).

XRF Analysis

1. Thoroughly homogenize the sample in the bag by kneading the soil.
2. If required, place a portion of the homogenized sample into an additional 1-quart resealable plastic bag so that is fits in the analyzer measurement stand.
3. Compact the material so that there is a flat surface on the area to be analyzed and visually inspect this area to ensure that only fines will be present in the XRF aperture.
4. Place the sample bag on the measurement stand and take the measurement.
5. Record the results for the selected metals on the XRF field data sheet (Appendix C).
6. Complete duplicate and replicate XRF analyses on at least 5% of the samples analyzed in the XRF unit.

The sampler will identify each sample and mark the sample bags as follows: operable unit, area, month, day, year, sample interval, and unique number. For example, BPSOU-XX-MMDDYY-0-2-X) where:

- BPSOU denotes Butte Priority Soils Operable Unit.
- XX denotes the specific area.
- MM denotes the month in which the sample was collected (07 for July, 08 for August, etc.).
- DD denotes the day of the month on which the sample was collected (01, 02, etc.).
- YY denotes the year in which the sample was collected (18 for 2018).
- 0-2, 2-6, 6-12 denotes sample interval (0-2 inches, 2-6 inches, 6-12 inches).
- X denotes the sample number (1, 2, 3, 4, etc.).

A sample marked as BPSOU-BO-091218-2-6-2 means the sample was collected in the BPSOU BO area on September 12, 2018, at the 2-6-inch level and it was sample #2.

3.2.2 Sampling Equipment

Resources and field equipment used for the soil sampling will include the following (at a minimum):

- Hard copy of the QAPP.
- Field notebook, pens, camera, batteries, and cell phone.
- Maps of sample locations.
- GPS unit.
- Nitrile gloves.
- Assorted shovels and breaker bars.
- Soil Probe.
- Disposable plastic scoops.
- #10 (2 mm) stainless steel screens.
- Disposable foil pans.
- 1-quart resealable plastic bags.

- Niton XL3 XRF Analyzer.
- Equipment and deionized water for decontamination.
- Sample coolers, ice, and tape.
- Required Level D Personal Protective Equipment (PPE) as detailed in the SSHASP.

Any problems due to equipment failures will be addressed by the Field Team Leader and resolved in a timely and orderly fashion. All actions will be documented in the field logbook.

3.2.3 Decontamination Procedures

Field personnel will decontaminate all non-disposable sampling equipment after use at each sampling location according to SOP-DE-02, Equipment Decontamination General (Appendix B). Disposable equipment and PPE intended for one-time use will not be decontaminated but will be packaged for appropriate disposal as a solid waste in the local landfill. Soil removed from holes during excavation will be returned to the sample holes.

Field personnel will decontaminate reusable sampling equipment within the site boundaries at a centralized location. Sampling equipment will be decontaminated using the procedure below. All equipment will also be decontaminated before leaving the site to prevent off-site transport of contaminants (refer to SOP-DE-02, Equipment Decontamination General).

- Rinse with water.
- Wash with non-phosphate detergent.
- Rinse three times with deionized water.
- Air dry.

For safety, all personnel will undergo decontamination procedures when leaving a contaminated area. Personnel decontamination includes routine practices as well as emergency decontamination. All personnel will follow SOP-DE-01, Personnel Decontamination Procedures General (Appendix B) protocols and take every measure possible to prevent the spread of potentially contaminated materials to clean areas.

3.2.4 Sample Containers and Handling

Soil samples will be collected in a labeled plastic bag, mixed, and analyzed using the field XRF. Individual soil samples will be placed in a cooler as soon as possible after sample collection and XRF analysis. If the laboratory requires different sample containers, the laboratory will provide the container and field personnel will handle the containers in such a way as to prevent accidental contamination. Field personnel will wear a new pair of nitrile gloves when transferring samples from the bag used for XRF analysis to the laboratory sample container.

Samples will be stored in insulated coolers with double-bagged ice as necessary to maintain a temperature of at less than 6 degrees Celsius (ºC) and then transported to the laboratory. Table 5 lists the required sample preservation, containers, and holding times. Sample holding times are established to minimize chemical changes in a sample prior to analysis or extraction. A holding time is defined as the allowable time between sample collection and analysis recommended to

ensure accuracy and representativeness of analysis results, based on the nature of the analytes of interest and chemical stability factors. The holding time for analyses of metals in soils is 180 days.

Table 5. Required Sample Preservation, Containers, and Holding Times

Media	Parameter	Analytical Method	Preservation	Holding Time	Sample Size	Sample Container
Solid	Total Metals*	EPA 6010, 7471B[1]	Ice to 4 °C	180 days	4 ounces	Ziplock bag or 4-ounce glass jar

* Arsenic, cadmium, copper, lead, and zinc.
1. EPA Method 6010D (EPA, 2014) and EPA Method 7471B (EPA, 2007) for mercury.
°C: degrees Celsius.

3.2.5 Sample Custody Protocols

Once the samples are collected, they will be maintained under strict protocols in accordance with SOP-SA-04, Chain of Custody Forms for Environmental Samples General (Appendix B). Field personnel will complete a CoC form (Appendix C) for each shipping container (e.g., cooler, ice chest, or other container) to be delivered to the laboratory. The sampler will be responsible for initiating and filling out the CoC form. The CoC form for a shipping container will list only the samples in that shipping container. Information contained on the form will include the following:

- Project name and identification number.
- Sampler's signature and affiliation.
- Date and time of collection.
- Sample identification number and matrix.
- Analyses requested.
- Remarks or additional notes to laboratory personnel (e.g., do not use for QC).
- Signature of persons relinquishing custody, dates, and times.
- Signature of persons accepting custody, dates, and times.

The sampler will cross out any blank spaces on the CoC form below the last sample number listed. Any documentation, including CoC forms, placed inside the cooler during sample shipment should be placed inside a reclosable plastic bag.

The sampling person whose signature appears on the CoC form is responsible for the custody of the samples from the time of sample collection until custody is transferred to a designated laboratory, a courier, or another project employee for the purpose of transporting the samples to the designated laboratory. The sample is considered to be *in custody* when the sample is:

- in the responsible individual's physical possession;
- in the responsible individual's visual range after having taken possession;
- secured by the responsible individual so that no tampering can occur;

- secured or locked by the responsible individual in an area in which access is restricted to authorized personnel; or
- transferred to authorized personnel.

A completed CoC form will be placed in a sealed zip lock bag and taped to the inside of the cooler lid. Custody seals will be attached to each cooler and samples will be delivered to the laboratory for analysis within the holding times specified for the test requested (Table 5).

The field sampler will file one copy of each CoC form with the project files as a temporary record of sample transfer. The original form will accompany the samples and be returned to the contractor as part of the laboratory QA/QC requirements. The original form will be filed as part of the project's permanent records.

3.2.6 Laboratory Sample Handling and Storage

When the laboratory receives the shipment, laboratory personnel will review the CoC form to verify it is complete and then the designated technician will sign and date it. Any broken custody seals, damaged sample containers, sample labeling discrepancies between container labels and the CoC form, or analytical request discrepancies will be noted on the CoC form. If any of these conditions exist, the laboratory will notify the Field Team Leader and CPM. The Field Team Leader and CPM will resolve discrepancies or non-conformance issues before the samples are analyzed. The laboratory will provide the Field Team Leader and CPM with a copy of the CoC form and the associated sample receipt information. The typical sample receipt information provided includes sample receipt date, sample identifications transcribed from the CoC forms, sample matrix type, and the list of analyses to be performed for each sample. The laboratory will be responsible for following their internal custody procedures from the time of sample receipt until sample disposal.

3.3 Analytical Methods

Surface and near-surface soil samples (0 to 12 inches below ground surface) will be analyzed using both field XRF and analytical laboratory methods described below. The target analytes are listed in Table 1. The samples will also be field checked for pH.

3.3.1 Field Analysis

Field personnel will use a Niton XL3 XRF for the XRF field analysis. A sample stand, which allows the samples to be analyzed in plastic bags, will be used during analysis to ensure consistent exposure times and position of the XRF aperture for each sample. Results for the analytes (listed in Table 1) will be recorded on the field data sheets. Samples will be tested for pH in the field using the Hanna Instruments, HI 99121 Soil pH Meter.

3.3.2 Sedimentation Analysis

The CPM will determine whether the site contributes metals-impacted sediment to waterways or existing infrastructure and rate the site impacts as marginal (little to no sediment impacts),

moderate (some impacts that may need maintenance efforts), or major (remediation necessary). Each site will be rated on the following criteria:

1. Presence of rills. If present, determine the amount of soil lost.
2. Concentrated outflow. Check outflow for soil loss.
3. Sediment in downstream infrastructure. Determine the amount of soil in the infrastructure and the last maintenance operation. If maintained, determine the amounts of material removed.
4. Determination as to whether the infrastructure is part of Superfund or Reclaimed areas. If Superfund, maintenance will be performed under an Operations and Maintenance Plan; if Reclaimed, opportunistic maintenance will be performed per a reclaimed area Monitoring and Maintenance Plan.
5. Condition of downstream infrastructure. Determine if flow rates are impeded by poor condition.
6. Sediment loading contributions. Check for contributing sediment loading above the site in question.
7. Linkage to Silver Bow Creek. Determine if the drainage links to Silver Bow Creek.

Information on each of the above criteria will be documented with photographs.

3.3.3 Laboratory Analysis

Personnel will evaluate field XRF data for each sampling area to determine potential source areas. Representative XRF samples of each source will be composited, and the composite sample analyzed on the field XRF. Confirmation samples will be submitted to the laboratory for analysis. The actual number of sample locations will be evaluated in the field based on environmental conditions of the site and after consultation with the Agencies. Rationale for laboratory sample submission will be based on the results obtained from the original XRF field analysis as well as 10% of all samples collected.

Selected samples will be submitted for laboratory analysis to confirm and expand on field XRF results. Confirmation samples will be analyzed for the analytes listed in Table 1. Samples will be prepared for metals analysis in accordance with the published laboratory procedures. Sample turnaround time is a maximum of two weeks from the submittal date. If Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES) methods are necessary, the laboratory will analyze the samples in accordance with EPA *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, also known as SW-846 Test Method 6010D: Inductively Coupled Plasma-Optical Emission Spectrometry* (ICP-AES), Revision 4 (EPA, 2014).

3.4 Laboratory Audit

The laboratory QA manager will conduct internal laboratory audits to evaluate compliance with the project requirements and this document. The laboratory will be responsible for verifying that QC procedures are followed and that the results of QC analyses are within the specified acceptance criteria, as well as for implementing corrective action if the QC acceptance criteria are not met.

3.5 Sample Disposal

Laboratory samples will be disposed of by the laboratory after all analyses have been completed. Field samples will be archived until confirmations have been completed and approved.

3.6 Quality Assurance/Quality Control

3.6.1 Field QC Samples

Field QC samples are used to identify any biases from transportation, storage, and field handling processes during sample collection and to determine sampling precision. All field QC samples will be delivered with field samples to the laboratory. This section includes brief descriptions of the QC samples to be collected during sampling activities along with frequency, collection, and analytical instructions. The measured values of a standard will be compared to the expected results and if a measured value falls outside this range, then the check sample will be reanalyzed. If the value continues to fall outside the acceptance range, the sampler will note this information on the XRF log. If any of the check sample results indicate that the XRF is not analyzing accurately, the XRF will be cleaned, turned off, and the energy calibration rerun. This information will be noted in the logbook and on the XRF field data sheet. The batch of samples analyzed prior to the unacceptable calibration verification check samples will be reanalyzed.

3.6.1.1 Equipment Rinsate Blanks

Field personnel will analyze equipment rinsate blanks to assess the efficiency of field equipment decontamination procedures in preventing cross contamination of samples. Equipment rinsate blanks will be created by pouring certified distilled or deionized water over or through decontaminated (clean) sampling equipment that has been used to collect investigative samples, and subsequently collecting this (poured) water in prepared sampling containers. Additives or preservatives will be included in the equipment rinsate blanks as required for analysis. The rinsate blank will be shipped with the associated field samples. Field blanks will not be designated for laboratory use in preparation of MS samples or analytical duplicate samples. Field blank samples will be submitted for the same analyses as the associated samples.

3.6.1.2 Field Duplicate

A field duplicate consists of 1 well-mixed and homogenized sample that is split in the field into 2 samples and placed in different sample containers for separate analyses. Each split will have its own sample number. Both split samples will be analyzed for identical chemical parameters. The

results of the field duplicate will be compared to determine laboratory and sampling precision. Field duplicate samples will be collected at a frequency of 1 per 20 samples or once per sampling event, whichever is more frequent.

3.6.2 Field XRF Quality Control Samples

3.6.2.1 Energy Calibration Check

Field personnel will run a preprogrammed energy calibration check on the equipment at the beginning of each working day. If the individual believes that drift is occurring during analysis, that individual will run the energy calibration check. The energy calibration check determines whether the characteristic X-ray lines are shifting, which would indicate drift within the instrument.

3.6.2.2 Blank Samples

The silicon dioxide sample, as provided by Niton, is a "clean" quartz or silicon dioxide matrix that contains concentrations of selected analytes near or below the XL3 XRF machine lower limit of detection. These samples are used to monitor for cross contamination. Field personnel will analyze this sample at the beginning of each day, once per every 20 samples, and at the end of each day's analysis. The sample information will be recorded as "SIO2" on the XRF field data sheets. This sample will also be analyzed whenever field personnel suspect contamination of the XRF aperture. Any elements with concentrations above the established lower limit of detection will be evaluated for potential contamination. If it is determined that the concentration is higher than that recorded at the start of the day, the probe window and the silicon dioxide sample will be checked for contamination. If it is determined that contamination is not a problem, and the concentration is significantly above the limit of detection, sample results will be qualified by the XRF operator as 'J' estimated, and the problem recorded on the XRF field data sheet and in the logbook. If the problem persists, the XRF will be returned to Niton for calibration.

3.6.2.3 Calibration Verification Check Samples

Calibration verification check samples help check the accuracy of the XL3 and assess the stability and consistency of the analysis for the analytes of interest. A check sample will be analyzed as one of the initial samples, once per every 20 samples and as the last analysis. Results for the check sample (standard reference material [SRM]) will be recorded on the individual site XRF field data sheets and identified as a check sample. There will be 3 Niton-provided SRM check samples for the project: NIST 2709a- Joaquin Soil, USGS SdAR-M2 (an SRM created by the U.S. Geological Survey [USGS]), and a Resource Conservation and Recovery Act (RCRA) sample. There will also be Niton-provided machine-specific expected results for several elements for the check samples. Pioneer has further refined the range of expected results for each SRM standard for each of the field XRFs in use. The measured values of a standard will be compared to the expected results and if a measured value falls outside this range, then the check sample will be reanalyzed. If the value continues to fall outside the acceptance range, this information will be noted on the XRF log. If any of the check sample results indicate that the XRF is not analyzing accurately, the XRF will be cleaned, turned off, and the energy calibration rerun. This

information will be noted in the logbook and on the XRF field data sheet. The batch of samples analyzed prior to the unacceptable calibration verification check samples will be reanalyzed.

3.6.2.4 Duplicate Samples

The XRF duplicate samples will be analyzed to assess reproducibility of field procedures and soil heterogeneity. To run a duplicate sample on the Niton XL3, field personnel will remove the sample bag from the analytical stand, knead it once or twice, and replace it in the stand to be analyzed a second time. Duplicate samples will be recorded on the XRF field data form with a D designator in the sample identification number. One duplicate sample will be analyzed at the rate of 1 per 20 samples.

3.6.2.5 Replicate Samples

Field personnel will analyze a replicate sample at the rate of 1 per 20 XRF samples. To run a replicate sample on the Niton XL3, once the primary sample analysis has been completed, requires restarting the XRF to analyze the same sample a second time with the same soil in the XRF aperture. Replicate samples help in assessing the stability and consistency of the XRF analysis. Replicate sample results will be recorded on the XRF field data form and designated with an R in the sample identification number.

3.6.2.6 Confirmatory Samples

The comparability of the field XRF analysis with laboratory samples will be determined by submitting field XRF-analyzed samples for analysis to the laboratory. The confirmatory analyses can be used to verify the quality of the field XRF data. All samples submitted to the laboratory will be analyzed using the field XRF prior to submittal. The samples analyzed by field XRF will be submitted to the laboratory for metals testing (Table 1) and the results will be used to verify field XRF results and to develop a statistical relationship to the laboratory XRF results.

3.6.3 Laboratory Quality Control Samples

Laboratory QC samples are introduced into the measurement process to evaluate laboratory performance and sample measurement bias. Laboratory QC samples may be prepared from environmental samples or generated from standard materials in the laboratory per the internal laboratory SOPs.

3.6.3.1 Laboratory Blanks

Method blanks will be used to monitor laboratory processes and performance. A method blank is a volume of deionized water or a specified weight of inert material for solid samples that is carried through the entire sample preparation and analyses procedures. The method blank volume or weight will be approximately equal to the sample volumes or sample weights being processed. Method blanks are used to monitor interference caused by constituents in solvents and reagents and on glassware and other sampling equipment. Blank results outside of specified

control limits will be re-run and/or flagged by the laboratory per the QC requirements of the analytical method.

3.6.3.2 Laboratory Control Samples

An LCS, or a blank spike, is an aqueous or solid control sample of known composition that is analyzed using the same sample preparation, reagents, and analytical methods employed for the project samples. The LCS is obtained from an outside source or is prepared in the laboratory by spiking reagent water or a clean solid matrix from a stock solution that is different from that used for the calibration standards. The LCS is the primary indicator of process control used to demonstrate whether the sample preparation and analytical steps are in control, apart from sample matrix effects. If the LCS recovery falls outside the specified control limits, the samples will be re-run and/or flagged by the laboratory per the QC requirements of the analytical method.

Calibration verification should be performed every 20 analyses and at the end of the last analytical run of each day, by analyzing a laboratory control sample and comparing the results to the established values. Control limits are plus or minus 35% of the reference value and the statistical criteria listed in Section 2.4.1. Failure will trigger corrective action and reanalysis of samples since the last in-control LCS measurement.

3.6.3.3 Analytical Duplicates

Analytical duplicates are samples that are split in the laboratory at some step in the measurement process and then carried through the remaining steps of the process. Duplicate analyses provide information on the precision of the operations involved. As the analytical duplicates are a pair of subsamples from a field sample taken through the entire preparation and analyses procedure, any difference between the results indicates the precision of the entire method in the given matrix. Analyses of analytical duplicates and MS duplicates monitor the precision of the analytical process. The frequency of analyses, precision goals, and corrective action information pertaining to analytical duplicates are included in example SOPs included in Appendix B. Information related to specific sites will be included in the individual site documents. If the analytical duplicate precision falls outside the specified control limits, the samples will be re-run and/or flagged by the laboratory per the QC requirements of the analytical method.

3.6.3.4 Matrix Spikes

Laboratory MS samples are used to evaluate potential sample matrix effects on the accurate quantitation of an analyte using the prescribed analytical method. The MS and MS duplicates are prepared by adding an analyte to a subsample of a field sample before sample preparation and analyses. A percent recovery is calculated from the concentrations of the analyte in the spiked and unspiked samples. If the percent recovery for the MS sample and the MS duplicate falls outside the control limits, the results are flagged by the laboratory that they are outside acceptance criteria along with the parent sample.

3.6.3.5 pH Calibration Check

The pH calibration check is performed immediately after calibration of the pH probe and should be within 0.10 pH units. If the acceptance criterion is not met, field personnel will terminate analysis, correct the problem, recalibrate the unit, and attempt a new pH calibration check.

3.7 Instrument Testing, Inspection, and Maintenance

3.7.1 Field Equipment

The Field Team Leader or designee will examine field equipment to certify that it is in proper operating order prior to its first use and at intermittent intervals during the day. Equipment, instruments, tools, and other items requiring preventative maintenance will be serviced in accordance with the manufacturer's specified recommendations. Any routine maintenance recommended by the equipment manufacturer will also be performed and documented in field logbooks or appropriate data sheets. Equipment will be inspected and the calibration checked, if applicable, before it is used. Should equipment deficiencies be found, including calibration failures, the equipment will be immediately removed from service and repaired. Specialized repair parts will be purchased from the manufacturer. Once equipment failure has been resolved and testing/calibration demonstrates proper equipment function, the particular piece of equipment will be returned to service. The Field Team Leader, or designee, will be responsible for field equipment checks and maintaining the Equipment Log.

3.7.2 Laboratory Equipment

Instruments used by the laboratory will be maintained in accordance with each laboratory's QA plan and analytical method requirements. All analytical measurement instruments and equipment used by the laboratory will be controlled by a formal calibration and preventive maintenance program. Required equipment for XRF analysis of soil samples is a drying oven, sieves, a grinder, and an x-ray fluorescence analyzer.

The laboratory will keep maintenance records and make them available for review, if requested, during laboratory audits. Laboratory preventive maintenance will include routine equipment inspection and calibration at the beginning of each day or each analytical batch, per the laboratory internal SOPs and method requirements.

3.8 Inspection/Acceptance for Supplies and Consumables

All supplies and consumables received for the project (e.g., sampling equipment, XRF blanks and SRMs, etc.) will be checked for damage and other deficiencies that would affect their performance. The types of equipment that will be needed to complete sampling activities are described in the relevant SOPs. The Field Team Leader or designee will inspect field supplies.

Per laboratory QA procedures, laboratory personnel will be responsible for inspecting laboratory supplies.

3.9 Data Management Procedures

The Contractor will maintain all project records, either electronic or hard copy, to include the following:

- Individual site maps (hard copy or scanned field drawings and electronic files).
- Project documents, with any approved modifications.
- Field documentation.
- Chain of custody forms.
- Laboratory documentation (results received from the laboratory will be documented both in report form and in an electronic format).
- Data summary reports (for each site sampling event).

Contractor will maintain the project field and laboratory records at a location in Butte, Montana. The CPM will be responsible for managing the project documents. The original field and laboratory documents will be filed chronologically and scanned into a Portable Document Format (PDF) file for future reference. The electronic versions of these records will be maintained on a central server system that is backed up daily.

4.0 ASSESSMENTS AND RESPONSE ACTIONS

Assessment and oversight of data collection and reporting activities are designed to verify that sampling and analyses are performed in accordance with the procedures established in this QAPP. The audits of field and laboratory activities include two independent parts: internal and external audits. Internal audits will be performed by the QAO and/or Atlantic Richfield QAM as necessary. External audits will be performed by the Agencies as necessary.

4.1 Corrective Actions

Assessment of sampling data will be performed during fieldwork on a daily basis. Any equipment malfunctions and data outliers will be reviewed by field technicians and reported to the CPM. All activities will be documented within the project logs. Equipment malfunctions will be remedied by following manufacturers' recommendations. Corrective actions during fieldwork will include replacing/repairing defective equipment and resampling to verify or negate original results. All field personnel and the CPM will have the authority to stop work until any issues are remedied.

Laboratory assessments and corrective actions will follow established procedures and published performance criteria common to accredited facilities and will be documented and reported by the laboratory to the CPM. If a performance criteria issue is unresolved by established laboratory procedures, the CPM, in consultation with the Agencies, will resolve the issue by reanalyzing or resampling. Any actions outside the scope of this QAPP will be reviewed and approved by the Agencies prior to work being completed.

4.2 Corrective Action during Data Assessment

The QAO may identify the need for corrective action during data assessment. Potential types of corrective actions could include resampling of an area by field personnel, reanalysis of samples by the laboratory, or resubmission of data packages with corrected clerical errors. The appropriate and feasible corrective actions will depend on the ability to mobilize field personnel and whether the data to be collected are necessary to meet the required QA objectives (e.g., the holding time for samples is not exceeded, etc.). Any corrective actions outside the scope of the project documents will be performed after consultation with the Agencies. Corrective actions of this type will be documented by the QAO and will be included in any subsequent reports.

4.3 Quality Assurance Reports to Management

The information to be reported to and retained by the CPM includes the following:

- Description of field activities.
- Physical characteristics of the study area.
- Field documentation.
- Field measurements/analysis.
- Equipment calibration and preventive maintenance activities.
- Results of data precision and accuracy calculations.
- Evaluation of data completeness and contract compliance.
- Field and laboratory QA issues and recommended or implemented corrective actions.
- Results of the data validation reviews.
- Deviations to the approved QAPP including an explanation for the deviation and the effect on data quality and usability, if any.

