UPPER COLUMBIA RIVER

FINAL
Quality Assurance Project Plan for the Plant Tissue Study

Prepared for

Teck American Incorporated
P.O. Box 3087
Spokane, WA 99220-3087

Prepared by

RAMBOLL
901 Fifth Avenue, Suite 2820
Seattle, WA 99222-3087

In Association and Consultation with

AECOM
and
Parametrix, Inc.

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SECTION A: PROJECT MANAGEMENT

A1 TITLE AND APPROVAL SHEET

QUALITY ASSURANCE PROJECT PLAN FOR THE PLANT TISSUE STUDY

EPA Project Manager	Monica Tonel	Monica Tonel	Date April 12, 2018
TAI Project Coordinator	Kris McCaig	Kris McCaig	Date April 12, 2018
TAI Assistant Project Coordinator	Denise Mills	Denise	Date April 11, 2018
EPA Region 10 Quality Assurance Manager	Donald Brown	Don Brown	Date 4-12-18
Principal Investigator	Dina Johnson	Dina L Johnson	Date April 11, 2018
Senior Technical Advisor	Rosalind Schoof	Rosalind Schoof	Date April 11, 2018
Task Manager	Lis Castillo Nelis	Lisa C Johnson FOR LIS	Date April 11, 2018
Field Supervisor	Jennifer Pretare	Jenny A Pretare	Date April 9, 2018
Task QA Coordinator	Rock Vitale	Rock Vitale	Date April 10, 2018
Analytical Chemistry Laboratory Coordinator	Cristy Kessel	Cristy Kessel	Date 4/10/18
Analytical Chemistry Laboratory Project Manager (ALS)	Mark Harris	Mark Harris	Date April 11, 2018
Analytical Chemistry Laboratory QA Manager (ALS)	Carl Degner	for Carl Degner	Date 4/11/18
Database Administrator	Randy O'Boyle	Randy O'Boyle	Date April 10, 2018

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ACRONYMS AND ABBREVIATIONS

Agreement	June 2, 2006, Settlement Agreement
ACG	analytical concentration goal
ALS	ALS Environmental
CCT	Confederated Tribes of the Colville Reservation
CLP	Contract Laboratory Program
COC	chain-of-custody
COI	contaminant of interest
DQO	data quality objective
DMP	data management plan
Ecology	Washington State Department of Ecology
EDD	electronic data deliverable
EPA	U.S. Environmental Protection Agency
ESI	Environmental Standards, Inc.
Exponent	Exponent, Inc.
FSP	field sampling plan
GIS	geographic information system
HHRA	human health risk assessment
IC	incremental composite
ID	identification
LCS	laboratory control sample
LOD	level of detection
Lodestone	Lodestone Environmental Consulting
MDL	method detection limit
MQO	measurement quality objective
MRL	method reporting limit
MS	matrix spike
MSD	matrix spike duplicate
PARCC	precision, accuracy or bias, representativeness, completeness, and comparability
QA	quality assurance
QA/QC	quality assurance and quality control
QAPP	quality assurance project plan
QC	quality control

RI/FS	remedial investigation and feasibility study
RPD	relative percent difference
RSD	relative standard deviation
SA	sampling area
SATES	Soil Amendment Technology Evaluation Study
SHSP	site health and safety plan
Site	Upper Columbia River site
SOP	standard operating procedure
SRM	standard reference material
S2BVM	Stage 2B data validation
S4VM	Stage 4 data validation
TAI	Teck American Incorporated
TAL	target analyte list
TDS	Total Diet Survey
UCR	Upper Columbia River

UNITS OF MEASURE

°C	degree(s) Celsius
dw	dry weight
g	gram(s)
in.	inch(es)
mg/kg	milligram(s) per kilogram
µg/dL	microgram(s) per deciliter
µm	micron(s)

A3 DISTRIBUTION LIST

EPA Project Manager	Monica Tonel
EPA Region 10 QA Manager	Donald Brown
TAI Project Coordinator	Kris McCaig
TAI Assistant Project Coordinator	Denise Mills
Principal Investigator	Dina Johnson
Task QA Coordinator	Rock Vitale
Senior Technical Advisor	Rosalind Schoof
Task Manager	Lis Castillo Nelis
Field Supervisor	Jennifer Pretare
Database Administrator	Randy O'Boyle
Analytical Chemistry Laboratory Coordinator	Cristy Kessel
Analytical Chemistry Laboratory Project Manager (ALS)	Mark Harris
Analytical Chemistry Laboratory QA Manager (ALS)	Carl Degner

A4 INTRODUCTION AND TASK ORGANIZATION

A4.1 Introduction

This document presents the quality assurance project plan (QAPP) for the plant tissue study (hereafter, the "study") of the Upper Columbia River (UCR; hereafter, the Site[1]). This study represents one of the tasks being completed as part of the remedial investigation and feasibility study (RI/FS) and baseline human health risk assessment (HHRA) being completed for the Site under an agreement between Teck American Incorporated (TAI) and the U.S. Environmental Protection Agency (EPA). The objective of the RI/FS is to investigate the nature and extent of contamination and potential for risk to humans and the environment. EPA is conducting the HHRA. TAI is conducting the RI/FS and the current plant study with EPA oversight.

This QAPP describes the organization, data quality objectives (DQOs), study design, analytical procedures, and quality assurance and quality control (QA/QC) procedures upon which the study will be based. The field sampling plan (FSP) describes field procedures and protocols that will be followed and is presented in Appendix A.

The primary objective of this study is to characterize the concentrations of metals in the tissues of wild upland plants sampled from tribal allotments in the study area. Data collection efforts will focus on obtaining information that will inform the exposure assessments for humans who consume or otherwise utilize plants from the study area. Chemistry data for plant parts of interest will be used in the HHRA to evaluate the potential for metal uptake into plants and subsequent exposure of people who harvest and consume or otherwise utilize plants.

The development of the requirements and design rationale for data collection activities presented in this QAPP was guided by meetings and telephone calls with the EPA's team on June 22, September 28, and November 9, 2017, and by the following additional documents or communications:

- A letter dated December 8, 2016 from Laura C. Buelow, EPA, to Kris McCaig, TAI, directing TAI to fund a UCR plant study and attached "Data Quality

[1] The Site as defined within the June 2, 2006 Settlement Agreement is the areal extent of hazardous substances contamination within the United States in or adjacent to the Upper Columbia River, including the Franklin D. Roosevelt Lake, from the U.S.-Canada border to the Grand Coulee Dam, and those areas in proximity to the contamination that are suitable and necessary for implementation of response actions.

Objectives for the Sampling of Terrestrial Plants and Laboratory Analysis of Tissues for Metals" for DQO steps 1 through 5 (USEPA 2016).

- A letter dated February 17, 2017 from Kris McCaig, TAI, to Laura C. Buelow, EPA, notifying EPA of TAI's dispute of the December 8, 2016 letter directive for TAI to fund a UCR plant study and documenting TAI's technical concerns regarding EPA's "Data Quality Objectives for the Sampling of Terrestrial Plants and Laboratory Analysis of Tissues for Metals" (TAI 2017).
- A letter dated June 14, 2017 from Laura C. Buelow, EPA, to Kris McCaig, TAI, documenting TAI's agreement to conduct limited plant tissue sampling focused on collection of plant tissue from the three tribal allotments sampled in the 2014 Residential Soil Study that had concentrations of lead in soil above the 700 mg/kg in addition to a reference area (USEPA 2017a).
- An undated letter and table transmitted via email on September 5, 2017 from Laura C. Buelow, EPA, to Kris McCaig, TAI, documenting EPA's responses to the technical concerns raised in TAI's dispute letter regarding EPA's directive to TAI to fund plant sampling (USEPA 2017b).
- Memoranda pertaining to prior plant reconnaissance efforts and cultural plant sampling recommendations prepared for the Confederated Tribes of the Colville Reservation (CCT) by Lodestone Environmental Consulting (Lodestone 2016a,b and 2017a,b).
- The UCR RI/FS Tribal Consumption and Resource Use Survey (Westat 2012).
- UCR Final Field Reconnaissance Plan for the Upper Columbia River Site Plant Tissue Study (Ramboll Environ 2017a).
- Draft Field Reconnaissance Summary Report for the Upper Columbia River Plant Tissue Study (AECOM 2017).
- Personal communication between D. Johnson, Ramboll Environ, and M. Stifelman, EPA, during a November 28, 2017 conference call approving removal of essential elements (calcium, magnesium, potassium, and sodium) in addition to mercury from the target analyte list (TAL[2]) (Johnson 2017).
- Personal communication between D. Mills, TAI, and M. Tonel, EPA, via an April 3, 2018 email documenting the addition of total mercury analysis for selected

[2] The original TAL for the study was provided in Table 5 of USEPA (2016).

plant targets (kinnikinnick leaves, wild rose leaves and stems, wild mint, willows, and tules only) and co-located soil/sediment samples when sufficient plant material is available to support analysis of both target analyte list (TAL) metals (except calcium, magnesium, potassium, and sodium) and mercury[3] (Mills 2018).

- Various literature and online sources as cited in this QAPP.

A4.2 Task Organization

This section presents the organizational structure for activities associated with the study, including task management and oversight, fieldwork, sample analysis, and data management. Contact information for team task members is provided in Table A4-1.

A4.2.1 EPA Organization and Responsibilities

EPA will oversee TAI activities associated with the study and will coordinate the comments and review of documents by the following parties: U.S. Department of the Interior, Washington State Department of Ecology (Ecology), and local tribes (i.e., the CCT and the Spokane Tribe of Indians). In addition, EPA, under Section 106 of the National Historic Preservation Act, has the primary responsibility for consulting with interested parties. EPA's project manager, Monica Tonel, will be responsible for ensuring that the work performed is consistent with all applicable EPA guidance. The EPA Region 10 quality assurance (QA) manager is Donald Brown. The responsibilities of the QA manager or QA designee will include review and approval of the QAPP and any subsequent addenda, as well as laboratory oversight as requested or necessary (i.e., data validation or laboratory observation).

A4.2.2 TAI Organization and Responsibilities

Kris McCaig will serve as TAI's project coordinator and will have the primary responsibility for ensuring that TAI meets all the requirements and associated deliverables specified within the June 2, 2006 Settlement Agreement (Agreement) (USEPA 2006a). Denise Mills will serve as TAI's assistant project coordinator working closely with Ms. McCaig to ensure the above.

[3] Where the quantity of plant material is limited, allocation of sample mass collected will be prioritized for analysis of TAL metals (except calcium, magnesium, potassium, and sodium).

A4.2.3 Key Task Personnel

TAI technical team members for the study and their respective responsibilities are identified below.

Principal Investigator—Dina Johnson (Ramboll) will serve as principal investigator and will oversee and approve all project activities, review QA reports, approve final project QA needs, and authorize necessary actions and adjustments needed to accomplish program QA objectives.

Senior Technical Advisor—Dr. Rosalind Schoof (Ramboll) will serve as senior technical advisor for the study. Dr. Schoof is responsible for providing technical oversight in the design, implementation, and data interpretation.

Task Manager—Dr. Lis Castillo Nelis (Ramboll) will lead technical design and data interpretation. Dr. Nelis will also provide onsite supervision as needed and ensure that proper tissue collection, preservation, storage, transport, and chain-of-custody (COC) procedures are followed. She will inform the principal investigator when problems occur and will communicate and document corrective actions taken.

Task QA Coordinator—Rock Vitale (Environmental Standards, Inc. [ESI]) is the task QA coordinator and is responsible for providing overall QA support for the study. Mr. Vitale will coordinate validation of laboratory data; communicate data quality issues to the analytical chemistry laboratory coordinator, and will work with the database administrator to address potential data limitations. Mr. Vitale will report directly to the analytical chemistry laboratory coordinator, and will work closely with the database administrator to ensure that the data are of the highest quality.

Analytical Chemistry Laboratory Coordinator—Cristy Kessel (TAI) is the analytical chemistry laboratory coordinator. She is responsible for ensuring that laboratory method selection and/or development is satisfactorily completed prior to the analysis of samples; coordinating with the testing laboratory and tracking the laboratory's progress; verifying that the laboratory has implemented the requirements of this QAPP; addressing QA issues related to the laboratory's analyses; ensuring that the laboratory's capacities are sufficient to undertake the required analyses in a timely manner; and addressing scheduling issues related to the laboratory's analyses. Ms. Kessel will report directly to TAI's project coordinator and will work closely with the principal investigator.

Database Administrator—Randy O'Boyle (Exponent) is the database administrator and will have primary responsibility for data management and database maintenance and development. Mr. O'Boyle is responsible for overseeing and/or conducting the following

activities: establishing storage formats and procedures appropriate for data collected; ensuring all data packages are complete and delivered in the correct format; maintaining the integrity and completeness of the database; and providing data summaries to data users for interpretation and reporting. Mr. O'Boyle will report directly to the TAI project coordinator and will work closely with the task QA coordinator and the laboratory.

Field Supervisor—Jennifer Pretare (AECOM) is the field supervisor responsible for overseeing the planning and coordination of the sampling efforts and for all aspects of upland plant tissue collection activities to ensure that appropriate sampling, QA, and documentation procedures are used. In the event that changes in the QAPP or FSP (Appendix A) are needed, the field supervisor will ensure that proposed changes are coordinated with EPA's project coordinators, its staff, and its authorized representatives in the field, and with TAI's project coordinator according to the established lines of communication among the TAI technical team, TAI, and EPA.

A4.2.4 Laboratory

ALS Environmental (ALS) will perform the sample processing and analyses for metals and conventional parameters (i.e., total mass and percent moisture). The following responsibilities apply to project and QA managers at ALS who will be available for this project.

Analytical Chemistry Laboratory Project Manager—Mark Harris (ALS) is responsible for the successful and timely completion of sample analyses at their laboratory, as well as the following:

- Ensuring that samples are received and logged correctly, are analyzed within specified holding times, that the correct methods and modifications are used, and that data are reported within specified turnaround times
- Reviewing analytical data to ensure that procedures were followed as required in this QAPP, the cited methods, and the laboratory standard operating procedures (SOPs)
- Apprising the TAI analytical chemistry laboratory coordinator of the schedule and status of sample analyses and data package preparation
- Notifying the TAI analytical chemistry laboratory coordinator if problems occur in sample receiving, analysis, or scheduling, or if control limits cannot be met
- Taking appropriate corrective action as necessary
- Reporting data and supporting QA information as specified in this QAPP

- Providing electronic data deliverables (EDDs) in a format consistent and compatible with the UCR electronic database.

Analytical Chemistry Laboratory QA Manager—Carl Degner (ALS) is responsible for overseeing QA activities in their laboratory and ensuring the quality of the data for this task. Specific responsibilities include the following:

- Overseeing and implementing the laboratory's QA program
- Maintaining QA records for the laboratory production unit
- Ensuring that QA/QC procedures are implemented as required for each method and providing oversight of QA/QC practices and procedures
- Reviewing and addressing or approving non-conformity and corrective action reports
- Coordinating responses to any quality control (QC) issues that affect this task with the analytical chemistry laboratory project manager.

A5 PROBLEM DEFINITION AND BACKGROUND

The current conceptual site model for the UCR HHRA provides a framework for considering the relationships among chemical sources, transport and uptake mechanisms into plant tissue, and exposure pathways from plant tissue to people (Figure A5-1). Members of the CCT consume and otherwise utilize (e.g., for weaving) terrestrial, wetland, and aquatic plants that may be contaminated with heavy metals. Other local residents and visitors to the Site may also utilize these resources. The UCR HHRA work plan (SRC 2009) identified metal concentrations in wild upland and riparian plants as a data need. To assess risk to humans posed by consuming or otherwise utilizing wild plants from the vicinity of the Site, information is needed on the concentration of metals in and on specific plants utilized by the CCT. This information will be used along with information about how members of the CCT prepare the target plants for use, how the plants are used, and the frequency and amount collected and consumed, mouthed, or otherwise utilized. With the exception of hazelnuts, pine nuts, and chokecherries, preparation of plant specimens will be limited to wiping and/or washing in the field prior to freezing. Frozen hazelnuts and pine nuts will be shelled in the laboratory prior to analysis. Frozen chokecherries will be pitted in the laboratory prior to analysis.

Elevated metal concentrations have been measured in surface soils by various studies conducted in the Columbia River Valley corridor south of the U.S.-Canada border, including:

- Upper Columbia River Upland Soil Sampling Study (Hart Crowser 2013)
- Final UCR Residential Soil Study Field Sampling and Data Summary Report (CH2M HILL 2016)
- Upper Columbia River, Final Soil Study Data Summary Report (Windward et al. 2015)
- Upper Columbia River, Final Residential Soil Study Data Summary Report (Ramboll Environ 2017b).

Plants may accumulate metals from soil (e.g., Carranza-Álvarez et al. 2008; Intawongse and Dean 2006), and metal uptake factors derived from prior studies are sometimes used to predict plant tissue concentrations from soil concentrations. EPA has determined that the uncertainty associated with available uptake factors for lead and other metals is high, and site-specific data are required to more accurately estimate the exposure point concentrations for contaminants of interest (COIs) in and on plant tissue for the UCR HHRA (USEPA 2016).

Preliminary COI screening of upland soil analytical results indicates lead and arsenic are key chemicals of potential concern for soil and plant ingestion pathways. It is anticipated that decisions based upon exposure information for the CCT may also be protective of other area residents and visitors who utilize these resources, because the modern subsistence exposure factors are likely to be greater than those for other non-tribal residents and visitors.

Delineation of the upland boundaries of the Site has not been determined. Therefore, based on agreement between EPA and TAI (USEPA 2017a), the study area for collection of plant tissues will be focused on three tribal allotment decision units where incremental composite samples from a 2014 residential soil study yielded lead concentrations greater than the time-critical removal action level of 700 mg/kg and one or more reference area. For this study, reference areas were selected based on consideration of a range of lead concentrations at tribal allotments where other decision units have been sampled during one of the three soil studies conducted as part of the UCR RI/FS. Because plant collection areas include areas of high metals concentrations, decisions based on plant tissue concentrations obtained within the study area should be protective of exposure to similar

upland plants at locations with comparable characteristics that may be delineated as part of the Site.

A6 DATA NEEDS

EPA (2016) derived plant uptake factors for various plant parts from several prior studies and estimated a soil lead concentration that would produce a 1 μg/dL increase in blood lead due to ingestion of wild plants that grow in lead-contaminated soil. Based on the available data for lead uptake, soil adherence and ingestion rates, the initial analysis indicated an increase of 1 μg/dL in children who consume vegetation grown in soils with lead concentrations less than 100 mg/kg. SRC reports this analysis conflicts with lead concentrations reported for various foods in the U.S. Food and Drug Administration (2014) Total Diet Survey (TDS) data for 2006 through 2011. For example, the concentration of lead in carrots is usually below the level of detection (LOD) of 0.007 mg/kg. The LOD is much lower than the concentration that would be estimated in carrots using the uptake factor approach. Using an uptake factor of 0.002 (for low soil lead concentration) and assuming the carrots sampled in the TDS Market Basket surveys were grown in soil that contained 10 mg/kg of lead, the estimated lead concentration in the carrot would be 0.02 mg/kg, which is much higher than the TDS LOD for lead.

EPA (2016) reports estimates of uptake factors are highly variable and subject to substantial measurement error. According to EPA (2016), in a study of lead, cadmium, and barium in vegetables grown in urban gardens, McBride et al. (2014) describes the effect of soil factors such as pH, organic matter content, and soil particle adherence to plants on the prediction of lead uptake in food crops. McBride et al. (2014) did not find a strong correlation between lead concentrations in soil (mean of 292 mg/kg) and vegetables (mean in fruit, leafy, herb, and root vegetables of 0.018, 0.099, 0.44, and 0.20 mg/kg, respectively) (McBride et al. 2014). EPA (2016) identifies a key uncertainty related to variation in metal uptake with concentration, with some studies reporting lower uptake at higher soil metal concentrations.

Taken together, these analyses suggest that lead uptake factors may yield overestimates of plant lead concentrations and could be misleading in indicating a potential risk where none exists. The same concerns may be applicable to uptake factors for arsenic. Data generated through sampling and analysis of plant tissue in the study area will help reduce the uncertainty in exposure estimates for the upland wild plant ingestion pathway.

A7 DATA QUALITY OBJECTIVES, CRITERIA, AND DESIGN RATIONALE

EPA's seven-step DQO process (USEPA 2006b) was used to guide the design rationale for the plant tissue study. Each step is described below.

A7.1 Step 1—State the Problem

As noted in Section A6, studies conducted to date have not provided sufficient data for evaluating potential risks to human receptors from the ingestion of upland wild plants from the study area. Accordingly, this study will characterize COI concentrations in plant tissue collected from tribal allotments where prior soil studies have measured a range of soil lead concentrations. Data collected during this study will be used to inform the HHRA.

A7.1.1 Team Members and Roles

Team members and their roles are described in Section A4.2 of this QAPP.

A7.1.2 Schedule

A field reconnaissance event was conducted by TAI with EPA oversight from August 14-18, 2017 to provide information necessary to refine DQOs for this study. It is anticipated that the field-collection portion of work will be conducted in three phases: the first during mid-April to early May 2018, the second during late June 2018, and the third during late August 2018. These time periods are needed to enable collection of plants that mature and are harvested during different times of the growing season (Lodestone 2017b).

Preliminary analytical chemistry results will be available approximately 60 days after ALS has received samples. Laboratory QA and data validation will be completed approximately 30 days after preliminary analytical results are available. Thus, validated data will be delivered to EPA within 90 days of completion of all field sampling activities as required in the Agreement (USEPA 2006a).

A7.2 Step 2—Identify the Goal of the Study

The primary goal of this study is to collect data to characterize the levels of lead, arsenic, and other metals in wild plant tissues within the study area that are consumed or mouthed or otherwise utilized from the Site by CCT members. The data will be used in the evaluation of potential risk to people. The principal study question to be addressed by this work is:

- Does exposure to total concentrations of TAL metals in wild plant tissues pose unacceptable risk to human consumers?

A secondary study question to be addressed by this work is:

- Do the chemical concentrations of TAL metals in wild plant tissues collected across a range of soil lead concentrations vary with concentrations of TAL metals in soil?

A7.3 Step 3—Identify Information Inputs

The third step of the DQO process (USEPA 2006b) requires consideration of the following:

- Types and potential sources of information (e.g., site characteristics or properties) that should be measured to provide estimates or resolve decisions
- Information to provide a basis for specifying performance or acceptance criteria
- Information on the performance of appropriate sampling and analysis methods.

Sources of information that may inform the study design include:

- Parts of plants consumed or mouthed or otherwise utilized by CCT members, plant preparation considerations, and seasonal use and harvesting information (Westat 2012; Lodestone 2016a,b and 2017a,b)
- 2017 Plant Tissue Study reconnaissance results (AECOM 2017)
- COI and other data from prior UCR RI/FS soil studies (Integral 2014; Windward et al. 2015; CH2M HILL 2016; Ramboll Environ 2017b)
- Soil Amendment Technology Evaluation Study (SATES) Phase 1 soil data (preliminary)
- Sample mass requirements from analytical laboratory

Data needs identified for this study include:

- COIs (TAL metals[4] except calcium, magnesium, potassium, and sodium) in plant tissues and co-located soil samples collected from sample areas (SAs) representing a range of soil lead concentrations
- Moisture content and total mass in plant tissues analyzed for COIs noted above.