This information will be included in the Data Summary Report at the completion of each project. A draft version of the report will be submitted to the Agencies for review and a final version will be submitted for approval. The CPM will be responsible for preparing the report

5.0 DATA REVIEW AND USABILITY

This section addresses the final project checks conducted after the data collection phase of the project is completed to confirm that the data obtained meet the project objectives and to estimate the effect of any deviations on data usability.

5.1 Data Review and Verification

This section describes the process to be used for reviewing and verifying field data and the internal laboratory data reduction process. Laboratory data reporting requirements, which describe how results are conveyed to data users, are also discussed.

5.1.1 Field Data Review

On a daily basis, field personnel will record all project information in bound field logbooks and on field data sheets. The Field Team Leader will review the data for accuracy and completeness before those records are considered final. The CPM will review the field data package to determine if the field records are complete and verify that the field team completed the measurements specified in this document and the specific site documents. Validation and review of laboratory and field measurement analytical data collected during the sampling event will be conducted as described in this section and preceding sections. Field and laboratory measurements will be tabulated and reviewed as part of the data validation and reduction efforts and maintained by the CPM.

5.1.2 Laboratory Data Review

Internal laboratory data reduction procedures will be according to the individual laboratory's quality management plan. At a minimum, paper records will be maintained by the analysts to document sample identification number and the sample tag number with sample results and other details, such as the analytical method used (e.g., method SOP #), name of analyst, the date of analysis, matrix sampled, reagent concentrations, instrument settings and the raw data. These records will be signed and dated by the analyst. Secondary review of these records by the Laboratory Supervisor (or designee) will take place prior to final data reporting to Atlantic Richfield. The laboratory will appropriately flag unacceptable data in the data package. If any deficiencies with the potential to change analytical results are found during laboratory review of previously reported data, Atlantic Richfield, or their representative, will be immediately notified, and a revised report and EDD will be issued.

5.1.3 Laboratory Data Reporting Requirements

The laboratory will prepare electronic data packages for transmittal of results and associated QC information to Atlantic Richfield or their designee. At a minimum, the data packages will include the case narrative and all sample results, units, and QC sample results. Standard data packages will be transmitted to Atlantic Richfield or their designee within 14 days of laboratory sample receipt.

The laboratory will prepare electronic data packages for transmittal of results and associated QC information to Atlantic Richfield, or their designee, in general accordance with the EPA CLP. Deviations from these specifications may be acceptable provided the report presents all the requested types of information in an organized, consistent, and readily reviewable format.

5.1.4 Laboratory Electronic Data Deliverable

Each electronic data package, as described in the previous section, will be accompanied by an EDD prepared by the laboratory. Additional laboratory QC data can be included in the EDD. As part of the data review process, the EDDs will be cross-checked against corresponding data reports to confirm consistency in results reported in the two separate formats.

5.1.5 Specific Quality Control/Assessment Procedures

The accuracy, precision, completeness, and representativeness of analytical data will be described relative to the project's control limits through a process of field and laboratory data quality review. Results from these reviews will be documented in a Data Quality Assessment Report prepared for all data users. Any qualification of the data resulting from that review will also be incorporated into the project's electronic database (Section 3.9) so that all data users are aware of any uncertainties.

5.2 Data Validation

The laboratory will complete the internal laboratory data validation checks and provide completed checklists with final EDDs and the Field Team Leader will review them. Both will use the following guidelines to review the data validation process. An independent data validation will be performed by an individual not involved with the sampling activities.

- Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020 (EPA, 1983).
- EPA Method 6200 Field Portable XRF Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment (EPA, 2007).

Components of the data validation checks will include an evaluation of the following:

- Holding times.
- Reported detection limits.
- Dilution factors.
- Method blanks.
- MS samples/MS duplicates.
- LCS/LCSD.
- Field blanks.
- XRF duplicates/replicates
- Confirmation sample results.

Data qualifiers will follow those used in the EPA *National Functional Guidelines for Inorganic Superfund Methods Data Review* (EPA, 2017). Contractor will complete a summary data review, which will be reviewed by the CPM, for all data received from the laboratory. The review will involve evaluating the data summary and QA/QC summary sheets provided by the laboratory with each data package.

The data validation review of each analytical data set will include evaluating the field procedures and QA/QC sample results. This will provide information regarding the potential introduction of artificial contaminants during the sample collection process, cross contamination, and field variability. If the introduction of contaminants is indicated by the QA/QC data, specific data will be flagged and qualified as appropriate.

5.3 Internal Data Review

Internal data review is the process of verifying that information generated relative to a given sample is complete and accurate. Review procedures will be performed for both field and laboratory operations as described in this section.

5.3.1 Field Quality Control Data

The results of field QC sample analyses associated with each laboratory data package will be reviewed to evaluate field blanks and other field QC samples and further indications of the data quality. If a problem is identified through the review process, all related field samples will be identified and, if possible, corrective actions will be instituted and documented. If corrective action requests are not in complete accordance with approved project planning documents, the EPA will be consulted and concurrence will be obtained before the change is implemented. If data are compromised due to a problem identified via field QC sample review, appropriate data qualifications will be used to identify the data for future data users. These qualifiers will be included with tabulated data presented in the data assessment section of the annual summary report.

The handling, preservation, and storage of samples collected during the sampling program will be monitored on an ongoing basis. The laboratory will document (log) receipt of the sample and record the condition of the containers and preservation times (if applicable). The sample receipt records (a required data package deliverable) as well as the CoC forms will be assessed during data review. A level A-B validation form included in Appendix C will be filled out for each sampling event.

5.3.2 Laboratory Chemistry Data

The second level of review will be performed by the QAO, or their designee, and will include a review of laboratory performance criteria and sample-specific criteria. All the data will be reviewed and validated. Data validation will follow criteria set forth by the EPA CLP. An additional responsibility of the QAO will be to determine whether the DQOs have been met and calculate the data completeness for the project.

The data quality review will include verification of the following:

- Compliance with the QAPP.
- Proper sample collection and handling procedures.
- Holding times.
- Field QC results.
- Instrument calibration verification.
- Laboratory blank analysis.
- Detection limits.
- Laboratory duplicates.

- MS and MS duplicate percent recoveries and RPDs.
- Data completeness and format.
- Data qualifiers assigned by the laboratory.

Qualifiers that may be applied to the data include the following:

U The analyte was analyzed for but was not detected above the reporting limit.

J The analyte was positively identified; the associated numerical value is an estimate of the concentration of the analyte in the sample.

UJ The analyte was not detected above the sample reporting limit. However, the reporting limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.

R The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet QC criteria. The presence or absence of the analyte cannot be verified.

A Data Quality Assessment (DQA) will be performed to determine whether the project-specific DQOs have been satisfied. The DQA consists of five steps that relate the quality of the results to the intended use of the data:

Step 1: Review DQOs and sampling design.

Step 2: Conduct preliminary data review.

Step 3: Apply statistical test(s) as described in this QAPP to the data set.

Step 4: Verify assumptions.

Step 5: Draw conclusions about the quality of the data (data report will not include interpretation of results but will state conclusions regarding the quality of the results).

During the DQA process, if it is determined that data do not satisfy all DQOs then corrective action(s) will be recommended and documented in the data reporting. Corrective actions include, but are not limited to, revision of the DQOs based on the results of the investigation or collection of more information. The review may also determine that corrective actions are not required, or the decision process may continue with the existing data with recognition of the data limitations.

Results of the QA review and/or validation will be included in any subsequent report, which will provide a basis for meaningful interpretation of the data quality and evaluate the need for corrective actions.

6.0 REFERENCES

Atlantic Richfield, 2016. Butte Priority Soils Operable Unit (BPSOU) Quality Management Plan (QMP). Atlantic Richfield Company, May 2016.

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Pioneer, 2011. Field Screening Criteria and Procedures Phase 7 and 8 Remedial Action, Streamside Tailings Operable Unit (SST OU) Subarea 4, Reaches R and S. Silver Bow Creek/Butte Area NPL Site. Pioneer Technical Services, Inc., March 2011.

Appendix A
Figures/Charts

Appendix A.1 BPSOU Area Map

Appendix A.2 Organizational Chart

Appendix A.3 Unreclaimed Area Decision Logic

Appendix A.4 Precision Calculations

LEGEND

BPSOU BOUNDARY

N

DISPLAYED AS:
PROJECTION/ZONE: MSP
DATUM: NAD 83
UNITS: INTN'L FT
SOURCE: PIONEER/ESRI

0 1,500 3,000 6,000
Feet

FIGURE 1

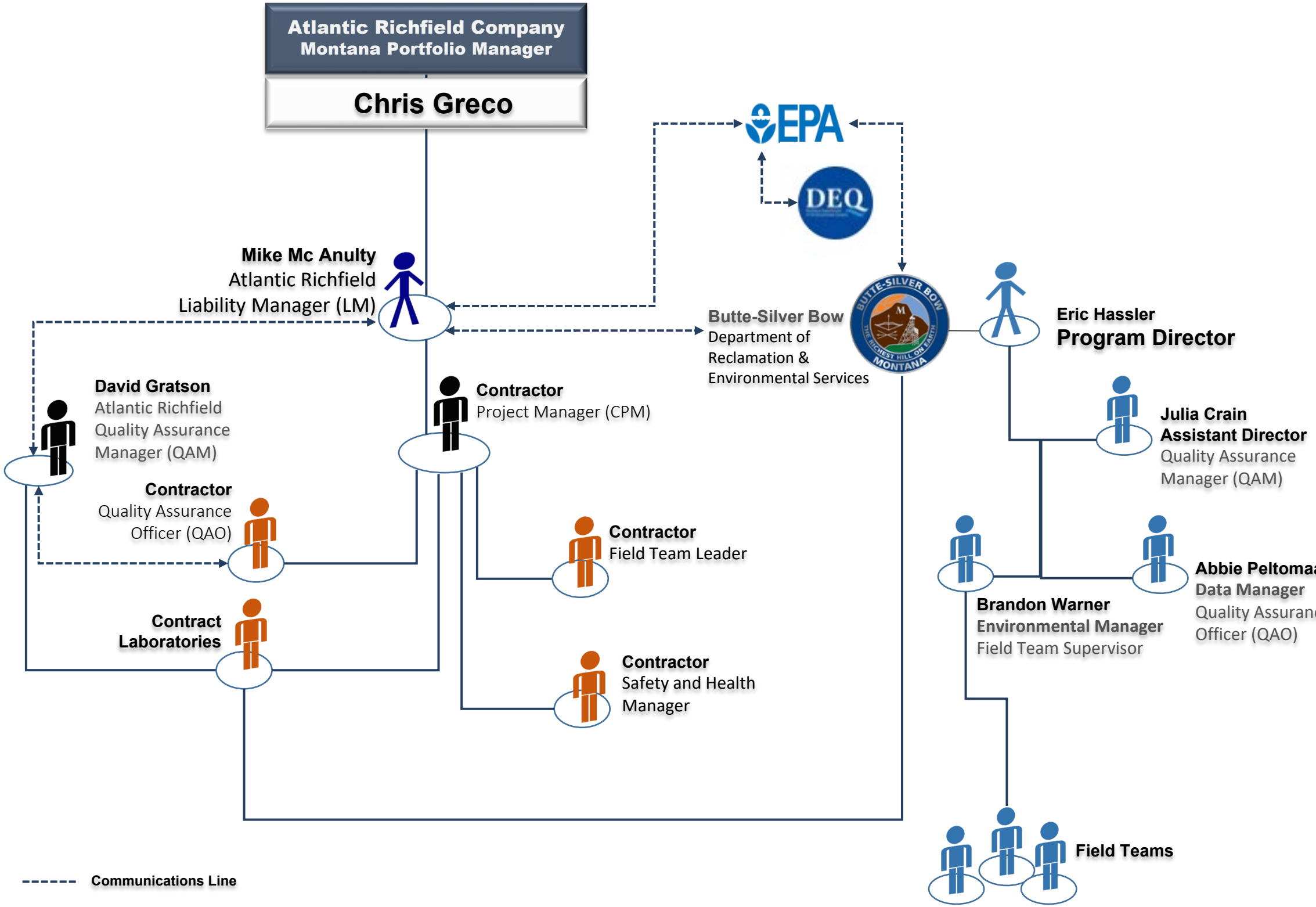
BPSOU AREA

PIONEER TECHNICAL SERVICES, INC.

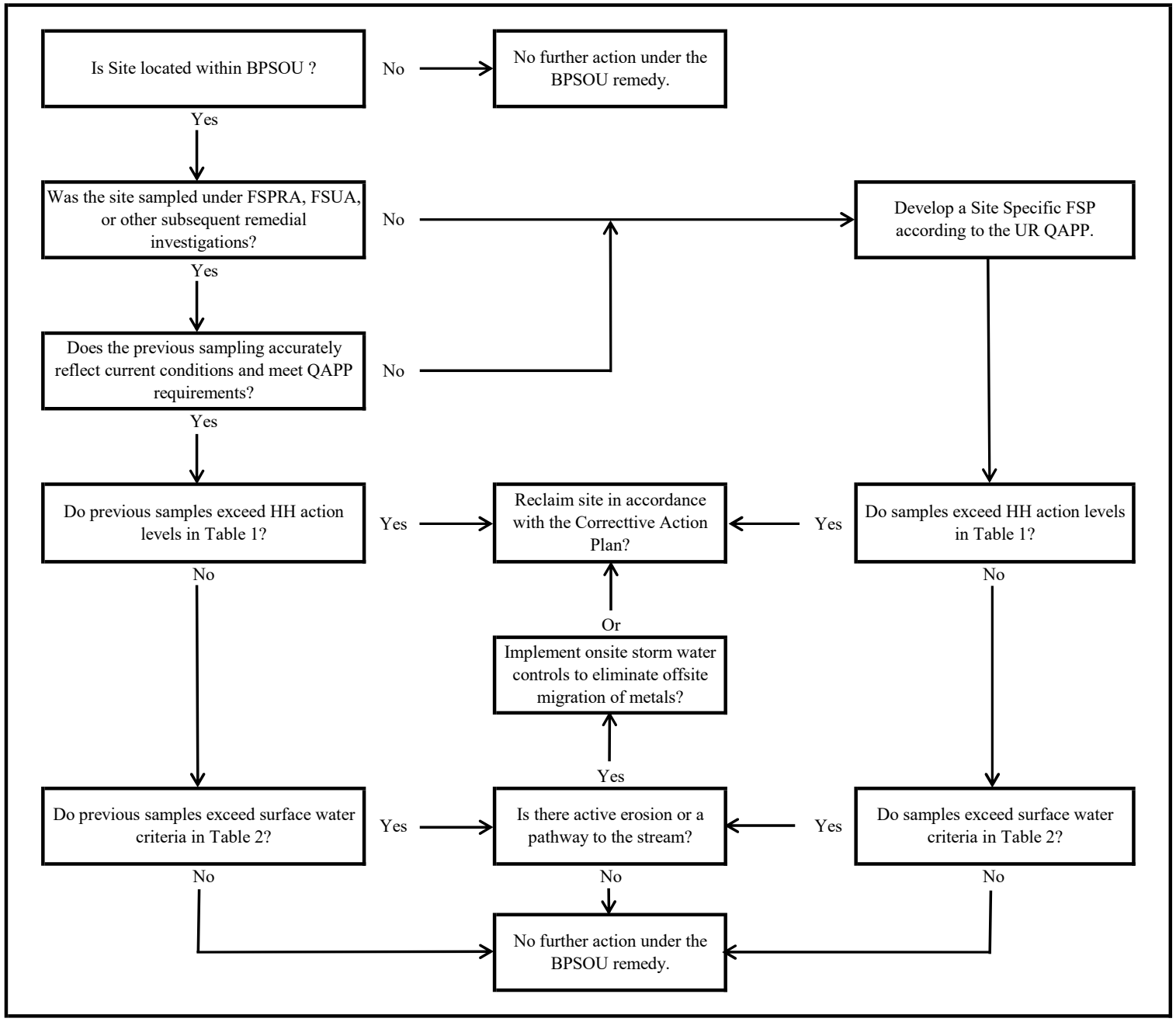
DATE: 1/6/2021

Path: Z:\Shared\Active Projects\ARCO\BPSOU_Misc\GIS\BPSOU_Bndry.mxd

Appendix A2. Program Organizational Chart



Appendix A.3:
Unreclaimed Area Decision Logic



PRECISION CALCULATIONS

Parameter	Formula	Definitions
Precision (as relative percent difference, RPD)	$RPD = (xi-xj)/((xi+xj)/2) * 100$	x_i, x_j: replicate values of x
Precision (as relative standard deviation, RSD, otherwise known as coefficient of variation)	$RSD = (\sigma/x') * 100$	σ: sample standard deviation x': sample mean
Accuracy (as percent recovery, R, for samples without a background level of the analyte, such as reference materials, laboratory control samples and performance evaluation samples)	$R = (x/t) * 100$	x: sample value t: true or assumed value
Completeness (as a percentage, C)	$C = (n/N) * 100$	n: number of valid data points produced N: total number of samples taken

Appendix B
Standard Operating Procedures

Decontamination Methods for Direct Sample Contact Equipment

Appendix B.1 SOP-S-01 Surface Soil Sampling General

Appendix B.2 SOP-SA-01 Soil and Water Sample Packaging General

Appendix B.3 SOP-SA-04 Chain of Custody Forms for Environmental Samples General

Appendix B.4 SOP-SA-05 Project Documentation General

Appendix B.5 SOP-SFM-01 Field Measurement of pH in Soil

Appendix B.6 SOP-SFM-02 Operating XL3-X-Ray Fluorescence Analyzer General

Appendix B.7 SOP-DE-01 Personal Decontamination Procedures General

Appendix B.8 SOP-DE-02 Equipment Decontamination General

Appendix B.9 S-MN-I-313 6010-200.7 Inductively Coupled Plasma Atomic Emission Spectroscopy

Appendix B.10 S-MN-I-359 Mercury in Liquid and Solid/Semi-Solid Waste

Appendix B.11 S-MN-I-460 Preparation of Solid Samples for Analysis by ICP and ICP-MS

Decontamination Methods for Direct Sample Contact Equipment

Routine decontamination steps for equipment that directly contacts samples (i.e. soil probes) are described below.

1. Physically remove gross contamination from equipment by abrasive scraping and/or brushing.
2. Wash equipment with non-phosphate detergent wipe.
3. Rinse with water
4. Air Dry

The above decontamination procedure is performed on the soil sampling probe prior to conducting soil sampling and at the conclusion of all soil sampling activities. The above procedure is also used to decontaminate soil probe between each residential property. Only gross contaminant removal occurs between different sampling sections of an individual residential property (such as between sampling of North Yard and South Yard).

SOP-S-01.
SURFACE SOIL SAMPLING-GENERAL

PURPOSE	To provide standard instructions for surface soil sampling for unreclaimed sites in the BPSOU area.
SCOPE	Work described in this procedure includes visual assessment and site documentation, sample collection and handling, and chain of custody protocol required to complete routine soil sampling tasks.
DEFINITIONS	Surface Sample: a surface sample is defined as a mineral soil sample collected from immediately beneath the vegetative mat. It generally includes some interval from the upper six inches of soil. Surface sampling under biased conditions may be selected after considering factors such as type of contaminant, length of time the area has been contaminated, the type of soil, and the past use of the area.

WORK INSTRUCTIONS

The following instructions are intended to provide sufficient guidance to perform the task in a safe, accurate, and reliable manner. Should these instructions present information that is inaccurate or unsafe, operations personnel must bring the issue to the attention of the Project Manager and the appropriate revisions made.

TASK	INSTRUCTIONS
Grab/Opportunistic Sample	
Visual Inspection and map	1. Verify utility locates have been performed and adjust sampling sites to avoid conflicts. 2. Inspect the area for possible hazards prior to sampling. 3. Visually inspect the site to determine the number test areas for composite sampling 4. Photograph and document the existing site conditions. 5. Draw a scaled map of the site if a pre-sampling map hasn't been completed
	Note: Sample collection devices include stainless steel scoops or trowels, stainless steel probes, and disposable Teflon trowels. For inorganic contaminants, disposable plastic scoops will be used. These procedures may be modified in the field based on field and site conditions after appropriate annotations have been made in the field log book.
	Identify site-specific hazards and verify utility locates. 1. Perform utility locates or verify utility locates have been performed. 2. Walk through the site and determine any site-specific hazards associated with the sampling area. Discuss findings with sampling crew and note in the field logbook. 3. Verify the utility locate information by identifying where natural gas pipes or other utilities enter any structures on the property or if yard lights or street lights are present with no overhead lines. Determine if an underground sprinkling system is present, where applicable. If sample locations have not been assigned in the Sampling Analysis Plan (SAP), note the already marked and/or probable locations of underground utilities and try to avoid those areas when choosing sample locations. Also, note the location of overhead lines and overhead hazards and avoid those areas, if possible. 4. If sample locations are identified in the SAP, use the appropriate survey method to locate and mark the sample locations.

Test Pit Sampling	
1. Dig a 6 to 12-inch square pit.	Dig a 6 to 12-inch square pit to a depth of approximately 6 inches. The size and depth of the sample pit required depends on the amount of material needed for sample analysis and the interval to be sampled. If a sod mat is present, separate the sod mat from the mineral soil surface with the chosen sampling tool. Shake and scrape the removed sod mat over the sample collection bowl to dislodge any mineral soil particles. Place all dislodged particles in the sample. If the surface material is coarse-grained material, free of intermixed materials (i.e., graveled driveway), collect the sample from the appropriate layer below the protective barrier. However, if the graveled driveway, alley or lot contains soil/dust material on the surface, collect the sample from the appropriate interval. If the sample area is unvegetated, collect the sample material from the designated interval inches below ground surface.
2. Measure and mark the interval to be sampled.	Measure the interval to be sampled (e.g., 0-2 inches or 0-6 inches) with a stainless steel tape measure or a ruler and mark the appropriate interval.
3. Scrape the walls of the sample pit.	Scrape the walls of the sample pit within the marked interval with a decontaminated stainless steel trowel or scoop, a Teflon scoop, or a disposable plastic scoop to expose a clean surface.
4. Collect the sample.	Once the wall of the test pit has been cleaned, collect the sample by scraping the appropriate interval on the cleaned face of the pit with the sampling tool and placing the material in a decontaminated stainless steel bowl, or a new cleaned foil pan.
5. Remove coarse fragments from the bowl.	Remove all coarse fragments greater than 0.5 inches from the bowl. Mix the remaining material in the bowl with the sampling tool.
6. Pack the samples.	Transfer the soil sample directly into the appropriate sample container according to SOP-SA-01 Soil and Water Sample Packaging and Shipping and store in a cooler at 4°C or less. Any remaining sample material will be returned to the sample holes. A sufficient quantity of soil will be collected in each sample container to provide for analysis with additional soil left over to be archived.
7. Record sampling information.	Record appropriate information about the sample collection in the field logbook.
8. Return all the removed dirt into the hole.	Return all the removed dirt into the hole and return the sample area to pre-sampling conditions.
9. Decontaminate the equipment.	Decontaminate sampling tools according to procedures outlined in SOP-DE-02 Equipment Decontamination.

Stainless steel probe opportunistic sampling	
1. Collect the sample	Collect sample as per probe manufacturers instructions
2. Pack the sample	Transfer the soil sample directly into the appropriate sample container according to SOP-SA-01 Soil and Water Sample Packaging and Shipping, label the samples, and store in a cooler at 4°C or less.
3. Record the sample	Record appropriate information about the sample collection in the field logbook.
4. Decontaminate sampling equipment	Decontaminate sampling tools according to procedures outlined in SOP-DE-02 Equipment Decontamination.
Composite Sampling/ Test Pits	
Note	In many situations, a composite sample is more appropriate for sample collection than a grab sample. Several types of composite samples can be collected. A sampler can collect a biased composite sample by identifying specific spots within the sample area that appear to be contaminated or not contaminated and digging sample pits in those locations. Composite samples can also be collected randomly as defined in a SAP. Sub samples shall be collected in a three-point (triangular) pattern. At each point, a subsample of predetermined depth is collected. The diagonal distance between the points is commonly ten feet, depending on the area of soil homogeneity. The precise method for compositing the sample will be discussed in the SAP. Each sub sample test hole will be prepared and sampled in the manner discussed above under the Grab Sample section.
1. Collect composite samples.	Composite samples will consist of discrete aliquots of equal amounts of soil from each subsample location. The soil aliquots will be collected into a stainless steel bowl and thoroughly mixed. The sampler may also "eyeball" an equal amount of sample material from each hole into a resealable plastic bag (i.e., Ziploc®). The sample material would be thoroughly mixed between each sub sample pit and prior to placing in the appropriate sample containers.
2. Remove coarse fragments.	Remove all coarse fragments greater than 0.5 inches from the bowl. Mix the remaining material in the bowl with the sampling tool.
3. Pack the samples.	Transfer the soil sample directly into the appropriate sample container according to SOP-SA-01 Soil and Water Sample Packaging and Shipping, label the samples, and store in a cooler at 4°C or less. Any remaining sample material will be returned to the sample holes. A sufficient quantity of soil will be collected in each sample container to provide for analysis with additional soil left over to be archived.
4. Record sampling information.	Record appropriate information about the sample collection in the field logbook.