[4] For selected plant tissues (kinnikinnick leaves, wild rose leaves and stems, wild mint, willows, and tules only) and co-located soil samples, total mercury analyses will also be conducted if sufficient plant tissue mass is available to support the additional mercury analysis. Where the quantity of plant material is limited, allocation of sample mass collected will be prioritized for analysis of TAL metals (except calcium, magnesium, potassium, and sodium).

The following subsections describe the SAs and numbers of samples, the chemicals that will be analyzed, and the types of plants targeted for collection in the study.

A7.3.1 SAs and Number of Samples

Evaluation of potential risks to human consumers requires that representative data be collected within areas appropriate for assessing potential risks to these consumers. The specific uses of these data will be determined in the baseline HHRA. CCT members utilizing wild plant resources from the north half of the Site[5] have been identified as the primary consumers of interest in this study. For CCT consumers, potential risk will be evaluated for areas where potential uptake of COIs from soil by wild plants is likely to result in the highest concentrations of COIs in plant tissues. Human foraging for and consumption of wild plants at other locations within the Site that are outside the boundaries of the targeted SAs may also occur.

Consequently, in June 2017, EPA directed TAI to conduct a study that will primarily be focused on collection of plant tissue from the three tribal allotments sampled in the 2014 Residential Soil Study (CH2M HILL 2016) that had bioavailability-adjusted incremental composite sample concentrations of lead in soil greater than 700 mg/kg (2014R-258, 2014R-401, and 2014R-441). EPA's June 2017 letter further specified that plant tissues from a reference area should be sampled; however, reference areas for the Site have not yet been determined. Therefore, based on further consultation with EPA during study planning, EPA approved collecting plant samples from the Site on tribal allotments where prior RI/FS soil studies have been collected and represent a range of lower soil lead concentrations as an alternative to the specified reference area. In addition, because the CCT has identified willows as a plant of cultural significance, and the species identified were not present on the three high lead tribal allotments or other lower lead tribal allotments surveyed during the August 2017 field reconnaissance phase of this study, potential SAs also include two areas of the Site that are located along the UCR and where sediment sampling was conducted as part of the RI/FS. While these additional SAs are not located on tribal allotments, willows grow in these additional areas and each potential SA is publicly accessible.

[5] EPA (2017b) defines the "north half" as "the area located between the U.S. Canada border and the (present day) northern boundary of the CCT Reservation, which previously extended up to the international border and includes area of highest soil contamination located 40-60 miles and one to two hours' drive from the closest point on the reservation."

Plant samples will be collected from the three high lead SAs and up to 13 additional lower lead SAs in the Site (Map A7-1 and Table A7-1). Maps A7-2 through A7-17 provide detailed aerial images of these SAs, along with other relevant information used to define the SAs (i.e., locations of previously sampled soil or sediment decision units, test plot locations for the SATES, etc.). Section B1.1 of this QAPP and Table A7-1 provide more detailed rationale for inclusion of these SAs in this study.

Specific sampling locations within each SA will be determined by the field crew based on the presence of targeted plant tissues sufficient to meet mass requirements for sample analysis. For each targeted plant tissue, the collection of sufficient mass from six individual plants will be targeted from the high lead SAs to address the principal study question. A co-located soil[6] sample will be collected with each plant tissue sample. In addition, as available, collection of sufficient mass from six individual plants will also be targeted from one or more lower lead SAs. These samples will be used to address the secondary study question; however, if a particular target species is only identified at a lower lead SA, or the measured soil lead concentration reported by the laboratory for a lower lead SA is higher than the concentrations measured at the high lead SAs[7], these samples may also be used to address the principal study question. Thus, a total of six high lead SA plant tissue and co-located soil samples and six lower lead SA plant tissue and co-located soil samples will be targeted for each plant tissue during this sampling effort (Table A7-2). Although collection of six samples per plant target is the goal, EPA has determined that three samples for each targeted tissue from the high lead SAs will be sufficient to support the HHRA.

While the objective is to collect each plant tissue sample from an individual plant, a composite sample of adjacent individual plants may be collected if insufficient mass is available from an individual plant. If mass limitations at an individual plant necessitate collection of a composite plant tissue sample, the co-located soil sample will also be collected as a composite of co-located soil samples for each individual plant contributing to the composite plant tissue sample.

Although the study will seek to obtain sufficient mass for six plant tissue samples of each tissue targeted, collection will not be limited to one tissue sample from an individual plant

[6] Throughout the remainder of this document, references to soil may include sediment, as applicable to a specific SA.

[7] Based on preliminary soil data collected as part of the SATES and the 2014 and 2016 UCR residential soil studies, variability in metals concentrations measured at discrete soil sample locations is expected to be greater than variability in metals concentrations measured in incremental composite soil samples collected over an entire sampling area.

if available plant material is sufficient to collect two or more additional samples for analysis as a field replicate or a potential EPA laboratory split sample.

A7.3.2 Chemical Analyses

Analyses of plant tissue and co-located soil samples for TAL metals (except calcium, magnesium, potassium, and sodium) will be conducted to support the HHRA (USEPA 2016; Johnson 2017). For selected plant tissues (kinnikinnick leaves, wild rose leaves and stems, wild mint, willows, and tules only) and co-located soil samples, total mercury analyses will also be conducted if sufficient plant tissue mass is available to support both types of analyses. Where the quantity of plant material is limited, allocation of sample mass collected will be prioritized for analysis of TAL metals (except calcium, magnesium, potassium, and sodium). Moisture content and total mass will also be measured for plant tissue samples. EPA methods for chemical analyses of tissue and soil are listed in Table A7-3. All results will be reported on a dry weight basis with percent moisture reported for all samples. Performance acceptance criteria and analyses required to evaluate data quality are discussed in Section A.7.6.1.

A7.3.3 Species

Twenty-one plant species have been targeted for sampling (Table A7-4). The target plant list includes those species found during the August 2017 field reconnaissance event, spring ephemeral species, and plant species that are important to CCT but do not have habitat within the areas surveyed during the August 2017 field reconnaissance event (AECOM 2017). All targeted plant species are not expected to be available for sampling during each of the three planned sampling events or at all SAs visited during any sampling event. Collection of each target species will be limited to one of three sampling events (spring, late June, or late August) with the exception of wild rose and black tree lichen. Wild rose will be a target species for two field events: spring to collect leaves and stems, and August to collect rose hips.[8] Black tree lichen will be targeted for sample collection during each field event or until mass requirements have been met or collection threshold has been reached.

[8] Therefore, a total of 22 plant tissues will be targeted for sampling.

A7.4 Step 4—Define the Boundaries of the Study

This step specifies the population of interest for the study, the geographical boundaries of the Site (including a description of the reference areas), and any temporal considerations that may be required.

A7.4.1 Target Populations for Risk Evaluation

CCT members utilizing wild plant resources from the north half of the Site have been identified as the primary consumers of interest in this study. As discussed in Section A5, CCT members consume and otherwise utilize (e.g., for weaving) terrestrial, wetland, and aquatic plants that may be contaminated with heavy metals. EPA (USEPA 2017b) has determined that it is reasonable to assume that tribal members living in the north half of the Site will obtain common plants close to their residence, and that plants documented to have been used by survey respondents will likewise be used by their north half counterparts. Other local residents and visitors to the UCR area may also utilize these resources.

A7.4.2 Geographic Boundaries of the Site

The Site, as defined in Section A4.1 of this document, is the areal extent of hazardous substances contamination within the United States in or adjacent to the Upper Columbia River, including the Franklin D. Roosevelt Lake, from the U.S.-Canada border to the Grand Coulee Dam, and those areas in proximity to the contamination that are suitable and necessary for implementation of response action. Within the Site, plant tissue samples and co-located soil samples will be collected from a subset of tribal allotments where prior RI/FS sampling efforts indicate a range of soil lead concentrations (including the three highest bioavailability-adjusted soil lead concentrations), and where target plant tissues of interest (or suitable habitat) were recorded during prior field reconnaissance efforts by CCT and TAI. Two additional SAs identified for this study are not located on tribal allotments, but are publicly-accessible areas that were sampled as part of the 2014 Upland Soil Study (Windward et al. 2015) and 2010 Beach Sediment Study (Integral 2014). These additional non-tribal areas are included to address the CCT's interest in collecting willows, which are not located on the tribal allotments previously sampled. The locations of all proposed SAs are shown on Map A7-1.

A7.4.3 Temporal Considerations

The appropriate time for collecting each plant species depends on the growing season for the target plant species and the target plant part. This information is compiled in Table A7-4 based on typical CCT plant collection times (Lodestone 2017b). Sample

collection will be conducted during three field sampling events in 2018: mid-April to early May, late June, and late August.

A7.5 Step 5—Define the Statistics and Types of Inferences

Step 5 of the DQO process provides data analysis approaches that will be used to evaluate the data and draw conclusions concerning plant tissue and co-located soil chemical concentrations, and to evaluate the associated unacceptable risks to people who consume and/or otherwise utilize wild plants from the Site. It is necessary to have a general understanding of the types of data analyses that will be conducted to ensure that the required parameters are measured, and that a sufficiently large data set is developed to provide the desired level of confidence in the statistics. This approach will ensure the generation of a data set that will be adequate for use in conducting the HHRA.

As discussed in Section A.7.3.1, the chemicals to be analyzed in tissue samples collected to support the HHRA are TAL metals, excluding calcium, magnesium, potassium, and sodium. EPA methods for chemical analyses of tissue are listed in Table A7-3, and the analytical concentration goals (ACGs) are presented in Table A7-5. As discussed in Section B5, data quality and conformance will be evaluated through third-party data validation of data quality indicators and laboratory QC procedures.

Across three high lead SAs (SA01, SA02, SA03; Map A7-1), six plant tissue and co-located soil samples are targeted for collection for each of the targeted plant tissues specific to a given field sampling event (Table A7-2). EPA has determined that a minimum of three high lead SA plant tissue and co-located soil samples for each target tissue will be necessary to support data analysis for the HHRA. Additionally, as available, six samples of each event-specific targeted plant tissue and co-located soil samples will also be collected across one or more of the other SAs (SA04 through SA16; Map A7-1). EPA has determined that a minimum of three plant tissue and co-located soil samples for each target tissue at these SAs will also be necessary to support data analysis for the HHRA.

A plant tissue sample will consist of sufficient sampling mass collected from available plant material at an individual plant or, if necessary, a composite sample of sufficient sampling mass made up of plant material from two or more adjacent individual plants. A co-located surface soil sample (0 to 3 in. below ground surface) for an individual plant tissue sample will be collected from beneath the sampled plant. This depth interval was selected because it is heavily used by plant root hairs, it is the most likely interval for people to come in contact with, and it is consistent with prior soil sampling conducted on the tribal allotments and in the upland aerial deposition areas. If the plant tissue sample

is collected as a composite of multiple individual plants, the co-located surface soil sample will be a composite made up of subsampled surface soil collected from beneath each of the individual plants included in the composite tissue sample.

The ALS laboratory methods for tissue and soil sample processing are discussed in ALS SOPs in Appendix B. Exposure point concentrations used in the HHRA for each non-lead chemical will be calculated as the 95th percentile upper confidence limit on the mean from the selected set of tissue and soil samples (e.g., the three to six tissue and co-located soil samples per target tissue in the highest lead SAs). For lead, the exposure point concentrations will be calculated at the arithmetic mean for the selected set of samples for each medium. From the three to six tissue and co-located soil samples per target tissue across all SAs, summary statistics (range, mean, median, etc.) for measured plant tissue and soil concentrations of each chemical and target plant tissue will be generated, and a statistical evaluation conducted, if possible, of the relationship between soil concentration and plant tissue concentration. In the event that fewer than three tissue samples of a target tissue are collected during the study, the maximum tissue concentration may be used as the exposure point concentration if the samples are judged to be adequately representative of expected exposure conditions. Alternatively, data for similar plants and tissues may be grouped prior to calculating exposure point concentrations.

A7.6 Step 6—Specify Performance or Acceptance Criteria

The goal of Step 6 is to define performance or acceptance criteria to minimize the possibility of either making erroneous conclusions or failing to keep uncertainty in estimates to within acceptable levels (USEPA 2006b). For this study, performance and acceptance criteria will apply to generating appropriate and acceptable data for use during risk assessment activities.

A7.6.1 Sampling Completeness

Collection success or sample size of target plant tissues cannot be reliably determined *a priori*. Target plants may be irregularly distributed across SAs representing a range of soil lead concentrations, or may not have the targeted tissue present in sufficient quantity during any one of the three sampling events. To mitigate such potential challenges, several SAs are included in the study based on prior CCT and TAI reconnaissance information regarding plant species presence and abundance as well as habitat suitability. Three field sampling events are also planned to capture several different periods of the growing season when targeted tissues are expected to be present. Reconnaissance of the three high lead SAs will be conducted at the beginning of each field sampling event to

refine the target tissue list for each event and aid in prioritization of visits to additional SAs for sample collection during that event.

The addition of mercury analysis for selected plant targets (kinnikinnick leaves, wild rose leaves and stems, wild mint, willows, and tules only) and their co-located soil samples requires more than two times the sample mass than is required for analysis of TAL metals excluding calcium, magnesium, potassium, sodium, and mercury. As described in Section 2.2 of Appendix A, the sampling team will be supported by a two-person survey team. The survey team will identify potential limitations regarding the availability of sufficient plant material for kinnikinnick leaves, wild rose leaves and stems, wild mint, willows, and tules to support analysis of mercury in addition to TAL metals. If available plant material is not expected to be sufficient to support both types of analysis, allocation of available material for analysis of TAL metals will be prioritized and the decision documented by the field team in consultation with EPA, CCT, and TAI field representatives.

The level of effort for each field sampling event is detailed in the FSP (see Section 2.2 of Appendix A). If the field sampling team cannot collect the targeted number of samples for a plant tissue target within the defined level of effort, further sampling for that target will not be attempted. Final determination of the study success will be evaluated against the DQOs.

A7.6.2 Data Quality

Sample collection must provide a sufficient mass of plant tissues and co-located soil samples for chemical analyses with the required detection limits (see Table A7-3 and Table A7-5). Determination of sufficiency of mass will be based on field observations of mass above minimum sample weights at the time of collection (see Section A7.6.1 and Appendix A, Table A2). Precision will be determined by repeatability of chemical measures in field replicate samples and EPA laboratory split samples (see below), which will require collection of additional sample mass.

DQOs are developed using EPA's DQO process (USEPA 2006b) to describe data and data quality needs. Data quality indicators, such as the precision, accuracy or bias, representativeness, completeness, and comparability (PARCC) parameters and analytical sensitivity, will be used to assess conformance of data with QC criteria (USEPA 2002a). Reporting limits and quantitation limits are included in Table A7-5.

QC samples will include equipment rinsate blanks, which will be used to identify possible contamination during sample processing in the laboratory. These blanks will be collected

at ALS by pouring deionized or distilled water over (or through) thoroughly cleaned sampling equipment and into a sample jar. Blank water will be treated similarly to tissue or soil samples as much as possible (e.g., grinding equipment will be operated during equipment blank generation). A small volume of rinse water will be used to minimize dilution of the rinsate. One equipment rinsate blank will be collected for each type of sampling equipment used on each day that samples are processed. Equipment rinsate blanks will be analyzed for TAL metals (excluding calcium, magnesium, potassium, and sodium).

Field replicate tissue and soil samples (i.e., field duplicates) will be submitted to ALS as independent field samples for analysis. The purpose of these samples is to evaluate the heterogeneity of field-collected tissue and soil samples at specific locations within a SA. Field replicate samples will be collected in the field at a target frequency of 5 percent of the total plant tissue samples collected during each field event. Field replicate samples will be collected opportunistically as individual plants are encountered with at least twice the target mass of the target tissue, enough to supply both the primary and field replicate sample. The targeted frequency of field replicate samples is not dependent upon the type of plant tissue. For each field replicate tissue sample collected, a field replicate co-located soil sample will also be collected. Field replicate samples will not be collected as composites of multiple individual plants.[9] Field replicate samples will be identified without distinction from primary field samples; thus, ALS will be "blind" to the status of field replicate samples received for processing.

EPA split samples (i.e., inter-laboratory splits) will also be prepared from the samples submitted by TAI to the laboratory with sufficient mass for analysis of the primary and laboratory split sample. Twice the target sample mass will be opportunistically collected in the field when individual plants with sufficient mass are encountered to analyze EPA split samples at a frequency of 5 percent, if possible.[10] The preparation of EPA split samples will be determined in consultation with EPA after the samples have been collected.

A matrix spike/matrix spike duplicate (MS/MSD) will be performed in the laboratory to assess the accuracy of the analyses. The MS/MSD will be performed according to the laboratory protocols and will occur at a frequency of once every 20 samples.

[9] Because of the increased mass required for analysis of mercury in addition to TAL metals, it is anticipated that available mass at individual plants targeted for collection of kinnikinnick leaves, wild rose leaves and stems, wild mint, willows, and tules may not be sufficient to support collection of field replicates.

[10] Ibid.

Method detection limits (MDLs) and method reporting limits (MRLs) are listed in Table A7-5.

A7.7 Step 7—Develop the Plan for Collecting Data

Detailed discussions of the various study components are presented in Section B1 of this QAPP. Because field sampling methods associated with this study may involve penetration and disturbance of soil or sediment, TAI and its technical team will work with potentially affected parties to assess the effects of the planned work and seek ways to avoid, minimize, or mitigate any adverse effects on historic properties as well as avoiding long-term adverse impact on plant health. A cultural resources coordination plan (Appendix C) has been prepared to provide relevant background information about Site-related cultural resources, define measures for protecting resources, and establish procedures for consulting with the appropriate state, federal, and tribal parties with interests in the cultural resources of the Site.

A8 SPECIAL TRAINING AND CERTIFICATION REQUIREMENTS

TAI has assembled a technical team with the requisite experience and technical skills to successfully complete the study. Minimum training and certification requirements for laboratory personnel are provided in the laboratory QA manual (Appendix B).

Sampling personnel will be familiar with the Site cultural resources coordination plan (Appendix C). Sampling personnel will report any materials that might be considered a cultural resource to cultural resource observers participating in the field sampling program.

A9 DOCUMENTATION AND RECORDS

This section identifies onsite and laboratory records to be maintained for this project, information to be included in project reports, data reporting format for data report packages, and document control procedures to be used. Critical records required for this study are identified below with descriptive or supporting information as appropriate. Records will include documents and electronic deliverables related to field sampling (field logbook, field forms, COC forms, etc.), as well as chemistry laboratory documentation (laboratory records, data packages, project reports, electronic deliverables, etc.), data validation, and data reports. Data reports will be made available through integration into the project database web tool. Briefly, this will be an electronic data management system that is accessible via an external website. The QAPP, FSP (Appendix A), site health and safety plan (SHSP) (TCAI 2007), and the general SHSP

addendum (Attachment A1 to Appendix A) will be provided to each person listed in Section A3 (distribution list). Any revisions or amendments to any of the documents that comprise the FSP will also be provided to these individuals.

A9.1 Field Documentation

The TAI technical team field supervisor will ensure that the field team receives the final approved version of the QAPP prior to the initiation of field activities. Minimum field records that will be maintained include the following:

- Field logbooks
- Photograph documentation
- Field forms
- Sample tracking/COC forms.

Additional content, information, and use of the above-listed documents are further described in the FSP (Appendix A).

A9.2 Chemistry Laboratory

Analyses for metals and conventional parameters will be conducted by ALS. Full laboratory data reports will be provided in electronic format to the task QA coordinator, who will oversee data verification and validation, as well as archiving the final data and data quality reports in the project file. EDDs will be prepared in spreadsheet format and will be compatible with the project database.

ALS will provide a data package for each sample delivery group or analysis batch that is comparable in content to a full Contract Laboratory Program (CLP) package. Documentation requirements are detailed in the analytical laboratory QA manual (Appendix B) and will, at a minimum, include the following:

- A cover letter discussing analytical procedures and any difficulties that were encountered
- A case narrative referencing or describing the procedures used and discussing any analytical problems and deviations from SOPs and this QAPP
- Sample receipt and analysis dates
- COC and cooler receipt forms
- Copies of sample processing documentation, including supporting documentation for sample extraction and digestion and analysis of percent moisture

- Final analyte concentrations, detection limits, and reporting limits
- Laboratory data qualifier codes appended to analyte concentrations, as appropriate, and a summary of code definitions
- Sample preparation, digestion, extraction, dilution, and cleanup logs
- Instrument run logs
- Initial and continuing calibration data, including instrument printouts and quantification summaries, for all analytes
- Results for method and calibration blanks
- Results and control limits for all applicable method QC checks, including laboratory control samples (LCSs; including blank spikes and standard reference materials [SRMs]), MS/MSDs, serial dilutions, interference checks, internal standards, recovery standards, surrogates, and any other QC procedures required by applicable method protocols and laboratory SOPs
- Original data quantification reports, printouts of mass spectra, and manual integration identification and reason codes for all analyses and samples, as applicable
- All laboratory worksheets and standards preparation logs
- A page of example calculations for each analytical method included in the data package
- A documented data deliverable for each analytical method performed and reported.

The analytical chemistry laboratory coordinator will oversee data verification and validation, and the data validator will be automatically notified via the web tool database (http://teck-ucr.exponent.com) that the data set is available and ready for review. Further details of data handling are in Section D.

A9.3 Data Quality Documentation

Data verification (i.e., confirming the accuracy and completeness of field and laboratory data) will be performed by the TAI technical team for data generated in the field, and by the laboratory for the analytical data that they generate. Data validation and data quality assessment for this task will be completed and results provided to the task QA coordinator.

Accuracy of the laboratory EDDs will be verified by, or under the direction of, the database administrator. All changes to data stored in the database will be recorded in the

database change log. Any data tables prepared from the database for data users will include all qualifiers that were applied by the laboratory and during data validation.

Data validation reports will be prepared and provided to the analytical chemistry laboratory QA manager. Any limitation to the usability of the data will be discussed in this report. Completed data validation checklists will also be provided to the task QA coordinator by the data validator.

SECTION B: DATA GENERATION AND ACQUISITION

B1 SAMPLING PROCESS DESIGN AND RATIONALE

This section presents the detailed design and rationale for the plant tissue study that will result in a data set that supports assessing potential risk to human receptors.

B1.1 Target Sampling Areas and Rationale

SAs within the Site (Map A7-1) were selected based on the following considerations:

1) Parts of plants consumed or mouthed or otherwise utilized by CCT members, plant preparation considerations, seasonal use and harvesting information (Westat 2012; Lodestone 2016a,b and 2017a,b)
2) 2017 Plant Tissue Study reconnaissance results (AECOM 2017)
3) COI and other data from prior UCR RI/FS soil studies (Integral 2014; Windward et al. 2015; CH2M HILL 2016; Ramboll Environ 2017b).

Plant samples will be collected from the three high lead SAs and up to 13 additional lower lead SAs in the Site (Maps A7-2 through A7-17). Average soil lead and soil arsenic concentrations reported for prior UCR RI/FS soil and sediment sampling studies at locations that overlap these SAs are summarized in Table A7-1. The rationale for selecting each of the SAs is also provided in Table A7-1 and summarized below:

- **SA01 (Map A7-2).** SA01 is one of the three high lead SAs. Sarvisberry, kinnikinnick, black tree lichen, ponderosa pine, chokecherry, wild rose, camas, and wild strawberry were recorded during the August 2017 field reconnaissance, as well as habitat for bitterroot, Lomatium, morel, shaggy mane, spring beauty, and Indian carrot.
- **SA02 (Map A7-3).** SA02 is one of the three high lead SAs. Sarvisberry, kinnikinnick, ponderosa pine, chokecherry, and hazelnut were recorded during the August 2017 field reconnaissance, as well as habitat for morel.
- **SA03 (Map A7-4).** SA03 is one of the three high lead SAs. Sarvisberry, kinnikinnick, ponderosa pine, chokecherry, wild rose, hazelnut, and wild strawberry were recorded during the August 2017 field reconnaissance, as well as habitat for morel.
- **SA04 (Map A7-5).** SA04 is one of the 13 lower lead SAs. Sarvisberry, kinnikinnick, ponderosa pine, chokecherry, wild rose, hazelnut, dwarf huckleberry, and wild

strawberry were recorded during the August 2017 field reconnaissance, as well as habitat for bitterroot, Lomatium, morel, shaggy mane, and Indian carrot.

- **SA05 (Map A7-6).** SA05 is one of the 13 lower lead SAs. Sarvisberry, kinnikinnick, black tree lichen, ponderosa pine, chokecherry, and wild rose were recorded during the August 2017 field reconnaissance, as well as habitat for bitterroot, Lomatium, shaggy mane, and Indian carrot.
- **SA06 (Map A7-7).** SA06 is one of the 13 lower lead SAs. Sarvisberry, kinnikinnick, wild rose, and hazelnut were recorded during the August 2017 field reconnaissance, as well as habitat for shaggy mane.
- **SA07 (Map A7-8).** SA07 is one of the 13 lower lead SAs. Sarvisberry, black tree lichen, ponderosa pine, chokecherry, and wild rose were recorded during the August 2017 field reconnaissance, as well as habitat for bitterroot, Lomatium, shaggy mane, and Indian carrot.
- **SA08 (Map A7-9).** SA08 is one of the 13 lower lead SAs. Sarvisberry, black tree lichen, ponderosa pine, chokecherry, wild rose, and hazelnut were recorded during the August 2017 field reconnaissance, as well as habitat for bitterroot, Lomatium, shaggy mane, and Indian carrot.
- **SA09 (Map A7-10).** SA09 is one of the 13 lower lead SAs. Sarvisberry, ponderosa pine, chokecherry, wild rose, red willow, hazelnut, and wild strawberry were recorded during the August 2017 field reconnaissance, as well as habitat for shaggy mane, spring beauty, and Indian carrot.
- **SA10 (Map A7-11).** SA10 is one of the 13 lower lead SAs. Sarvisberry, kinnikinnick, ponderosa pine, puffball, and wild rose were recorded during the August 2017 field reconnaissance, as well as habitat for bitterroot, Lomatium, shaggy mane, and Indian carrot.
- **SA11 (Map A7-12).** SA11 is one of the 13 lower lead SAs. Sarvisberry, black tree lichen, ponderosa pine, wild rose, and hazelnut were recorded during the August 2017 field reconnaissance, as well as habitat for bitterroot, Lomatium, shaggy mane, and Indian carrot.
- **SA12 (Map A7-13).** SA12 is one of the 13 lower lead SAs. Sarvisberry, black tree lichen, chokecherry, and hazelnut were recorded during the August 2017 field reconnaissance, as well as habitat for shaggy mane, spring beauty, and Indian carrot.

- **SA13 (Map A7-14).** SA13 is one of the 13 lower lead SAs. Sarvisberry, chokecherry, and wild rose were recorded during the August 2017 field reconnaissance, as well as habitat for bitterroot, Lomatium, shaggy mane, and Indian carrot.
- **SA14 (Map A7-15).** SA14 is one of the 13 lower lead SAs. Sarvisberry, black tree lichen, wild rose, hazelnut, wild mint, and tule were recorded during the August 2017 field reconnaissance, as well as habitat for bitterroot, Lomatium, shaggy mane, and Indian carrot.
- **SA15 (Map A7-16).** The location represented by SA15 was not visited during the August 2017 field reconnaissance. This SA was added based on the possible presence of willows in a previously sampled area with moderately high concentrations of lead in relict floodplain soil.
- **SA16 (Map A7-17).** The location represented by SA16 was not visited during the August 2017 field reconnaissance. This SA was added based on the possible presence of willows in a previously sampled area with low concentrations of lead in beach sediment.

Collection of each target species will be limited to one of three sampling events (spring 2018, late June 2018, or late August 2018) with the exception of wild rose and black tree lichen. Wild rose will be a target species for two field events: spring to collect leaves and stems, and August to collect rose hips. Black tree lichen will be targeted for sample collection during each field event or until mass requirements have been met or collection threshold has been reached. Field collection teams will collect plant tissues by hand. The precise location of sampled plants will be recorded via a hand-held global positioning system unit.

For each targeted plant tissue, the collection of sufficient mass from six individual plants will be targeted from the high lead SAs to address the principal study question. A co-located soil sample will be collected with each plant tissue sample. In addition, as available, collection of sufficient mass from six individual plants will also be targeted from one or more lower lead SAs. These samples will be used to address the secondary study question; however, if a particular target species is only identified at a lower lead sampling area, or the measured soil lead concentration reported by the laboratory for a lower lead sampling area is higher than the concentrations measured at the high lead sampling areas, these samples may also be used to address the principal study question. Thus, a total of six high lead sampling area plant tissue and soil samples and six lower lead sampling area plant tissue and soil samples will be targeted for each plant tissue during this sampling

effort (Table A7-2). Specific sampling locations within each sampling area will be determined by the field crew based on the presence of targeted plant tissues sufficient to meet mass requirements for sample analysis (Table A7-3).

Ideally, each plant tissue sample will come from one individual plant with a co-located soil sample taken from directly below the plant. When possible, individual samples will be taken from physically distant individuals of the same species. Physically distant plants are less likely to be closely genetically related, are less likely to share nutrients through connected root tissues, and are more likely to uptake nutrients from soil with different conditions (pH, organic matter, etc.) and COI concentrations. If all individuals of a target species occur within a patch, individuals as far from one another as possible will be selected for sampling.

Some plant species may not have enough of the targeted plant material to obtain the mass required for a single sample from one individual plant. In that case, plant material may be collected from multiple adjacent individuals located in proximity and combined into a single composite plant tissue sample. Individual plants located in proximity are more likely to be genetically related, more likely to be sharing nutrients through connected root networks, and more likely to uptake nutrients from soil with similar COI concentrations and soil conditions (pH, organic matter, etc.).

A co-located surface soil sample (0 to 3 in. below ground surface) will be collected with each plant tissue sample. The soil sample location will be collected within the expected root zone of each individual plant sampled. If more than one plant species targeted for sampling are growing together at the same location, a single co-located soil sample may be collected and associated with each of the plant tissue samples collected at that location. If mass limitations at an individual plant necessitate collection of a composite plant tissue sample from more than one plant, the co-located soil sample will also be collected as a composite of co-located soil samples for each individual plant that is part of the composite plant tissue sample.

B2 SAMPLING METHODS

Field sampling methods for collection of plant tissue and soil are described in the FSP (Appendix A). The FSP includes the following topics:

- Sampling location positioning (Section 2.2.2)
- Field equipment and supplies (Section 2.2.3)
- Sample collection methods (Section 2.2.4)

- Sampling contingencies (Section 2.2.5)
- Location and sample event identification (ID) (Section 2.2.9)
- Sample IDs for individual and composite samples (Section 2.2.10)
- Equipment decontamination procedures (Section 2.2.11)
- Sample handling (Section 2.3)
- Cultural resources (Section 2.4)
- Sample packaging and transport (Section 2.5)
- Study-derived waste (Section 2.6)
- Field documentation and procedures (field logbooks, photograph documentation, COC forms) (Sections 3.1 and 3.2).

SOPs for each sampling method are provided in Attachment A2 of the FSP (Appendix A).

In the event that unanticipated or changed circumstances occur in the field, the field supervisor, in consultation with EPA or its representatives in the field, will institute the necessary corrective actions, complete a corrective action record, and ensure that the appropriate procedures are followed. If corrective actions require a departure from the FSP, these changes will be documented on a field change request form (refer to Attachment A3 of Appendix A for examples of these and other forms) and submitted to the TAI and EPA project coordinators for review and approval. In any other circumstances where sampling conditions are unexpected, the appropriate sampling actions consistent with this task's objectives will be conducted. This change will be noted by the field supervisor in the field logbook, and a change request form will be completed for the project files and submitted to EPA. Any problems that cannot be easily resolved or that affect the final quality of the work product will be brought to the attention of the principal investigator, TAI project coordinator, and EPA (including EPA's representatives in the field). EPA will be notified of any problems that may affect the final outcome of this task. Additional information regarding corrective actions and related documentation is provided in Section C1.

B3 SAMPLE HANDLING AND CUSTODY

Principal documents used to identify samples and to document possession will be field logbooks, field forms, and COC records. Custody will be documented for all samples at all stages of the collection or transfer process. COC procedures for sample handling prior to delivery to the laboratory are outlined in the FSP (Appendix A).

Soil and plant tissue samples will be transported from the field to the analytical chemistry laboratory via priority overnight delivery service or courier service. Requirements for storage temperature and holding times are summarized in Table A7-3 and detailed in the FSP (Appendix A). For soil samples submitted for analysis of TAL metals except mercury, the EPA Regional Quality Assurance Manager has approved storage in the field and transport of the soil samples without refrigeration or use of ice (Tonel 2017). Soil samples submitted for analysis of mercury in addition to TAL metals will be stored in the field and transported to the laboratory on ice or with refrigeration. Upon receipt of samples at the laboratory, the physical integrity of containers and custody seals will be checked, and samples will be inventoried by comparing sample labels to those on the COC forms. The laboratory will include the COC and shipping container receipt forms in the data package. Any breaks in the COC or non-conformances will be noted and reported in writing to the analytical chemistry laboratory coordinator within 24 hours of receipt of samples. A laboratory-specific QA manual is provided in Appendix B. The analytical chemistry laboratory project manager will ensure that a sample-tracking record is maintained that follows each sample through all stages of sample processing at the laboratory. Samples will be stored in accordance with protocols listed in Table A7-3 (soil samples will be stored at room temperature or on ice/with refrigeration, depending on specific chemical analyses ordered, and plant tissue samples will be stored frozen to approximately -20°C). The laboratory will maintain COC records and documentation of proper storage conditions for the entire time that the samples are in their possession. The laboratory will not dispose of samples from this task until receiving written authorization to do so by EPA. After authorization is obtained, the laboratory will dispose of samples, as appropriate, based on matrix, analytical results, and information received from the client.

B4 SAMPLE PROCESSING AND ANALYTICAL METHODS

In the field, plant tissue will be enclosed in resealable plastic bags, labeled, placed in a second resealable plastic bag, and stored in coolers with wet ice. Plants will be kept on wet ice for no longer than 20 hours before being frozen in a freezer or in a cooler with dry ice. Plant tissue samples will be shipped via priority overnight delivery service or courier service on dry ice to ALS, where they will be stored at -20°C until processing. At ALS, tissues will be processed according to ALS SOPs (Appendix B) and procedures specified in Table A7-3.

Sub-aliquots of freeze-dried tissues will be prepared by ALS. The benefits of using freeze-dried material are that tissue loss will be minimized during sample processing and

homogenization will be more thorough resulting in freeze-dried sub-aliquots that are more representative of the bulk sample compared to wet tissue sub-aliquots. Table A7-3 summarizes the preparation and analytical methods for each analyte.

In the field, soil samples will be sealed into wide-mouthed jars and stored in a secure area until being shipped via priority overnight delivery service or courier service to ALS, where they will be stored at room temperature or in refrigeration prior to processing by ALS according to the specific chemical analyses ordered for each soil sample, ALS SOPs (Appendix B), and procedures specified in Table A7-3. Soil samples will be dried, sieved, and subsampled in the laboratory. Physical disaggregation of soil samples will be conducted by breaking aggregates apart by hand but not through pounding with a mortar and pestle. This process is also expected to break down the dried vegetation such that the attached soil particles will be knocked loose. Grinding will not be conducted without prior approval from EPA. Samples will be air dried and passed through a No. 10 sieve (2.0 mm) in the laboratory to remove large debris (e.g., sticks, stones) present in the sample. The resulting material will be weighed and sieved through a No. 100 sieve to isolate the target particle size of <150 μm. This particle size fraction is intended to represent the fraction expected to adhere to skin via dermal contact (Ruby and Lowney 2012). No additional subsampling will be done once the laboratory subsample (2 g of <150 μm soil) is placed in the jar. If laboratory replicate samples or split samples are required from a particular sample, additional jars will be required and 2 g of soil will be placed in each jar.

B5 QUALITY CONTROL

This section describes the laboratory QC procedures and the data quality indicators that will be used to assess the conformance of data with QC criteria.

B5.1 Analytical Laboratory Quality Control

Extensive and detailed requirements for laboratory QC procedures are provided in the EPA methods that will be used for this study (see Table A7-3). Every method protocol includes descriptions of QC procedures, and many incorporate additional QC requirements by reference to separate QC sections. QC requirements include control limits and requirements for corrective action in many cases. QC procedures will be completed by the laboratory, as required in each protocol and their internal SOPs, and as indicated in this QAPP.

The frequency of the preparation and analysis of LCSs (i.e., blank spikes or SRMs), MS samples, and method blanks will be 1 for every 20 samples or 1 per extraction or analysis

batch, whichever is more frequent. Names and control limits of applicable SRMs are included in Table B5-1. Calibration procedures will be completed at the frequency specified in each method description. Equipment rinsate blanks will be created by pouring water over clean sample processing implements (e.g., cutting boards, knives, blenders, and bowls) and into a sample bottle. Blank water will be treated similarly to tissue samples as much as possible (e.g., grinding equipment will be operated during equipment blank generation). ALS will document the volume of rinsate water used to rinse the equipment, as well as the volume of rinsate water collected and used for analysis.

As required for EPA SW-846 methods (USEPA 2008), performance-based control limits have been established by the laboratory. These and all other control limits specified in the method descriptions will be used by the laboratory to establish the acceptability of the data or the need for reanalysis of the samples. Laboratory control limits for recoveries of QC samples applicable to each method (e.g., LCSs, MS/MSDs, field split samples, serial dilutions, interference checks, internal standards, recovery standards, surrogates, and any other QC required by applicable method protocols and laboratory SOPs), acceptable concentration ranges (e.g., SRMs), and for relative percent differences (RPDs) of MS/MSDs, are provided in the analytical chemistry laboratory's QA manual (Appendix B).

B5.2 Data Quality Indicators

The overall quality objective for this task is to develop and implement procedures that will ensure the collection of representative data of known and acceptable quality. QA procedures and measurements that will be used for this task are based on EPA guidance. Data quality indicators such as the PARCC parameters and analytical sensitivity will be used to assess the conformance of data with QC criteria (USEPA 2002b). Measurement quality objectives (MQOs) for the quantitative PARCC parameters are provided in Table B5-2. Data quality indicators and QC objectives are described in this section.

Precision reflects the reproducibility between individual measurements of the same property. Precision will be evaluated using the results of laboratory replicates. Precision is expressed in terms of the RPD for two measurements. The following equation is used to calculate the RPD between measurements:

$$RPD = \frac{|C_1 - C_2|}{(C_1 + C_2)/2} \times 100$$

Where: RPD = relative percent difference

C_1 = first measurement
C_2 = second measurement

For three or more measurements, the relative standard deviation (RSD) is used to evaluate precision. The RSD is calculated as the ratio of the standard deviation of three or more measurements to the average of the measurements, expressed as a percentage.

Accuracy or bias represent the degree to which a measured concentration conforms to a reference value. Results for matrix spikes, LCSs, field blanks, and method blanks will be reviewed to evaluate accuracy or bias of the data. The following calculation is used to determine percent recovery for a matrix spike sample:

$$\%R = \frac{M - U}{C} \times 100$$

Where: %R = percent recovery
M = measured concentration in spiked sample
U = measured concentration in unspiked sample
C = concentration of added spike

Percent recovery for an LCS is calculated as follows:

$$\%R = \frac{M}{C} \times 100$$

Where: %R = percent recovery
M = measured concentration in reference sample
C = established reference concentration

When SRMs are used, laboratory results will be compared to an acceptable range of concentrations statistically generated from historical data. Applicable concentration ranges by analyte and SRM are provided in Table B5-1.

Results for equipment rinsate and method blanks can reflect systematic bias that results from contamination of samples during processing or analysis. Detection of any target analytes at concentrations greater than the MRLs in field or method blanks will be evaluated as potential indicators of bias.

QC samples and procedures are specified in each method protocol (analytical methods are presented in Table A7-3). All QC requirements will be completed by the analytical chemistry laboratory as described in the protocols, including the following (as applicable to each analysis):

- Initial calibration
- Initial calibration verification
- Continuing calibration
- Calibration or instrument blanks
- Method blanks
- Equipment rinsate blanks
- LCSs, including blank spikes and SRMs
- MS/MSDs
- Serial dilutions
- Interference checks
- Internal standards
- Recovery standards
- Surrogates.

To alert data users of possible bias or imprecision, data qualifiers will be applied to reported analyte concentrations when associated QC samples or procedures do not meet laboratory internal control limits (Appendix B).

ACGs provide the target concentration required for the chemical analysis. Methods selected for this study are expected to provide sufficient sensitivity to yield ACGs for most chemicals that are below the lowest reference value for this study (Table A7-5). For six chemicals, targeted plant tissue ACGs exceed the human health risk-based concentration for plant ingestion. The risk-based concentrations for both media were derived using highly conservative exposure assumptions for a traditional tribal scenario. EPA has determined that the targeted ACGs are suitable for use in this study. Potential uncertainty related to targeted ACGs that exceed risk-based screening concentrations will be considered by EPA, as needed, in development of the human health risk assessment.

ALS has determined an MDL for each analyte, as required by EPA (USEPA 2010). MDLs are statistically derived and reflect the concentration at which an analyte can be detected in a clean matrix with 99 percent confidence that a false positive result has not been reported. ALS established MRLs at levels above the MDLs. The MRLs are based on the laboratory's experience analyzing environmental samples and reflect the typical sensitivity obtained by the analytical system; they represent the level of analyte above which concentrations are accurately quantified. MDLs and MRLs for each analyte are summarized in Table A7-5.

ALS will quantify analytes at concentrations above the MRL. Analytes detected at concentrations between the MDL and MRL will be flagged with a "J" qualifier to indicate that the value is an estimate (i.e., the analyte concentration is greater than or equal to the MDL and less than the MRL). Analytes that are not detected will be reported as the MDL and will be flagged with a "U" qualifier. MDLs and MRLs will be adjusted by ALS as necessary to reflect sample dilution or matrix interference. All results will be reported on a dry weight basis (with percent moisture reported for all samples).

Representativeness is the degree to which data represent a characteristic of an environmental condition. In the field, representativeness will be addressed primarily in the sampling design by the selection of sampling sites and sample collection procedures. In the laboratory, representativeness will be ensured by the proper handling and storage of samples, the use of standard performance-based methods, and initiation of analyses within holding times.

Comparability is the qualitative similarity of one data set to another (i.e., the extent to which different data sets can be combined for use). Comparability will be addressed through the use of field and laboratory methods that are consistent with methods and procedures recommended by EPA.

Completeness is a measure of the amount of valid data obtained from the analytical measurement system and the complete implementation of defined field procedures. The target completeness objective will be 90 percent; the actual completeness may vary depending on the intrinsic nature of the samples. Completeness of the data will be assessed during QC reviews.

Completeness is defined as follows for all measurements:

$$\%C = \frac{V}{T} \times 100$$

Where: %C = percent completeness
V = number of measurements judged valid
T = total number of measurements.

B6 INSTRUMENT AND EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE

Analytical instrument testing, inspection, maintenance, setup, and calibration will be conducted by the analytical chemistry laboratory in accordance with requirements identified in the laboratory SOPs and manufacturer instructions. In addition, each of the

specified analytical methods provides protocols for proper instrument setup, tuning, and critical operating parameters. Instrument maintenance and repair will be documented in the laboratory's maintenance logs or record books.

B7 INSTRUMENT AND EQUIPMENT CALIBRATION AND FREQUENCY

Before beginning each analysis, laboratory instruments will be properly calibrated, and the calibration will be verified with appropriate check standards and calibration blanks for each parameter. Instrument calibration procedures and schedules will conform to analytical protocol requirements and descriptions provided in the laboratory's QA manual.

Calibration standards will be obtained from a commercial vendor, and the laboratory will maintain traceability back to the National Institute of Standards and Technology. Stock standards will be used to establish intermediate standards and calibration standards. Special attention will be given to expiration dating, proper labeling, proper refrigeration, and prevention of contamination. Documentation relating to the receipt, mixing, and use of standards will be recorded in a laboratory logbook. All calibration and spiking standards will be checked against standards from another source, as specified in the analytical methods and the laboratory QA manual (Appendix B).

B8 INSPECTION AND ACCEPTANCE OF SUPPLIES AND CONSUMABLES

The quality of supplies and consumables used during sample collection and laboratory analysis can affect the quality of the data. All equipment that comes into contact with the samples and extracts must be sufficiently clean to prevent detectable contamination, and the analyte concentrations must be accurate in all standards used for calibration and QC purposes.

The quality of laboratory water used will be documented at the analytical chemistry laboratory. All containers will be visually inspected prior to use, and any suspect containers will be discarded.

Reagents of appropriate purity and suitably cleaned laboratory equipment will also be used for all stages of laboratory analyses. Details of acceptance requirements for supplies and consumables at the laboratory are provided in the laboratory SOPs and QA manual (Appendix B). All supplies will be obtained from reputable suppliers with appropriate documentation or certification. Supplies will be inspected to confirm that they meet use

requirements, and certification records will be retained by the field supervisor (i.e., for supplies used in the field) or the laboratory QA manager (i.e., for supplies used in the laboratory).

B9 DATA MANAGEMENT

Data for this study will be generated both in the field and at the analytical chemistry laboratory. The final repository for sample information will be the relational database housed at http://teck-ucr.exponent.com. Procedures used to transfer data from the point of generation to the database are described in this section.