5. Return all the removed dirt into the hole.	Return all the removed dirt into the hole and return the sample area to pre-sampling conditions.
6. Decontaminate the equipment.	Decontaminate sampling tools according to procedures outlined in SOP-DE-02 Equipment Decontamination.
Composite Sampling Stainless Steel Probe	
1. Collect composite samples	Collect in the same triangular pattern and mix as described above. Collect samples as per probe manufacturers instructions
1. Pack the samples	Transfer the soil sample directly into the appropriate sample container according to SOP-SA-01 Soil and Water Sample Packaging and Shipping, label the samples, and store in a cooler at 4°C or less.
2. Record sampling information	Record appropriate information about the sample collection in the field logbook.
3. Decontaminate the equipment	Decontaminate sampling tools according to procedures outlined in SOP-DE-02 Equipment Decontamination.

ADDITIONAL HSSE CONSIDERATIONS This section to be completed with concurrence from the Safety and Health Manager.	
Required PPE	**Personnel Protection Equipment** (PPE): Hard hat, safety glasses, high-visibility work shirt or vest, long pants, work boots, nitrile gloves, and leather gloves.
Applicable SDS	**Safety Data Sheets** (SDSs) will be maintained based on-site characterization and contaminants.
Required Permits/Forms	Per site/project requirements.
Additional Training	Per site/project requirements.

DRAWINGS, DOCUMENTS, AND TOOLS/EQUIPMENT The following documents should be referenced to assist in completing the associated task.	
Drawings	Map with site location and sample locations.
Related SOPs/ Procedures/ Work Plans	SOP-SA-01 Soil and Water Sample Packaging and Shipping and SOP-DE-02 Equipment Decontamination.
Tools	Sampling tools: stainless steel scoops or trowels, stainless steel probes, disposable Teflon trowels, disposable plastic scoops (for inorganic contaminants), stainless steel tape measure or a ruler, decontaminated stainless steel bowl or cleaned foil pan, one-quart plastic bag, sampling containers, and cooler. Field logbook.
Forms/Checklists	

PURPOSE	To provide standard instructions for soil and water sample packaging and shipping for unreclaimed sites in the BPSOU area.
SCOPE	Work described in this procedure includes instruction on the correct methods to package, ship and Chain of Custody documentation.

WORK INSTRUCTIONS

The following instructions are intended to provide sufficient guidance to perform the task in a safe, accurate, and reliable manner. Should these instructions present information that is inaccurate or unsafe, operations personnel must bring the issue to the attention of the Project Manager and the appropriate revisions made.

TASK	INSTRUCTIONS
1. Place the sample containers in Ziploc bags.	Based on the analytes requested (e.g., low level mercury, low level chromium, etc.), it may be necessary to place each filled sample container in separate Ziploc bags to prevent cross contamination; keep the container clean, dry, and isolated; and protect the sample label. In most cases, all sample containers collected from a specific sample location are placed in a large Ziploc bag and shipped together.
2. Package the samples.	Place samples in a cooler, which has been previously lined with a plastic bag. Surround the samples with non-contaminating packaging materials to reduce movement and absorb any leakage. Double bag the ice and place it in the cooler. Seal the plastic bag in the cooler to contain the samples, packing material, and ice.
3. Review and sign Chain of Custody forms.	The Field Team Leader or their designated representative will double check the Chain-of-Custody (CoC) forms to assure those samples recorded on the CoC forms are in the cooler. The Field Team Leader or the designated representative will then sign the CoC form to relinquish custody. One copy of the signed CoC form will remain with the Field Team Leader. Make a photocopy of the completed forms, if there are no carbon copies available.
4. Tape paperwork to cooler.	Place paperwork in a sealed Ziploc bag and tape it to the inside of the cooler lid.
5. Bag samples for separate analytical batches.	If the shipping cooler contains more samples than can be analyzed in one analytical batch, the laboratory may request that the samples in the cooler be bagged for separate analytical batches. This may be necessary so that the appropriate Quality Control/ Quality Assurance samples are included in each analytical batch. In this case, fill out separate COC forms for each batch and include the forms in the appropriate plastic bags. Place the COC forms for each batch in a sealed Ziploc bag. The COC forms for each batch should be placed at the top of the plastic bag so that they are clearly visible to laboratory personnel when they open the plastic bags.
6. Label the cooler.	Label the cooler with the appropriate labels to describe the content of the cooler (e.g., NOS, flammable liquids, flammable solids, this side up, fragile, etc.). Close the cooler and place the appropriate shipping labels (e.g., overnight shipping from Federal Express, UPS, or the U.S. Postal Service or equivalent) on the lid of the cooler.
7. Sign CoC seals.	The Field Team Leader or the designated representative will sign CoC seals and place the signed seals over the opening edge of the cooler.

8. Tape the cooler.	Place tape over the custody seals and around the cooler.
9. Transport the cooler.	Transport the cooler(s) to a secure storage, to the shipping agent, or directly to the laboratory. If shipping the cooler, follow established federal and state regulations depending on cooler content.
Note:	Bagging of samples and lining of coolers is not necessary, if samplers transport the samples directly to the laboratory.

DRAWINGS, DOCUMENTS, AND TOOLS/EQUIPMENT **The following documents should be referenced to assist in completing the associated task**.	
P&IDS	
Drawings	
Related SOPs/ Procedures/ Work Plans	As per individual site SAPs.
Tools	Plastic bags, Ziploc bags, non-contaminating packaging materials, tape, COC seals, ice, and cooler
Forms/Checklist	Chain of Custody forms.

PURPOSE	This Standard Operating Procedure (SOP) establishes the requirements for documenting and maintaining environmental sample chain of custody from point of origin to receipt of sample at the analytical laboratory. This procedure will apply to all types of air, soil, water, sediment, biological, and/or core samples collected in environmental investigations by Pioneer Technical Services, Inc. (Pioneer). It is applicable from the time of sample acquisition until custody of the sample is transferred to an analytical laboratory.
SCOPE	Pioneer prepared this practice for the workforce and this SOP applies to all work performed by and on behalf of Pioneer. All members of the Pioneer workforce who conduct the work shall be trained and competent (as defined by OSHA) in the risk-assessed procedure described below before performing the work.
DEFINITIONS	**Chain of custody** is an unbroken trail of accountability that ensures the physical security of samples, data, and records. Custody refers to the physical responsibility for sample integrity, handling, and/or transportation. Custody responsibilities are effectively met, if the samples are: • In the responsible individual's physical possession; • In the responsible individual's visual range after having taken possession; • Secured by the responsible individual so that no tampering can occur (usually for shipping); or • Secured or locked by the responsible individual in an area in which access is restricted to authorized personnel only.

WORK INSTRUCTIONS

The following instructions provide guidance to perform the task in a safe, accurate, and reliable manner. If these instructions present information that is inaccurate or unsafe, personnel must notify the Project Manager, Safety Manager, and the SOP Technical Author to initiate appropriate revisions. Personnel will perform all work under this SOP in a manner that is consistent with procedures and policies described in the appropriate Operation, Maintenance, and Monitoring (O&M) Plan (where applicable), appropriate Site-Specific Health and Safety Plans (SSHASP), and Pioneer Corporate Health and Safety Plan (HASP).

TASK	INSTRUCTIONS
Project Manager's Responsibilities	The Project Manager is responsible for overall management of environmental sampling activities, designating sampling responsibilities to qualified personnel, and reviewing any changes to the sampling plan.
Field Team Leader's Responsibilities	The Project Manager may act as the Field Team Leader or may choose to appoint a Field Team Leader. The Field Team Leader is responsible for general supervision of field sampling activities and ensuring proper storage/transportation of samples from the field to the analytical laboratory. The Field Team Leader is also responsible for maintaining sample custody as defined above until the sample has been properly relinquished as documented on the chain of custody form.

	The Field Team Leader will review chain of custody forms for accuracy and completeness to preserve sample integrity from collection to receipt by an analytical laboratory. The review of chain of custody forms may be delegated to qualified personnel.
Field Sampler's Responsibilities	The Field Sampler is responsible for sample acquisition in compliance with technical procedures, initiating the chain of custody, and checking sample integrity and documentation prior to transfer. Field samplers are also responsible for initial transfer of samples consisting of physical transfer of samples directly to the internal laboratory or transferred to a shipping carrier, (e.g., United Parcel Service or Federal Express) for delivery.
Laboratory Technician's Responsibilities	The receiving Laboratory Technician is responsible for inspecting transferred samples to ensure proper labeling and satisfactory sample condition. Unacceptable samples will be identified and segregated. The Laboratory Project Manager will be notified. The Laboratory Technician will review the chain of custody for completeness and file as part of the project's permanent record.
Fill out Chain of Custody Forms	The Field Team Leader or designated Field Sampler will initiate the chain of custody form for the initial transfer of samples. A chain of custody form will be completed and accompany every sample set. Only those samples included in the shipping container (cooler or box) should be listed on the chain of custody form included in the container. All chain of custody forms must be completed and include the following information: • Project code. • Project name. • Sampler's signature. • Sample identification. • Date sampled. • Time sampled. • Analysis requested. • Remarks column should contain information about a sample that the laboratory might need. Examples of remarks that should be included: ▪ If samples could have very high or low expected concentrations (outside of normal instrument calibration range). ▪ DO NOT USE FOR QA/QC (quality assurance/quality control) should be indicated for field blanks, bottle blanks, or equipment rinsate blanks. ▪ If a sample should be held for later analysis (i.e., if sample being analyzed requires results from another sample to determine analysis status).

	▪ The sample should be archived after initial analysis by the laboratory for potential additional analysis in the future. ▪ Requires filtering (if not completed in the field). ▪ Requires preservation (if not completed in the field). ▪ Any other sample specific information that will aid the laboratory in completing the appropriate analysis. • Relinquishing signature, data, and time. • Receiving signature, date, and time. Laboratory-provided chain of custody forms should be used if provided, and all required fields should be filled out. Pioneer also has generic chain of custody forms that can be used if no laboratory forms are available. Make sure that the above required information is on the form and include the laboratory name and address to which the samples are being shipped. The Field Sampler relinquishing custody and the responsible individual accepting custody will sign, date, and note the time of transfer on the chain of custody form. Note: if the transporter is not an employee of Pioneer, the Field Sampler may identify the carrier and reference the bill of lading number in lieu of the transporter's signature. One copy of the chain of custody form will be filed as a temporary record of sample transfer by the Field Sampler. The original form will accompany the sample set and will be returned to Pioneer as part of the contracted laboratory QA/QC requirements. The original form and the transporter's receipt will be filed as part of the project's permanent records. The Project Manager (or designee) will track the chain of custody to ensure timely receipt of samples by an analytical laboratory. Shipping information, including date shipped, laboratory shipped to, transporter's identity (i.e., Federal Express), and tracking number should be recorded in the field logbook. If more than one sample shipment occurs during a project, the associated samples per shipment should be referenced (sample numbers or samples collected on these dates).
Sample Handling.	All samples will be collected and handled in accordance with SOP-SA-01 Soil and Water Sample Packaging and Shipping and SOP-SA-02 Sample Preservation and Containerization for Aqueous Samples, or methods described in the Sampling and Analysis Plan (SAP) or Work Plan (WP). Samples will be transported in insulated coolers with ice as necessary to maintain a temperature of 4 degrees Celsius (°C) plus or minus 2 °C until receipt by the analytical laboratory. Alternate shipping containers can be used if the analytical method, SAP, or WP does not have temperature requirements for the samples.

HEALTH SAFETY SECURITY ENVIRONMENT (HSSE) CONSIDERATIONS

This section to be completed with concurrence from the Safety and Health Manager.

SOURCE	*HAZARDS*	*WHERE*	*HOW, WHEN, RESULT*	*CONTROLS*
CHEMICAL	Potential contact with contaminated water/soil samples.	Outside of bottles.	Inadvertent exposure to contaminated water/soil samples could lead to adverse health effects.	Personnel will practice proper personal hygiene – wash hands prior to eating/drinking and when leaving the site. Personnel will wear nitrile gloves and safety glasses when handling sample containers.
	Preservatives (HCL, HNO_3, H_2SO_4, Zinc, Acetate, and NaOH).	Outside of bottles.	Inadvertent exposure to preservatives could lead to adverse health effects.	Safety Data Sheets for each preservative chemical are available to all Personnel on the Pioneer company web site. Personnel will wear nitrile gloves and safety glasses when handling the bottles. Refer to the Chemical Flushing Guidelines available inside vehicle's first aid kit for first-aid procedures in case of contact with preservatives.
NOISE	Not applicable.			
ELECTRICAL	Not applicable.			
BODY MECHANICS	Improper lifting.	Sites.	Back injuries and muscle/back strains could result when using improper techniques to lift and carry packaged samples and coolers.	Personnel will use proper lifting techniques – get a good grip, keep the load close to the body, lift with legs and not with back, and avoid lifting loads above shoulder's height. Two workers will lift/carry packaged samples and coolers, if needed.

HEALTH SAFETY SECURITY ENVIRONMENT (HSSE) CONSIDERATIONS

This section to be completed with concurrence from the Safety and Health Manager.

SOURCE	*HAZARDS*	*WHERE*	*HOW, WHEN, RESULT*	*CONTROLS*
GRAVITY	Falls from slips and trips.	Uneven terrain, slick/muddy/wet surfaces and steep slopes.	Walking/working on slick/muddy/ wet and uneven terrain could cause slips and trips resulting in falls and injuries.	Personnel will wear work boots with good traction and ankle support. Personnel will be aware of working/walking surfaces and choose a path to avoid hazards. Keep work areas as dry as possible.
WEATHER	Not applicable.			
RADIATION	Not applicable.			
BIOLOGICAL	Not applicable.			
MECHANICAL	Not applicable.			
PRESSURE	Not applicable.			
THERMAL	Not applicable.			
HUMAN FACTORS	Inexperienced and improperly trained personnel.	Sites.	Inexperienced personnel and improper training could cause incidents resulting in adverse health effects and/or property damage.	Personnel will be properly trained in this procedure and other applicable procedures. Personnel will implement stop work procedures, if necessary.
SIMOPS (Simultaneous Operations)	Not applicable.			

ADDITIONAL HSSE CONSIDERATIONS This section to be completed with concurrence from the Safety and Health Manager.	
REQUIRED PPE	**Personal Protection Equipment** (PPE): Safety glasses, high-visibility work shirt or vest, long pants, work boots, and nitrile gloves.
APPLICABLE SDSs	**Safety Data Sheets** (SDSs): HCL, HNO_3, H_2SO_4, Zinc, Acetate, and NaOH. Safety Data Sheets are available to Pioneer employees at the link below: https://pioneertechnicalservices.sharepoint.com/Safety/SafetyDataSheets
REQUIRED PERMITS/ FORMS	Per site/project requirements.
ADDITIONAL TRAINING	Per site/project requirements.

DRAWINGS, DOCUMENTS, AND TOOLS/EQUIPMENT The following documents should be referenced to assist in completing the associated task.	
DRAWINGS	
RELATED SOPs/ PROCEDURES/ WORK PLANS	SOP-SA-01 Soil and Water Sample Packaging and Shipping and SOP-SA-02 Sample Preservation and Containerization for Aqueous Samples.
TOOLS/ EQUIPMENT	Seals and labels, chain of custody forms, chain of custody seals (provided by contracted laboratory), packing and shipping materials, cooler, and ice.
FORMS/ CHECKLIST	Chain of custody forms.

APPROVALS/CONCURRENCE

By signing this document, all parties acknowledge the completeness and applicability of this SOP for its intended purpose. Also, by signing this document, it serves as acknowledgement that I have received training on the procedure and associated competency testing.

SOP TECHNICAL AUTHOR	DATE
Julie Flammang **Julie Flammang**	**11/12/2020**
SAFETY AND HEALTH MANAGER	**DATE**
Tara Schleeman **Tara Schleeman**	**11/12/2020**

PURPOSE	This SOP establishes the requirements for documenting and maintaining field logbooks and photographs. These procedures apply from the time field work begins until site activities are completed.
SCOPE	This practice has been prepared as a basic guide for project documentation.

WORK INSTRUCTIONS

The following instructions are intended to provide sufficient guidance to perform the task in a safe, accurate, and reliable manner. Should these instructions present information that is inaccurate or unsafe, operations personnel must bring the issue to the attention of the Project Manager and the appropriate revisions made.

TASK	INSTRUCTIONS
Logbooks	A designated field logbook will be used for each field project. If requested by the Project Manager, use a separate field logbook for each field task within a larger project. Label each logbook with the project name, dates that it covers, and logbook number. Use a waterproof marker, such as a Sharpie©, to write down the information. The logbooks will be bound and have consecutively numbered pages. The information recorded in these logbooks shall be written in ink. Begin a new page for each days notes. Write on every line of the logbook. If a blank space is necessary for clarity, such as a change of subject, skip one line before beginning the new subject. Do not skip any pages or parts of pages unless a day's activity ends in the middle of a page. Draw a diagonal line on any blank spaces of four lines or more to prevent unauthorized entries. The author will initial and date entries at the end of each day. All corrections will consist of a single line-out deletion in ink, followed by the author's initials and the date. Information not related to the project should not be entered in the logbook. The language used in the logbook should be factual and objective. These bound logbooks shall include the following entries: 1. A description of the field task. 2. Time and date fieldwork started. 3. Location and/or a description of the work areas including sketches, if needed, any maps or references needed to identify locations, and sketches of construction activities. If the location has been documented in the logbook during/prior visits, only changes in conditions should be noted. 4. Names and company affiliations of field personnel. 5. Name, company affiliation or address, and phone number of any field contacts or official site visitors. 6. Meteorological conditions at the beginning of fieldwork and any ensuing changes in these conditions. 7. Details of the fieldwork performed and reference to field data sheets, if used. 8. Deviation from the task-specific Sampling and Analysis Plan (SAP), Work Plan (WP), or Standard Operating Procedures (SOP). 9. All field measurements made.

	10. Any field laboratory analytical results. 11. Personnel and equipment decontamination procedures, if appropriate. For any field sampling work, the following entries should be made: 1. Sample location and number. 2. Sample type and amount collected. 3. Date and time of sample collection. 4. Type of sample preservation. 5. Split samples taken by other parties. Note the type of sample, sample location, time/date, name of person for whom the split was collected, that person's company, and any other pertinent information. 6. Sampling method, particularly any deviations from the SOP. 7. Documentation or reference of preparation procedures for reagents or supplies that will become an integral part of the sample, if available. This information may not be available for water or soil sampling bottles that come preserved from the laboratory or for preservatives provided by the laboratory. Bottle blanks will need to be used to evaluate the provided reagents. 8. The laboratory where the samples will be sent. No bound field logbooks will be destroyed or thrown away even if they are illegible or contain inaccuracies that require a replacement document.
Photographs	Take photographs of field activities using a digital camera. Photographs should include a scale in the picture when practical. Telephoto or wide-angle shots will not be used, since they cannot be used in enforcement meetings. The following items shall be recorded in the bound field logbook or on a field data sheet for each photograph taken: 1. The photographer's name, the date, the time of the photograph, and the general direction faced. 2. A brief description of the subject and the fieldwork portrayed in the picture. 3. Sequential number of the photograph. An electronic copy and/or a hard copy of the photographs shall be placed in task files in the field office after each day of field activities. Supporting documentation from the bound field logbooks or field data sheets shall be photocopied and placed in the task files to accompany the photographs once the field activities are complete

DRAWINGS, DOCUMENTS, AND TOOLS/EQUIPMENT The following documents should be referenced to assist in completing the associated task.	
P&IDS	
Drawings	
Related SOPS/ Procedures/ Work Plans	
Tools	Field logbook, Sharpie©, black pen, digital camera, and field data sheets.
Forms/Checklist	

STANDARD OPERATING PROCEDRE

FIELD LABORATORY DETRMINATION OF SOIL PH USING HI 99121 SOIL PH METER

February 10, 2012

Field Laboratory Procedure

1. **Operation of Device**
    a. To turn the device on or off Press: On/Off.
    b. To Freeze the device Press: Set/Hold.

2. **Calibrate the PH Meter**
    a. Connect the PH probe to the meter.
    b. Hold the On/Off button until Calibration is visible on the screen.
    c. Put the probe in 7.01 calibration solution.
    d. The meter will recognize the solution and calibrate.
    e. Once the calibration is recognized and stable, press: On/Off

3. **To Take a Measurement**
    a. Connect the probe when the device is off.
    b. Remove the protective cap from the probe.
    c. Insert the probe into the sample.
    d. Wait until the "not stable" read out has turned off; and
    e. Record the measurement.

4. **Direct Ground Measurement of PH**
    a. Verify that the Meter is calibrated.
    b. Dig a small hole, discarding the top 5 centimeters (2 inches) of soil.
    c. Perforate the soil with the included soil drill to a depth of at least 20 centimeters (8 inches).
    d. If the soil is dry, moisten with a small amount of distilled water.
    e. Rinse the probe with tap water (not distilled).
    f. Insert the probe slightly into the soil, making sure that it is in contact with the soil surfaces.
    g. Once the readings have stabilized record the measurement.
    h. Remove the probe from the hole, gently clean off loose soil with your fingers (avoid using a rag or cloth) and then rinse the probe with tap water;
    i. Repeat this procedure in several locations; then
    j. Average the results.

5. **Measurement of Soil PH Solution**
    a. Verify that the Meter is calibrated.
    b. Collect a soil sample:
        i. Collect a minimum of one sample per 0.25 acres if the area is homogeneous (soil type, vegetation type, slope etc.).
            1. A minimum of 2 subsamples are recommended for each sample.
            2. If the area is considered "contaminated" collect all samples for that composite within that area.
            3. Collect a similar quantity for each subsample.
        ii. Dig a small hole, discarding the top 5 centimeters (2 inches) of soil, collect the sample from the hole. Complete this step for each subsample.
        iii. Thoroughly mix the subsamples for each sample together, discarding vegetation and aggregates.
        iv. Spread the sample on a sheet (paper, foil or aluminum pan) and allow to dry in a shaded area or place in an oven to dry. Discard sheet when done drying.
    c. Measuring PH of the Soil Sample
        i. Sift the soil sample through a clean #10 screen.
        ii. Measure 10 grams of the sample and place it in a beaker.
        iii. Measure 25 milliliters of Soil Solution HI 7051 into the beaker.

iv. Mix for 30 seconds.
v. Let the mixture sit 5 minutes.
vi. Mix again; and
vii. Place probe in mixture and wait for reading to stabilize. Record the measurement.
viii. Rinse the probe with tap water prior to next use. If needed remove any remaining soil on the probe using a finger (avoid using a rag or cloth).

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PURPOSE	To provide standard instructions for operating XL3 X-Ray Fluorescence (XRF) analyzer
SCOPE	This practice has been prepared for task trained personnel conducting work on unreclaimed sites within the BPSOU area. The tasks are general and are to be used in conjunction with published manufacturer and internal practices.

WORK INSTRUCTIONS

The following instructions are intended to provide general guidance to perform the task in a safe, accurate, and reliable manner. Should these instructions present information that is inaccurate or unsafe, operations personnel must bring the issue to the attention of the Project Manager and the appropriate revisions made. All work carried out under this SOP will be consistent with procedures and policies described within appropriate internal policies.