The data management plan (DMP) and its draft amendment (Exponent 2010) establish standard procedures for the management of all documents and environmental data (field and laboratory) generated during the RI/FS. The DMP describes data management procedures relating to the creation, acquisition, handling, storage, and distribution of task-related data. Data management systems and procedures described below are intended to establish and maintain an efficient organization of large volumes of complex environmental information for a diverse combination of data types. To accomplish this task, the following four management systems will be used to provide organized and efficient data management and retrieval:

- **Project database**. Stores environmental sampling and analysis data, information pertaining to geographic information system (GIS) files, and citations of documents related to collection, analysis, or interpretation of environmental data stored in the database. Both current and historical data are stored in the project database. Access to the data is password controlled, with various levels of access available to users on a "need-to-know" basis, as determined by the project manager.
- **GIS**. Stores spatial data and enables the cartographic presentation of data trends and patterns.
- **Hard copy files**. Maintains a record and archive of documents from field studies and resulting reports.
- **Website** (http://www.ucr-rifs.com). Makes available draft documents and other project information via the secure domain. Users with appropriate privileges are able to download documents.

Study activities will use spatial data sets and analyses for planning, data interpretation, decision support, and data presentation. Links between data in the project database and

GIS files will be established via common identifiers for sampling locations and other geographic features.

B9.1 Field Data

Data that are generated during the study will be manually entered into the field logbook, field data forms, and COC forms. Data from these sources will be entered into the project database directly from the field logbook and field data forms. These data include sample collection coordinates (World Geodetic System of 1984), station identification numbers, sampling dates, sample identifiers and numbers, and additional station and sample information. All entries will be reviewed for accuracy and completeness by a second individual, and any errors will be corrected before the data are approved for release to data users.

B9.2 Analytical Laboratory Data

A variety of manually entered and electronic instrument data will be generated at the laboratory. Data will be manually entered into the following:

- Standard logbooks
- Storage temperature logs
- Balance calibration logs
- Instrument logs
- Sample preparation and analysis worksheets
- Maintenance logs
- Individual laboratory notebooks.

All manual data entry into the laboratory information management system will be proofed at the analytical chemistry laboratory. Data collected from each laboratory instrument, either manually or electronically, will be reviewed and confirmed by analysts before reporting. A detailed description of procedures for laboratory data management and data review and verification is provided in the laboratory QA manual (Appendix B). Analytical data packages will be comprehensive Tier 4 CLP packages that will allow for a full Stage 4 (S4VM) data validation (reference Section D2 below for data verification and validation methods for this study).

SECTION C: ASSESSMENT AND OVERSIGHT

This task will rely on the knowledge and expertise of the TAI technical team. The field team and laboratory will stay in close verbal contact with the principal investigator and the task QA coordinator during all phases of this task. This level of communication will serve to keep the management team apprised of activities and events, and will allow for informal but continuous task oversight.

C1 ASSESSMENTS AND RESPONSE ACTIONS

Assessment activities will include readiness reviews prior to sampling and prior to release of the final data to the data users, as well as internal review while work is in progress. A technical systems audit may be conducted by either EPA or TAI if problems are encountered during any phase of this task.

Readiness reviews typically are conducted to ensure that all necessary preparations have been made for efficient and effective completion of each critical phase of work. The first readiness review will be conducted prior to field sampling. The field supervisor will verify that all field equipment is ready for transfer to the Site. The field supervisor will also verify that the field team and subcontractors, as required, have been scheduled and briefed (including review of the SHSP and the cultural resources coordination plan), and that the contract for the subcontractor has been signed by both parties. Any deficiencies noted during this readiness review will be corrected prior to initiation of sampling activities.

The second readiness review will be completed before final data are released for use. The database administrator will verify that all results have been received from the laboratory, data validation and data quality assessment have been completed for all of the data, and data qualifiers have been entered into the database and verified. Any deficiencies noted during this review will be corrected by the database administrator, the task QA coordinator, or their designees. Data will not be released for final use until all data have been verified, validated, and approved by EPA. No written report will be prepared in conjunction with the readiness reviews.

Technical review of intermediate and final work products generated for this task will be completed throughout the course of all sampling and laboratory activities, data validation, data management, and data interpretation to ensure that every phase of work is accurate and complete and follows the QA procedures outlined in this QAPP. Any problems that are encountered will be resolved between the reviewer and the person completing the work. Any problems that cannot be easily resolved or that affect the final quality of the work product will be brought to the attention of the TAI technical team

coordinator and TAI project coordinator. EPA will be notified of any problems that may affect the final outcome of this task, according to the Agreement. EPA assessment and/or oversight of sampling and laboratory processing or analysis will be conducted as directed by the EPA project manager.

The laboratory will be required to have implemented a review system that serves as a formal surveillance mechanism for all laboratory activities. Each phase of work will be reviewed by a supervisor before it is approved for release. Details are provided in the laboratory QA manual (Appendix B).

Technical system audits may be conducted if serious problems are encountered during sampling or analysis operations. If completed, these audits will be conducted by the task QA coordinator or designee, or by the laboratory, as appropriate. These audits may consist of onsite reviews of any phase of field or laboratory activities or data management. Results of any technical systems audits will be provided in the draft data summary report and verbally to the project manager.

Any task team member who discovers or suspects a non-conformance is responsible for reporting the non-conformance to the principal investigator, the task QA coordinator, or the laboratory project or QA manager, as applicable. The task QA coordinator will ensure that no additional work dependent on the non-conforming activity is performed until a confirmed non-conformance is corrected. Any confirmed non-conformance issues will be communicated to the TAI project coordinator and to EPA. In addition, during corrective actions, communication among the field personnel and the laboratory relative to the accuracy and completeness of the COC documents will follow the procedures for corrective action.

C2 REPORTS TO MANAGEMENT

The laboratory will keep the appropriate technical team coordinators and QA managers apprised of their progress on a regular basis. The laboratory will provide the following information:

- Inventory and status of samples held at the laboratory in spreadsheet format by sample delivery group
- Summaries of out-of-control laboratory QC data that resulted in a requirement for corrective action and a description of the corrective actions implemented
- Descriptions and justification for any significant changes in methodology or QA/QC procedures.

The analytical chemistry laboratory coordinator and QA manager will provide this information to the task QA coordinator who, in turn, will provide this information to the TAI technical team coordinator.

The laboratory will be required to have implemented routine systems of reporting non-conformance issues and their resolutions. These procedures are described in the laboratory QA manual (Appendix B). Laboratory non-conformance issues will also be described in the data summary report if the data validator determines that they affect any of the data quality indicators discussed in Section B5.2 of this QAPP.

Data packages and EDDs will be prepared by the analytical chemistry laboratory upon completion of analyses for each sample delivery group. The case narrative will include a description of any problems encountered, control limit exceedances (if applicable), and a description and rationale for any deviations from protocol. Copies of corrective action reports generated at the laboratory will also be included with the data package.

Validated data will be provided electronically to EPA. These data will also be provided with the data summary report containing an overview of the field event, a sampling location map, sample collection methods, and rationale for any deviations from the FSP and/or QAPP according to the Agreement.

SECTION D: DATA VALIDATION AND USABILITY

Data generated in the field and at the laboratory will be verified and validated according to criteria and procedures described in this section. Data quality and usability will be evaluated and a discussion of the findings will be included in the data validation report.

D1 DATA REVIEW, VERIFICATION, AND VALIDATION

Field and laboratory data for this task will undergo a formal verification and validation process. All entries into the database will be verified. All errors found during the verification of field data, laboratory data, and the database will be corrected and documented prior to release of the final data.

Data verification and validation will be completed according to methods described in the following EPA guidance documents for data validation and criteria in this QAPP.

- Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use (EPA 540-R-08-005, January 2009) (USEPA 2009)
- National Functional Guidelines for Inorganic Superfund Data Review (USEPA 2017c)

Data will be qualified as estimated, as necessary, if results for applicable method QC (e.g., LCSs, MS/MSDs, serial dilutions, interference checks, internal standards, recovery standards, surrogates, or any other QC required by applicable method protocols and laboratory SOPs) do not meet method-specified control limits, including performance-based control limits. Results for other QC procedures will be qualified if they do not meet control limits outlined in EPA's national functional guidelines and SOPs for data validation (USEPA 2009, 2017c). Data will be qualified as undetected based on concentrations of target analytes detected in laboratory or field blanks, according to EPA's national functional guidelines and SOPs for data validation. The data summary report will include a list of definitions of qualifiers applied by the laboratory and the data validator.

Performance-based control limits are established periodically by the laboratory as required for the selected methods. Current values will be provided in the laboratory QA manual, as applicable.

Equipment rinsate blank concentrations will be expressed on a mass basis calculated from the volume of water used to rinse the laboratory equipment and the average mass of tissue processed by the equipment. ALS will document the volume of rinsate water collected and analyzed, and the average mass of tissue used in the mass basis calculation. Data

qualifiers will be applied for equipment rinsate blanks in the same manner as method blanks, as described in the national functional guidelines for data review (USEPA 2017c). Data will be rejected if control limits for acceptance of data are not met, as described in the national functional guidelines for inorganic data review (USEPA 2017c).

D2 VERIFICATION AND VALIDATION METHODS

Field data will be verified during preparation of samples and COC forms. Field notebook entries (including field taxonomy), field data forms, and COC forms will be reviewed daily by the field supervisor or designee. After field data are entered into the project database, 100 percent verification of the entries will be completed to ensure the accuracy and completeness of the database. Any discrepancies will be resolved before the final study data are released for use.

Approximately 10 percent of the chemistry data will undergo Stage 4 (S4VM) validation. The remaining data will undergo Stage 2B (S2BVM) validation with the understanding that more detailed validation will be performed on the S2BVM data if issues are identified in the S4VM validation. If problems or questions are encountered during validation, the laboratory will be contacted for resolution. An additional full validation will be completed, if required, to fully assess the quality of the data or to verify that laboratory errors have been addressed.

Procedures for verification and validation of laboratory data and field QC samples will be completed as described in the national functional guidelines (USEPA 2017c) and SOPs for data validation and summarized in Section D1 above. Accuracy and completeness of each data set will be verified at the laboratory when EDDs are prepared and again as part of data validation. Ten percent of entries into the database from the laboratory EDDs will be checked against the hard-copy data packages. Data validation will be completed by ESI.

ESI will provide definitions of qualifiers applied by the laboratory and data validator. In addition to verification of field and laboratory data and information, data qualifier entries into the database will be verified. Any discrepancies will be resolved before the final database is released for use.

MRL goals for this task are provided in Table A7-5. Reporting limits for non-detects will be compared to the MRL goals to evaluate method sensitivity for each sample. Any exceedance of actual MRLs over the target MRLs will be discussed in the data validation report.

D3 RECONCILIATION WITH USER REQUIREMENTS

The goal of data validation is to determine the quality of each data result and to identify those that do not meet the task MQOs. Non-conforming data may be qualified as estimated (i.e., a "J" qualifier will be applied to the result) or rejected as unusable (i.e., an "R" qualifier will be applied to the result) during data validation if criteria for data quality are not met. Data may also be qualified as undetected during validation based on laboratory and field blank results. Rejected data will not be used for any purpose. A summary of the qualified data and the reasons for qualification will be included in the data validation report.

Data qualified as estimated will be used for all intended purposes and will be appropriately qualified in the final project database. However, these data are less precise or less accurate than unqualified data. Data users, in coordination with the principal investigator and task QA coordinator, are responsible for assessing the effect of the inaccuracy or imprecision of the qualified data on statistical procedures and other data uses. The data quality discussion in the data validation report will include information regarding the direction or magnitude of bias or the degree of imprecision for qualified data to facilitate the assessment of data usability. Data validation reports will also include a discussion of data limitations and their effect on data interpretation activities.

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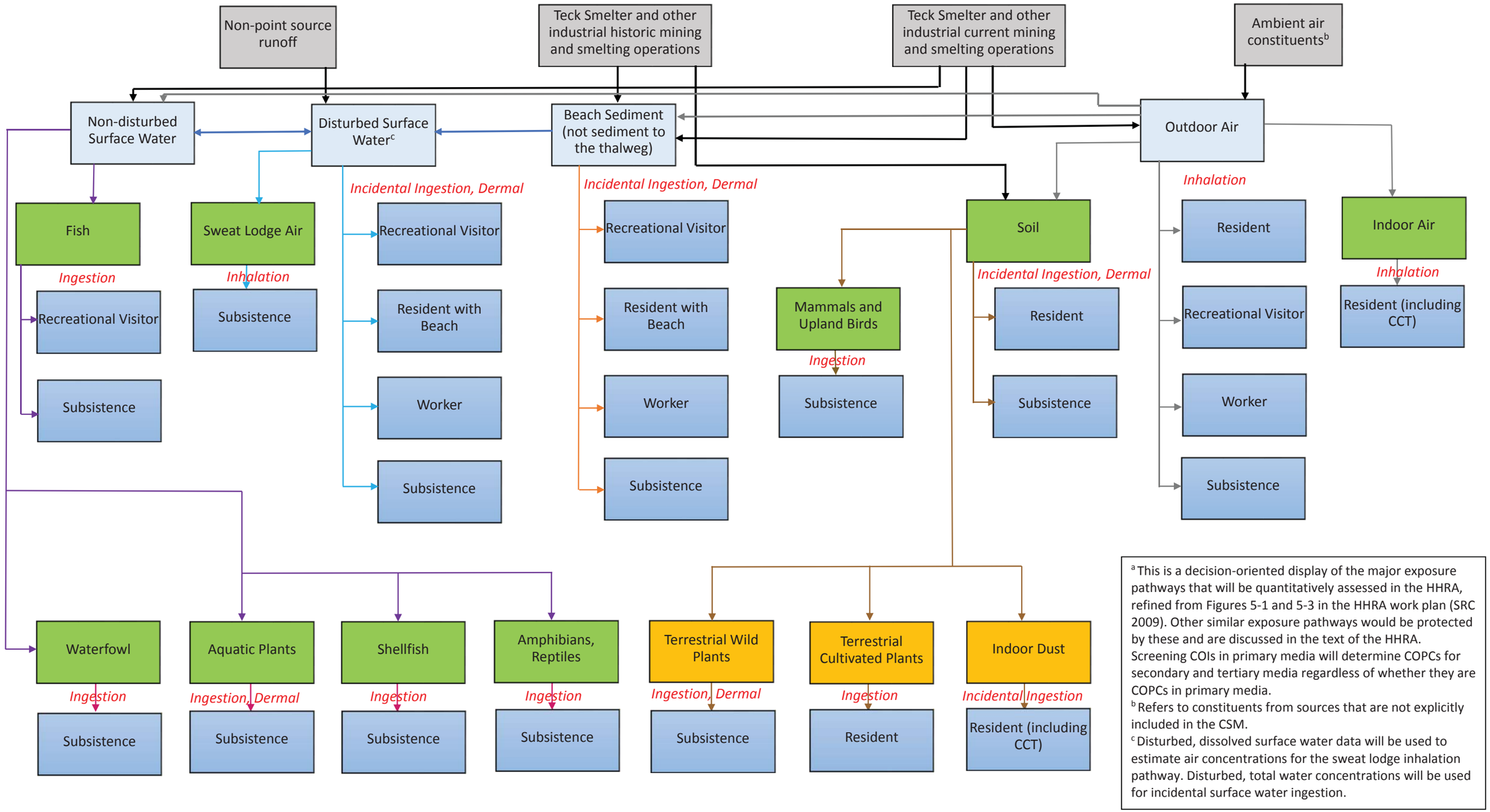
FIGURE

Quantitative Conceptual Site Model for the UCR Human Health Risk Assessment as of November 6, 2017[a]

Primary Media
Secondary Media
Tertiary Media



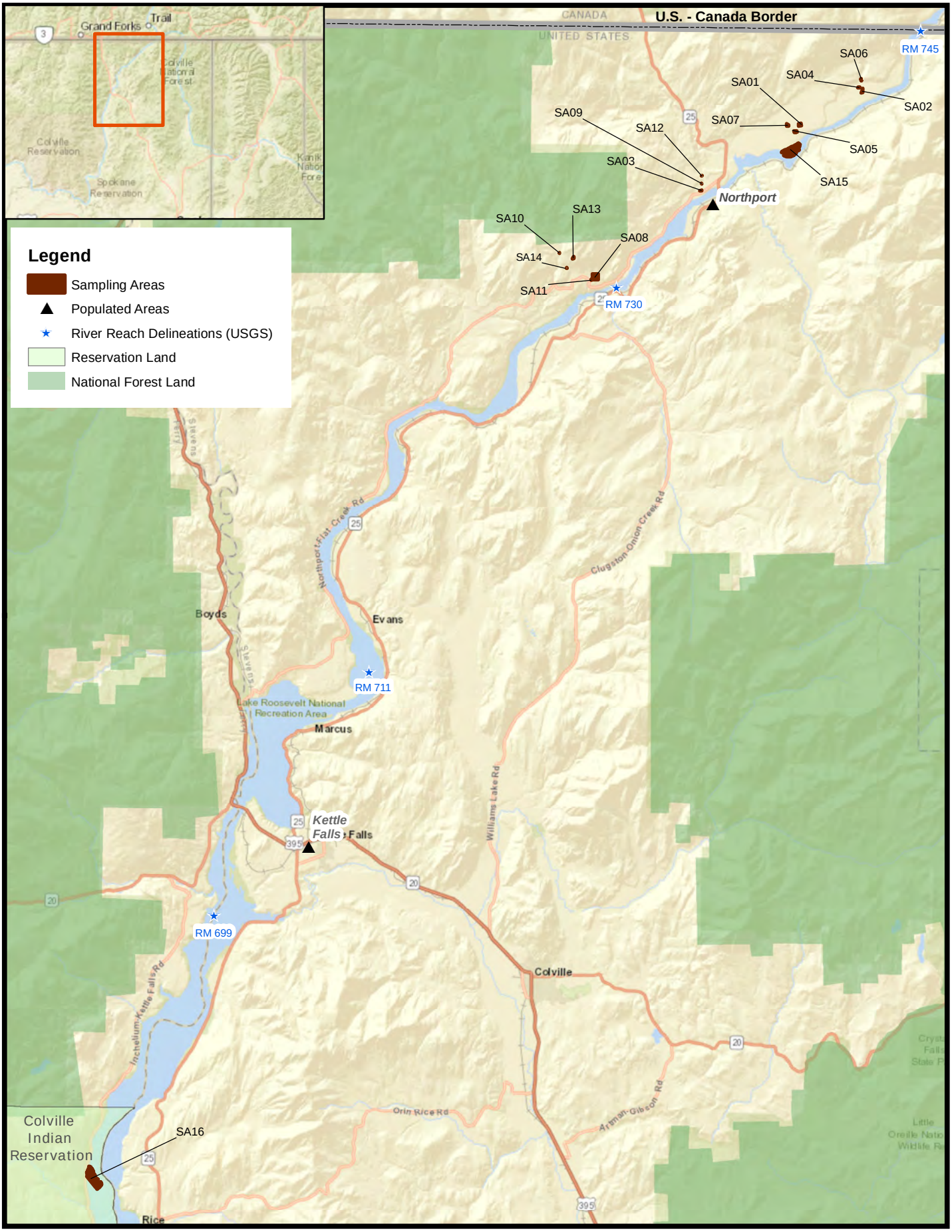
[a] This is a decision-oriented display of the major exposure pathways that will be quantitatively assessed in the HHRA, refined from Figures 5-1 and 5-3 in the HHRA work plan (SRC 2009). Other similar exposure pathways would be protected by these and are discussed in the text of the HHRA. Screening COIs in primary media will determine COPCs for secondary and tertiary media regardless of whether they are COPCs in primary media.

[b] Refers to constituents from sources that are not explicitly included in the CSM.

[c] Disturbed, dissolved surface water data will be used to estimate air concentrations for the sweat lodge inhalation pathway. Disturbed, total water concentrations will be used for incidental surface water ingestion.