TASK	INSTRUCTIONS
1. Assemble XRF stand.	a. Open the case containing the stand and insert 4 legs into base of stand. b. Place stand on a solid, level surface.
2. Prep XRF sample for analysis.	a. Wearing latex or nitrile gloves, remove any large aggregate from the sample and place in a separate bag for disposal. For gravel or rocky soils, a sieve can be used to remove the large aggregates. If a sieve is used, it needs to be decontaminated between samples. Refer to SOP General Equipment Decontamination for instructions. b. Consolidate the sample into the bottom of the baggie. c. Open the lid to the XRF stand and place sample inside, making sure that sample is flush against the opening on the inside of the XRF stand. d. Close the lid to the XRF stand.
3. Turn on XRF case.	a. Open the XRF case and remove XRF gun from case. b. Slide XRF battery onto bottom of XRF gun handle. c. Press and hold power button ( ) until XRF gun turns on and wait for system to start. d. Press where it says 'press to logon.' A warning message appears asking to verify that the user is aware of the radiation source in the XRF unit. e. Press 'Yes' to continue.
4. Log in and calibrate detector.	a. Type in appropriate password when prompted. b. Click ‘E’ to log in. After logging in, a screen appears with 7 icons appears, this is the Main Menu screen. c. Tap the ‘System Check’ icon. d. Tap ‘Yes.’ e. The XRF unit will then go through an internal calibration. f. When the calibration is done, tap ‘CLOSE’ on the XRF gun to return to the Main Menu screen. **The detector should be calibrated at the start of each day of operation.**
5. Set up XRF run test.	a. Set parameters (e.g., analysis types, time, and analytes) required for the analysis as detailed in the XL3 user’s manual, Sampling and Analysis Plan (SAP), or Work Plan. b. Once logged into XRF system, tap the ‘Analyze’ icon on XRF screen. A screen appears. c. On the next screen tap ‘Soils.’ d. On the next screen tap ‘Data Entry.’ A Data Entry screen appears showing several options (Sample Name, Sampler, Date, etc.).

	e. In the upper right-hand corner, next to the 'Sample Name' icon, click the symbol that looks like a miniature keyboard to display a keyboard on the screen. f. Type in the sample name (do not press return yet). g. Insert XRF gun into the bottom of the XRF stand with the XRF gun handle pointing away from you. Be sure that the XRF gun is securely in place in the bottom of the stand. h. Press 'return' in the lower right corner of the keyboard screen. i. To activate the unit, pull the trigger on the gun handle. The analysis will take approximately 2 minutes to complete.
6. Record data.	a. After the XRF analysis is complete, results from the analysis will appear on the screen. b. Record the results and Test Number displayed on the screen; use the up and down arrows on the XRF gun to scroll through data. c. Open the lid on the XRF stand and remove the sample. d. Mark the sample baggie as "RAN" so that sample does not get analyzed twice. Place ran samples in a labeled box for storage and record keeping.
7. Run additional samples.	a. With the XRF gun still in the XRF stand, press the return button (↵) on the XRF gun. This will display the 'Data Entry' screen. b. On the Data Entry Screen, press the keyboard symbol located to the right of 'Sample Name' to display the keyboard. c. Type the next sample name (do not press return yet). d. Place the sample into the XRF stand and close the lid to the stand (as discussed in Task 2). e. Repeat the steps in Task 5 to activate the XRF unit. f. Repeat Tasks 6 and 7 until all samples are analyzed.
8. Turn off XRF.	a. After all samples have been analyzed, remove the XRF gun from the bottom of the stand (press and hold buttons on the side of the stand to allow XRF gun to be removed from stand). b. Press the return button (↵) on the XRF gun until the Main Menu screen appears. c. Press and hold the power button (⏻) until the XRF turns off. d. Remove the battery from the gun and place these items back into the appropriate case. e. Disassemble the XRF stand and place back into the appropriate case.

Quality Assurance/ Quality Control (QA/QC) Requirements.	**Required QA/QC tasks:** 1. Run the Niton-supplied XRF blanks and NIST standards at the start of each day. 2. Record the results in the field logbook or on the XRF field datasheet or equivalents. If the results are not within the ranges supplied by NITON in the user manual, initiate troubleshooting tasks on the analyzer (refer to the user's manual). 3. Run the blank and one standard QA/QC samples during sample analysis at the rate of 1 for every 20 samples analyzed. QA/QC includes analyzing a replicate sample every 20 samples and a duplicate sample (see the steps below). **Analyze a replicate sample (1 for every 20 samples analyzed)** 1. After recording the initial reading for a sample, DO NOT remove the sample from the holder. 2. Restart the XRF gun and rerun the sample. 3. Record the information on the field data form or logbook as a replicate (or R sample). Replicates samples help track the precision of the XRF.

	Analyze a duplicate sample (after every 20 samples analyzed) 1. After every 20 samples, analyze a duplicate sample by recording the results of the 20th sample. 2. Remove the sample bag from the XRF stand, remix the sample, and replace it in the XRF stand. 3. Reanalyze the sample. 4. Record the results as a duplicate (or D sample). Duplicates help to determine the precision of the XRF analysis as well as the homogeneity of the sample matrix. 5. Run a NITON-supplied blank or NIST standard after the replicate/duplicate QA/QC samples to monitor the accuracy of the XRF results.

DRAWINGS, DOCUMENTS, AND TOOLS/EQUIPMENT **The following documents should be referenced to assist in completing the associated task**.	
Drawings	
Related SOPs/ Procedures/ Work Plans	SOP-DE-02 General Equipment Decontamination.
Tools	XRF and hand tools.
Forms/Checklist	Private Property Access Agreement, if required.

APPROVALS/CONCURRENCE

By signing this document, all parties acknowledge the completeness and applicability of this SOP for its intended purpose. Also, by signing this document, it serves as an acknowledgement that I have received training on the procedure and associated competency training

Manager	**Date**
Lead Operator	**Date**
Operator	**Date**

SOP-DE-01. PERSONAL DECONTAMINATION PROCEDURES - GENERAL	REVISION: 0 PAGE 1 of 2

PURPOSE	To provide standard instructions for decontamination of all personnel leaving a contaminated area.
SCOPE	This practice has been prepared for task trained personnel conducting work on unreclaimed sites within the BPSOU area. The tasks are general and are to be used in conjunction with published manufacturer and internal practices.

WORK INSTRUCTIONS

The following instructions are intended to provide general guidance to perform the task in a safe, accurate, and reliable manner. Should these instructions present information that is inaccurate or unsafe, operations personnel must bring the issue to the attention of the Project Manager and the appropriate revisions made. All work carried out under this SOP will be consistent with procedures and policies described within appropriate internal policies.

TASK	INSTRUCTIONS
1. Wash/ Remove outer contaminated items.	Remove nitrile or latex gloves by grasping the outside of the opposite glove near the wrist. Pull and peel the glove away from the hand, turning the glove inside out with the contaminated side now on the inside. Hold the removed glove in the opposite gloved hand. Slide one or two fingers of the ungloved hand under the wrist of the remaining glove. Peel glove off from the inside, creating a bag for both gloves. If wearing protective coveralls such as Tyvec suites, brush built up material off the suit, only if in designated decontamination zone. Unzip the coverall and begin rolling that outwards, rolling it down over your shoulders. Place both hands behind your back and pull down each arm until completely removed. Sit down and remove each shoe then roll the coveralls down (ensuring the contaminated side is not touched or comes into contact with clothing) over your knees until completely removed. If there is not a designated decontamination zone, remove personal protective equipment (PPE) carefully to contain material and place it in the appropriate disposal container. For instructions to remove additional PPE not described in this document, refer to the project's HASP. Wash with soap (nonphosphate) and tap water the outer, more heavily contaminated items, such as boots. Rinse the items in tap water.
2. Wash inner contaminated items.	If necessary, wash with soap (nonphosphate) and tap water the inner, less contaminated items. Rinse the items in tap water.
3. Store/ transport items.	Store/transport contaminated items in a separate designated area to prevent cross contamination prior to disposal.
4. Dispose of contaminated items.	Dispose of contaminated clothing and equipment in accordance with site/project, client, and/or federal and state requirements.
5. Contact the Safety and Health Manager.	For contaminants other than those found typically at uncontrolled hazardous waste sites, such as asbestos, PCB, PCE, etc. see the Safety and Health Manager.

Information about Emergency Decontamination	
1. During life-saving process.	If the decontamination procedure is essential to the life-saving process, decontamination must be performed immediately.
2. During heat-related illness.	If heat-related illness develops, protective clothing should be removed as soon as possible. Wash, rinse, and/or cut off protective clothing/equipment.
3. When medical treatment is needed.	If medical treatment is required to save a life, decontamination should be delayed until the victim is stabilized. Wrap the victim to reduce contamination of others.

DRAWINGS, DOCUMENTS, AND TOOLS/EQUIPMENT The following documents should be referenced to assist in completing the associated task.	
Drawings	
Related SOPS/ Procedures/ Work Plans	
Tools	In general, the following items will be needed: soap, tap water, tarps, decontamination tubs, brushes, and sprayer. The Sampling and Analysis Plan (SAP) will describe additional items needed for decontamination, if required.
Forms/Checklist	

APPROVALS/CONCURRENCE

By signing this document, all parties acknowledge the completeness and applicability of this SOP for its intended purpose. Also, by signing this document, it serves as an acknowledgement that I have received training on the procedure and associated competency training

Manager	**Date**
Lead Operator	**Date**
Operator	**Date**

PURPOSE	To provide standard instructions for equipment decontamination.
SCOPE	Pioneer Technical Services, Inc. (Pioneer) prepared this practice for the workforce and this Standard Operating Procedure (SOP) applies to all work performed by and on behalf of Pioneer. All members of the Pioneer workforce who conduct the work shall be trained and competent (as defined by OSHA) in the risk-assessed procedure described below before performing the work.
NOTES	All equipment leaving the contaminated area of a site must be decontaminated. Decontamination methods include removal of contaminants through physical, chemical, or a combination of both methods. Decontamination procedures are to be performed at the same level of protection used in the contaminated area of a site. In some cases, decontamination personnel may be sufficiently protected by wearing one level lower protection. The information for site-specific equipment decontamination and personnel protection levels, as detailed in the Sampling and Analysis Plan (SAP), work plan (WP), and Site-Specific Health and Safety Plan (SSHASP), should be followed. The following decontamination procedures are for typical uncontrolled hazardous waste sites. For a specific or unusual contaminant, such as dioxins, see the SSHASP and consult with the Safety and Health Manager. Decontamination procedures should be used in conjunction with methods to prevent contamination of sampling and monitoring equipment. If practical, particularly with organic contaminants, one-time-use equipment should be used and disposed of in accordance with the SAP, WP, and SSHASP. This SOP covers all equipment decontamination EXCEPT for submersible pumps. Decontamination of pumps is detailed in SOP-DE-02A – Equipment Decontamination - Pumps for Well Sampling.

WORK INSTRUCTIONS

The following instructions provide guidance to perform the task in a safe, accurate, and reliable manner. If these instructions present information that is inaccurate or unsafe, personnel must notify the Project Manager, Safety Manager, and the SOP Technical Author to initiate appropriate revisions. Personnel will perform all work under this SOP in a manner that is consistent with procedures and policies described in the appropriate Operation, Maintenance, and Monitoring (O&M) Plan (where applicable), appropriate Site-Specific Health and Safety Plans (SSHASP), and Pioneer Corporate Health and Safety Plan (HASP).

TASK	INSTRUCTIONS
1. Set up decontamination station.	a. Review the SAP or WP and determine if decontamination fluids need to be contained and the need for special decontamination requirements (i.e., chemical rinse). b. If the fluids require containment, set up the decontamination station so that it is located within a small plastic swimming pool or on plastic sheeting with turned up edges to contain water that may slop over during the decontamination process.

	c. If pressurized or gravity flow water is available, attach a hose or piping to reach the decontamination area. If no water is available, bring 5-gallon containers of tap and deionized water (DI) to the decontamination area to clean the equipment. d. Label empty 5-gallon buckets: *gross wash, soap wash, DI rinse, final rinse, and chemical rinse* (if required). e. Lay out clean plastic or foil to place cleaned equipment on to allow for air drying. f. If a chemical rinse is required, fill a spray bottle with the appropriate chemical and label the spray bottle with the chemical's name. g. Pour approximately 2.5 to 3 gallons of tap water into the buckets labeled: *gross wash* and *soap wash*. h. Add a **few drops** (1-3 drops) of Liquinox© soap to the bucket marked *soap wash*. i. Pour 2.5-3 gallons of DI water into the buckets labeled: *DI rinse* and *final rinse.* If a chemical rinse is required, pour DI water into the bucket labeled: *chemical rinse.*
2. Remove gross contamination.	Remove gross contamination using pressurized or gravity flow tap water, if available. If not, manually scrub the equipment using the 5-gallon bucket of water marked *gross wash* and a stiff brush (dedicated to the gross wash step).
3. Wash equipment.	Move the equipment to the 5-gallon bucket marked *soap wash*. Wash equipment with a stiff brush (dedicated to the soap wash step).
4. Triple rinse equipment.	In the bucket marked *DI rinse*, triple rinse the equipment with DI water to remove any soap residue.
5. Second rinse with deionized water.	Using DI water, triple rinse the equipment again in the bucket marked *final rinse* if a chemical rinse is not required.
6. Rinse equipment with chemicals.	In many cases, the tap water and DI water rinses will be sufficient. However, if specified in the SAP, WP, or SSHASP, chemical rinses of the equipment may be required. For inorganic contaminants, a mixture of 10:1 nitric acid in distilled water (10 parts water to 1 part nitric acid) may be specified. A methanol rinse may be required for some organic contaminants, such as hydrocarbons. Spray bottles, clearly marked with the appropriate chemical name, are an acceptable means of rinsing most equipment. **To perform the chemical rinse:** a. Hold the equipment over a collection container (5-gallon bucket or bowl). b. Make sure that all personnel and vehicles are upwind of the spray. c. Spray the piece of equipment inside and out starting at the top and working down to the bottom. d. Dispose of the contained chemicals as described in the SAP, WP or SSHASP. The Safety and Health Manager and/or Project Manager must approve the disposal method used.

7. Rinse equipment with deionized water.	After a required chemical rinse, rinse the equipment again with the DI water in the bucket marked *chemical rinse*. This DI water will need to be retained (i.e., do not dispose of this water on the site), tested, and disposed of according to federal and state requirements for the chemical used. The Safety and Health Manager and/or Project Manager must approve the disposal method used. After the rinse in the *chemical rinse* bucket, triple rinse the equipment again in the bucket marked *final rinse.*
8. Air dry equipment.	Place equipment on plastic sheeting or foil to air dry.
9. Transport/ store equipment.	Wrap equipment in foil or plastic wrap to transport or store.
10. Clean decontamination equipment.	a. Triple rinse equipment from the *gross wash* and *soap wash* (brushes and buckets) with clean tap water, preferably with pressurized water. Soap can be used on particularly dirty equipment. b. Triple rinse all decontamination equipment with DI water, including *DI rinse* and *final rinse* buckets. c. Store decontamination equipment, labeled and in a clean location so they are used only for decontamination purposes.
11. Dispose of decontamination solutions.	Storage of contained decontamination fluids as required by the SAP, QAPP, or WP or of residue from a chemical rinse should have been arranged on site prior to sampling. Once the sampling and associated decontamination is complete, sampling of the stored fluids for hazardous waste criteria will be required. If the fluids are determined to be hazardous (e.g., meet the characteristics of a hazardous waste [ignitability, corrosivity, reactivity, or toxicity] or contain listed wastes from title 40 of the Code of Federal Regulations [CFR] in part 261.4), dispose of them according to federal and state requirements. The Safety and Health Manager and/or Project Manager must approve the disposal method used. Note: when using other than the above-mentioned solutions, check with the Safety and Health Manager and the Project Manager.
12. Measure effectiveness of procedures.	Measure the effectiveness of the decontamination procedures using field equipment rinsate blanks as discussed in the SAP, QAPP, or WP.

HEALTH SAFETY SECURITY ENVIRONMENT (HSSE) CONSIDERATIONS
This section to be completed with concurrence from the Safety and Health Manager.

SOURCE	*HAZARDS*	*WHERE*	*HOW, WHEN, RESULT*	*CONTROLS*
CHEMICAL	Potential contact with contaminated items and resulting water from decontamination procedures.	Sites.	Inadvertent exposure to contaminated items and water resulting from decontamination procedures could lead to adverse health effects.	Personnel will practice proper personal hygiene (wash hands prior to eating/drinking and when leaving the site); follow decontamination procedures as described above; and wear nitrile gloves and safety glasses when handling contaminated items.
	Chemical rinse (e.g., dilute nitric acid, methanol, and hexane).	Sites.	Personnel could be exposed to chemicals via ingestion and skin/eye contact when decontaminating equipment. Exposure could cause irritation of skin/eye and adverse health effects.	Personnel will check and follow safety procedures as outlined in the chemical-specific Safety Data Sheets. Personnel will prevent skin/eye contact with chemicals and they will wear nitrile gloves and eye protection when handling chemicals. Personnel will practice proper personal hygiene (wash hands prior to eating/drinking, after decontaminating equipment, and when leaving the site). All personnel and vehicles will stand upwind when spraying equipment with chemicals. Refer to the Chemical Flushing Guidelines available inside any Pioneer vehicle's first aid kit for first-aid procedures in case of contact with chemicals.
NOISE	Not applicable.			
ELECTRICAL	Not applicable.			

HEALTH SAFETY SECURITY ENVIRONMENT (HSSE) CONSIDERATIONS
This section to be completed with concurrence from the Safety and Health Manager.

SOURCE	*HAZARDS*	*WHERE*	*HOW, WHEN, RESULT*	*CONTROLS*
BODY MECHANICS	Improper lifting.	Sites.	Back injuries and muscle/back strains could result when using improper techniques to lift and carry 5-gallon containers.	Personnel will use proper lifting techniques: get a good grip, keep the load close to the body, lift with legs and not with back, and avoid lifting loads above shoulder's height. Two people will lift awkward/heavy tools and equipment.
GRAVITY	Falls from slips and trips.	Areas designated for decontamin-ation procedures.	Slips and falls could occur while performing decontamination procedures due to slippery surfaces resulting in bruises, scrapes, or broken bones.	Personnel will wear work boots with good traction and ankle support. Personnel will also be aware of working/ walking surfaces and choose a path to avoid hazards, keep work areas as dry as possible, and wear muck boots as necessary.
WEATHER	Cold/heat stress.	Sites.	Exposure to cold climates may result in cold burns, frostbites, and hypothermia. Exposure to high temperatures may result in heat cramps, heat exhaustion, or heat stroke.	Training on signs and symptoms of cold/heat stress is required. Personnel will wear appropriate clothing when working outdoors, remain hydrated, and have sufficient caloric intakes during the day. Personnel will also follow procedures outlined in applicable SSHASP and/or Pioneer corporate HASP.
	Hypothermia/ frostbite.	Sites where air temperature is 35.6 °F (2 °C) or less.	Personnel whose clothing becomes wet during decontamination procedures may be exposed to hypothermia and/or frostbite.	Personnel will change clothing if it becomes wet.

HEALTH SAFETY SECURITY ENVIRONMENT (HSSE) CONSIDERATIONS
This section to be completed with concurrence from the Safety and Health Manager.

SOURCE	*HAZARDS*	*WHERE*	*HOW, WHEN, RESULT*	*CONTROLS*
	Lightning.	Outdoor sites.	Electrocution, injury, death, or equipment damage could be caused by lightning strike.	Personnel will follow the 30/30 rule during lightning storms.
RADIATION	Ultraviolet (UV) radiation.	Outdoors.	Personnel could be exposed to UV radiation during summer months causing sun burns, skin damage, and eye damage.	Personnel will wear safety glasses with tinted lenses, long-sleeve work shirts, and long pants. Personnel should wear sunscreen, if necessary.
BIOLOGICAL	Plants, insects, and animals.	Sites.	Exposure to plants, insects, and/or animals may cause rashes, blisters, redness, and swelling.	Training on the signs and symptoms of exposure to plants, insects, and animals is required. Personnel will avoid contact with plants, insects, and animals. First-aid kits will be available on the site. Personnel with allergies will notify their supervisor.
MECHANICAL	Not applicable.			
PRESSURE	Not applicable.			
THERMAL	Contact with hot surfaces.	Foil and decontamination equipment.	If foil and decontamination equipment are placed directly in the sun, they could get hot. Contact with hot surfaces could result in personal injury.	Personnel will not set decontamination stations directly in the sun.

HEALTH SAFETY SECURITY ENVIRONMENT (HSSE) CONSIDERATIONS This section to be completed with concurrence from the Safety and Health Manager.				
SOURCE	***HAZARDS***	***WHERE***	***HOW, WHEN, RESULT***	***CONTROLS***
HUMAN FACTORS	Inexperienced and improperly trained personnel.	Sites.	Inexperienced personnel and improper training could cause incidents resulting in injuries and/or property damage.	Personnel will be properly trained in this procedure and other applicable procedures. Personnel will implement stop work procedures, if necessary.
SIMOPS (Simultaneous Operations)	Not applicable.			

ADDITIONAL HSSE CONSIDERATIONS This section to be completed with concurrence from the Safety and Health Manager.	
REQUIRED PPE	**Personnel Protection Equipment** (PPE)**:** Safety glasses, high-visibility work shirt or vest, long pants, work boots, and nitrile gloves.
APPLICABLE SDSs	**Safety Data Sheets** (SDSs) for corresponding chemicals used during chemical rinse will be maintained based on the site characterization and contaminants. Safety Data Sheets are available to Pioneer personnel at the link below: https://pioneertechnicalservices.sharepoint.com/Safety/SafetyDataSheets
REQUIRED PERMITS/ FORMS	Per site/project requirements.
ADDITIONAL TRAINING	Per site/project requirements.

DRAWINGS, DOCUMENTS, AND TOOLS/EQUIPMENT The following documents should be referenced to assist in completing the associated task.	
DRAWINGS	
RELATED SOPs/ PROCEDURES/ WORK PLANS	

TOOLS/ EQUIPMENT	Five empty 5-gallon buckets, tap water, stiff brushes, Liquinox soap, four 5-gallon containers of DI (or distilled water if DI water is not available), chemicals for chemical rinse (if required), small plastic swimming pool/plastic sheeting or foil, tarps, and sprayers (if available). If additional items for decontamination are needed, they will be listed on the SAP.
FORMS/ CHECKLIST	

APPROVALS/CONCURRENCE

By signing this document, all parties acknowledge the completeness and applicability of this SOP for its intended purpose. Also, by signing this document, it serves as acknowledgement that I have received training on the procedure and associated competency testing.

SOP TECHNICAL AUTHOR	**DATE**
Julie Flammang **Julie Flammang**	**09/08/2020**
SAFETY AND HEALTH MANAGER	**DATE**
Tara Schleeman **Tara Schleeman**	**09/08/2020**

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STANDARD OPERATING PROCEDURE

INDUCTIVELY COUPLED PLASMA ATOMIC EMISSION SPECTROSCOPY

Reference Methods: EPA 6010B, 6010C, 6010D, and EPA 200.7

Local SOP Number:	S-MN-I-313-rev.30
Effective Date:	Date of Final Signature
Supersedes:	S-MN-I-313-rev.29

APPROVALS

Laboratory General Manager — 14 Apr 2017 — Date

Laboratory Quality Manager — 14 Apr 2017 — Date

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

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1. Purpose/Identification of Method

1.1. The purpose of this SOP is to establish a procedure for the determination of metals by inductively coupled plasma atomic emission spectroscopy (ICP-AES) as delineated in EPA Method 6010B (Dec. 1996), 6010C (Feb. 2007), 6010D (Jul. 2014) or 200.7 (Rev. 4.4).

2. Summary of Method

2.1. Prior to analysis, samples must be solubilized or digested using appropriate sample preparation methods.

2.2. This method describes the determination of elements by ICP-AES. The method measures element-emitted light by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific atomic-line emission spectra are produced by a radio-frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the lines are monitored by a charge coupled device detector (CCD).

2.3. All data is collected by simultaneous measurement. Software is used to measure and apply corrections due to background or inter-element interferences using a variety of techniques. Alternate wavelengths are also monitored for confirmation or to use in correction equations.