Figure A5-1. Site-Wide Conceptual Site Model

Maps



RAMBOLL ENVIRON

0 1 2 4 Kilometers

0 1 2 4 Miles

N

Map A7-1. Proposed Sampling Areas
Upper Columbia River, WA

Map A7-2. Detail for Sampling Area 01
Upper Columbia River, WA

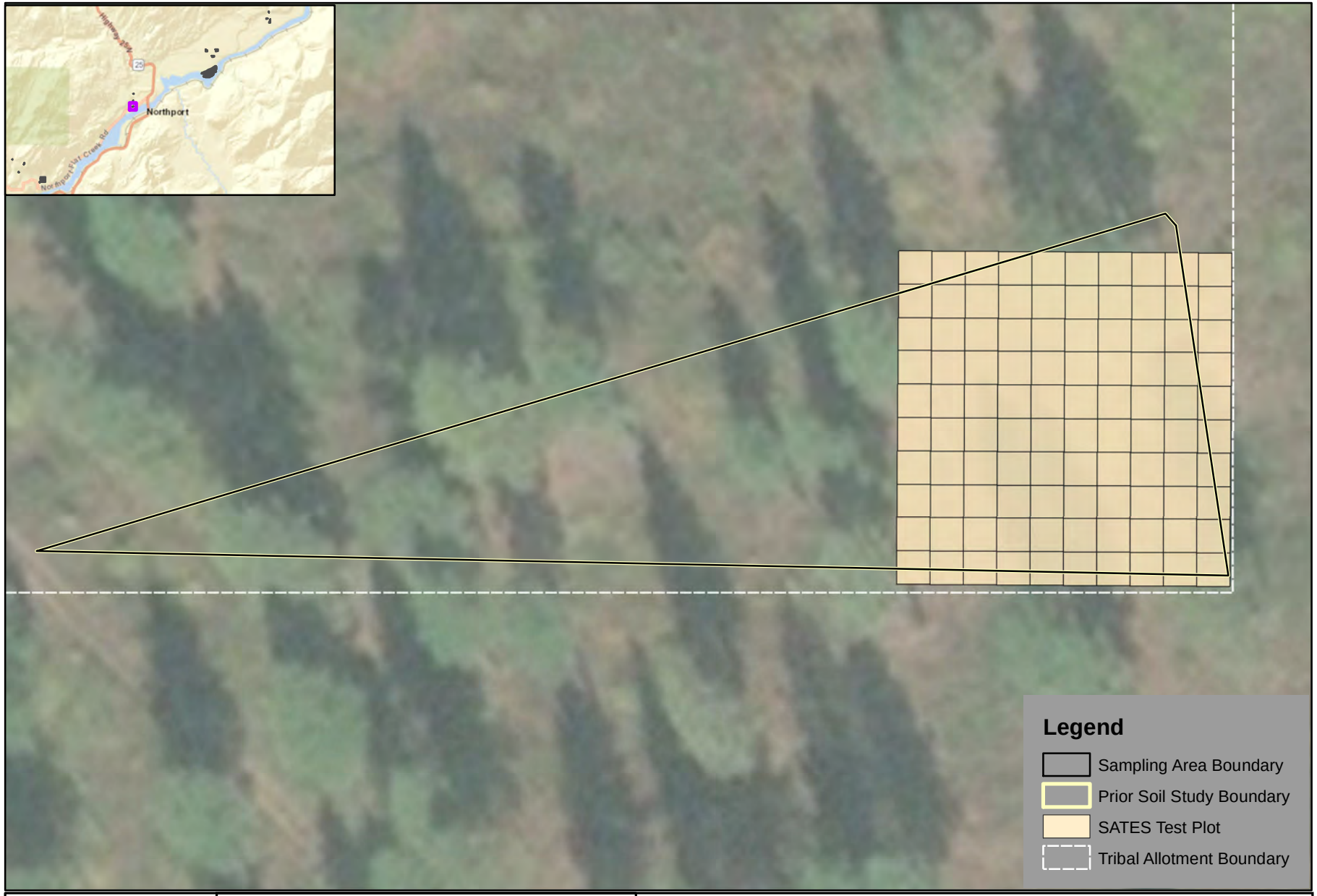
RAMBOLL ENVIRON

0 10 20 40
Meters

0 50 100 200
Feet

N

Map A7-3. Detail for Sampling Area 02
Upper Columbia River, WA



RAMBOLL ENVIRON

Map A7-4. Detail for Sampling Area 03
Upper Columbia River, WA

RAMBOLL ENVIRON

0 5 10 20
Meters
0 25 50 100
Feet
N

Map A7-5. Detail for Sampling Area 04
Upper Columbia River, WA

RAMBOLL ENVIRON

Map A7-6. Detail for Sampling Area 05
Upper Columbia River, WA

RAMBOLL ENVIRON

0 5 10 20 Meters

0 25 50 100 Feet

N

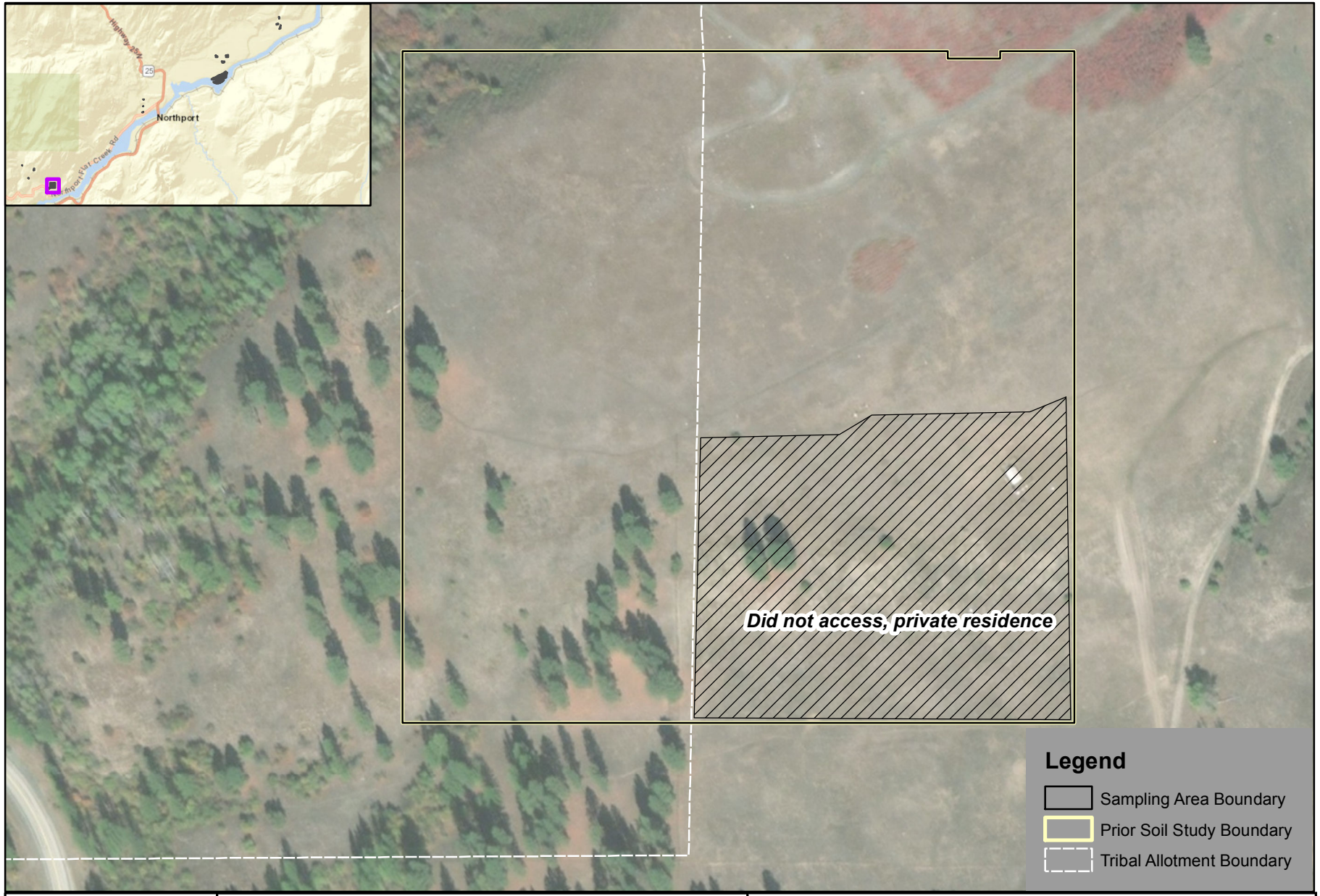
Map A7-7. Detail for Sampling Area 06
Upper Columbia River, WA

RAMBOLL ENVIRON

0 5 10 20
Meters
0 25 50 100
Feet

N

Map A7-8. Detail for Sampling Area 07
Upper Columbia River, WA



RAMBOLL ENVIRON

Map A7-9. Detail for Sampling Area 08
Upper Columbia River, WA

RAMBOLL ENVIRON

0 5 10 20 Meters

0 25 50 100 Feet

N

Map A7-10. Detail for Sampling Area 09
Upper Columbia River, WA

Map A7-11. Detail for Sampling Area 10
Upper Columbia River, WA

RAMBOLL ENVIRON

0 5 10 20 Meters

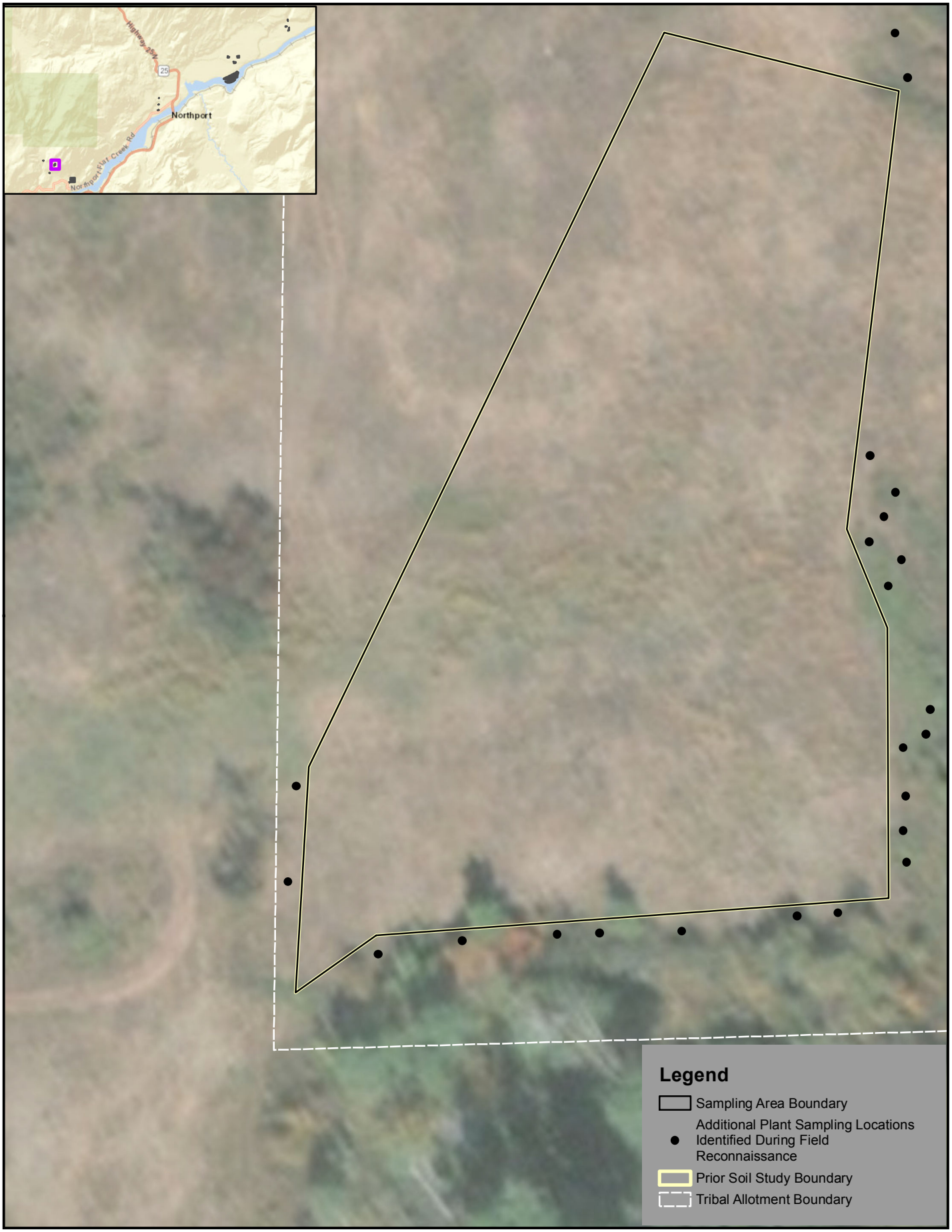
0 25 50 100 Feet

N

Map A7-12. Detail for Sampling Area 11
Upper Columbia River, WA

RAMBOLL ENVIRON

Map A7-13. Detail for Sampling Area 12
Upper Columbia River, WA



RAMBOLL ENVIRON

0 5 10 20 Meters

0 25 50 100 Feet

N

Map A7-14. Detail for Sampling Area 13
Upper Columbia River, WA

RAMBOLL ENVIRON

Map A7-15. Detail for Sampling Area 14
Upper Columbia River, WA

Map A7-16. Detail for Sampling Area 15
Upper Columbia River, WA

RAMBOLL ENVIRON

0 50 100 200
Meters
0 250 500 1,000
Feet
N

Map A7-17. Detail for Sampling Area 16
Upper Columbia River, WA

TABLES

Table A4-1. Technical Team Task Member Information

Name	Task Role	Phone	Email
Teck American Incorporated			
Kris McCaig	TAI Project Coordinator	(509) 623-4501	Kris.McCaig@teck.com
Cristy Kessel	Analytical Chemistry Laboratory Coordinator	(509) 496-1160	Cristy.Kessel@teck.com
Denise Mills	Assistant Project Coordinator	(509) 623-4515	denise.mills@teck.com
U.S. Environmental Protection Agency			
Monica Tonel	EPA Project Manager	(206) 553-0323	tonel.monica@epa.gov
Marc Stifelman	EPA Human Health Risk Assessment Lead	(206) 553-6979	stifelman.marc@epa.gov
Donald Brown	EPA Region 10 QA Manager	(206) 553-0717	Brown.DonaldM@epa.gov
Jennifer Crawford	EPA QA Chemist	(206) 553-6261	crawford.jennifer@epa.gov
Don Matheny	EPA QA Chemist	(206) 553-2599	matheny.don@epa.gov
Consultant Team			
Dina Johnson	Principal Investigator	(206) 336-1662	DLJohnson@ramboll.com
Rosalind Schoof	Senior Technical Advisor	(206) 336-1653	rschoof@ramboll.com
Lis Nelis	Task Manager	(206) 336-1659	lnelis@ramboll.com
Jenny Pretare	Field Team Manager	(206) 438-2175	jennifer.pretare@aecom.com
Jeff Walker	Field Team Botanist	(206) 438-2351	jeff.walker@aecom.com
Rock Vitale	Task QA Manager	(610) 935-5577	rvitale@envstd.com
Randy O'Boyle	Database Administrator	(425) 519-8727	roboyle@exponent.com
Laboratories			
Mark Harris	Analytical Chemistry Laboratory Project Manager - ALS	(360) 501-3376	mark.harris@alsglobal.com
Carl Degner	Analytical Chemistry Laboratory QA Manager - ALS	(360) 501-3270	Carl.Degner@alsglobal.com

Notes:
ALS - ALS Environmental
QA - quality assurance
TAI - Teck American Incorporated

Table A7-1. Proposed Sampling Areas

Sampling Area ID	Overlap with Prior UCR RI/FS Sampling Area[a]	Type of Property Ownership/ Management	Rationale for Inclusion	Average IC Soil Lead (mg/kg)[a]	Average IC Soil Arsenic (mg/kg)[a]
SA01	2014R-258	Tribal allotment	High soil lead. Sarvisberry, kinnikinnick, black tree lichen, ponderosa pine, chokecherry, wild rose, camas and wild strawberry recorded during August 2017 field reconnaissance, as well as habitat for bitterroot, lomatium, morel, shaggy mane, spring beauty, and Indian carrot.	678	46.8
SA02	2014R-401	Tribal allotment	High soil lead. Sarvisberry, kinnikinnick, ponderosa pine, chokecherry, and hazelnut recorded during August 2017 field reconnaissance, as well as habitat for morels.	1120	80.8
SA03	2014R-441	Tribal allotment	High soil lead. Sarvisberry, kinnikinnick, ponderosa pine, chokecherry, wild rose, hazelnut, and wild strawberry recorded during August 2017 field reconnaissance, as well as habitat for morels.	624	43.6
SA04	2014R-402	Tribal allotment	Sarvisberry, kinnikinnick, ponderosa pine, chokecherry, wild rose, hazelnut, dwarf huckleberry, and wild strawberry recorded during August 2017 field reconnaissance, as well as habitat for bitterroot, lomatium, morel, shaggy mane, and Indian carrot.	542	34
SA05	2014R-410	Tribal allotment	Sarvisberry, kinnikinnick, black tree lichen, ponderosa pine, chokecherry, and wild rose recorded during August 2017 field reconnaissance, as well as habitat for bitterroot, lomatium, shaggy mane, and Indian carrot.	370	35
SA06	2014R-403	Tribal allotment	Sarvisberry, kinnikinnick, wild rose, and hazelnut recorded during August 2017 field reconnaissance, as well as habitat for shaggy mane.	394	26.1

Table A7-1. Proposed Sampling Areas

Sampling Area ID	Overlap with Prior UCR RI/FS Sampling Area[a]	Type of Property Ownership/ Management	Rationale for Inclusion	Average IC Soil Lead (mg/kg)[a]	Average IC Soil Arsenic (mg/kg)[a]
SA07	2014R-259	Tribal allotment	Sarvisberry, black tree lichen, ponderosa pine, chokecherry, and wild rose recorded during August 2017 field reconnaissance, as well as habitat for bitterroot, lomatium, shaggy mane, and Indian carrot.	226	19.7
SA08	2014U-ADA-023	Tribal allotment	Sarvisberry, black tree lichen, ponderosa pine, chokecherry, wild rose, and hazelnut recorded during August 2017 field reconnaissance, as well as habitat for bitterroot, lomatium, shaggy mane, and Indian carrot.	151	18.3
SA09	2014R-442	Tribal allotment	Sarvisberry, ponderosa pine, chokecherry, wild rose, red willow, hazelnut, and wild strawberry recorded during August 2017 field reconnaissance, as well as habitat for shaggy mane, spring beauty, and Indian carrot.	243	21.7
SA10	2016R-808-O2	Tribal allotment	Sarvisberry, kinnikinnick, ponderosa pine, puffball, and wild rose recorded during August 2017 field reconnaissance, as well as habitat for bitterroot, lomatium, shaggy mane, and Indian carrot.	42.6	6.98
SA11	2016R-804-O1	Tribal allotment	Sarvisberry, black tree lichen, ponderosa pine, wild rose, and hazelnut recorded during August 2017 field reconnaissance, as well as habitat for bitterroot, lomatium, shaggy mane, and Indian carrot.	121	7.74
SA12	2014R-440	Tribal allotment	Sarvisberry, black tree lichen, chokecherry, and hazelnut recorded during August 2017 field reconnaissance, as well as habitat for shaggy mane, spring beauty, and Indian carrot.	136	9.12
SA13	2016R-801-O3	Tribal allotment	Sarvisberry, chokecherry, and wild rose recorded during August 2017 field reconnaissance, as well as habitat for bitterroot, lomatium, shaggy mane, and Indian carrot.	37	15.1

Table A7-1. Proposed Sampling Areas

Sampling Area ID	Overlap with Prior UCR RI/FS Sampling Area[a]	Type of Property Ownership/ Management	Rationale for Inclusion	Average IC Soil Lead (mg/kg)[a]	Average IC Soil Arsenic (mg/kg)[a]
SA14	2016R-805-O2	Tribal allotment	Sarvisberry, black tree lichen, wild rose, hazelnut, wild mint, and tule recorded during August 2017 field reconnaissance, as well as habitat for bitterroot, lomatium, shaggy mane, and Indian carrot.	54.1	5.31
SA15[b]	RFA-001, RFA-002, RFA-003, RFA-004, RFA-005	Washington Department of Natural Resources	Possible presence of willows in a previously sampled area with moderately high concentrations of lead in relict floodplain soil.	389[c]	15.8[c]
SA16[b]	Barnaby Island Campground	National Park Service	Possible presence of willows in a previously sampled area with low concentrations of lead in beach sediment.	46.2[d]	1.99[d]

Notes:

[a] Based on the UCR 2010 Beach Sediment Study (Integral 2014), the UCR 2014 Residential Soil Study (CH2M Hill 2016), the UCR 2014 Upland Soil Study (Windward et al. 2015), or the UCR 2016 Residential Soil Study (Ramboll Environ 2017b).

[b] Sampling areas were not visited during the August 2017 field reconnaissance event. Areas are publicly-accessible and are included for potential sampling of willows.

[c] These averages represent the average of pre-averaged replicates for each of the five decision units sampled.

[d] Averages are based on sample results for <63 µm, 63 to 125 µm, and 125 to 250 µm size fraction samples.

IC - incremental composite

Table A7-2. Target Number of Samples to be Collected[a]

Sampling Area	Target Number of Samples to Address Principal Study Question[b]		Target Number of Samples to Address Secondary Study Question[c]	
	Plant Tissue (see Table A7-4)	Co-Located Soil/Sediment	Plant Tissue (see Table A7-4)	Co-Located Soil/Sediment
SA01 SA02 SA03	Collect 6 samples of each plant tissue targeted for the specific field sampling event (minimum 3 samples per tissue targeted) from across the three high lead SAs.	Collect 1 sample for each tissue sample collected.	Addressed by Principal Study Question	
SA04 SA05 SA06 SA07 SA08 SA09 SA10 SA11 SA12 SA13 SA14 SA15 SA16	Augment sample collection for tissues sampled at SA01, SA02, and/or SA03, if fewer than 6 samples collected. Also, for tissues not sampled from SA01, SA02, and/or SA03, collect 6 samples of each plant tissue targeted for the specific field sampling event (minimum 3 samples per tissue sampled).	Collect 1 sample for each tissue sample collected.	Collect 6 samples of each plant tissue collected to address the Principal Study Question for the specific field sampling event (minimum 3 samples per tissue sampled) from across the lower lead SAs.	Collect 1 sample for each tissue sample collected.
Maximum Number of Samples	66 to 132 plant tissue samples[d]	1 co-located soil/sediment sample for each plant tissue sample[e]	66 to 132 plant tissue samples[d]	1 co-located soil/sediment sample for each plant tissue sample[e]
Field Replicates	To be collected at a frequency of 5 percent (dependent on availability of sufficient excess plant mass of an individual plant).			
EPA Splits	To be processed by the laboratory at a frequency of 5 percent (dependent on availability of sufficient excess plant mass of an individual plant).			

Notes:

[a] As stated in Section A7.3.1, the objective of the study is to collect each sample from an individual plant; however, a composite sample of adjacent individual plants may be collected if insufficient mass is available at an individual plant. If mass limitations at an individual plant necessitate collection of a composite plant tissue sample, the co-located soil sample will also be collected as a composite of co-located soil samples for each individual plant contributing to the composite plant tissue sample.

[b] As stated in Section A7.2, the principal study question is: "Does exposure to total concentrations of target analysis list (TAL) metals in wild plant tissues pose unacceptable risk to human consumers?"

[c] As stated in Section A7.2, the secondary study question is: "Do the chemical concentrations of TAL metals in wild plant tissues collected across a range of soil lead concentrations vary with concentrations of TAL metals in soil?"

[d] A total of 22 plant tissues are targeted for collection over three field sampling events. This range reflects the maximum number of samples to address the study question if 3 to 6 samples per target is collected by the end of the field sampling program. If the minimum (3) or targeted number of samples (6) for all targeted tissues are collected from both the high lead sampling areas (SAs) and one or more lower lead SAs, the maximum number of tissue samples for the field program would range from 132 to 264, respectively.

[e] In some cases, targeted plant tissue for two plant species growing at the same location may be sampled. If this occurs, the same co-located soil/sediment sample may be associated with each plant tissue target. Therefore, the total number of soil/sediment samples collected will be up to the total number plant tissue samples collected.

Table A7-3. Methods and Sample Mass Requirements

Analyte	Sample Preparation		Quantitative Analysis		Holding Time[a]	Sample Mass Required for Analysis (g dw)	
	Protocol	Procedure	Protocol	Procedure		Soil/Sediment	Plant Tissue
Conventional Parameters - Plant Tissue							
Total Mass	NA	NA	NA	NA	NA	NA	8-12[b]
Percent moisture	ALS SOP MET-TISP	Gravimetric: dry at 60°C[c], freeze-dry[d]	ALS SOP MET-TISP	Gravimetric: dry at 60°C[c], freeze-dry[d]	1 year at -20°C	NA	NA[e]
TAL Metals/Metalloids - Plant Tissue							
TAL metals (except calcium, magnesium, potassium, and sodium)	ALS SOP MET-TDIG	Acid digestion	EPA 6020A MET-6020	ICP-MS	180 days at -20°C	NA	0.3[f]
Total mercury	ALS SOP MET 1631	Acid digestion	EPA 1631E	CVAFS	1 year at -20°C	NA	0.4[f]
Total Target Sample Mass						NA	2.1[g]
TAL Metals/Metalloids - Soil/Sediment							
TAL metals (except calcium, magnesium, potassium, and sodium)	MET-3050B	Acid digestion	EPA 6020A MET-6020	ICP-MS	180 days at room temperature[h]	2[e]	NA
Total mercury	ALS SOP MET 1631	Acid digestion	EPA 1631E	CVAFS	1 year at < -15°C	NA[i]	NA
Total Target Sample Mass						2[i]	NA

Notes:

Sample masses do not include additional mass for field splits, laboratory duplicates, or re-extraction. Field splits will be prepared on the targeted number of samples if double the sample mass is available (i.e., sufficient mass to conduct all analyses twice). If insufficient sample mass is available for the targeted number of field splits, laboratory duplicates will be analyzed in an attempt to meet the same targeted frequency for each as field splits, for each analyte. Laboratory duplicates will be analyzed as sample mass allows, in order of priority.

Tissue freeze-dried after compositing will be stored at ambient temperature. There are no standard holding times for freeze-dried tissue, however, EPA has approved a holding time of 2 years from the time of freeze-drying for this study (Tonel 2017).

[a] Holding time based on applicable standard operating procedure.

[b] Wet weight mass in grams.

[c] For typical plant tissue

[d] For berries

[e] Percent moisture will be analyzed with TAL metals; no additional sample mass required

[f] The target sample mass for analysis listed achieves the reporting limits in Table A7-5

[g] The total target sample mass allows for ALS to have three aliquots of each sample

[h] Note that the EPA Regional Quality Assurance Manager has approved shipment and storage of the soil/sediment samples analyzed for metals other than mercury without refrigeration or use of ice; however, soil/sediment samples designated for mercury analysis will be iced or refrigerated immediately upon sampling and a temperature of ≤2°C will be maintained during shipment to the laboratory.

[i] The total target sample mass for TAL metals (except calcium, magnesium, potassium, and sodium) in soil/sediment will be sufficient for additional analysis of mercury in soil/sedime

ALS - ALS Environmental
CVAFS - cold vapor atomic fluorescence spectrometry
ICP-MS - inductively-coupled plasma - mass spectrometry
ICP-OES - inductively-coupled plasma - optical emission spectrometry
MET-TISP - tissue sample preparation
MET-TDIG - sample preparation of biological tissue for metals analysis by ICP-OES and ICP-MS
NA - not applicable
SOP - standard operating procedure
TAL - target analyte list

Table A7-4. Target Plant Tissue List by Field Sampling Event

Field Event	Sample Type	Plant Scientific Name	Target Plant Tissue
All	Black tree lichen	*Bryoria fremontii*	Lichen
Mid-April to Early May	Camas	*Camassia quamash*	Bulbs
	Kinnikinnick	*Arctostaphylos uva-ursi*	Leaves
	Wild rose (stems and leaves)	*Rosa* spp.	Leaves
	Puffball	*Calvatia gigantea*	Fruiting body
	Bitterroot	*Lewisia rediviva*	Root
	Lomatium	*Lomatium* spp.	Roots
	Morel	*Morchella esculenta*	Fruiting body
	Shaggy mane	*Coprinus comatus*	Fruiting body
	Spring beauty/ Indian potato	*Claytonia lanceolata*	Corm
	Indian carrot[a]	*Perideridia gairdneri*	Roots
	Green willow	*Salix exigua*	Inner bark
	Red willow/red-osier dogwood	*Cornus sericea*	Inner bark
Late June	Sarvisberry	*Amelanchier alnifolia*	Berries
	Wild strawberry	*Fragaria vesca & F. virginiana*	Berries
Late August	Chokecherry	*Prunus virginiana*	Cherries
	Hazelnut	*Corylus cornuta var. californica*	Nuts
	Ponderosa pine	*Pinus ponderosa*	Nuts
	Wild rose (hips)	*Rosa* spp.	Rose hips
	Huckleberry	*Vaccinium* spp.	Berries
	Wild mint	*Mentha arvensis*	Leaves
	Tule	*Schoenoplectus acutus*	Culms

Notes:

[a] Ideally gathered in May or June, before flowering.

Table A7-5. Plant Tissue and Soil/Sediment Target Analyte List and Analytical Concentration Goals

	Plant Tissue				Soil/Sediment			
		Laboratory				Laboratory		
		Tissue (mg/kg-dw)				Soil/Sediment (mg/kg-dw)		
Analyte	Human Health RBCs (mg/kg-dw)[a]	MRL[b]	MDL[b]	ACG[c]	Human Health RBC (mg/kg)	MRL[b]	MDL[b]	ACG[c]
Conventional Parameters								
Total Mass	na	na	na	na	N/A	N/A	N/A	N/A
Moisture Content	na	na	na	na	na	na	na	na
Metals/Metalloids								
Aluminum	28	2	0.6	28	5,000	2	0.6	5,000
Antimony	0.01	0.05	0.02	0.05	2	0.05	0.02	2
Arsenic	0.0004	0.5	0.2	0.5	0.29	0.5	0.2	0.5
Barium	5.6	0.05	0.02	5.6	1,000	0.05	0.02	1,000
Beryllium	0.06	0.02	0.005	0.06	10	0.02	0.005	10
Cadmium	0.03	0.02	0.009	0.03	5	0.02	0.009	5
Chromium	42	0.2	0.07	42	7,500	0.2	0.07	7,500
Cobalt	0.008	0.02	0.009	0.02	1.5	0.02	0.009	1.5
Copper	1.1	0.1	0.04	1.1	200	0.1	0.04	200
Iron	19	1	2	19	3,500	4	2	3,500
Lead	0.09	0.02	0.02	0.09	143	0.05	0.02	143
Manganese	3.9	0.05	0.02	3.9	120	0.05	0.02	120
Mercury	0.008	0.001	0.00009	0.008	1.5	0.001	0.00009	1.5
Nickel	0.56	0.2	0.04	0.56	100	0.2	0.04	100
Selenium	0.14	1	0.2	1	25	1	0.2	25
Silver	0.14	0.02	0.005	0.14	25	0.02	0.005	25
Thallium	0.0003	0.02	0.002	0.02	0.05	0.02	0.002	0.05
Vanadium	0.14	0.2	0.08	0.2	25	0.2	0.08	25
Zinc	8.3	0.5	0.2	8.3	1,500	0.5	0.2	1,500

Notes:

[a] Risk-based concentrations (RBCs) for human health are based on exposure assumptions and calculation methods specified in the 2016 plant tissue data quality objectives (DQOs) (USEPA 2016). The RBC shown represents the lower of the non-cancer child RBC or the cancer RBC based on a time-weighted average child and adult.

[b] Method reporting limits (MRLs) and method detection limits (MDLs) for metals were obtained from ALS Environmental (ALS).

[c] Analytical concentration goals (ACGs) represent the RBC value for human health. If the RBC is lower than the MRL, the MRL will be used as the ACG. Values are shaded if the ACG exceeds the RBC.

na - not available

N/A - not analyzed

Table B5-1. Standard Reference Materials by Analyte

Analyte	SRM 1	Control Limits for SRM 1[a] (concentration range [mg/kg-dw])	SRM 2	Control Limits for SRM 2[a] (concentration range [mg/kg-dw])	SRM 3	Control Limits for SRM 3 (concentration range [mg/kg-dw])
Conventional Parameters						
Total Mass	NA	NA	NA	NA	NA	NA
Percent moisture	NA	NA	NA	NA	NA	NA
Metals/Metalloids[b]						
Aluminum	1547	193-308	1573a	469-732	D087-540	3090-12,800
Antimony	1547	NA	1573a	0.045-0.0828	D087-540	21.4-267
Arsenic - total	1547	0.034-0.094	1573a	0.086-0.192	D087-540	68.3-143
Barium	1547	96-154	1573a	NA	D087-540	228-388
Beryllium	1547	NA	1573a	NA	D087-540	48.6-83.4
Cadmium	1547	0.018-0.035	1573a	1.18-1.87	D087-540	107-185
Chromium	1547	NA	1573a	1.54-2.46	D087-540	129-236
Cobalt	1547	NA	1573a	0.44-0.708	D087-540	120-203
Copper	1547	2.64-4.92	1573a	3.65-5.81	D087-540	79-133
Iron	1547	163-278	1573a	289-450	D087-540	5120-23,600
Lead	1547	0.672-1.08	1573a	NA	D087-540	94.2-165
Manganese	1547	76-121	1573a	190-305	D087-540	313-508
Mercury - total	1547	0.024-0.0456	1573a	0.024-0.0456	D087-540	3.64-10.6
Nickel	1547	0.48-0.936	1573a	1.22-1.99	D087-540	109-189
Selenium	1547	0.089-0.155	1573a	0.04-0.068	D087-540	104-204
Silver	1547	NA	1573a	NA	D087-540	27-54.7
Thallium	1547	NA	1573a	NA	D087-540	120-229
Vanadium	1547	0.272-0.48	1573a	0.66-0.845	D087-540	62.9-131
Zinc	1547	14-22	1573a	24.2-37.9	D087-540	133-249

Notes:

[a] Standard reference materials (SRMs) 1 and 2 will be used to analyze target analyte metals in plant tissue, and are not species-specific.

[b] SRMs for metals are prepared and analyzed with every batch of 20 samples or at a frequency of 5%, whichever is greater. For metals with no available SRM, a blank spike is prepared at the same frequency as the SRMs.

1547 - National Institue of Standards and Technology (NIST) standard reference material for trace elements in peach leaves

1573a - NIST standard reference material for trace elements in tomato leaves

D087-540 - ERA reference material for metals in soil

NA - not applicable

Table B5-2. Measurement Quality Objectives for Plant Tissue and Soil/Sediment

Analysis Type	Bias[a] (percent)	Precision[b] (RPD)	Completeness (percent)
Plant Tissue			
Total mass	NA	40	90
Percent moisture	NA	40	90
TAL metals (except calcium, magnesium, potassium, and sodium)	75-125	40	90
Total mercury	70-130	40	90
Soil/Sediment			
TAL metals (except calcium, magnesium, potassium, and sodium)	75-125	20	90
Total mercury	70-130	20	90

Notes:

[a] The bias criteria applies to matrix spike/matrix spike duplicate analyses. For laboratory control samples, the method specified criteria will be utilized. See Table B5-1 for control limits for standard reference materials.

[b] Precision criteria applies to relative percent difference (RPD) of laboratory duplicate results. Control limits for RPDs are based on the laboratory specified criteria.

NA - not applicable

TAL - target analyte list

APPENDIX A

FIELD SAMPLING PLAN FOR THE PLANT TISSUE STUDY

CONTENTS

LIST OF MAPS

LIST OF TABLES

ACRONYMS AND ABBREVIATIONS

CCT	Confederated Tribes of the Colville Reservation
COC	chain-of-custody
COI	contaminant of interest
EPA	U.S. Environmental Protection Agency
EPCs	exposure point concentrations
FSP	field sampling plan
GPS	global positioning system
HHRA	human health risk assessment
ID	identification number
MS	matrix spike
MSD	matrix spike duplicate
QA/QC	quality assurance and quality control
QAPP	quality assurance project plan
QC	quality control
RI/FS	remedial investigation and feasibility study
SATES	Soil Amendment Technology Evaluation Study
SHSP	site health and safety plan
Site	Upper Columbia River site
SOP	standard operating procedure
SA	sample area
TAI	Teck American Incorporated
TAL	target analyte list
UCR	Upper Columbia River
WGS84	World Geodetic System of 1984

UNITS OF MEASURE

dw	dry weight
°C	degree(s) Celsius
g	gram(s)
in.	inch(es)
m	meter(s)
µm	microgram(s) per meter
mg/kg	milligram(s) per kilogram

1 INTRODUCTION

This document presents the field sampling plan (FSP) for the plant tissue study (hereafter referred to as the "study") for the Upper Columbia River (UCR, hereafter the Site[1]). Information collected in this study will be used to support the remedial investigation and feasibility study (RI/FS) and the human health risk assessment (HHRA) for the Site. Both the RI/FS and HHRA are being completed under an agreement between Teck American Incorporated (TAI) and the U.S. Environmental Protection Agency (EPA). The objective of the RI/FS is to investigate the nature and extent of contamination and potential for risk to humans and the environment. EPA is conducting the HHRA; TAI is conducting the RI/FS and this study with EPA oversight.

The primary objective of this study is to characterize the concentrations of metals in the tissues of wild upland plants sampled from tribal allotments in the study area. Data collection efforts will focus on obtaining information that will inform the exposure assessments for humans who consume or otherwise utilize plants from the study area. Chemistry data for plant parts of interest will be used in the HHRA to evaluate the potential for metal uptake into plants and subsequent exposure of people who harvest and consume or otherwise utilize plants. This FSP describes how and where plant tissues will be collected for chemical analyses.

The requirements and design rationale for data collection activities presented in this FSP were developed in consultation with EPA and representatives of the Confederated Tribes of the Colville Reservation (CCT).

1.1 OVERVIEW

Members of the CCT consume and otherwise use (e.g., for weaving) terrestrial, wetland, and aquatic plants within the vicinity of the Site. Other local residents and visitors to the Site may also use these resources. The UCR HHRA work plan (SRC 2009) identified metal concentrations in upland and riparian plants as a data need. Plants may accumulate metals from soil (e.g., Carranza-Álvarez et al. 2008; Intawongse and Dean 2006). Metal uptake factors derived from prior studies are sometimes used to predict plant tissue concentrations from soil concentrations; however, EPA has determined that the uncertainty associated

[1] The UCR Site as defined within the June 2, 2006 Settlement Agreement is the areal extent of hazardous substances contamination within the United States in or adjacent to the Upper Columbia River, including the Franklin D. Roosevelt Lake, from the U.S.-Canada border to the Grand Coulee Dam, and those areas in proximity to the contamination that are suitable and necessary for implementation of response actions.

with available uptake factors for lead and other metals is high, and site-specific data are required to more accurately estimate the exposure point concentrations (EPCs) for contaminants of interest (COIs) in plant tissue for the UCR HHRA (USEPA 2016).

Data on chemical concentrations in the tissues of plants consumed, mouthed, or otherwise used from the Site by CCT members have not been collected. Thus, plant tissues (described herein) will be collected to fill these data gaps. The principal study question to be addressed with this work is:

- Does exposure to total concentrations of target analyte list (TAL) metals[2] in wild plant tissues pose unacceptable risk to human consumers?

A secondary study question to be addressed by this work is:

- Do the chemical concentrations of TAL metals in wild plant tissues collected across a range of soil lead concentrations vary with concentrations of TAL metals in soil?

This FSP describes field methods that will be used to collect plant tissues and co-located soil or sediment[3] for the study. Section 2 of this FSP describes field sampling procedures that will be followed. Section 3 describes procedures for field documentation. References cited in this document are listed in Section 4.

Attachments to this FSP are:

- **Attachment A1—General Site Health and Safety Plan (SHSP) Addendum.** Describes site-specific requirements and procedures to minimize the safety risk to personnel who carry out the field study program.[4]

[2] For this study, calcium, magnesium, potassium, and sodium are excluded from the list of TAL metals. Mercury is included but only for kinnikinnick leaves, wild rose leaves and stems, wild mint, willows, and tules, and their co-located soil samples at locations where the quantity of plant material is sufficient to support analysis of both TAL metals and mercury. Where the quantity of plant material is limited, allocation of sample mass collected will be prioritized for analysis of TAL metals (except calcium, magnesium, potassium, and sodium).

[3] Throughout the remainder of this document, references to "soil" may include sediment, as applicable to a specific SA.

[4] Subcontractors that are contracted to perform field work associated with the RI/FS may adopt the general SHSP and this Addendum or develop and follow their own SHSPs; however, subcontractor SHSPs must be consistent with the provisions outlined in the Addendum and the general SHSP, and any discrepancies will follow the most protective practices. Ramboll may provide oversight of plant tissue and soil sampling events conducted by a TAI field contractor for this study; however, Ramboll personnel will not participate in the actual collection of plant tissue and soil samples.

- **Attachment A2—Standard Operating Procedures (SOPs).** Detailed field procedures to be used include
    - SOP-1 – Sample Area Selection
    - SOP-2 – Recording Plant Tissue Sample Collection Locations
    - SOP-3 – Sample Labeling
    - SOP-4 – Plant Tissue Surveying and Sample Collection
    - SOP-5 – Field Documentation
    - SOP-6 – Digital Camera Use and Documentation Procedures
    - SOP-7 – Sample Packaging and Shipping
    - SOP-8 – Decontamination of Sampling Equipment
    - SOP-9A – Discrete Soil Sample Collection
    - SOP-9B – Composite Soil Sample Collection
    - SOP-9C – Sediment Sample Collection
    - SOP-10 – Handling and Reporting of Cultural Resources
    - SOP-11 – Sample Custody.
- **Attachment A3—Examples of Various Field Forms.** Contains examples of various forms that will be used during field sampling (e.g., location, reconnaissance, plant collection, soil/sediment collection; photograph logging forms); change request form; protocol modification form; a chain-of-custody (COC) form; sample tracking form; and shipment tracking form.
- **Attachment A4—Archaeological Monitoring Protocol.** Provides study-specific procedures to be followed if any archaeological objects or resources are discovered during sampling activities.

2 SAMPLE COLLECTION AND PROCESSING

This section describes procedures and methods that will be used during the study, including sampling procedures, record keeping, sample handling, storage, and field quality control (QC) procedures. Sample collection and processing will be conducted in accordance with the SOPs provided in Attachment A2. Depending on field conditions, procedures specified in the referenced SOPs may be modified, if necessary, in consultation with EPA and documented in a Protocol Modification Form or Change Request Form (Attachment A3).

2.1 SAMPLING AREAS

In June 2017, EPA directed TAI to conduct a study that will primarily be focused on collection of plant tissue from the three tribal allotments sampled in the 2014 Residential Soil Study (CH2M HILL 2016), which had bioavailability-adjusted incremental composite concentrations of lead in soil greater than 700 mg/kg (2014R-258, 2014R-401, and 2014R-441; USEPA 2017). EPA's June 2017 letter further specified that plant tissues from a reference area should be sampled; however, reference areas for the Site have currently not been determined. Therefore, based on further consultation with EPA during study planning, EPA approved collecting plant samples from the Site on tribal allotments where prior RI/FS soil studies have been collected, and represent a range of lower soil lead concentrations as an alternative to the specified reference area. In addition, because willows have been identified by the CCT as a plant of cultural significance and the species identified were not present on the three high lead tribal allotments or other lower lead tribal allotments surveyed during the August 2017 field reconnaissance phase of this study (AECOM 2017), potential sample areas (SAs) also include two areas of the Site that are located along the UCR and were sampled previously for sediment as part of the RI/FS. Although these additional SAs are not located on tribal allotments, each potential SA is publicly accessible.

Plant samples will be collected from the three high lead SAs and up to 13 additional lower lead SAs in the Site (Map A1 and Table A1). Maps A2 through A17 provide detailed aerial photographs of these SAs, along with other relevant information used to define the SAs (e.g., locations of previously sampled decision units, locations of test plots for the Soil Amendment Technology Evaluation Study [SATES], etc.).

In consultation with EPA oversight or the agency's authorized representative, a brief reconnaissance of the three high lead SAs will be conducted at the start of each field sampling event to identify specific plant tissues targeted for sampling during that event and to guide prioritization of the additional lower lead SAs by field crews during that event

(see SOP-1 in Attachment A2). Specific sampling locations within each SA will be determined by the field crew based on the presence of targeted plant tissues sufficient to meet mass requirements for sample analysis.

For each targeted plant tissue, the collection of sufficient mass from six individual plants[5] will be targeted from across the high lead SAs to address the principal study question. A co-located soil sample[6] will be collected with each plant tissue sample. In addition, as available, collection of sufficient mass from six individual plants will also be targeted from across one or more lower lead SAs. These samples will be used to address the secondary study question; however, if a particular target species is only identified at a lower lead SA or the measured soil lead concentration reported by the laboratory for a lower lead SA is higher than the concentrations measured at the high lead SAs, these samples may also be used to address the principal study question. Thus, a total of six high lead SA plant tissue and co-located soil samples, and six lower lead SA plant tissue and co-located soil samples will be targeted for each plant species and tissue type during this sampling effort.

2.2 FIELD SURVEY AND SAMPLING METHODS

It is anticipated that sampling will be carried out by one team. This sampling team will be supported by a two-person survey team. The primary role of the survey team will be to prioritize the additional SAs to be visited by the sampling team. Based on the initial reconnaissance of the high lead SAs and identification of the specific target plant tissues for sampling during that field sampling event, the survey team will deploy to the lower lead SAs ahead of the sampling team to identify the availability of target plant tissues for sampling survey, and to identify potential access issues for mobilization by the sampling team as well as other personnel (e.g., EPA oversight representatives, cultural resource monitors, etc.) to each SA. Mobilization to SAs will be via vehicles suitable for traversing unmaintained dirt roads.

The sampling team vehicles will need to have space large enough to accommodate sampling team members, in addition to field sampling equipment, sample packaging

[5] Collection of six samples for each targeted tissue is the goal for this study; however, EPA has determined that three samples for each targeted tissue from the high lead SAs will be sufficient to support the HHRA. Furthermore, while the objective is to collect each sample from an individual plant, a composite sample of adjacent individual plants may be collected if insufficient mass is available from an individual plant.

[6] If mass limitations at an individual plant necessitate collection of a composite plant tissue sample, the co-located soil sample will also be collected as a composite of co-located soil samples for each individual plant contributing to the composite plant tissue sample.

supplies, coolers, and multiple sampling equipment boxes containing sample containers and other ancillary equipment. The survey team vehicle will need to have space to accommodate the survey team and equipment. The field team leader, in consultation with the survey team, will also oversee access to SAs to ensure the safety of the field teams and to minimize impacts to potential SAs.

2.2.1 Task Schedule

Subject to EPA approval, field sampling is expected to be conducted during three field sampling events during the spring and summer of 2018. The first event will take place between mid-April and early May. The second field event will take place in late June, and the third event will take place late August. Each sampling event will take approximately 1 week. Thirty days prior to field sampling activities, a detailed schedule will be prepared by the field sampling crew to facilitate planning and scheduling of EPA technical and cultural oversight.

2.2.2 Sampling Location Positioning

Borders of pre-determined SAs will be uploaded onto handheld global positioning system (GPS) units. The position of each individual plant sampled will be recorded using the GPS units. The procedure for recording sampling locations is detailed in SOP-2 (Attachment A2). The standard projection method to be used during field activities will be the horizontal datum of the World Geodetic System of 1984 (WGS84).

2.2.3 Field Equipment and Supplies

Field equipment and supplies anticipated for this study include garden shears, handheld spades, small saws, hand trowels, buckets, scale or analytic balance, decontamination supplies, sample containment supplies (e.g., resealable bags), coolers, shipping containers, cameras, field logs and forms (or electronic tablet), personal protective equipment, waders, personal gear, and first aid supplies (see SOPs in Attachment A2 for details). Protective wear (e.g., gloves) is required to minimize the possibility of cross-contamination between SAs.

Sample containers, distilled or deionized water, coolers, and packaging material for samples will be supplied by the analytical laboratory. Details on required sample mass for the analysis of plant tissue and soil/sediment samples are provided in Table A2 and discussed further in Section 2.2.4. Sample containers will be clearly labeled at or prior to the time of sampling. Completing as much labeling as possible prior to the field work (especially electronic labeling) can be advantageous because it may reduce errors stemming from inconsistent naming, handwriting legibility, and label adhesion that may occur when labeling in field conditions. Labels will include the task name, sample location, sample

identification number (ID), samplers' initials, analyses to be performed, and sample date and time. Sample labeling procedures are detailed in SOP-3 (Attachment A2).

2.2.4 Sample Collection Methods

This section describes plant tissue and soil/sediment sampling methods that will be implemented in the field. These methods are supported by SOPs and summarized in the following subsections.

Plant Tissue Sampling

A target plant tissue list that includes 21 plant species has been developed for this study in consultation with EPA and CCT representatives (Table A2). The target plant list includes those species found during the August 2017 field reconnaissance study (AECOM 2017), spring ephemeral species, and plant species that are important to CCT members but do not have habitat within the decision units surveyed in the field reconnaissance study. Plants will be identified by trained field botanists who are familiar with local plant species. Plant identification methods will be based on recognition of shared and divergent morphological characteristics. Questions about plant identity will be resolved using the Flora of the Pacific Northwest key (Hitchcock and Cronquist 1973), as amended by current taxonomic classification updates.