3. Scope and Application

3.1. **Personnel**: The policies and procedures contained in this SOP are applicable to all personnel involved in the analytical method or non-analytical process.

3.2. **Parameter**s: This SOP applies to the elements listed in Attachment I.

4. Applicable Matrices

4.1. This SOP is applicable to drinking water, ground water, aqueous samples, liquid samples, leachates, industrial wastes, soils, sludges, sediments, and other solid wastes.

5. Limits of Detection and Quantitation

5.1. The reporting limit (RL) / Limit of Quantitation (LOQ) for all analytes is listed in Attachment I. All current method detection limits (MDL) are listed in the LIMS and are available by request from the Quality Manager.

6. Interferences

6.1. Spectral Interferences are caused by background emission from continuous or recombination phenomena, stray light from the line emission of high concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra.

6.1.1. Spectral overlap can be compensated by computer-correcting the raw data after monitoring and measuring the interfering element. Unresolved overlap requires selection of an alternate wavelength. Background contribution and stray light can usually be compensated for by a background correction adjacent to the analyte line.

6.2. Physical Interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. A high solids nebulizer is used on all instruments. Internal standards are also used to monitor and correct for physical effects.

6.3. Chemical interferences include molecular compound formation, ionization effects and solute vaporization effects. Normally, these effects are not significant with the ICP technique, but if observed, can be minimized by careful selection of operating conditions, use of an ionization buffer, or by matrix matching of standards and samples.

6.4. Memory interferences result when analytes in a previous sample contribute to the signals measured in the new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer and from buildup of sample material in the plasma torch and spray chamber. Regular maintenance and awareness of samples with high concentrations minimize these interferences.

7. Sample Collection, Preservation, Shipment and Storage

7.1. Table 7.1 – Sample Collection, Preservation, Shipment and Storage

Sample type	Collection per sample	Preservation	Storage	Hold time
Liquid	Polyethylene containers. Collect dissolved metal samples and filter them immediately through a 0.45-micron filter on-site by the sampler before adding preservative. If samples are filtered at the laboratory, use a polyethylene container and preserve after filtration with HNO_3. Collect total metal samples into a nitric acid (HNO_3) preserved bottle.	Preserve immediately with HNO_3 to bring the pH to <2 For samples received with a pH>2, additional nitric acid must be added upon receipt to dissolve the metals that may have adhered to the sample container. Sample receiving personnel add the additional acid, labels the samples with the amount of acid added, the lot number of the acid, date, time and initials of person that added the acid. NOTE: Do not add HNO3 to exceed 1% of the total volume of the sample container (example: 2.5 mL for 250 mL bottle, 10 mL for 1000 mL bottle). The samples must not be analyzed for 24 hours from acid addition per the Method Update Rules. The final pH is checked and recorded prior to sample preparation.	Store total and dissolved metal samples at room temperature.	The maximum sample holding time for metals is 180 days from sample collection.
Solid	Glass or polyethylene container	N/A	Above freezing but below 6°C.	The maximum sample holding time for metals is 180 days from sample collection.

8. Definitions

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary Section.

9. Equipment and Supplies (Including Computer Hardware and Software)

9.1. Table 9.1 – Equipment and Supplies

Supply	Description	Vendor/Item #/Description
Simultaneous ICP-AES	CCD Detector, full wavelength region	Agilent 720 or Agilent 5100/5110.
Desktop computer and printer	Optimized per instrument	Various –matched with instrument

Auto-sampler	Optimized per instrument	Cetac 520, Agilent SPS3, or Agilent SPS4.
Peristaltic pump tubing	Various – including but not limited to blue-blue, red-red, white-white, black-black, orange-blue, orange-green	Fisher, Agilent, Environmental Express, SCP, Perkin-Elmer, or equivalent
Refrigerated Circulator	One per instrument	PolyScience or equivalent
Argon gas supply	high-purity grade, 99.99%	House Argon
Mechanical pipettes, and metals-free disposable pipet tips	Various	Eppendorf, Fisherband, or equivalent
Glassware / Plastic ware	Class A volumetric flasks or calibrated non-class A plastic ware	Fisher or equivalent
Disposable digestion cups	50 mL or 100 mL	Environmental Express or equivalent
Epic Pro	Data reporting software	See master list for current version
LimsLink	Data transmission software	See master list for current version
Agilent ICP Expert Software	Agilent Control & Data	See master list for current versions
Filtermate Plunge filters	2 um PTFE	Environmental Express, SC0408

10. Reagents and Standards

10.1. Table 10.1 – Reagents and Standards

Reagent/Standard	Concentration/Description	Requirements/Vendor/Item #
De-ionized Water	ASTM Type II	House E-pure DI water (>17.5 MOhm)
Concentrated Hydrochloric acid (HCl)	Trace Metals grade	Fisher or equivalent
Concentrated Nitric Acid (HNO3)	Trace Metals grade	Fisher or equivalent
Calibration Standard Stock Solutions	Custom blend	Inorganic Ventures or equivalent
Initial Calibration Verification (ICV) Stock Standard solutions	Custom blend. Must be separate stock from the calibration standards.	Spex Certiprep or equivalent
– Cesium Ionization Buffer for use with Agilent 720	50,000 PPM	High Purity Standards P/N 1B-CS-B5 or equivalent.
Wavelength Cal Solution - Agilent	Various analytes	Agilent P/N 6610030100
Internal Standards	Yttrium	Inorganic Ventures or equivalent

10.2. Table 10.2 - Working Standard Dilutions and Concentrations

11. Calibration and Standardization

11.1. Table 11.1 – Calibration and Standardization

Calibration Metric	Parameter/Frequency	Criteria	Comments
Initial Calibration (ICAL)	Instruments must be calibrated at a minimum once every 24 hours or prior to use. The instrument standardization date and time must be included in the raw data. See Attachment VI for an example run sequence.	A calibration curve must consist of a blank and at least one calibration standard.	If not met, remake standards, recalibrate, and verify before sample analysis.

Second Source Verification Standard (ICV)	Immediately after the calibration standards have been analyzed, the accuracy of the initial calibration shall be verified and documented for every analyte by the analysis of an ICV Solution at each wavelength used for analysis. The Initial Calibration Verification (ICV) Stock Solution(s) must be obtained from a different source than the calibration standards.	± 10% for method 6010B,6010C and 6010D or ± 5% for method 200.7 The RSD of the standards must be below 5% for 6010B, 6010C,and 6010D and below 3% for 200.7.	If the ICV fails, take the following action. Remake the standard accordingly if that is the cause. Re-inject the ICV one more time; if it fails stop all analysis. Perform all necessary instrument maintenance and recalibrate the instrument. Only two injections are allowed back to back, and then the system must be recalibrated.
Continuing Calibration Verification (CCV)	To ensure calibration accuracy during each analytical run, a CCV standard must be analyzed after no more than 10 samples and at the end of the run for each wavelength. The ICV solution can be utilized as the CCV.	For method 6010B, 6010C, 6010D and 200.7, the CCV must be within ± 10% of the true value. The RSD of the CCV must be below 5% for 6010B.	If the requirements for continuing calibration are not met, review for preparation error or instrument drift. A CCV may be repeated once, but a second failure requires re-analysis of any samples bracketed by a failing CCV with the following exception: If the samples bracketed are non-detect and the CCV is biased high, data may be reported as there is no impact from the high bias. If the samples associated are non-detect and the only detections are associated with the batch QC (LCS/LCSD/MS/MSD) but the QC is within limits, the data can be reported. The QC should be flagged indicating that there was bias but that there was no impact to the associated samples.
6010B/200.7 - Contract Required Detection Limit Sample (CRDLA)	The CRDLA must be analyzed at the beginning of each run for every analyte of interest. The CRDLA is analyzed at or below the RL.	± 40% (or specified by the client)	If the CRDLA fails, the system must be stopped. The CRDLA may be repeated once. If it fails again, perform any necessary maintenance and recalibrate accordingly.
6010C/D – Low Level Initial/Continuing Calibration Verification (LLICV/LLCCV)	The LLICV/LLCCV is named CRDLA for purposes of consistency. It is the same solution. The CRDLA must be analyzed following the ICV at a concentration at or below the RL. Additionally, the CRDLA must be analyzed after samples to cap them for method 6010C or client request. This frequency varies by client. In some cases it is every 10 samples and some cases by batch. The method requires that it be run at least once capping samples. The CRDLA need not be bracketed by a CCV/CCB to be considered valid in most	For method 6010C, must be within ± 30% For method 6010D, must be within .± 20%	If the CRDLA fails, it may be repeated once. If it fails again, samples bracketed by a failing CRDLA must be re-analyzed with the following exception. If the samples bracketed are non-detect and the CRDLA is biased high, data may be reported as there is no impact from the high bias. If the samples associated are non-detect and the only detections are associated with the batch QC (LCS/LCSD/MS/MSD) but the QC is within limits, the data can be reported. The QC should be flagged

	cases. In some instances the CRDLA must be bracketed by a valid CCV/CCB depending on the QAPP. It is best to bracket the CRDLA.		indicating that there was bias but that there was no impact to the associated samples.
Initial Calibration Blank (ICB)	An ICB must be analyzed immediately following an ICV for each element of interest.	All elements of interest must be evaluated to a criteria of < ½ the absolute value of the RL for method 6010D. All elements of interest must be evaluated to absolute value of the RL for method 6010B,6010C and 200.7. Criteria to be evaluated to method criteria unless otherwise specified by client	If the ICB fails, re-pour the sample and analyze a second time. If the ICB is still out of control, re-calibrate.
Continuing Calibration Blank (CCB)	A CCB must be analyzed, for each element of interest after every CCV.	All elements of interest must be evaluated to a criteria of < than the absolute value of the RL for 200.7, 6010B, and 6010C.. Depending on the data quality objective of individual client's different criteria may apply. For example data may need to be evaluated to ½ the RL or to the MDL. For 6010D, data must be evaluated to an absolute value of ½ the RL	If the absolute value of an analyte of interest is greater than the RL (or 1/2 RL, if specified), the CCB may be repeated once. If it fails again, all samples bracketed by the failed CCB must be re-analyzed with the following exception. If the sample concentration is greater than 10 times the CCB concentration, or the result is non-detect, the result may be reported. If associated projects are evaluated to the method detection limits per data quality objectives, the detections must be evaluated for data impact and the system evaluated for necessary corrective actions.
Spectral Interference Check Solutions (SIC)	SIC solutions are single-element solutions used to evaluate and correct IEC factors. Specific elements evaluated are listed in specific instrument methods.	SIC absolute value must be less than RL.	If SIC fails, re-calculate IEC and re-process data. If sample level exceeds an SIC level and the interfering element affects target analytes, then: a) run a higher SIC or b) dilute the sample.
Interelement Correction Standard A (ICSA)	A solution containing high concentrations of Al, Ca Fe and Mg is analyzed at the beginning of each sample run sequence.	Acceptance criteria for the spiked interferent elements are ± 20% and ± the	If the initial ICSA fails criteria, it may be re-analyzed once. Also, the ICSA can be re-processed after

	In some specific client requirements the ICSA must bracket the run or the analytical batch.	RL for target analytes.	appropriate SIC solutions are analyzed and the IECs are recalculated. If ICSA passes, continue. If a bracketing ICSA fails, it may be repeated once, and if it fails again, all affected samples must be re-analyzed.
Interelement Correction Standard AB (ICSAB)	A solution containing high concentrations of Al, Ca, Fe and Mg and low to mid-range concentrations of target analytes as outlined in ILM5.3. This is analyzed following the ICSA when requested. This is required by certain clients. It is not a method requirement and need be analyzed only for clients specifying this in the QAPP	The acceptance criteria are ± 20% for all interferent elements and target elements and +/- the RL for non-spiked analytes.	If the initial ICSAB fails criteria, it may be re-analyzed once. Also, the ICSAB can be re-processed after appropriate SIC solutions are analyzed and the IECs are recalculated. If ICSAB passes, continue... If a bracketing ICSAB fails, it may be repeated once, and, if it fails again, all affected samples must be re-analyzed.

12. Procedure

12.1. Instrument Set up and Operation. Set up and calibrate the instrument per the manufacturer's instruction and individual training provided by senior staff. It is outside the scope of this SOP to provide detailed instruction on instrument and/or software operation. The following highlights common items that are critical to success. Method conditions are stored with each instrument run.

12.1.1. Perform maintenance as needed and record in the daily maintenance log.

12.1.1.1. Ensure rinse is above ¼ full.

12.1.1.2. Ensure waste is below ½ full. Back pressure can cause problems.

12.1.1.3. Clean nebulizer once per month or more if drifting / clogging occurs.

12.1.2. ICP Ignition / Warm-up.

12.1.2.1. Check the gas supply.

12.1.2.2. Confirm the water circulator/chiller is on.

12.1.2.3. Leave instrument on and software up when not in use. Turning things off and on are bad for the instruments.

12.1.2.4. A periodic re-boot of the PC is necessary and recommended at least once per month.

12.1.2.5. Adjust the pump-tubing in such a way to ensure proper flow prior to igniting the plasma.

12.1.2.5.1. Decrease flow to where flow of bubble actually stops or barely moves.

12.1.2.5.2. Turn knob 2 full turns.

12.1.2.6. Ignite plasma while tubing is in a rinse solution.

12.1.2.7. Allow plasma to warm up at least 30 minutes and preferably 60-90 minutes.

12.1.3. Use the warm up time to create the sequence and pour samples.

12.1.3.1. Use Horizon Uploader to copy labels into the sequence.

12.1.3.2. Label all sample tubes so that each sample can be uniquely identified on the rack.

12.1.3.2.1. Alternate red, blue, and black Sharpie so that batches can be readily identified.

12.1.3.3. If any samples in a batch need to be filtered because of suspended material, use an Environmental Express Filtermate. The Method Blank and LCS must also be filtered if any samples are. Record the ID of the Filtermates used.

12.1.4. Pour the standards and start the calibration of the instrument.

12.1.5. Set the system to either a) shut down once complete or b) leave plasma and pump on.

12.1.5.1. If option b is used it is critical that someone be present when run completes – be very careful using this option.

12.1.6. Monitor all initial QC checks. One re-analysis of QC checks is allowed. If initial QC fails twice, make instrument modifications and recalibrate. If checks pass criteria, continue with sample analysis.

12.1.6.1. If ICSA fails, analyze SIC 1-4 and re-process IECs based on the data gathered.

12.1.7. During the sample analysis or after the analysis is completed, transfer valid data into LIMS system using LIMS LINK.

12.1.7.1. Export data from instrument to CSV file.

12.1.7.2. Open LIMSLINK

12.1.7.3. Click open instrument, select CSV file from list, data will import

12.1.7.4. Highlight QC + samples, select "Get LIMS Info"

12.1.7.4.1. Run QC will prompt for Q-Batch # plus standard selection

12.1.7.4.2. Sample data will prompt for SD/PDS source sample.

12.1.7.5. Right click on samples to select/de-select elements

12.1.7.5.1. Be sure to make the appropriate selections in LIMSLNK rather than post-editing in EPIC. This provides for a much smoother experience and minimizes chance for error. If edits must be done in EPIC be sure to make edits prior to uploading new data from LIMSLINK, as this, again minimizes error due to confusion.

12.1.7.6. Highlight samples to upload and select "Export Run to Epic Pro".

12.1.7.7. When complete select "Excel bench sheet"

12.1.7.8. Save the Excel Bench sheet to the instrument folder marked "LIMSLINK RAW DATA and to the DATA REVIEW FOLDER (see below) Use convention of run date (e.g. 032917ICP5) Note discrepancies in the notes section of the run log (including dilutions, QC issues, re-runs, etc).

12.1.8. In LIMS system make final adjustments and add any required footnotes. Print validation lists and complete checklist. Turn data in for validation.

12.2. Documentation for Data Review /Daily File

12.2.1. Documentation is a mix of electronic and paper files. Key data must be stored electronically so that data review may be performed from any location. Some documents are stored in the physical daily folder and archived for easy reference.

12.2.2. Label a physical file with the date.

12.2.3. Record the file name, Q-Batch, and all prep batches on the folder for each run that day (example: 032917ICP5 and 032917ICP5B..

12.2.4. Store printed copies batch worklist reports, prep bench sheets, the original checklist, a printed copy of the IEC Form 10-IN generated from Gandolf, and a printed copy of the run log from LIMSLINK file in this folder. If the data reviewer requests additional printed information place it in this folder as well.

12.2.5. On the G: drive created a new folder for the run labeled using the run name. This folder is created under G:\METALS\Instrument Data\10ICP5\DATA REVIEW\032917ICP5. Substitute ICP4 when using ICP4 and use the file name current to the data being used.

12.2.6. In this folder store electronic copies of the LIMSLINK Raw Data File, Validation Reports from LIMS (labeled using the LIMS Batch # first), scanned copies of the completed checklists (labeled using the LIMS Batch # first), the Instrument QC Report, the IEC Form 10-IN, and a copy of the raw data that is the same as that stored on the X: Drive.

12.2.7. For data validated off-site the electronic copy is signed and data validated in LIMS. The original copies of the checklists are signed off by the data review chemist the next time he or she is in the office and archived in the physical folder.

12.2.8. Label the daily file folder with date, Q-Batch, and instrument information.

12.3. Calibration Standards. There may be some exceptions due to client specific requests for non-routine analysis; however, for most samples the following calibration standards apply.

12.3.1. Calibration levels are set at the levels denoted in attachments II (calibration) and III (calibration verification).

12.3.2. CRDLA is prepared at the RL/LOQ listed in attachment I.

12.3.3. ICSA and ICSAB levels are noted in attachments IV and V and follow the definitions given in ILM05.3.

12.3.4. The acid matrix is typically 4% nitric acid and 5% hydrochloric acid.

13. Quality Control

13.1. Table 13.1 – Quality Control

QC Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	DI water for liquid samples Resin beads for solid samples	Prepared and analyzed with each group of samples digested. Carried through the appropriate steps of the analytical process. These steps may include, but are not limited to, prefiltering, digestion, dilution, filtering and analysis.	All elements of interest must be evaluated to a criteria of the absolute value being < ½ the RL for method 6010D. All elements of interest must be evaluated to a critera of the absolute value being< the RL for method 6010,6010B,6010C and 200.7 If the method blank does not contain target analytes at a level that interferes with project-specific DQOs, then the method blank would be considered acceptable.	If MB fails, one reanalysis allowed. If it fails again, affected samples should be re-prepared and re-analyzed with following exceptions: If sample ND, report sample without qualification; If sample result >10x MB detects, report the data as it is not impacted by the blank detections; If sample result <10x MB detects and cannot be reprepared/reanalyzed, report sample with appropriate B-flag qualifier to indicate an estimated value. Client must be alerted and authorize this condition.
Laboratory Control Sample (LCS)	DI water for liquids and resin beads for solids, spiked with analytes of interest at same	Prepared and analyzed for every batch of 20 or less samples digested	80-120% for 6010B,6010C and 6010D 85-115% for 200.7	If the percent recovery for the LCS falls outside the control limits of 80-120% for 6010B, 6010C,and 6010Dor 85-115% for 200.7, one reanalysis of the LCS is allowed. . If reanalysis of the LCS

	level as MS/MSD			fails, all samples affected by the failing LCS elements need to be re-digested and re-analyzed. ***EXCEPTION:*** if LCS fails high and samples are ND, the data may be reported with appropriate qualification.
Matrix Spike (MS) / Matrix Spike Duplicate (MSD)	The spike is added to a well-mixed aliquot of a selected sample before the digestion (i.e., prior to the addition of other reagents).	One MS/MSD per batch. If batch consists of more than10 samples for 200.7, an additional MS is required. Clients may have requirements that create a higher frequency of MS/MSD samples.	75-125% for 6010B, 6010C, and 6010D 70-130% for 200.7 % RPD: 20%	If the percent recovery for the MS and MSD fall outside the control limits, the results are flagged that they are outside acceptance criteria along with the parent sample. If the RPD exceeds the acceptance criteria, the MSD sample and associated parent sample need to be flagged. If MS or MSD fails and spike amount is less than 4 times the native concentration in the sample, remove M1 flag and replace with P6 flag. For Minnesota Admin Contract clients – all MS/MSD failures require reanalysis of the MS/MSD and the original sample. If it is still out of control, investigate and document the cause in the associated narrative as well as qualifying appropriately.
Post Digestion Spike (PDS)	Spike is added to the native QC sample at the same concentration as the MS but at the instrument.	Method suggestion / Pace policy if reporting by 6010B, 6010C, 6010D and MS/MSD fail outside 75-125%	75-125% for 6010B and 80-120% for 6010C.	Data is provided to data package clients for their evaluation
Internal Standard	The same concentration should be used for standards and samples throughout the entire analytical run.	Introduced automatically with every sample.	70-130% of its true concentration	If the recovery is outside the criteria, sample is reanalyzed at a 5X dilution. If it fails at a 5X dilution, higher dilutions are made until result is within specification.
Serial Dilution	A 1:5 dilution of the sample used for the QC. This is performed at the bench.	One SD per batch. Method suggestion / Pace Policy, if reporting by 6010B, 6010C, or 6010D.	6010B/C: SD should agree within +/- 10% of the original result when the original sample is greater than 10x the RL. The SD test is not applicable to sample concentrations < 10x the RL. 6010D: 1:5 Dilution of MS, or concentrations 25x > LLOQ in parent sample,	Data is provided to data package clients for their evaluation.

			results within +/- 20%	
Laboratory Filter Blank (FB)	A filtered aliquot of reagent water treated and prepared exactly as all samples when lab filtration is requested.	Analyzed only with batches of lab filtered dissolved metals, one per batch of 20 or less.	All elements of interest must be evaluated to a criteria of the absolute value of the result being < ½ the RL for method 6010D. All elements of interest must be evaluated to a criteria of the absolute value of the result being < the RL for method 60106010B,6010C and 200.7 . . If the FBlank does not contain target analytes at a level that interferes with project-specific DQOs, then the FB would be considered acceptable.	If FB fails, one reanalysis allowed. If it fails again, affected samples should be re-analyzed with following exceptions: If sample ND, report sample without qualification; If sample result >10x FB detects, report sample as not impacted by the blank contamination; If sample result <10x FB detects and sample cannot be reanalyzed, report sample with appropriate qualifier to indicate an estimated value. Client must be alerted and authorize this condition.

14. Data Analysis and Calculations

14.1. Inter-element Correction Factor (IEC) = Concentration of apparent concentration (observed) in mg/L / Concentration of Interferent in mg/L.

14.2. The percent recovery of the spike is calculated from the following equation:

$$\% \text{ Recovery} = \frac{(SSR-SR) \times 100}{ST}$$

Where: SSR = Spiked Sample Result, ug/L or mg/kg dry
SR = Sample Result, ug/L or mg/kg dry
ST = Spike Target, ug/L or mg/kg dry

14.3. The relative percent difference between the MS/MSD can be calculated as follows:

$$RPD = \frac{|(S-D)| \times (100)}{(S+D)/2}$$

Where: RPD = Relative Percent Difference
S = Original Spiked Sample Value, ug/L or mg/kg dry
D = Second Spiked Sample Value, ug/L or mg/kg dry

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. See tables in section 11 and 13.

16. Corrective Actions for Out-of-Control Data

16.1. See tables in section 11 and 13.

17. Contingencies for Handling Out-of-Control or Unacceptable Data

17.1. If not specifically listed in the tables in section 11 or 13, the contingencies are as follows. If there is no additional sample volume to perform re-analyses, all data will be reported as final with applicable qualifiers. If necessary, an official case narrative will be prepared by the Quality Manager or Project Manager.

18. Method Performance

18.1. All applicable personnel must read and understand this SOP with documentation of SOP review maintained in their training files.

18.2. **Method Detection Limit (MDL) Study**: An MDL study must be conducted annually (per the method) per S-MN-Q-269 – Determination of Limit of Detection and Limit of Quantitation (or equivalent replacement) for each matrix per instrument.

18.3. **Instrument Detection Limit (IDL) Study**: An IDL study must be conducted quarterly per S-MN-Q-269 – Determination of Limit of Detection and Limit of Quantitation (or equivalent replacement).

18.4. **Demonstration of Capability (DOC)**: Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC) per S-ALL-Q-020 - Training Procedures (or equivalent replacement).

18.5. **Periodic performance evaluation (PE)** samples are analyzed to demonstrate continuing competence per SOP S-MN-Q-258 – Proficiency Testing Program (or equivalent replacement). Results are stored in the QA office.

18.6. **Linear Dynamic Range (LDR) study:** The upper limit of the LDR must be established annually for each instrument and each wavelength utilized. The LDR is determined by analyzing progressively higher standard concentrations of the analyte until the observed analyte concentration remains within +/- 10% of the known concentration of the study. Method 6010D requires that a LDR check sample be analyzed daily prior to any samples. Data is reported up to 90% of the LDR. When evaluating interferences use values up to the full LDR for the interferent.

19. Method Modifications

19.1. There is considerable variability and confusion in the reference methods concerning blank evaluation. In order to provide consistency, blanks are evaluated as the absolute value compared to either the reporting limit (RL), ½ the RL, or to the method detection limit depending on the method or client QAPP. Data is rejected and re-prepared/re-analyzed based on either the RL or ½ the RL only. If data is being evaluated to the MDL, data is not rejected based on the MDL but rather on the RL or ½ the RL. However, data is qualified with a B-Flag if the absolute value is greater than the MDL in the case where data is evaluated to the MDL and passed criteria for the RL or 1/2 RL.

20. Instrument/Equipment Maintenance

20.1. All maintenance activities are listed daily in maintenance logs that are assigned to each separate instrument.

21. Troubleshooting

21.1. Not applicable for this SOP.

22. Safety

22.1. **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. **Samples**: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in S-MN-S-003 - Waste Handling and Management (or equivalent replacement).

23.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (e.g., before a reagent expires).

24. Pollution Prevention

24.1. The company wide Chemical Hygiene and Safety Manual contains information on pollution prevention.

25. References

25.1. Pace Quality Assurance Manual- most current version.

25.2. National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, "Quality Systems"- most current version.

25.3. The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems"- most current version.

25.4. Test Methods for Evaluating Water and Solid Waste, SW-846 3rd Edition, Final Update III, Method 6010B.

25.5. Test Methods for Evaluating Water and Solid Waste, SW-846, Method 6010C Update IV, Feb. 2007.

25.6. Test Methods for Evaluating Water and Solid Waste, SW-846, Method 6010D Update V, July 2014.

25.7. Method 200.7 Revision 4.4 , Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-atomic Emission Spectrometry.

25.8. US EPA Contract Laboratory Program Statement of Work ILM05.3, March 2004.

26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. Attachment I - Target Analyte List and Reporting Limits (PRL)

26.2. Attachment II - ICP Working Calibration Standard

26.3. Attachment III - ICP Calibration Verification Standard

26.4. Attachment IV - ICSA

26.5. Attachment V - ICSAB

26.6. Attachment VI - Sample Run Sequence

26.7. Attachment VII – Wisconsin Procedures for 3020A/3050B

26.8. Attachment VIII – ICP Linear Ranges

27. Revisions

Document Number	Reason for Change	Date
S-MN-I-313-rev.30	Updated to LLC throughout document Removed uncontrolled Updated attachments II/III/IV/V/VII/VIII Updated sections 1/2/3/4/5/12/14/18/19/25 & Tables 7.1/9.1/10.1/11.1/13.1 Removed form F-MN-I-412-rev.01	14Apr2017

ATTACHMENT I – Target Analyte List and Reporting Limits (PRL)

Element	Water PRL (ug/L)	Soil PRL (mg/kg)
Aluminum	200	10
Antimony	20	1.0
Arsenic	20	1.0
Barium	10	0.50
Beryllium	5.0	0.25
Boron	150	7.5
Cadmium	3.0	0.15
Calcium	500	25
Chromium	10	0.50
Cobalt	10	0.50
Copper	10	0.50
Iron	50	2.5
Lead	10	0.5
Magnesium	500	25
Manganese	5.0	0.25
Molybdenum	15	0.75
Nickel	20	1.0
Potassium	2500	125
Selenium	20	1.0
Silver	10	0.50
Sodium	1000	50
Sulfur	500	25
Thallium	20	1.0
Tin	75	3.75
Titanium	25	1.25
Vanadium	15	0.75
Zinc	20	1.0
Hardness	3300	N/A

ATTACHMENT II –ICP Working Calibration Standard

Element	Stock Conc. (mg/L)	Aliquot (mL)	Final Volume (mL)	Cal STD Final Conc. (mg/L)
Ag	100	1.0	50	2
Al	2,000	0.5	50	20
As	200	1.0	50	4
Ba	200	1.0	50	4
Be	200	1.0	50	4
Ca	2000	0.5	50	20
Cd	200	1.0	50	4
Co	200	1.0	50	4
Cr	200	1.0	50	4
Cu	200	1.0	50	4
Fe	2000	0.5	50	20
K	2000	0.5	50	20
Mg	2000	0.5	50	20
Mn	200	1.0	50	4
Na	2000	0.5	50	20
Ni	200	1.0	50	4
Pb	200	1.0	50	4
S	10000	0.1	50	20
Sb	200	1.0	50	4
Se	200	1.0	50	4
Tl	200	1.0	50	4
V	200	1.0	50	4
Zn	200	1.0	50	4
Mo	200	1.0	50	4
B	200	1.0	50	4
Sn	200	1.0	50	4
Ti	200	1.0	50	4
Si	1000	1	50	20
Li	200	1	50	4
P	200	1	50	4
Sr	200	1	50	4

ATTACHMENT III –ICP Calibration Verification Standard

Element	Stock Conc. (mg/L)	Aliquot in (mL)	Final Volume (mL)	Final Conc. (mg/L)
Ag	50	1.0	50	1
Al	1000	0.5	50	10
As	100	1.0	50	2
Ba	100	1.0	50	2
Be	100	1.0	50	2
Ca	1000	0.5	50	10
Cd	100	1.0	50	2
Co	100	1.0	50	2
Cr	100	1.0	50	2
Cu	100	1.0	50	2
Fe	1000	0.5	50	10
K	1000	0.5	50	10
Mg	1000	0.5	50	10
Mn	100	1.0	50	2
Na	1000	0.5	50	10
Ni	100	1.0	50	2
Pb	100	1.0	50	2
S	10000	0.05	50	10
Sb	100	1.0	50	2
Se	100	1.0	50	2
Tl	100	1.0	50	2
V	100	1.0	50	2
Zn	100	1.0	50	2
Mo	100	1.0	50	2
B	100	1.0	50	2
Sn	100	1.0	50	2
Ti	100	1.0	50	2
Si	500	1	50	10
Li	100	1	50	2
P	100	1	50	2
Sr	100	1	50	2

ATTACHMENT IV – ICSA

Table 1

Source: CLP SOW ILM 5.3 for 200.7, 6010B, 6010C

Element	Stock Conc. (mg/L)	Aliquot in (mL)	Final Volume (mL)	Final Conc. (ug/L)
Al	5000	5	100	250000
Ca	5000	5	100	250000
Fe	2000	5	100	100000
Mg	5000	5	100	250000

Table 2

For 6010D, may be used with 200.7, 6010B, 6010C

Element	Stock Conc. (mg/L)	Aliquot in (mL)	Final Volume (mL)	Final Conc. (ug/L)
Al	5000	10	100	500000
Ca	5000	10	100	500000
Fe	2000	10	100	200000
Mg	5000	10	100	500000

ATTACHMENT V – ICSAB

Source: CLP SOW ILM 5.3 for 200.7, 6010B, 6010C

Element	Stock Conc. (mg/L)	Aliquot in (mL)	Final Volume (mL)	Final Conc. (ug/L)
Ag	20	1.0	100	200
Al	5000	5.0	100	250000
As	10	1.0	100	100
Ba	50	1.0	100	500
Be	50	1.0	100	500
Ca	5000	5.0	100	250000
Cd	100	1.0	100	1000
Co	50	1.0	100	500
Cr	50	1.0	100	500
Cu	50	1.0	100	500
Fe	2000	5.0	100	100000
Mg	5000	5.0	100	250000
Mn	50	1.0	100	500
Ni	100	1.0	100	1000
Pb	5	1.0	100	50
Sb	60	1.0	100	600
Se	5	1.0	100	50
Tl	10	1.0	100	100
V	50	1.0	100	500
Zn	100	1.0	100	1000

For 6010D, substitute values for Al, Ca, Fe, and Mg from Attachment IV, Table 2 above. This may also be used with 200.7, 6010B, and 6010C.

ATTACHMENT VI – Sample Run Sequence

1. **CAL0**
2. **CAL1**
3. **ICV**
4. **ICB**
5. **CRDLA**
6. **ICSA**
7. **ICSAB**
8. **SIC-1 Fe**
9. **SIC-2 Ca**
10. **SIC-3 Al**
11. **SIC-4 Mg**
12. **CCV**
13. **CCB**
14. **SAMPLE 1**
15. **SAMPLE 2**
16. **SAMPLE 3**
17. **SAMPLE 4**
18. **SAMPLE 5**
19. **SAMPLE 6**
20. **SAMPLE 7**
21. **SAMPLE 8**
22. **SAMPLE 9**
23. **SAMPLE 10**
24. **CCV**
25. **CCB**
26. **SAMPLE 11**
27. **SAMPLE 12**
28. **SAMPLE 13**
29. **SAMPLE 14**
30. **SAMPLE 15**
31. **SAMPLE 16**
32. **SAMPLE 17**
33. **SAMPLE 18**
34. **SAMPLE 19**
35. **SAMPLE 20**
36. **CCV**
37. **CCB**
38. **CRDLA**
39. **CCV**
40. **CCB**

ATTACHMENT VII – Procedure for Wisconsin Samples 3020A/3050B

	Document Name: Procedure for Wisconsin Samples – 3010A/3020A/3050B	Document Revised: 03Feb2016 Page 1 of 1
Pace Analytical	Document No.: F-MN-I-411-Rev.02	Issuing Authority: Pace Minnesota Quality Office

For Wisconsin solid samples only

ICPMS/ICP Metals (1.0-1.5 grams sample)

1. Add at least 5mL of conc. HNO3 (or 10mL 1:1) to the samples
2. Heat at 95 'C for at least 10 minutes, covered with reflux cap for refluxing
3. Add at least 5mL conc. HNO3
4. Heat at 95 'C for at least 30 min, covered with reflux cap for refluxing
5. Check for brown fumes
    a. If no brown fumes - continue to step 6
    b. If brown fumes, add at least 5mL conc. HNO3
    c. Heat
    d. If no brown fumes - continue to step 6
    e. If brown fumes, add more conc. HNO3 and heat
    f. Continue step e until brown fumes no longer exist
6. Heat for at least 2 hrs, covered with reflux cap for concentrating
7. Add 2mL H2O and 3mL of 30% H2O2
8. Heat to 95 'C and add 1mL increments of H2O2 until effervescence subsides, covered with reflux cap for refluxing
9. Heat for at least 2 hrs, covered with reflux cap for concentrating
10. Add at least 10mL of conc. HCl, heat to 95 'C for at least 15 min, covered with reflux cap for refluxing.
11. Dilute to 50mL
12. Match standards to final acid concentrations

Note: Method 3050B section 4.2 states: "Vapor recovery device (e.g., ribbed watch glasses, appropriate refluxing device, appropriate solvent handling system) We have opted to use a reflux cap as the appropriate refluxing device as stated rather than the ribbed watch glass.

For Wisconsin water samples only

ICPM/ICP Metals (50mL sample)

Transfer 50mL of well-mixed sample into a labeled digestion tube.

Add 1.5mL concentrated nitric acid to each digestion tube. Place the tubes into the block digester which has been preheated to achieve a temperature of 95°C (+/- 3°C) in the digestion tubes and cover with ribbed watch glass.

Evaporate without boiling to <10mL. Do not allow samples to go dry.

If digestate is generating brown fumes, add another 2.5mL concentrated nitric acid and reflux gently. Continue heating and adding acid as necessary, until the digestion is complete, generally indicated when the digestate is light in color and brown fumes are no longer generated.

Evaporate without boiling to approximately 5mL. Do not allow samples to go dry.

Cool the samples then add 2mL concentrated hydrochloric acid, return the samples to the hot block and heat for 15 minutes to dissolve any precipitate then allow samples to cool.

Dilute the digestates to 50mL in the digestion tube with reagent water. If necessary, filter the digestates to remove particulates using a plunger filter. If any sample digestates in a batch are filtered, the Method Blank and LCS must also be filtered.

Attachment VIII – ICP Linear Ranges

10ICP5		10ICP4	
Wavelength	**LDR (PPM)**	**Wavelength**	**LDR (PPM)**
Ag 328	5	Ag 328	2.5
Al 237	1500	Al 308	500
As 188	50	As 188	50
B 249	50	B 249	50
Ba 455	30	Ba 585	50
Ba 493*	30		
Be 234	20	Be 234	30
Ca 370	3000	Ca 370	3000
Cd 228	30	Cd 228	50
Co 228	200	Co 228	200
Cr 267	50	Cr 267	50
Cu 327	50	Cu 327	50
Fe 261	200	Fe 261	200
Fe 273*	3000	Fe 273	3000
K 766	500	K 766	50
Li 610	10	Li 610	20
Mg 383	1500	Mg 383	1000
Mn 257	30	Mn 257	30
Mn 293*	200	Mn 293	50
Mo 204	50	Mo 204	50
Na 589	500	Na 589	50
Ni 231	50	Ni 231	50
P 213	50	P 213	50
Pb 220	200	Pb 220	200
S 181	300	S 181	300
Sb 206	50	Sb 206	50
Se 196	50	Se 196	50
Si 251	200	Si 251	200
Sn 189	50	Sn 189	50
Sr 421	5	Sr 421	5
Ti 334	50	Ti 334	50
Tl 190	50	Tl 190	50
V 292	50	V 292	50
Zn 206	80	Zn 206	100

***Used for Interference Correction Only**

PLACEHOLDER PAGE FOR S-MN-I-359-Rev 26 SOP

Pace Analytical Services, LLC
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Minneapolis, MN 55414

Phone: 612.607.1700
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STANDARD OPERATING PROCEDURE

MERCURY IN LIQUID AND SOLID/SEMI-SOLID WASTE

Reference Methods: EPA SW-846 7470A/7471/7471B and EPA 245.1

Local SOP Number:	S-MN-I-359-Rev.27
Effective Date:	Date of Final Signature
Supersedes:	S-MN-I-359-Rev.26

APPROVALS

Laboratory General Manager — Date: 01 Mar 2018

Laboratory Quality Manager — Date: 26 Feb 2018

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date

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TABLE OF CONTENTS

SECTION **PAGE**

1. Purpose/Identification of Method

1.1. The Standard Operating Procedure provides a detailed description, based on method SW-846 7470/7470A/7471/7471B and EPA 245.1, of sample preparation and analysis for determining the concentration of mercury in liquid and solid/semi-solid environmental samples using manual cold vapor atomic absorption (CVAA).

2. Summary of Method

2.1. The method, a cold-vapor atomic absorption technique, is based on the absorption of radiation at the characteristic wavelength of 253.7 nm by mercury vapor. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance is measured as a function of mercury concentration.

2.2. Chemical Reactions - Organic mercury compounds are decomposed by digestion with potassium permanganate in acid solution. The mercuric ions are then reduced to the elemental state with stannous chloride and mercury vapor is produced.

3. Scope and Application

3.1. Personnel: The policies and procedure contained in this SOP are applicable to all personnel involved in the analytical method or non-analytical process.

3.2. Parameters: This procedure is applicable for determining the concentration of mercury in mobility procedure extracts, aqueous wastes, ground waters, soils, sediments, bottom deposits, and sludge-type materials.

4. Applicable Matrices

4.1. This SOP is applicable to liquid and solid/semi-solid matrices.

5. Limits of Detection and Quantitation

5.1. The default reporting limit (RL) or Limit of Quantitation (LOQ) for mercury in liquid is 0.2 µg/L. The default reporting limit for mercury in soil is 0.02 mg/kg. Reporting limits may vary based on the nature of the individual sample matrix. All current RLs and MDLs (Method Detection Limit) are listed in the LIMS and are available by request from the Quality Manager. For certain clients we use a separate instrument method optimized for sensitivity in which the reporting limit is 0.010 µg/L. This is for aqueous samples only.

6. Interferences

6.1. Potassium permanganate is added during digestion of samples to break down organo-mercury compounds which would otherwise not respond to the cold vapor technique. A heating step is required for methyl mercuric chloride when present in or spiked to a natural system. Possible sulfide interferences are also eliminated by the addition of potassium permanganate. EPA studies indicate concentrations as high as 20 mg/L of sodium sulfide do not interfere with the recovery of added inorganic mercury from distilled water.

6.2. Copper has also been reported to interfere; however, EPA studies indicate copper concentrations as high as 10 mg/L had no effect on recovery of mercury from reagent water.

6.3. Sea waters, brines and industrial effluents high in chlorides require additional permanganate. During the oxidation step, chlorides are converted to free chlorine which will also absorb radiation of 253 nm.

Care must be taken to assure that free chlorine is absent before the mercury is reduced and swept into the cell. The design of the dedicated mercury analyzer assures that this does not occur.

7. Sample Collection, Preservation, Shipment and Storage

7.1. Sample Collection Table

Sample Type	Collection per Sample	Preservation	Storage	Hold Time
Liquid	Samples should be collected in plastic or glass containers	Acidified with nitric acid to a pH<2 at the time of collection	Room temperature	Max hold time is 28 days
Solid	Glass jars	n/a	Above freezing but below 6°C until sample preparation	Max hold time is 28 days

8. Definitions

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary Section.

9. Equipment and Supplies (Including Computer Hardware and Software)

9.1. Equipment and Supplies Table

Supply	Description	Vendor/Item #/Description
Mercury analyzer, computer controlled	Cold Vapor Atomic Adsorption	Cetac M-7500 or M-7600 or equivalent
Autosampler	Cetac or compatible with analyzer	Cetac ASX 520 or equivalent
Peristaltic pump tubing	Various sizes	Fisher Scientific or equivalent
Argon gas supply	high-purity grade, 99.99%	House argon
Metals-free digestion cups	50 mL	Environmental Express UC-475 NL or equivalent
Hot Block digester	54 place block or equivalent	Environmental Express SC154 or equivalent
Volumetric pipettes	various sizes, class A	Fisher Scientific or equivalent
Volumetric flasks	various sizes, class A	Fisher Scientific or equivalent
Analytical balance	accurate to at least 20 mg	Ohaus Explorer Pro
Analytical balance	accurate to at least 200 mg	A&D EK-610i
Mechanical pipettes	various sizes and metals free disposable tips	Fisher Scientific or equivalent
Resin Pellets	Metals free, solid matrix	Environmental Express SC400 or equivalent
Cetac Quicktrace Software	Data acquisition software	See master list for current version
LIMSLINK Software	Interface between Quicktrace and Horizon Epic	See master list for current version
Horizon Epic Pro	Database and Reporting Software	See master list for current version

10. Reagents and Standards

10.1. Reagents and Standards

Reagent/Standard	Concentration/ Description	Requirements/ Vendor/ Item #
Reagent water	Deionized water	Interference-free and analyte free (ASTM Type II)
Nitric acid	Concentrated, trace metal grade	Store at room temperature. Expires as specified by manufacturer. Fisher Scientific P/N A509-P212 or equivalent
Sulfuric acid	Concentrated, trace metal grade	Store at room temperature. Expires as specified by manufacturer. Fisher Scientific P/N A510-P212 or equivalent
Hydrochloric acid	Concentrated, trace metal grade	Store at room temperature. Expires as specified by manufacturer. Fisher Scientific P/N A508-P212 or equivalent
Potassium permanganate solution	Dissolve 50 g potassium permanganate in a minimum volume of water and dilute to 1000 mL with reagent water.	Store the reagent at room temperature in either a plastic or glass container. This solution expires 3 months from preparation date. Fisher Scientific brand reagents or equivalent.
Sodium chloride-Hydroxylamine hydrochloride solution	Dissolve 120 g sodium chloride and 120 g hydroxylamine hydrochloride in reagent water and dilute to 1000 mL with reagent water.	Store the standard at room temperature in either a plastic or glass container. Solution expires 1 month from preparation date. Fisher Scientific brand reagents or equivalent.
Potassium persulfate solution (5%)	Dissolve 100 g of potassium persulfate in reagent grade water and dilute to 2000 mL.	This solution expires 3 months from the preparation date. Fisher Scientific brand reagents or equivalent.
Rinse solution	Add 48mL concentrated hydrochloric acid to 800mL water, add 24 mL concentrated nitric acid and dilute to 1 liter with reagent water.	Store in 5L Nalgene container at room temperature. The solution expires 1 week from preparation date.
Stannous Chloride	Add 70 mL concentrated hydrochloric acid and 100 grams SNCl2-2H20 to 1000 mL DI water.	Different amounts may be made based on need. Store in bottle marked “Stannous Chloride” at the instrument. Fisher Scientific brand reagents or equivalent.
Aqua Regia	Mix 3 parts concentrated hydrochloric acid with 1 part concentrated nitric acid	Use fresh daily, expires within 24 hours
Mercury Calibration Stock Solution	1000 mg/mL, NIST traceable standard	Store at room temperature. Expires as specified by manufacturer. Inorganic Ventures or equivalent.
ICV/CCV Mercury Stock Solution	1ug/mL, NIST traceable standard	Must be from a separate source than the mercury calibration stock source. Spex-Certiprep or equivalent.
Low Level Mercury Calibration Stock Solution	10 mg/L, NIST traceable standard	Store at room temperature. Expires as specified by manufacturer. Inorganic Ventures or equivalent.
Low Level ICV/CCV Mercury Stock Solution	10 mg/L, NIST traceable standard	Must be from a separate source than the mercury calibration stock source. Inorganic Ventures or equivalent.

10.2. Working Standard Dilutions and Concentrations

Standard	Standard(s) Used	Standard(s) Amount (mL)	Solvent	Solvent Volume (mL)	Final Total Volume (mL)	Final Concentration (µg/L)
Mercury Calibration Intermediate.	Mercury Stock (10 ug/mL)	5 mL	Reagent water	985 mL	1000 mL	50µg/L
	concentrated nitric acid	10 mL				
Standard 1	Intermediate standard (50 µg/L)	0.12	Reagent water	29.88	30 mL	0.2
Standard 2		0.6		29.4		1.0
Standard 3		1.8		28.2		3.0
Standard 4		3.0		27		5.0
Standard 5		6.0		24		10
CRDL		0.12		29.88		0.2
ICV/CCV	Mercury Stock 1000 mg/mL	0.15	Reagent water	29.85	30	5.0
Low Level Mercury Calibration Intermediate Standard Prepare every 6 months	Calibration Mercury Stock (10 mg/L)	0.100 mL	Reagent water	984.9 mL	1000 mL	1.0 µg/L
	Concentrated nitric acid	5.0 mL				
	Concentrated hydrochloric acid	10 mL				
Standard 1	Intermediate Standard (1.0 µg/L)	0.30	Reagent Water	29.7	30 mL	0.01
Standard 2		0.75		29.25		0.025
Standard 3		1.5		28.5		0.050
Standard 4		3.0		27		0.100
Standard 5		6.0		24		0.200
CRDL		0.30		29.7		0.01
Low Level Mercury ICV/CCV **Intermediate** Standard. Prepare every 6 months	ICV/CCV Mercury Stock (10 mg/L)	0.4 mL	Reagent water	184.6 mL	200 mL	20 µg/L
	Concentrated nitric acid	5.0 mL				
	Concentrated hydrochloric acid	10 mL				
Low Level Mercury ICV/CCV	Low Level Mercury ICV/CCV Intermediate (75 µg/L)	0.15	Reagent water	29.85	30 mL	0.10 µg/L

10.2.1. Mercury Calibration Intermediate Standard to be prepared every 6 months or as needed. Mercury Calibration Standards: Prepare dilutions of the intermediate standard solution to be used as calibration standards at the time of analysis. The calibration standards are prepared using the same type of acid and reagents, at the same concentration range as the samples to be analyzed. SW-846 series methods for mercury require that calibration standards are processed like samples including heating while EPA 245.1 specifically prohibits the calibration standards from being heated. Daily calibration records are documented in the electronic preplog.

10.3. Note: when processing samples following sample preparation procedures described in S-MN-I-306 and S-MN-I-490 relating to fluorescent light bulb materials refer to the preparation of calibration and QC standards described there in.