Plants will be collected from SAs based on information obtained during prior studies and field reconnaissance efforts[7]. Visitation of lower lead SAs for possible sample collection will be prioritized based on the likelihood that a potential target plant species will be found in sufficient abundance. Lower lead SAs that have the highest likelihood of providing plant biomass will be sampled first, according to the results of the field reconnaissance effort from August 2017 (AECOM 2017).

Collection of each target species will be limited to one of three sampling events (spring, late June, or late August) with the exception of wild rose (*Rosa* spp.) and black tree lichen (*Bryoria fremontii*). Wild rose will be a target species for two field events: spring to collect leaves and stems, and August to collect rose hips. Black tree lichen will be targeted for sample collection during each field event or until mass requirements have been met or collection threshold has been reached. Field collection teams will collect plant tissues by hand. The precise location of sampled plants will be recorded via a handheld GPS unit.

[7] September 20, 2017 Lodestone Memorandum (Lodestone 2017a), and the August 2017 field reconnaissance (AECOM 2017), which was in turn informed by the prior CCT field reconnaissance efforts (Lodestone 2016a,b, 2017b) and the CCT Tribal Consumption and Resource Use Survey (Westat 2012).

For each targeted plant tissue, the collection of sufficient mass from six individual plants will be targeted from the high lead SAs. As available, collection of sufficient mass from six individual plants will also be targeted from one or more of the other lower lead SAs.

For each plant tissue sample submitted for analysis of TAL metals without mercury, a minimum of 1 g of each tissue (dry weight) will be required for laboratory analysis; 2 g is preferred. For plant tissue samples submitted for analysis of both TAL metals and mercury,[8] a minimum of 2.1 g of each tissue (dry weight) will be required (4.2 g is preferred). The corresponding sample mass required to achieve this analytical sample mass varies by species, tissue, and analyses as summarized in Table A2. The quality of tissue collected is unknown and the actual amount of dry mass that will be obtained after sample processing for analysis is estimated. Therefore, sampling will not be limited to the minimum wet weight required for each species when additional sample mass from an individual plant can be collected without harm to the health of the plant or population.

Ideally, each plant tissue sample will come from one individual plant. The required tissue from the plant (e.g., leaves, stems, berries, etc.) will be collected and weighed in the field according to the required weight for laboratory analysis of the appropriate species and tissue (Table A2), as described in SOP-4 (Attachment A2). When possible, discrete samples will be taken from physically distant individuals of the same species. Physically distant individuals are less likely to be closely genetically related, are less likely to be sharing nutrients through connected root tissues, and are more likely to uptake nutrients from soil with different COI concentrations. If all individuals of a target species occur within a patch, individuals as far from one another as possible will be selected for sampling.

Some plant species may not have enough of the targeted plant material to obtain the mass required for a single sample from one individual plant. In that case, plant material may be collected from multiple adjacent individuals located in proximity and combined into a single composite plant tissue sample. Individual plants located in proximity are more likely to be genetically related, more likely to be sharing nutrients through connected root networks, and are more likely to uptake nutrients from soil with similar COI concentrations. GPS coordinates will be collected for each plant sampled as part of a composite sample if they are farther apart than 3 m (GPS accuracy limitations would preclude meaningful interpretation of points within 3 m). Individual plants within a

[8] Analysis of mercury will be limited to the following plant targets: kinnikinnick leaves, wild rose leaves and stems, wild mint, willows, and tules. Where the quantity of plant material is not sufficient for analysis of TAL metals and mercury, allocation of sample mass collected for these targets will be prioritized for analysis of TAL metals (except calcium, magnesium, potassium, and sodium).

composite must be collected from within the same SA. If a composite is collected from all available plants within one SA but remains below the minimum sample mass, one of three actions will take place: root or bulb samples will be replanted roughly where they were collected so they can re-sprout; berry, leaf, or stem samples will be submitted to the laboratory as a sample if they are within 10 percent of the minimum sample mass because they may weigh enough to constitute a sample once dried; berry, leaf, or stem samples will be left in the SA for natural decomposition if they are less than 10 percent of the minimum sample mass. If fewer than three samples are collected for any given species, these samples will be archived at the laboratory pending EPA guidance.

The addition of mercury analysis for selected plant targets (kinnikinnick leaves, wild rose leaves and stems, wild mint, willows, and tules only) and their co-located soil samples requires more than two times the sample mass required for analysis of TAL metals excluding calcium, magnesium, potassium, sodium, and mercury. At each SA, the survey team will identify potential limitations regarding the availability of sufficient plant material for kinnikinnick leaves, wild rose leaves and stems, wild mint, willows, and tules to support analysis of mercury as well as TAL metals. If available plant material is not expected to be sufficient to support both types of analysis, allocation of available material for analysis of TAL metals will be prioritized and the decision documented by the field team in consultation with EPA, CCT, and TAI field representatives.

Collection of additional plant material designated for field replicates or EPA split samples will occur opportunistically from robust individual plants that have sufficient target plant material to collect two to three times the target sample mass without harming the plant.[9] For replicates, that will be an additional sample. For splits, it will be a sample with twice the mass. Field replicate samples will not be obtained for target plant tissue that would require dividing a bulb or root. Laboratory split samples can be made from any type of tissue, provided sufficient sample mass is available to create a comparable split sample. Replicates of composite samples will not be collected. The identification of field replicate sample status will be withheld from the analytical laboratory. Samples designated as potential EPA splits will be identified as such and submitted to the laboratory for further processing and splitting.

Individual plant sample characteristics to be recorded in the field forms include taxonomic identification, GPS unit used to record location of an individually sampled plant

[9] For kinnikinnick leaves, wild rose leaves and stems, wild mint, willows, and tules, allocation of limited plant material from an individual plant will be prioritized as follows: analysis for TAL metals, then analysis for mercury, then collection of a field replicate.

contributing to a composite sample that are more than 3 m apart, and sample IDs of other plants collected from the same patch (see SOP-5 in Attachment A2). Examples of field forms used to enter data are provided in Attachment A3. Each sampled plant will be photographed (either individually or as a group), and the photograph ID will be documented in the field so that the photograph can be subsequently labeled with location, date, and time of sample (SOP-6). All plant tissue samples will be placed in a resealable plastic bag. Plant materials that exceed the dimensions of the plastic bag will be cut down to size or stored in multiple bags, as necessary. For root samples, soil adhering to the roots will be shaken off into the sample location to limit overestimating root mass contributed by soil. Bagged plant tissue and the associated label will be placed in a second resealable plastic bag and then placed in a cooler with ice, and shipped according to SOP-7. Plant tissues that require further processing will be processed in the laboratory prior to analysis (e.g., removing pine nuts from pine cones).

Co-Located Soil/Sediment Sampling

A co-located surface soil/sediment sample from 0 to 3 in. below ground surface will be collected with each plant tissue sample. The soil/sediment sample location will be collected within the expected root zone of each individual plant sampled; the exact location in relation to each plant species is dependent on plant species and is detailed in SOP-4. If more than one plant species targeted for sampling are growing together at the same location, a single co-located soil/sediment sample may be collected and associated with both plant tissue samples. If mass limitations at an individual plant necessitate collection of a composite plant tissue sample, the co-located soil sample will also be collected as a composite of co-located soil samples for each individual plant contributing to the composite plant tissue sample.

Each co-located soil/sediment sample will be dried and sieved in the laboratory to a target particle size of less than 150 μm. At least 200 g of soil/sediment must be collected for each co-located soil/sediment sample in order to have sufficient mass for analysis after sieving.

SOP-9A through SOP-9C provide more details about co-located soil/sediment sample collection procedures including adjustment of locations, as needed, in the event of rocks or other obstructions. Sample collection details for each sample will be recorded in the field form. A digital photograph of the sample location will be taken and the photograph ID will be documented in the field so that the photograph can be subsequently labeled with location, date, and time of sample (SOP-6).

Each discrete soil sample and each soil component contributing to a composite soil sample will be placed into a quart-sized re-sealable plastic bag for inspection in the field by a

cultural resources representative according to SOP-9A and SOP-9B, respectively. Each sediment sample will be placed into a decontaminated stainless-steel mixing bowl for inspection by a cultural resources representative according to SOP-9C. Sediment samples are not examined in plastic bags because the high moisture content of sediment makes it difficult to see potential artifacts through the plastic of the bag; the plastic can become muddy and opaque. Soil and sediment samples must pass inspection by the cultural resources representative prior to being transferred into a laboratory-supplied sample jar. Samples designated for analysis of mercury will then be placed in a cooler with ice for further processing, packaging, and shipping according to SOP-7. Samples designated for analysis of TAL metals without mercury will be processed, packaged, and shipped without refrigeration or ice, according to SOP-7. In the event that a soil/sediment sample does not pass cultural resources review, sampling will stop and procedures documented in the Archaeological Monitoring Protocol (Attachment A4) will be followed.

2.2.5 Sampling Contingencies

During the course of sampling, field conditions or circumstances may adversely affect sampling success. Such conditions or circumstances may include, but are not necessarily limited to, the presence of cultural resources (refer to the cultural resources coordination plan, Appendix C of the quality assurance project plan [QAPP]), absence of targeted plant tissues, sample area access issues, and presence of wildlife (e.g., bears, venomous snakes, etc.) and other animals (e.g., open range cattle). Multiple potential sampling areas have been identified for this sampling effort to help accommodate such circumstances. Decision trees will assist the field sampling crew in deciding which SA to visit given their need for tissues from certain plant species (SOP-1). The field sampling crew will sample between 6 to 12 SAs of the possible 16 SAs in any one field event to reach the maximum level of effort (10 SAs in spring and late June, 12 in late August; see SOP-1). The field sampling crew will also select specific sampling locations within the SAs based on plant presence.

If the targeted number of plant tissue samples for a given target tissue is not met after the maximum level of effort identified in this FSP has been completed, the number of samples collected will be less than what was targeted.

2.2.6 Sample Mass Requirements

Sampling for plant tissue will be conducted to inform the HHRA. To conduct the desired analyses for the refined list of chemicals identified in the QAPP, species-specific target sample masses have been determined and are summarized in Table A2. Note that these minimum tissue needs are based on estimated dry weight of the target plant tissues.

2.2.7 Sample Acceptability and Quality Assurance

To ensure that a minimal sample quality is achieved, acceptance criteria will be applied to each individual sample that is collected, as described in Section 2.2.4. Field personnel will apply the criteria for plant tissue collection using their experience and best professional judgment.

2.2.8 Quality Control Samples

QC samples will include equipment rinsate blanks, field replicate tissue and soil/sediment samples (i.e., field duplicates), EPA split samples (i.e., inter-laboratory splits), and matrix spike/matrix spike duplicate (MS/MSD) analyses. Laboratory QC samples will be used to assess sample representativeness, completeness, and variability and evaluate potential sources of contamination, as well as to assess laboratory precision, accuracy or bias, and comparability. Given the likelihood that insufficient tissue may be collected for some target plant tissues, allocating tissue to chemical analyses of primary field samples will be prioritized. For field samples designated for EPA splits, the analytical laboratory will be instructed to process the EPA split sample at a targeted frequency of 5 percent of the total number of samples, or one sample per analytical batch (whichever is greater). Field replicate samples will be submitted to the analytical laboratory as independent samples and will be processed the same as field samples. Detailed information on quality assurance and quality control (QA/QC) procedures, limits, and reporting is provided in the QAPP.

2.2.9 Location and Sample Event IDs

Location and sample event IDs will identify the sample area and sample event from which each sample was collected. All high and lower lead SAs were selected *a priori* to this study based on prior information[10]. Sample events were selected *a priori* to the current study based on CCT harvest seasons[11]. These location and sample event IDs will consist of the following parts:

- Four-digit location identification codes with sequential numbers (e.g., SA01, SA02, SA03). The first three SAs, SA01 to SA03, represent the high lead sample areas; SA04 to SA16 represent lower lead sample areas.

[10] September 20, 2017 Lodestone Memorandum (Lodestone 2017a), and the August 2017 field reconnaissance (AECOM 2017), which was in turn informed by the prior CCT field reconnaissance efforts (Lodestone 2016a,b, 2017b) and the CCT Tribal Consumption and Resource Use Survey (Westat 2012).

[11] September 20, 2017 Lodestone Memorandum (Lodestone 2017a).

- Two-digit sample event designation—SP for spring, JU for late June, and LA for late August.

Examples:

SA04-SP = lower lead SA number 4 sampled in the spring
SA01-LA = high lead SA number 1 sampled in late August.

These location and sample event IDs will be used to document sampling locations and times within the SAs.

2.2.10 Sample IDs for Individual and Composite Samples

Each individual sample will be assigned a unique identifier, whether that sample is from a single plant or is a composite. The sample ID will include the location and sample event ID (as described in Section 2.2.9), the individual number, and the soil/sediment or plant code as shown below.

- Six-digit code that combines the SA ID, and sampling event code—see Table A1 for the four-digit SA IDs
- Two-digit sequential number to indicate location of sample
- One-digit code to designate a plant or co-located soil/sediment sample—P for plant, S for soil/sediment
- Two-digit number to indicate if more than one specimen was collected from that location.

Examples:

SA04-SP05-P01 = First plant tissue sample collected from lower lead SA number 4, sampled in the spring from location 5.

SA01-LA02-S02 = Second co-located soil sample collected in high lead SA number 1 sampled in late August from location 2.

Individual plant tissue samples will be double-bagged, and a label will be included between the two bag layers to ensure that all samples can be individually tracked and are sufficiently protected (see SOP-3 and SOP-7). Soil/sediment samples will be placed into wide-mouthed jars after inspection by a cultural resources representative (SOP-9).

2.2.11 Equipment Decontamination Procedures

All sampling equipment coming into direct contact with samples will be decontaminated prior to beginning field work, between sampling locations (except in the case of composite samples), and at the conclusion of the field effort as outlined in SOP-8 (Attachment A2). Nitrile or latex gloves will be used for handling samples and will be discarded in between

sampling locations (except in the case of composite samples) to avoid transfer of potential contaminants.

2.3 SAMPLE HANDLING

Records will be maintained to document all activities and data associated with field sampling and chemical analyses. Results of data verification and validation activities will also be documented. Procedures for documenting field activities are described herein (see SOP-5); laboratory procedures are presented in Appendix B of the QAPP.

Planning and documentation of all activities are emphasized to ensure that sample identity and integrity are preserved during all stages of the field operation (SOP-11). The following documentation will be provided with samples:

- A field form that contains information about each sampling location and sampling event
- Sample coordinates (recorded on the field form and/or recorded electronically)
- Photograph documentation (SOP-6)
- A sample identification label that accompanies and identifies each individual sample
- A COC form that provides continuous tracking information for all samples
- A COC label that seals each shipping container.

The following information will be handwritten on the sample label at the time of collection with an indelible marker (or preprinted sample labels may be used, see SOP-3):

- Sample ID
- Location ID (e.g., SA06-SP)
- Sampler's initials
- Date
- Time.

If necessary, corrections will be made on the sample labels by drawing a single line through the error and entering the correct information with an indelible marker. All corrections will be initialed and dated by the person performing the correction. If possible, the individual who made the error will correct it.

Plant tissue samples will be placed inside resealable plastic bags. The bagged sample and sample label will then be placed inside a second resealable plastic bag. This process will ensure that when the samples are prepared for shipment, the label will remain with the

correct plant sample. Soil samples will be placed in wide-mouthed jars after they have been inspected by a cultural resources representative.

The following characteristics of individual samples will be recorded on the specimen collection form:

- Taxonomic identification of collected specimens (for plant tissues only)
- Location and sampling event collection information (e.g., location and sampling event ID, date, and time)
- Note to identify which GPS unit recorded the sampling location details (plant tissue samples and co-located soil samples will be collected in proximity; therefore, a single GPS location will be collected to represent both sample types at each location)
- Photograph ID (photographs will be taken of all plants sampled and all locations where soil samples were extracted)
- ID numbers of other samples taken from the same patch
- Whether the tissue and co-located soil sample is from one individual plant or is a composite of tissue or soil from more than one individual plant location.

2.4 CULTURAL RESOURCES

A cultural resources coordination plan has been prepared for the RI/FS to provide relevant background information about site-related cultural resources, define measures for protecting resources, and define procedures for consulting with the appropriate state, federal, and tribal parties with interests in the cultural resources of the Site. Because field sampling methods associated with this investigation involve ground disturbance, TAI and its technical team will work with the CCT to assess the effects of the planned work and seek ways to avoid, minimize, or mitigate any adverse effects on historic properties. SOPs for each sampling method are provided in Attachment A2 to this FSP. Handling and reporting of cultural resources is provided in Attachment A2 as SOP-10.

In accordance with the cultural resources coordination plan, an archaeological monitor and/or tribal representative will be present on the site when sampling or sampling-related activity occurs (but not when the survey team is scouting because no ground-disturbing activities will occur at that time). The archaeological monitor and/or tribal representative will visually examine the area prior to collection of each sample. The archaeological monitor and/or tribal representative will not make physical contact with the sample unless artifacts or other cultural deposits are present. If artifacts or potential archaeological deposits are present, the archaeological monitor or tribal representative will record the location of the materials and photograph the materials in place in such a manner to provide information

on provenience. The artifacts and other archaeological materials will then be re-deposited at their original location. At the discretion of the archaeological monitor or tribal representative, a specific sample location may be relocated from the location of the discovery. Such relocation will be coordinated with the field supervisor and documented in the field logbook. These procedures, collectively referred to as the Archaeological Monitoring Protocol, are summarized in the cultural resources coordination plan and also reproduced in Attachment A4 to this FSP.

2.5 SAMPLE PACKAGING AND TRANSPORT

This section describes procedures for handling samples prior to shipping to the analytical laboratory (see SOP-7 in Attachment A2). Packaging and transport procedures vary for plant tissue samples and soil samples. For soil samples that will not be submitted for mercury analysis, the EPA Regional Quality Assurance Manager has approved storage and shipment of the soil samples without refrigeration or use of ice (Tonel 2017). Storage and shipment with refrigeration is required for soil samples designated for analysis of mercury in addition to TAL metals.

After completing each day of sampling, all plant tissue samples will be transferred from the coolers with wet ice into a freezer or cooler with dry ice, and held there until preparation for shipment to the laboratory. Samples will be frozen within 20 hours of collection; typically as soon as the field crew returns to the location where samples are being held until shipment to the laboratory. The temperature of the cooler/freezer will be recorded in the logbook twice daily (both in the morning and evening).

In the field, soil samples will be sealed into wide-mouthed jars and placed in individual sealable plastic bags prior to storage. Soil samples designated for analysis of TAL metals without mercury will be stored at 4 ±2°C until being shipped to the laboratory.

A secure area will be available for sample holding and preparing plant tissue and soil samples for shipment to the laboratory (SOP-7 in Attachment A2).

The following steps will be taken to prepare samples for shipment:

1) Review field logs regarding sample characteristics
2) Leave the original sample label with the appropriate sample
3) Ensure that appropriate SOPs have been followed regarding sample identification
4) Further prepare sample for shipment to the analytical laboratory (as needed) and complete the COC forms.

The storage temperature will be maintained for plant tissue samples and soil samples designated for analysis of mercury in a secure area while they are awaiting shipping. Prior to shipping to the analytical laboratory, plant samples will be packed in hard-sided coolers on dry ice. Sealed soil sample jars enclosed in individual sealable plastic bags will be packed in hard-sided coolers to prevent breakage and separated in the cooler by bubble wrap or other shock-absorbent material. Separate coolers will be used for soil samples that require refrigeration (those designated for mercury analysis) and those that do not require refrigeration (no mercury analysis). Wet ice in sealed plastic bags will be placed in the cooler of soil samples that require refrigeration to maintain a temperature of approximately 4 ±2°C. All samples will be shipped via priority overnight delivery service or courier service so that they arrive at ALS within 48 hours from the time of sample shipment.

For plant tissue samples, appropriate shipping containers will be selected for dry ice, and appropriate shipping labels indicating the use of dry ice will be affixed to the containers. Sufficient plant tissue samples will be placed in each laboratory-supplied hard-sided plastic cooler to occupy approximately 60 to 70 percent of the cooler volume, and the remaining space in the cooler will be filled with dry ice. Plant tissue samples will be placed inside a large plastic bag (e.g., sturdy garbage bag or drum liner); the bag will be tied closed and sealed at the tied area with a custody seal to ensure that custody is maintained if the cooler is opened for inspection during shipment. Completed COC forms will be placed in resealable plastic bags and included in each cooler. After each cooler is packed with samples and ice, it will be secured at both ends with nylon strapping tape and the following items will be attached:

- Address label for processing laboratory
- Two custody seals
- Overnight shipping airbill (unless transported via courier service)
- Perishable goods label and at least one of the following labels: "This End Up," "Fragile," or "Handle With Care."

For soil samples, appropriately sized coolers will be selected for packaging and the inside cleaned of gross contamination. The inside of each cooler will be lined with bubble wrap. A large opened plastic bag (e.g., sturdy garbage bag or drum liner) will be placed inside the bubble wrap-lined cooler before placing any soil samples inside the cooler. Soil samples (which at the sample collection site have already been placed in individual sealable plastic bags) will be individually wrapped with bubble wrap and placed inside the bag-lined cooler. For coolers with soil samples designated for mercury analysis, the wrapped sample jars will be placed in a designated laboratory-supplied hard-sided plastic cooler to occupy approximately 60 to 70 percent of the cooler volume, and the remaining space in the cooler

will be filled with wet ice in sealed plastic bags and bubble wrap to keep samples from shifting during transport. The bag will be tied closed and sealed at the tied area with a custody seal to ensure that custody is maintained if the cooler is opened for inspection during shipment. Completed COC forms will be placed in resealable plastic bags and included in each cooler. After the cooler is sufficiently packed to prevent shifting of the containers, it will be secured at both ends with nylon strapping tape and the following items will be attached:

- Address label for processing laboratory
- Two custody seals
- Overnight shipping airbill (unless transported via courier service)
- At least one of the following labels: "This End Up," "Fragile," or "Handle With Care."

2.6 STUDY-DERIVED WASTE

All disposable materials and supplies used for sample collection and processing (e.g., paper towels, gloves) will be placed in heavyweight garbage bags or other appropriate containers. This waste will be placed in a normal refuse container for disposal at a solid waste landfill.

3 FIELD DOCUMENTATION

The integrity of each sample from the time of collection to the point of data reporting must be maintained. Proper record-keeping and COC procedures will be implemented to allow samples to be traced from collection to final disposition. Representative photographs will be taken of each type of sampling activity performed during the study. Site photographs from various angles and views of the specific sampling locations within the SAs will be collected.