11. Calibration and Standardization

11.1. Calibration and Standardization Table

Calibration Metric	Parameter / Frequency	Criteria	Comments
Calibration Curve Fit	Linear Regression	$r \geq 0.995$	If any of the criteria are not met, terminate analysis, fix the problem and recalibrate.
Initial Calibration Verification (ICV)	Immediately after each initial calibration	± 10% for SW-846 7000 series methods and ± 5% for 245.1 May be re-analyzed once if first analysis fails.	If criteria are not met, terminate analysis, fix the problem and recalibrate. The ICV solution must be obtained from a different source than the calibration standards.
Initial Calibration Blank (ICB)	Immediately following the ICV	Result must be less than the absolute value of the Reporting Limit (LOQ). May be reanalyzed once if first analysis fails. NC requires blanks to be clean to ½ RL.	Depending on the data quality objective of the associated projects, more stringent blank criteria may apply. If an analyte of interest is greater than the RL, the sample concentration must be greater than 10 times the blank concentration or the sample cannot be reported. If associated projects are evaluated to the method detection limits per data quality objects, the detections must be evaluated for data impact and the system evaluated for necessary corrective action.
Contract Required Detection Limit Sample (CRDL)	At the beginning of each run	± 30% (or specified by the client) May be reanalyzed once if first analysis fails.	The CRDL is at or below the RL. Depending on data quality objectives it may be required that a CRDL bracket samples.
Continuing Calibration Verification (CCV)	Prior to the analysis of any samples and after every 10 samples. Samples must be bracketed with a closing CCV standard.	CCV must be within ± 10% of the true value. May be reanalyzed once if first analysis fails.	If criteria are not met, any sample data the failing CCV brackets on either side may not be accepted. The ICV solution can be utilized as the CCV.
Continuing Calibration Blank (CCB)	Immediately following all CCVs	Result must be less than the absolute value of the Reporting Limit (LOQ).	Depending on the data quality objective of the associated projects, more stringent blank criteria may apply. If an analyte of interest is greater than the RL, the sample concentration must be

		May be reanalyzed once if first analysis fails. NC requires blanks to be clean to ½ RL.	greater than 10 times the blank concentration or the sample cannot be reported. If associated projects are evaluated to the method detection limits per data quality objects, the detections must be evaluated for data impact and the system evaluated for necessary corrective action.

11.2. Consult the instrument manufacturer's user manuals for specific operational instructions.

11.3. Instrument Calibration - Instrumental calibration is to be performed in accordance with the manufacturer's specifications

11.4. Instruments must be calibrated at a minimum of once every 24 hours or prior to use. The instrument standardization date and time must be included in the raw data.

11.5. Verify calibration by reviewing the curve fit, ICV, ICB, and CRDL before running any samples. Make sure all criteria are met before proceeding.

12. Procedure

12.1. Sample Preparation- Water samples: The following is an example of dilutions used to make standards and to obtain a final concentration for the solutions used. Other dilutions may be utilized as long as the final concentration is at the specified concentration. If the dilution is documented and the final concentration is the same, no further corrective action or explanation is necessary.

12.1.1. Prepare a method blank by transferring 30 mL of reagent grade water to a new 50 mL digestion cup. Label with the LIMS batch number and sample number.

12.1.2. Prepare a laboratory control sample (LCS) by transferring a 0.15 mL aliquot of the stock mercury standard to a 50 mL cup. For Low Level Mercury Samples, transfer 0.15 mL aliquot of the Low Level Mercury Intermediate standard. Bring the total volume to 30 mL with reagent water. Label with the LIMS batch number and sample number. See section 10.2.3 for high sensitivity method.

12.1.3. Shake sample to achieve homogeneity. Maximum sample volume is 30 mL. Use this or a smaller volume diluted to 30 mL. Place the sample into the 50 mL cup labeled with the corresponding LIMS sample number. Record sample volume in the Hg CVAA Sample Preparation Log.

12.1.4. Prepare an MS/MSD by transferring 0.15 mL aliquot of the stock mercury standard to 50 mL cups. For Low Level Mercury samples, transfer 0.15 mL aliquot of the Low Level Mercury Intermediate standard. Bring the total volume of each to 30 mL with sample. See section 10.2.3 for high sensitivity method.

12.1.5. To all samples (including QC) add 1.5mL concentrated sulfuric acid and 0.75mL concentrated nitric acid, mixing well after each addition.

12.1.6. To all samples (including QC) add 5mL potassium permanganate. If the purple color disappears, the sample is re-batched and re-prepped at a lower volume.

12.1.7. To all samples (including QC) add 2.5 mL of potassium persulfate solution and swirl to mix.

12.1.8. Loosely cap each cup with the green caps and place cups into the digestion block, maintained at a temperature of 95°C ± 2°C and heat for two hours. Measure the initial temperature and time in the block and record in the Hg CVAA Sample Preparation Log. Daily temperature recording, thermometer information, and well monitoring information documented in each Hot Block Monitoring Log, Documents.

12.1.9. After the two-hour digestion, remove the samples from the block and cool. Record the time the samples were removed from the block, as well as the final temperature of the block in the Hg CVAA Sample Preparation Log.

12.1.10. To all samples (including QC) add 1.8 mL of hydroxylamine hydrochloride to reduce the excess permanganate. The permanganate is reduced when the purple color dissipates. If the purple color does not dissipate, add additional hydroxylamine hydrochloride until the color dissipates. Note this on the preparation log and adjust in LIMS. For example: if an additional mL is needed, then add 1 mL to the final volume.

12.2. Sample Preparation- Solid/Semi-solid samples

12.2.1. Prepare a method blank by weighing 0.3 g of resin pellets (SC400) in a 50 mL cup.

12.2.2. Prepare a LCS by weighing 0.3 g of resin pellets (SC400) in a 50 mL cup and spiking with a 0.15 mL aliquot of the ICV/CCV working mercury standard.

12.2.3. Weigh a representative 0.3-0.36 g portion of sample in a 50 mL cup.

12.2.4. Weigh two additional samples for MS/MSD and spike carefully to get these samples as close to the weight of the unspiked sample used for QC, as possible. Spike both the MS and MSD with 0.15 mL of the mercury ICV/CCV working standard.

12.2.5. To all samples (including QC) add 3 mL DI water.

12.2.6. To all samples (including QC) add 3 mL aqua regia (see 10.1 above).

12.2.7. Place in hot block, maintained at 95°C ± 2°C, and heat for 2 minutes.

12.2.8. Remove from hot block and allow to cool.

12.2.9. Bring all samples (including QC) up to a volume of 30 mL with DI water.

12.2.10. To all samples (including QC) add 9 mL potassium permanganate. If the purple color disappears, re-prepare the sample, method blank, and laboratory Control Sample with less DI and the corresponding amount of potassium permanganate added so that final volume does not exceed 30mL. Additional permanganate is noted as a comment on the prep form.

12.2.11. Loosely cap each cup and place in Hot Block™ digester, maintained at a temperature of 95°C ± 2°C and heat for 30 minutes. Measure the initial temperature of the Hot Block and record that, as well as the time, in the Hg CVAA Preparation Logbook. Daily temperature recording, thermometer information, and well monitoring information documented in each Hot Block Monitoring Log, Documents.

12.2.12. Remove the samples from the block and allow to cool.. Record the time and the temperature that the samples are removed from the block on the HG CVAA Preparation Log.

12.2.13. To all samples (including QC) add 3.6 mL of hydroxylamine hydrochloride to reduce the excess permanganate. The permanganate is reduced when the purple color dissipates. If the purple color does not dissipate, add additional hydroxylamine hydrochloride until the color dissipates. Note this on the preparation log and adjust in LIMS. For example: if an additional mL is needed, then add 1 mL to the final volume.

12.3. Sample Preparation – ground glass and end caps from fluorescent light bulb recycling

12.3.1. Refer to SOP S-MN-I-306 for sample preparation of ground glass samples from fluorescent light bulbs.

12.3.2. Refer to SOP S-MN-I-490 for sample preparation of aluminum end cap samples from fluorescent light bulbs.

12.4. Instrument Operation

12.4.1. Turn on, or ‘wake up’ the instrument and allow the lamp to warm up for about 90 minutes from a cold shut down (lamp off, main power off and gas off) and 5 minutes from standby (lamp off, main power on and gas off).

12.4.2. Prepare any necessary reagents and record the appropriate information (volumes, manufacturer, lot numbers, etc.) in the standard solution log.

12.4.3. Check instrument waste, and empty as needed.

12.4.4. Perform any routine maintenance as needed and record in maintenance log

12.4.5. Check the $KMnO_4$ trap at the back of the instrument to make sure it is filled with crystalline $KMnO_4$, and not wet or spent (the brown MnO_2 color approaches the open end of the trap).

12.4.6. Fill the rinse solution container with rinse solution, if needed, and move the probe down into the rinse well.

12.4.7. Check peristaltic pump tubing installation, make sure tension is adjusted if needed, and turn pump on.

12.4.8. Place the $SnCl_2$ line in DI water.

12.4.9. Initialize the wetting of the GLS by selecting 'wet the gas liquid separator post' option in the software. This increases the gas flow to 300-350 mL/min and ramps the pump speed to 100%. Pinch the waste line tubing shut with your fingers. Watch the bubbles and ensure that 1-2 bubbles completely propels to the top of the chamber, wetting the entire post and the top. As soon as this happens, open the waste line tubing so the GLS can drain.

12.4.10. Inspect the GLS to make sure it is draining completely and liquid is not pooling.

12.4.11. Attach the sample gas line to the nafion dryer cartridge.

12.4.12. Fill the stannous chloride bottle with stannous chloride.

12.4.13. Place the $SnCl_2$ line into the $SnCl_2$ solution bottle.

12.4.14. Create a worksheet for analysis by selecting 'new from' in the file menu. Enter the name, ie 20Aug15 (DDMMMYY), a, b, c etc. (if more than one run is performed that day) soil or water to indicate sample matrix, and instrument ID number.. The program will then go to the Method Editor page.

12.4.14.1. In the conditions page in the Method Editor, check the instrument settings including the time profile (baseline correction and read time delays). To do this, read a standard and move the baseline correction window and read time window accordingly if needed.

12.4.14.2. Check the Standards page to ensure the correct calibration parameters and standards are entered.

12.4.14.3. Check the QC tests page to make sure the correct test solutions and parameters are entered if the software is to calculate recoveries during analysis.

12.4.15. Create a sequence in the sequence editor tab and enter sample IDs or import them from LimsLink.

12.4.16. Start analysis, monitor all initial QC checks. If initial QC fails, make adjustments if needed and recalibrate. If checks pass criteria, continue with sample analysis.

12.4.17. After analysis, print out a report and transfer valid data into LIMS system via LimsLink.

12.4.18. After completing sample analysis for the day, shut down the instrument.

12.4.18.1. Place the $SnCl_2$ line in 10% HNO_3 and run for ~10 minutes. After this move the probe up out of the rinse well and place the $SnCl_2$ line in DI water and run for 2-5 minutes. Remove from DI and allow the line to run dry. Turn off pump, disconnect the clamps, and loosen pump tubing.

12.4.18.2. Disconnect the sample gas line from the nafion dryer cartridge.

12.4.18.3. Turn off the gas and the lamp.

12.4.18.4. If the instrument will be used in the next day or two, leave it in the stand-by mode. If not, do a cold shut down and turn off the software, instrument, auto sampler and auto diluter.

13. Quality Control

13.1. Quality Control Table

QC Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Method Blank (MB)	For 7470A/7470/245.1: DI water For 7471/7471B: Resin Pellets (SC400) weighed to 0.3 g.	Prepared and analyzed with each batch (20 or less) of samples digested.	Less than the absolute value of the reporting limit. NC requires blanks to be clean to ½ RL.	Re-analyze associated samples. ***Exceptions:*** If sample ND, report sample without qualification; If sample result >10x MB detects, report the data as it is not impacted by the blank detections; If sample result <10x MB detects and cannot be reprepared/reanalyzed, report sample with appropriate qualifier to indicate an estimated value. Client must be alerted and authorize this condition.
Laboratory Control Sample (LCS) / Laboratory Control Sample Duplicate (LCSD)	For 7470A/7470/245.1: DI water spiked so that the final concentration of mercury is 5µg/L. For 7471/7471B: Resin Pellets weighed to 0.3 g spiked to give a final concentration of 0.5 mg/kg.	Prepared and analyzed with each batch (20 or less) of samples digested. A LCSD is sometimes required.	80-120% for 7470/7470A and 7471/7471B 85-115% for 245.1	The LCS and/or LCSD may be re-analyzed once. If the LCS is not within acceptance criteria, the batch must be re-prepared.
Matrix Spike (MS), Matrix Spike Duplicate (MSD)	Client samples spiked with the analytes of interest. Spiked with the exact same spiking solution and with the same spike amount as the LCS/LCSD above.	Prepared and analyzed with each batch (20 or less) of samples digested. For 245.1 if there are more than 11 samples then an additional MS is required. Clients may request that specific samples be used for the MS/MSD. These are designated	80-120% for 7470/7470A and 7471/74/1B 70-130% for 245.1 %RPD: 20%	Check for errors in calculations, sample and standards preparation and spiking procedure, or problems with the instrument performance. If the recovery is outside of the control limits, data is auto-flagged. Verify auto-flag is correct. If matrix interferences are suspected to be the cause of the noncompliant recovery, report the data and flag all affected project sample with a comment discussing the interference. The footnote only applies to samples within the same batch containing the sample used for the MS analysis. If the native sample concentration is greater than 4 times the spike concentration added, then the measurement is not applicable. Remove auto-flag and use appropriate qualifier on data. If the RPD is outside the limit, report the data and footnote the samples with precision outliers. The footnote only applies to samples within the same batch containing the sample used for the MS and MSD analyses.

		"RQS" in LIMS.		For Minnesota Admin Contract Clients - all MS/MSD failures require reanalysis of the MS/MSD and the original sample. If it is still out of control, investigate and document the cause in the associated narrative as well as qualifying appropriately.
Laboratory Filter Blank (FB)	A filtered aliquot of reagent water treated and prepared exactly as a sample when lab filtration is requested.	Analyzed only with batches of lab filtered dissolved metals, one per batch of 20 or less.	Less than the absolute value of the reporting limit. NC requires blanks to be clean to ½ RL.	Re-analyze associated samples. ***Exceptions:*** If sample ND, report sample without qualification; If sample result >10x MB detects, report sample as not impacted by the blank contamination; If sample result <10x MB detects and sample cannot be reanalyzed, report sample with appropriate qualifier to indicate an estimated value. Client must be alerted and authorize this condition.

14. Data Analysis and Calculations

14.1. The percent recovery in the LCS is calculated using Equation 1:

Equation 1

$$\%\ Recovery = \frac{SR}{SA} \text{ x } 100$$

Where:

SR = LCS result (ug/L or mg/kg)

SA = spike added, ug/L or mg/kg

14.2. The percent recovery of mercury in the matrix spike and matrix spike duplicate is calculated using Equation 2:

Equation 2

$$\%\ Recovery = \frac{(SSR - SR)}{SA} x100$$

Where:
SSR = Spiked sample result, mg/L or mg/kg
SR = Sample result, mg/L or mg/kg
SA = Spike added, mg/L or mg/kg

14.3. Calculate the Relative Percent Difference (RPD) between the matrix spike and matrix spike duplicate using Equation 3:

Equation 3

$$\%RPD = \frac{|S - D|}{(S + D)/2} x100$$

Where:
S = Sample result, mg/L or mg/kg
D = Duplicate sample result, mg/L or mg/kg

14.4. The corrected dry weight concentration can be calculated using the following:

$$corrected\ dry\ wt\ conc = \frac{\left(c \times \frac{v_f}{wt_i}\right)}{\%\ dry\ wt}$$

Where:
c = concentration on instrument, µg/L
v_f = final volume, L
wt_i = initial weight, g

$$\%\ \text{Dry weight} = \frac{\text{Sample Dry Weight}}{\text{Sample Wet Weight}} \times 100$$

14.5. Refer to ground glass and end cap SOPs for calculations for each matrix.

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. See tables in section 11 & 13.

16. Corrective Actions for Out-Of-Control Data

16.1. See tables in section 11 & 13.

17. Contingencies for Handling Out-Of-Control or Unacceptable Data

17.1. See tables in section 11 & 13.

18. Method Performance

18.1. All applicable personnel must read and understand this SOP with documentation of SOP review maintained in their training files.

18.2. **Method Detection Limit (MDL) Study**: An MDL study must be conducted annually (per the method) per S-MN-Q-269 – Determination of Limit of Detection and Limit of Quantitation (or equivalent replacement) for each matrix per instrument.

18.3. **Demonstration of Capability (DOC)**: Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC) per S-MN-Q-279 - Training and Employee Orientation (or equivalent replacement).

18.4. **Periodic performance evaluation (PE)** samples are analyzed to demonstrate continuing competence per SOP S-MN-Q-258 – Proficiency Testing Program (or equivalent replacement). Results are stored in the QA office.

19. Method Modifications

19.1. ICV/CCV criteria is ± 10% while the 7000 series indicates ± 20%, the tighter criteria is applied to allow for instrumentation to be utilized for any mercury method throughout an analytical shift.

19.2. Currently we process a CRDL at ± 30% while the method has no low end criteria.

19.3. Use of Block Digestor- Heating is conducted with a hot block digestion as the heating equivalent mentioned in SW 846 7471B (section 6.10) and SW 846 7470. This is also compliant with method 245.1 under the Clean Water Act method flexibility in 40CFR section 136.6 (b) (4) (iii).

19.4. The lab utilizes a 30 mL final volume, all solid weights and reagent ratios are conducted based on the 0.3 g versus the 0.5 g initial weight accordingly.

19.5. This lab carries certification in multiple states that recognize different versions of the EPA methods.

20. Instrument/Equipment Maintenance

20.1. Please refer to the Cetac Mercury Analyzer 7500 and 7600 User Manual.

20.2. All maintenance activities are listed daily in maintenance logs that are assigned to each separate instrument.

21. Troubleshooting

21.1. Refer to instrument operation manuals.

22. Safety

22.1. **Standards and Reagents**: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound is be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats, and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions are prepared in a hood whenever possible.

22.2. **Samples**: Take precautions when handling samples. Samples arealways be treated as potentially hazardous “unknowns”. The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

22.3. Mercury fumes are highly toxic. Care is taken to insure proper ventilation of analysis area.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in S-MN-S-003 - Waste Handling and Management (or equivalent replacement).

23.2. In order to minimize the amount of waste generated during this procedure, the analyst should prepare reagents in an amount which may be used in a reasonable amount of time (e.g., before a reagent expires).

24. Pollution Prevention

24.1. The company wide Chemical Hygiene and Safety Manual contains information on pollution prevention.

25. References

25.1. Pace Quality Assurance Manual- most current version.

25.2. National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, “Quality Systems”- most current version.

25.3. The NELAC Institute (TNI); Volume 1, Module 2, “Quality Systems”- most current version.

25.4. Test Methods for Evaluating Water and Solid Waste, Physical/Chemical Methods, SW-846, Method 7470A, 1994.

25.5. Test Methods for Evaluating Water and Solid Waste, Physical/Chemical Methods, SW-846, Method 7471A, 1994.

25.6. Test Methods for Evaluating Water and Solid Waste, Physical/Chemical Methods, SW-846, Method 7000a, Revision 1, July 1992.

25.7. Test Methods for Evaluating Water and Solid Waste, Physical/Chemical Methods, SW-846, Method 7471B, Revision 2, Feb 2011.

25.8. Methods for Chemical Analysis of Water and Wastes, Method 245.1. Rev.3.0, 1994

26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. Not applicable to this SOP.

27. Revisions

Document Number	Reason for Change	Date
S-MN-I-359 Rev.26	Updated to LLC throughout document Removed uncontrolled Updated attachment I to rev.10 Added "Copies without a distribution number below are considered uncontrolled" to the statement of copyright.	5Jul2017
S-MN-I-359 Rev.27	Table 9.1: updated Item # for Metals-free digestion cups. 10.2.1: removed duplicating "Standards", added "reagents", removed "Process in the same manner as samples with the exception of the heating steps," added "SW-846 series methods…preplog." 12.1.2 and 12.1.4: 0.100 changed to 0.15mL aliquot for Low-level Replaced reference to corporate training SOP with local SOP S-MN-Q-279 in Section 18.3. Removed Section 19.1. Removed attachment (and reference to attachment in 19.6) because retired form.	15Feb2018

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STANDARD OPERATING PROCEDURE

PREPARATION OF SOLID SAMPLES FOR ANALYSIS BY ICP AND ICP-MS

Reference Methods: EPA 3050B

Local SOP Number:	S-MN-I-460-rev.19
Effective Date:	Date of Final Signature
Supersedes:	S-MN-I-460-rev.18

APPROVALS

Laboratory General Manager — Date: 31 Jul 2017

Laboratory Quality Manager — Date: 17 Jul 2017

PERIODIC REVIEW

SIGNATURES BELOW INDICATE NO CHANGES HAVE BEEN MADE SINCE PREVIOUS APPROVAL.

Signature	Title	Date

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TABLE OF CONTENTS

1. Purpose/Identification of Method

1.1. The purpose of this SOP is to establish a procedure for the digestion of solid samples to be analyzed by ICP and ICP-MS as described in EPA Method 3050B.

2. Summary of Method

2.1. Inductively coupled plasma atomic emission spectroscopy (ICP-AES) and inductively coupled plasma mass spectrometry (ICP-MS) are utilized for the determination of metals in solution. The method is applicable to a large number of matrices.

2.2. The samples are digested in concentrated nitric acid, hydrochloric acid and hydrogen peroxide. After digestion, samples are filtered (unless ICPMS) and brought to volume.

3. Scope and Application

3.1. **Personnel**: The policies and procedures contained in this SOP are applicable to all personnel involved in the analytical method or non-analytical process.

3.2. **Parameter**s: Not applicable to this SOP.

4. Applicable Matrices

4.1. This SOP is applicable to solid samples.

5. Limits of Detection and Quantitation

5.1. Not applicable to this SOP.

6. Interferences

6.1. Not applicable to this SOP.

7. Sample Collection, Preservation, Shipment and Storage

7.1. Table 7.1 – Sample Collection, Preservation, Shipment and Storage

Sample type	Collection per sample	Preservation	Storage	Hold time
Solid	Plastic or glass containers. Pre-cleaned containers are purchased from a supplier.	N/A	Above freezing but below 6°C until digested if samples are to be tested for mercury too	Must be analyzed within 6 months of collection.

8. Definitions

8.1. Definitions of terms found in this SOP are described in the Pace Analytical Services Quality Manual, Glossary Section.

9. Equipment and Supplies (Including Computer Hardware and Software)

9.1. Table 9.1 – Equipment and Supplies

Supply	Description	Vendor/Item #/Description
Mechanical pipettes	Various sizes	Fisher Scientific or equivalent
Digestion Cups	50 mL	Environmental Express or equivalent
Filtermate Plunge filters	2 um PTFE SC0408	Environmental Express
Hot Block ™	54 Place Hot Block	Environmental Express
Reflux Caps	Caps with a center hole	Environmental Express or equivalent

Analytical Balance	Ability to weigh to the nearest 0.01g	Fisher Scientific or equivalent
Resin beads	For solid matrix QC	Environmental Express or equivalent

10. Reagents and Standards

10.1. Table 10.1 – Reagents and Standards

Reagent/Standard	Concentration/Description	Requirements/Vendor/Item #
De-ionized (DI) water	ASTM Type II	Verify that background levels of volatile compounds are acceptable by analysis
Hydrogen Peroxide	30% ACS Grade	Fisher brand
Hydrogen Peroxide	30%, Optima Grade for tin only	Fisher brand
Concentrated nitric acid (HNO_3)	Trace Metal grade	Fisher brand
Concentrated hydrochloric acid (HCl)	Trace Metal grade	Fisher brand
Metals Spike - Stock solution standards for LCS and MS/MSD	The solution identifications are PACE-67Aand Pace-67B. See 10.1.1.	Purchased from Inorganic Ventures (or equivalent). Store at room temperature. Expires as specified by manufacturer.

10.1.1. Metals Stock Standards Table

PACE-67B		PACE-67A	
Element	(mg/L)	Element	(µg/L)
Ca	4000	Si	1000
Fe	4000	Sb	200
Mg	4000	Mo	200
K	4000	Sn	200
Na	4000	Ti	200
Se	200		
Al	4000		
Ba	200		
Be	200		
Bi	200		
B	200		
Cd	200		
S	4000		
Cs	200		
Cr	200		
Co	200		
Cu	200		
As	200		
Li	200		
P	200		
Mn	200		
Pb	200		
Ni	200		
Ag	100		
Sr	200		
Tl	200		

V	200		
Zn	200		
U	200		
Pd	40		
Pt	40		

11. Calibration and Standardization

11.1. Calibrate variable and fixed volume pipettes as specified in SOP S-MN-Q-264 – Support Equipment (or equivalent replacement). Calibration records are kept in the QA Office.

11.2. Calibrate the thermometer as specified in SOP S-MN-Q-264 – Support Equipment (or equivalent replacement). Calibration records are kept in the QA Office.

12. Procedure

12.1. Sample Preparation

12.1.1. Mix the sample thoroughly to achieve homogeneity. For each digestion procedure, weigh a 1-1.1g portion of sample (to the nearest 0.01g) and transfer to a 50 mL digestion cup. Alternative sample volume may be used based on sample matrix. Weigh out 3 aliquots for the batch QC sample (background, matrix spike (MS), and matrix spike supplicate (MSD) being sure to weigh them as close to the same weight as possible.

12.1.1.1. Create a method blank and a laboratory control sample (LCS) by weighing out 1 gram of resin beads for each.

12.1.1.2. Spike the LCS, MS/MSD using 0.25 mL of each PACE-67A and PACE-67B.

12.1.2. Add 10mL of DI water to each sample.

12.1.3. Add 7.5mL of concentrated HNO3, mix the slurry, and cover with a reflux cap. Heat the sample to 95 +/- 2°C and reflux for 70 minutes without boiling. Observe the sample during heating for brown fumes indicating oxidation of the sample. If this occurs, add up to an additional 5 mL HNO3 and re-heat. Repeat this process until no fumes are given off during heating. Record on the digestion log to what samples and how much additional acid was added.

Note: record initial Hot Black temperature in the digestion log.

12.1.4. Cool the sample 10 minutes. Add 2.5mL of 30% hydrogen peroxide. Cover with reflux cap and return to the Hot Block for warming which will start the peroxide reaction. Care must be taken to ensure that losses do not occur due to vigorous effervescence. Heat until effervescence subsides for a total of 10 minutes. Cool the samples in the plastic cups.

Note: use Optima grade hydrogen peroxide if the analysis of tin (Sn) is required. Tin is used as a stabilizer in the ACS grade of hydrogen peroxide.

12.1.4.1. If effervescence does not subside, continue to add 30% hydrogen peroxide in 1mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged. Note in the comments section of prep sheet the additional aliquots.

12.1.4.1.1. NOTE: Do NOT add more than a total of 10mL hydrogen peroxide.

12.1.5. Add 5mL of concentrated HCl, return the sample to the Hot Block and reflux for an additional 15 minutes without boiling.

12.1.6. Remove samples from Hot Block and record final temperature in digestion log. Allow samples to cool. Bring samples up to a final volume of 50 ml with DI water. Invert several times for good mixing. FOR ICP-MS sample prep, cap and label samples for analysis – do not filter if analyzed by ICPMS.

12.1.7. For ICP-AES, samples may be allowed to sit overnight while solid materials settle out or samples may be filtered. If filtered, use FilterMate plunge filters following manufacturers instructions. If

samples are filtered all QC samples including the method blank and laboratory control sample (LCS) must also be filtered.

12.1.7.1. Note: The method modifications that have been utilized have been defined in the above process have been demonstrated effective in MDLs, DOCs, successful PTs, and ongoing precision and accuracy data samples.

12.2. Documentation

12.2.1. Digestion Logbook

12.2.1.1. Record the necessary information in the digestion log book including sample ID, initial and final volumes, prep date, prep analyst, supporting equipment, and lot numbers of solutions used, including spike solutions and LCS solutions.

12.2.1.2. Also include any additional comments if needed.

12.2.2. Temperature Logbook

12.2.2.1. Record the temperature of each hot block daily in the temperature logbook.

12.2.2.2. Use a NIST-traceable thermometer inserted into a digestion cup filled with 50mL of DI to measure the temperature of the hot block. The temperature should be checked in different wells of the Hot Blocks such that all wells are evaluated over a period of time.

13. Quality Control

13.1. Table 13.1 – Quality Control

QC Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Preparation Blank	A clean matrix similar to the samples. For solids, 1.0 grams of resin beads. For wipes, use a new Ghost Wipe.	Prepared with each batch	See appropriate analysis SOP.	See appropriate analysis SOP.
Laboratory Control Sample (LCS)	For solids, weigh 1.0 gram of resin beads. Spike with appropriate spiking solutions.	Prepared with each batch	See appropriate analysis SOP.	See appropriate analysis SOP.
Matrix Spike (MS) / Matrix Spike Duplicate (MSD)	Weigh out similar amounts of soil as the parent sample; be sure to weigh QC sample and MS/MSD samples as close as possible. Spike with appropriate spike solutions and record in digestion log.	Prepared with each batch of samples. Client specific requirements may result in a greater number of MS or MS/MSD sets in a batch.	See appropriate analysis SOP.	See appropriate analysis SOP.
Duplicate (DUP)	In some cases the client may request a duplicate in lieu of an MSD. This is weighed out in similar amount (as close as possible) to the background sample.	As requested.	See appropriate analysis SOP.	See appropriate analysis SOP.

14. Data Analysis and Calculations

14.1. Not applicable to this SOP.

15. Data Assessment and Acceptance Criteria for Quality Control Measures

15.1. See table in section 13.

16. Corrective Actions for Out-Of-Control Data

16.1. See table in section 13.

17. Contingencies for Handling Out-Of-Control or Unacceptable Data

17.1. If not specifically listed in the table in section 13, the contingencies are as follows. If there is no additional sample volume to perform re-analyses, all data will be reported as final with applicable qualifiers. If necessary, an official case narrative will be prepared by the Quality Manager or Project Manager.

18. Method Performance

18.1. All applicable personnel must read and understand this SOP with documentation of SOP review maintained in their training files.

18.2. **Method Detection Limit (MDL) Study**: An MDL study must be conducted annually (per the method) per S-MN-Q-269 – Determination of Limit of Detection and Limit of Quantitation (or equivalent replacement) for each matrix per instrument.

18.3. **Demonstration of Capability (DOC)**: Every analyst who performs this method must first document acceptable accuracy and precision by passing a demonstration of capability study (DOC) per S-ALL-Q-020 - Training Procedures (or equivalent replacement).

18.4. **Periodic performance evaluation (PE)** samples are analyzed to demonstrate continuing competence per SOP S-MN-Q-258 – Proficiency Testing Program (or equivalent replacement). Results are stored in the QA office**.**

19. Method Modifications

19.1. The preparation method has been modified in terms of the amounts of reagents used and the individual heating times. The chemistry is maintained. Part of the reason for this modification is better performance for silver and antimony. PT samples are analyzed regularly to validate that the modifications are effective. Per the method, the nitric acid and peroxide amounts are varied based on the sample reaction and this is the case with the Pace method. Overall, the Pace digestion ends up with a higher total acid concentration.

19.2. The final volume for the Pace method is 50 mL, opposed to 100 mL for the reference method.

19.3. Samples are processed using the Hot Block digestion system employing metals free disposable plastic ware rather than glass beakers.

20. Instrument/Equipment Maintenance

20.1. Please refer to the specific manufacturer's instrument manual for maintenance procedures performed by the lab.

20.2. All maintenance activities are listed daily in maintenance logs that are assigned to each separate instrument.

20.3. Logs are kept daily for each hot block, monitoring temperature. The temperature probe is varied daily so that each individual hot block sample cell is monitored to ensure consistency across the block.

21. Troubleshooting

21.1. Not applicable to this SOP.

22. Safety

22.1. Standards and Reagents: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound should be treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Material Safety Data Sheets (MSDSs) are on file in the laboratory and available to all personnel. Standard solutions should be prepared in a hood whenever possible.

22.2. Samples: Take precautions when handling samples. Samples should always be treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

23. Waste Management

23.1. Procedures for handling waste generated during this analysis are addressed in S-MN-S-003 - Waste Handling and Management (or equivalent replacement).

23.2. In order to minimize the amount of waste generated during this procedure, analyst should prepare reagents in an amount which may be used in a reasonable amount of time (e.g., before a reagent expires).

24. Pollution Prevention

24.1. The company wide Chemical Hygiene and Safety Manual contains information on pollution prevention.

25. References

25.1. Pace Quality Assurance Manual- most current version.

25.2. National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, "Quality Systems"- most current version.

25.3. The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems"- most current version.

25.4. Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846, Third Edition. Method 3050B

26. Tables, Diagrams, Flowcharts, and Validation Data

26.1. Attachment I – Wisconsin Procedure 3020A/3050B

27. Revisions

Document Number	Reason for Change	Date
S-MN-I-460-Rev.19	"And Wipes" removed from SOP title throughout. Section 4.1: "and wipes" deleted. Table 9.1: "or equivalent" added to vendor item description of digestion cups and reflux caps. Ghost wipes row deleted. Filtermate plunge filters description updated. Table 10.1: ICP spike updated as metals spike with updated solution identification information provided. ICP MS Spike row deleted. Table 10.1.1, ICP Stock Standards Table, deleted. Table 10.1.2 renumbered, and renamed "Metals Stock Standards Table, with updated element and unit of measurement values. Table 10.2 deleted. Section 10.2.1 deleted. Section 12.1.1: sample portion changed from 1.5 g to 1.1 g. Section 12.1.1.1 deleted Text regarding wipes removed from 12.1.1.1.1, as renumbered. Section 12.1.1.2, previously 12.1.1.1.2, appended with "MS/MSD using 025 mL of each PACE-67A and PACE-67B. Section 12.2.1 deleted. Subsequent sections renumbered. Section 12.2.1.1: "supporting equipment" added following "prep analyst". Table 13.1: instructions regarding Ghost wipe use deleted from LCS row. Attachment I – updated to current revision Updated LLC Removed "uncontrolled" Added "Copies without a distribution number below are considered uncontrolled" to the statement of copyright.	19June2017

Attachment I – Wisconsin Procedure for 3020A and 3050B

Pace Analytical®	Document Name: Procedure for Wisconsin Samples – 3010A/3020A/3050B	Document Revised: 03Feb2016 Page 1 of 1
	Document No.: F-MN-I-411-Rev.02	Issuing Authority: Pace Minnesota Quality Office

For Wisconsin solid samples only

ICPMS/ ICP Metals (1.0-1.5 grams sample)

1. Add at least 5mL of conc. HNO3 (or 10mL 1:1) to the samples
2. Heat at 95 'C for at least 10 minutes, covered with reflux cap for refluxing
3. Add at least 5mL conc. HNO3
4. Heat at 95 'C for at least 30 min, covered with reflux cap for refluxing
5. Check for brown fumes
    a. If no brown fumes - continue to step 6
    b. If brown fumes, add at least 5mL conc. HNO3
    c. Heat
    d. If no brown fumes - continue to step 6
    e. If brown fumes, add more conc. HNO3 and heat
    f. Continue step e until brown fumes no longer exist
6. Heat for at least 2 hrs, covered with reflux cap for concentrating
7. Add 2mL H2O and 3mL of 30% H2O2
8. Heat to 95 'C and add 1mL increments of H2O2 until effervescence subsides, covered with reflux cap for refluxing
9. Heat for at least 2 hrs, covered with reflux cap for concentrating
10. Add at least 10mL of conc. HCl, heat to 95 'C for at least 15 min, covered with reflux cap for refluxing.
11. Dilute to 50mL
12. Match standards to final acid concentrations

Note: Method 3050B section 4.2 states: "Vapor recovery device (e.g., ribbed watch glasses, appropriate refluxing device, appropriate solvent handling system) We have opted to use a reflux cap as the appropriate refluxing device as stated rather than the ribbed watch glass.

For Wisconsin water samples only

ICPM/ICP Metals (50mL sample)

Transfer 50mL of well-mixed sample into a labeled digestion tube.

Add 1.5mL concentrated nitric acid to each digestion tube. Place the tubes into the block digester which has been preheated to achieve a temperature of 95°C (+/- 3°C) in the digestion tubes and cover with ribbed watch glass.

Evaporate without boiling to <10mL. Do not allow samples to go dry.

If digestate is generating brown fumes, add another 2.5mL concentrated nitric acid and reflux gently. Continue heating and adding acid as necessary, until the digestion is complete, generally indicated when the digestate is light in color and brown fumes are no longer generated.

Evaporate without boiling to approximately 5mL. Do not allow samples to go dry.

Cool the samples then add 2mL concentrated hydrochloric acid, return the samples to the hot block and heat for 15 minutes to dissolve any precipitate then allow samples to cool.

Dilute the digestates to 50mL in the digestion tube with reagent water. If necessary, filter the digestates to remove particulates using a plunger filter. If any sample digestates in a batch are filtered, the Method Blank and LCS must also be filtered.

Appendix C
Forms

Appendix C.1 Chain of Custody

Appendix C.2 XRF Field Data Sheet

Appendix C.3 Level A-B Validation Form

Appendix C.4 Corrective Action Template

Laboratory Management Program (LaMP) Chain of Custody Record

Soil, Sediment and Groundwater Samples

BP Site Node Path: _____ **Req Due Date (mm/dd/yy):** _____ Rush TAT Yes _____ No ____x

BP/RM Facility No: _____ **Lab Work Order Number:** _____

Lab Name:	BP/ARC Facility Address:	
Lab Address:	City, State, ZIP Code:	Consultant/Contractor Project No:
Lab PM:	Lead Regulatory Agency:	Address:
Lab Phone:	California Global ID No.:	Consultant/Contractor PM:
Lab Shipping Accnt:	Enfos Proposal No:	Phone: Email:
Lab Bottle Order No:	Accounting Mode: Provision _____ OOC-BU _____ OOC-RM _____	Send/Submit EDD to:
Other Info:	Stage Activity OMM	Invoice To: BP-RM _____ BP-Other _____

BP/RM PM:	Sample Details	Requested Analyses	Report Type & QC Level
			Limited (Standard) Package ____
PM Phone:		Filt	Limited Plus Package ____
PM Email:		Pres	Full Package ____

Lab No.	Sample Description	Date	Time	Field Matrix	Start Depth	End Depth	Depth Unit	Grab (G) or Composite (C)	Total Number of Containers	Analysis	Comments

Sampler's Name:	Relinquished By / Affiliation	Date	Time	Accepted By / Affiliation	Date	Time
Sampler's Company:						
Ship Method: Ship Date:						
Shipment Tracking No:						

Special Instructions:

THIS LINE - LAB USE ONLY: Custody Seals In Place: Yes / No | Temp Blank: Yes / No | Cooler Temp on Receipt: _____°F/C | Trip Blank: Yes / No | MS/MSD Sample Submitted: Yes / No

XRF SAMPLES

XRF - Thermo Fisher Scientific
Nitron XL3 X-Ray Based

Soil Action/Screening Levels (mg/kg)

Residential	250	71	3,100	1,200	
Non-Residential				2,300	
Recreational	1,000	20	1,000		1,000
Commercial	500				

XRF Reading	Sample Name	Date collected	Date analyze	As mg/kg	Cd mg/kg	Cu mg/kg	Pb mg/kg	Zn mg/kg	Depth	Pass/Fail

Level 2 Data Validation Checklist XRF Sample Analysis

Site:	**Case No:**	**Laboratory:**
Project:	**Sample Matrix:**	**Analyses:**
Sample Date(s):	**Analysis Date(s):**	
Data Validator:	**Validation Date(s):**	

1. Holding Times

Analyte	Laboratory	Matrix	Method	Holding Times	Collection Date(s)	Analysis Date(s)	Holding Time Met (Y/N)	Affected Data Flagged (Y/N)

*Reference for Holding Times –

Were any data flagged because of holding time? Y [ ] N [X]
What sample preparation steps were performed (i.e. drying, sieving etc.)?
Were the samples prepped according to the SAP/QAPP? Y [X] N [ ]

Describe Any Actions Taken:

Comments:

2. Energy Calibration (System Check)

Was the energy calibration performed at the frequency of once per day? Y [ ] N [ ]
Was the energy calibration Resolution below 195? Y [ ] N [ ]
Did the energy calibration run for at least 50 seconds? Y [ ] N [ ]

Describe Any Actions Taken:

Comments:

3. SiO_2 Standards

Was the SiO_2 Standard analyzed at the beginning of analysis? Y [ ] N [ ]
Was the SiO_2 Standard analyzed at the frequency of 1 per 20 natural samples? Y [ ] N [ ]
Were the SiO_2 Standard results within the control limits? Y [ ] N [ ]
Were any data flagged because of the SiO_2 Standard results? Y [ ] N [ ]

Describe Any Actions Taken:

Comments:

4. Calibration Check Samples

Were the appropriate Calibration Check Samples (CCS) analyzed at the beginning of analysis? Y [ ] N [ ]
Were the appropriate CCS analyzed at the frequency of 1 per 20 natural samples? Y [ ] N [ ]
Were CCS results within the control limits? Y [ ] N [ ]
Were any data flagged because of CCS problems? Y [ ] N [ ]

Describe Any Actions Taken:

Comments:

5. Duplicate Sample Results

Were Duplicate Samples analyzed at the frequency of 1 per 20 natural samples?	Y ☐	N ☐
Were Duplicate Sample results within the control window?	Y ☐	N ☐
Were any data flagged because of duplicate sample results?	Y ☐	N ☐

Describe Any Actions Taken:

Comments:

6. Replicate Sample Results

Were Replicate Samples analyzed at the frequency of 1 per 20 natural samples?	Y ☐	N ☐
Were replicate sample results within the control window?	Y ☐	N ☐
Were any data flagged because of replicate sample results?	Y ☐	N ☐

Describe Any Actions Taken:

Comments:

7. Overall Assessment

Are there analytical limitations of the data that users should be aware of?	Y ☐	N ☐

If so, explain:

Comments:

8. Authorization of Data Validation

Data Validator

Name: **Reviewed by:**

Signature: _____ _____

Date: _____ _____

Level A/B Assessment Checklist

1. General Information

Site:
Project:
Client:
Sample Matrix:

2. Screening Result

Data are:

1. Unusable ____
2. Level A ____
3. Level B ____

I. **Level A**

Criteria – The following must be fully documented.	**Yes/No**	**Comments**
1. Sampling date		
2. Sampling team or leader		
3. Physical description of sampling location		
4. Sample depth (soils)		
5. Sample collection technique		
6. Field preparation technique		
7. Sample preservation technique		
8. Sample shipping records		

II**. Level B**

Criteria – The following must be fully documented.	**Yes/No**	**Comments**
1. Field instrumentation methods and standardization complete		
2. Sample container preparation		
3. Collection of field replicates (1/20 minimum)		
4. Proper and decontaminated sampling equipment		
6. Field custody documentation		
7. Shipping custody documentation		
8. Traceable sample designation number		
9. Field notebook(s), custody records in secure repository		
10. Completed field forms		

Level 2 Data Validation Checklist for Sample Analysis

Site: **Case No:** **Laboratory:**
Project: **Sample Matrix:** **Analyses:**
Sample Date(s): **Analysis Date(s):**
Data Validator: **Validation Date(s):**

1. Holding Times

Analyte	Laboratory	Matrix	Method	Holding Times	Collection Date(s):	Analysis Date(s)	Holding Time Met (Y/N)	Affected Data Flagged (Y/N)

Were any data flagged because of holding time? Y ☐ N ☐
Were any data flagged because of preservation problems? Y ☐ N ☐

Describe Any Actions Taken:

Comments:

2. Blanks

Were Method Blanks (MBs) analyzed at the frequency of 1 per analytical batch? Y ☐ N ☐
Were MBs within the control window? Y ☐ N ☐
Were any data flagged because of blank problems? Y ☐ N ☐

Describe Any Actions Taken:

Comments:

3. Laboratory Control Samples

Were Laboratory Control Samples (LCS) analyzed at the frequency of 1 per batch? Y ☐ N ☐
Were LCS results within the control window? Y ☐ N ☐
Were any data flagged because of LCS problems? Y ☐ N ☐

Describe Any Actions Taken:

Comments:

4. Duplicate Sample Results

Were Laboratory Duplicate Samples (LDS) analyzed at the frequency of 1 per batch? Y ☐ N ☐
Were LDS results within the control window? Y ☐ N ☐
Were any data flagged because of LDS problems? Y ☐ N ☐

Describe Any Actions Taken:

Comments:

5. Matrix Spike Sample Results

Were Laboratory Matrix Spike Samples (LMS) analyzed at the frequency of 1 per batch? Y ☐ N ☐
Were LMS results within the control window? Y ☐ N ☐
Were any data flagged because of LMS problems? Y ☐ N ☐

Describe Any Actions Taken:

Comments:

6. Field Blanks

Were field blanks submitted as specified in the QAPP?	Y		N		N/A	
Were field blanks within the control window?	Y		N		N/A	
Were any data qualified because of field blank problems?	Y		N		N/A	

Describe Any Actions Taken:

Comments:

7. Field Duplicates

Were field duplicates submitted as specified in the QAPP?	Y		N		N/A	
Were results for field duplicates within the control window?	Y		N		N/A	
Were any data qualified because of field duplicate problems?	Y		N		N/A	

Describe Any Actions Taken:

Comments:

8. Overall Assessment

Are there analytical limitations of the data that users should be aware of? Y ☐ N ☐

If so, explain:

Comments:

9. Authorization of Data Validation

Data Validator

Name: **Reviewed by:**

Signature: _____ _____

Date: _____ _____

Corrective Action Report/ Corrective Action Plan

Project ID	Project Name	Document ID

Preparer's Signature/Submit Date	Submitted to:

Description of the requirement or specification	
Reason for the Corrective Action	
Location, affected sample, affected equipment, etc. requiring corrective action	
Suggested Corrective Action	(Continue on Back)
Corrective Action Plan	(Continue on Back) ☐ Approval signature/date:_____
	Approval of corrective actions required by EPA? ☐ Yes ☐ No ☐ EPA approval name/date:_____ ☐ Corrective actions completed name/date:_____
Preventative Action Plan	(Continue on Back) ☐ Preventative actions completed name/date:_____

Corrective Action Report/
Corrective Action Plan

Suggested Corrective Action (Continued)	
Corrective Action Plan (Continued)	
Preventative Action Plan (Continued)	

Appendix D
Summary of Revisions and Bibliography of Data Summary Reports

Summary of Revisions
Bibliography of Completed Sites and Executive Summaries

Appendix D.1
Summary of Revisions

Rev. No.	Year	Description
1	2021	Distribution lists: Updated to current distribution list. Updated text to reference BPSOU CD and Field Sampling Plans (FSPs) rather than sampling and analysis plans (this affected Section 2). Section 2.1: Updated Project Organization and Responsibilities • Updated Atlantic Richfield QAM to David Gratson • Updated Atlantic Richfield Liability Manager Title (Mike Mc Anulty Atlantic Richfield Company) • Updated Operations Manager (Eric Hassler) • Added Brandon Warner as BSB Field Team Supervisor Section 2.2 and Section 2.3: Updated text to reference the BPSOU CD and specify metals-impacted sediment. Section 2.4. Updated Step 2: Identify the Goals of the Study to include: Are contaminants, if present on site, the result of historic mining operations or related activities? Minor word changes in Step 4 and Step 7 for clarification. Section 2.6.7: Added metals-impacted to clarify type of sediments. Added Section 3.1 Site Evaluation Objectives, which changed all the section 3 headings after it. Section 3.3.2 Sedimentation Analysis (previously Section 3.2.2): Added metals-impacted to clarify type of sediments. Section 6 References: added the BPSOU CD information. Appendix A: Figures/Charts • Updated A.1 – Updated BPSOU Area Map to revised BPSOU boundary in the Consent Decree • Updated A.2 – Organization Chart • Updated A.3 – Decision Logic Appendix B: SOP Updates • SOP-SA-04 – revised 11/12/2020 • SOP-DE-02 – revised 09/08/2020 Appendix C: Updated forms Appendix D: Added changes to previous revision.