3.1 FIELD LOGBOOK

All field activities and observations will be noted in a field log (SOP-5). The field log will be either a bound document containing individual field and sample log forms or an electronic tablet (backed up daily) containing the same documentation. Information will include personnel, date, time, SA and sample event, sampler, types of samples collected, and general observations. Any changes that occur during sampling (e.g., personnel, responsibilities, deviations from the FSP) and the reasons for these changes will be documented in the field log. The log will identify onsite visitors (if any) and the number of photographs taken at each sampling location (if this information is not recorded on the sampling forms). The field supervisor is responsible for ensuring that the field log and all field data forms are correct; if electronic records are kept, the field supervisor will upload those to the secure project website on a daily basis, or as often as practical. Requirements for keeping logbooks include the following:

- If paper logbooks are used:
  - They will be bound all-weather paper, with consecutively numbered pages.
  - Removal of any pages, even if illegible, will be prohibited.
  - Entries will be made legibly with black (or dark) waterproof ink.
  - Corrections will be made by drawing a single line through the original entry, with the corrected entry written alongside the original. Corrections will be initialed and dated and may require a footnote for explanation.
- Each day's first entry will be made on a new, blank page.
- Easy to understand, descriptive language will be used.
- Entries will be made while activities are in progress or as soon afterward as possible (the date and time that the notation is made should be noted, as well as the time of the observation itself).

- Blank lines on a page or blank pages in the logbook will be lined out to indicate that they were intentionally left blank.
- The date and time, based on a 24-hour clock (e.g., 0900 for 9:00 am and 2100 for 9:00 pm), will appear on each page.
- The field supervisor must sign and date the last page of each daily entry in the field logbook (either electronic or hardcopy).

In addition to the preceding requirements, if a paper logbook is used, the person recording the information must initial and date each page of the field logbook. If more than one individual makes entries on the same page, each recorder must initial and date each entry. The bottom of the page must be signed and dated by the individual who makes the last entry. The field supervisor, after reading the day's entries, also must sign and date the last page of each daily entry in the field logbook.

The type of information that may be included in the field logbook and/or field forms includes the following:

- Task name and sampling locations within each SA
- Sample event (e.g., spring, late June, late August)
- Task start date and end date
- Weather conditions
- Name of person making entries and other field staff (including EPA oversight)
- Onsite visitors, if any
- Date and collection time of each sample
- The sampling location name
- Coordinates of plant and soil/sediment samples
- Specific information on each type of sampling activity (including whether the quantity of plant material for kinnikinnick leaves, wild rose leaves and stems, wild mint, willows, or tules is too limited to collect the additional mass required for mercury analyses as well as TAL metals)
- Observations made during sample collection
- Number of photographs taken at each sampling location
- A record of site health and safety meetings, updates, and related monitoring
- Any deviation from the sampling plan and reasons for deviation.

In addition, a sampling location map will be updated during sampling and will be maintained throughout the sampling event. This map will include the specific plant species

and plant parts collected in each SA. All logs must be completed at the time any observations are made. It is advisable to, when possible, photocopy each day's entries to provide a backup copy that can be kept at a secure location (e.g., field laboratory or hotel room). When field activities are complete, the logbooks and all forms will be retained by TAI and its technical team as hardcopy and/or pdf files. These documents will be entered into the TAI technical team project file.

3.2 CHAIN-OF-CUSTODY PROCEDURES

Samples are in custody if they are in the custodian's view, stored in a secure place with restricted access, or placed in a container secured with custody seals. Samples will not be outside of designated personnel's custody unless the samples have been transferred to a secure area (i.e., locked up and custody sealed) or transferred to the laboratory. If the samples cannot be placed in a secure area, then a field team member must physically remain with the samples at all times (e.g., at meal times). A COC record will be signed by each person who has custody of the samples and will accompany the samples at all times. Copies of the COC form will be included in laboratory and QA/QC reports. Attachment A3 contains an example of the COC form that will be used during the study, with directions for how to fill out the form in SOP-7 (Attachment A2).

The COC form will be either paper or electronic and, at a minimum, will include the following information:

- Project name
- Field supervisor's name and team members responsible for collection of the listed samples
- Sample identification number
- Collection date and time for each sample
- Sample type (e.g., plant tissue, soil, or sediment sample)
- Number of sample containers (e.g., coolers) shipped
- Requested analyses for each sample (as shown in Table A7-3 of the QAPP)
- Name, date, time, and signature of the relinquishing and receiving personnel (this does not include commercial shipment carriers).

The field supervisor, as the designated field sample custodian, will be responsible for all sample tracking and COC procedures for samples in the field. The field sample custodian will be responsible for final sample inventory and will maintain sample custody documentation. The field sample custodian will complete the COC form prior to removing

samples from the field. Upon transferring the samples to the laboratory, sample custodian, or shipping courier, the field supervisor will sign, date, and note the time of transfer on the COC form. The original COC form will be transported with the samples to the laboratories. All samples will be shipped to the testing laboratories in coolers sealed with custody seals.

Each laboratory will designate a sample custodian who will be responsible for receiving samples and documenting their progress through the laboratory analytical process. The sample custodian for each laboratory will confirm the integrity of the custody seals upon sample arrival at the laboratory. The laboratory sample custodian will also ensure that the COC and sample tracking forms are properly completed, signed, dated, and initialed upon receipt of the samples.

Upon receipt of the samples by the laboratory, the laboratory sample custodian will measure the internal cooler temperature and inventory the samples by comparing sample labels (i.e., number of samples and sample IDs) to those on the COC form. For plant samples or soil samples designated for mercury analysis, if sample temperatures fall outside the temperature range specified for sample preservation (i.e., the internal temperature of the cooler is >0°C for plant samples or outside the 4°C ±2°C range specified for soil and sediment samples designated for mercury analysis), the field supervisor will be alerted immediately and field personnel will increase the amount of ice used in the field and shipped with subsequent samples. The laboratory sample custodian will enter the sample numbers into a laboratory tracking system by task code and sample designation. The custodian will assign a unique laboratory sample identifier to each sample number and will be responsible for distributing the samples to the appropriate analyst or for storing samples at the correct temperature in an appropriate and secure area.

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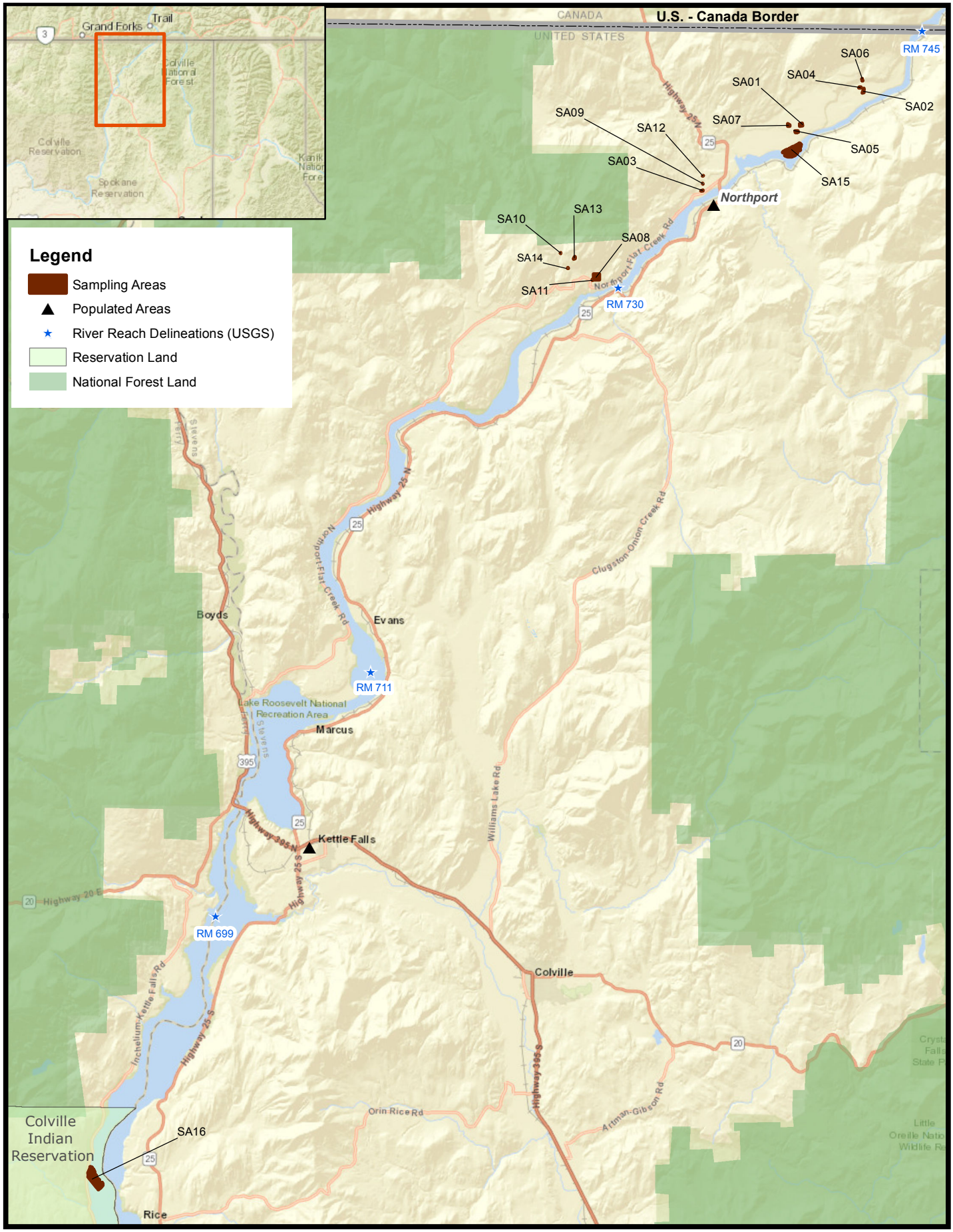
Maps



RAMBOLL ENVIRON

0 1 2 4 Kilometers

0 1 2 4 Miles

N

Map A1. Proposed Sampling Areas

Upper Columbia River, WA

RAMBOLL ENVIRON

Map A2. Detail for Sampling Area 01
Upper Columbia River, WA

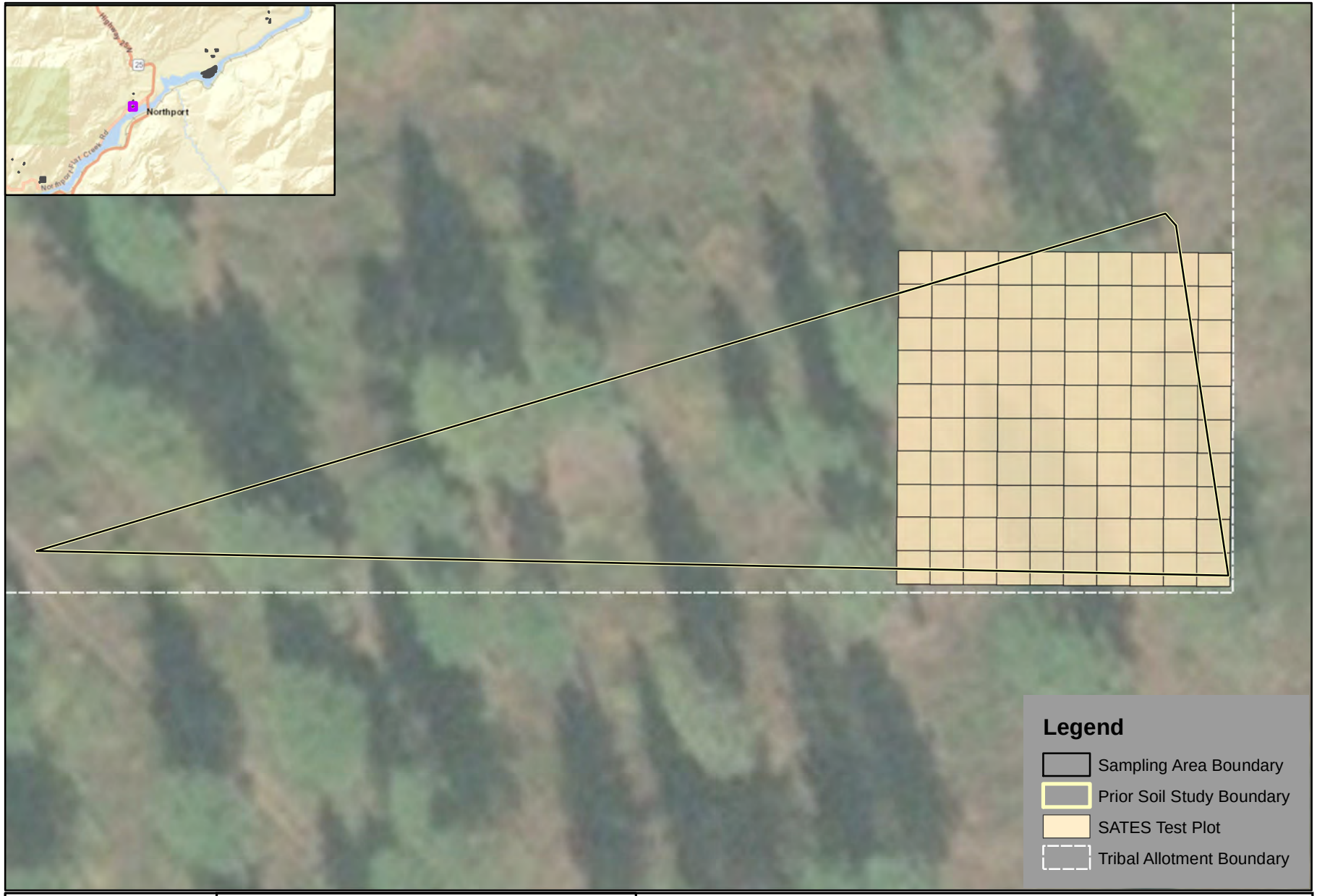
RAMBOLL ENVIRON

0 10 20 40
Meters

0 50 100 200
Feet

N

Map A3. Detail for Sampling Area 02
Upper Columbia River, WA



RAMBOLL ENVIRON

Map A4. Detail for Sampling Area 03
Upper Columbia River, WA

RAMBOLL ENVIRON

Map A5. Detail for Sampling Area 04
Upper Columbia River, WA

RAMBOLL ENVIRON

Map A6. Detail for Sampling Area 05
Upper Columbia River, WA

RAMBOLL ENVIRON

0 5 10 20 Meters

0 25 50 100 Feet

N

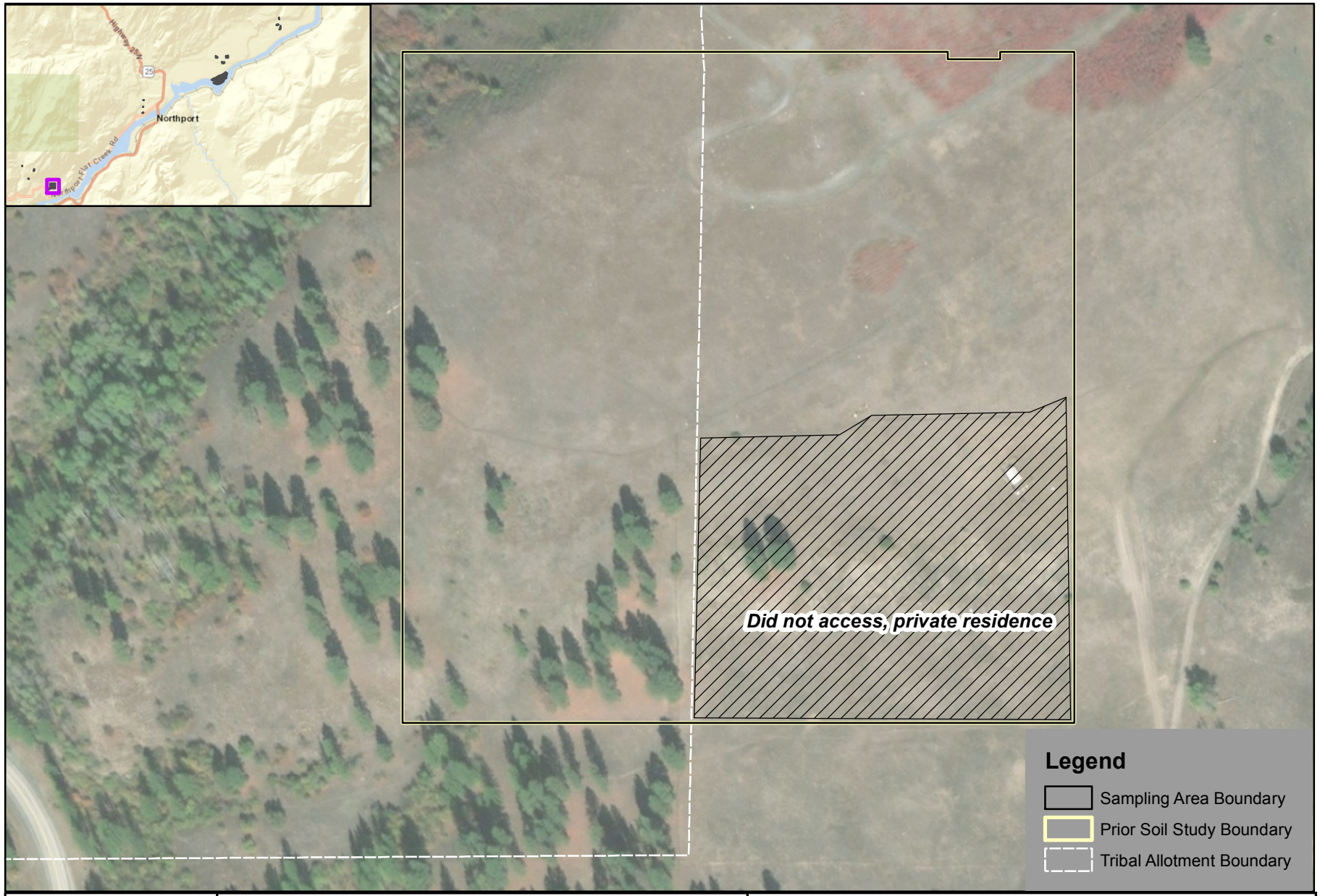
Map A7. Detail for Sampling Area 06
Upper Columbia River, WA

RAMBOLL ENVIRON

0 5 10 20
Meters
0 25 50 100
Feet

N

Map A8. Detail for Sampling Area 07
Upper Columbia River, WA



RAMBOLL ENVIRON

Map A9. Detail for Sampling Area 08
Upper Columbia River, WA

RAMBOLL ENVIRON

0 5 10 20 Meters

0 25 50 100 Feet

N

Map A10. Detail for Sampling Area 09
Upper Columbia River, WA

Map A11. Detail for Sampling Area 10
Upper Columbia River, WA

RAMBOLL ENVIRON

0 5 10 20 Meters

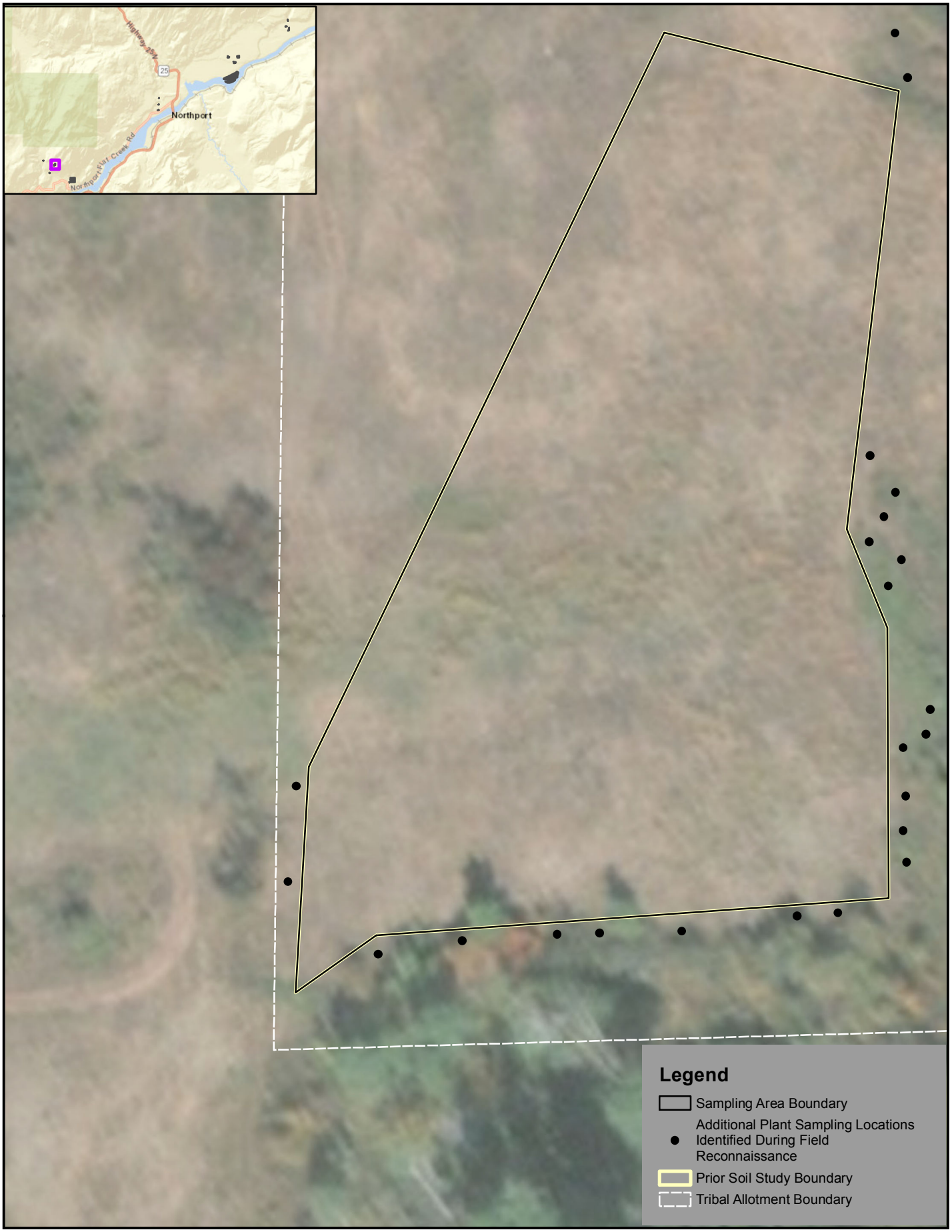
0 25 50 100 Feet

N

Map A12. Detail for Sampling Area 11
Upper Columbia River, WA

RAMBOLL ENVIRON

Map A13. Detail for Sampling Area 12
Upper Columbia River, WA



RAMBOLL ENVIRON

0 5 10 20
Meters
0 25 50 100
Feet
N

Map A14. Detail for Sampling Area 13
Upper Columbia River, WA

RAMBOLL ENVIRON

Map A15. Detail for Sampling Area 14
Upper Columbia River, WA

RAMBOLL ENVIRON

Map A16. Detail for Sampling Area 15
Upper Columbia River, WA

RAMBOLL ENVIRON

0 50 100 200
Meters
0 250 500 1,000
Feet
N

Map A17. Detail for Sampling Area 16
Upper Columbia River, WA

TABLES

Table A1. Proposed Sampling Areas

Sampling Area ID	Overlap with Prior UCR RI/FS Sampling Area[a]	Type of Property Ownership/ Management	Rationale for Inclusion	Average IC Soil Lead (mg/kg)[a]	Average IC Soil Arsenic (mg/kg)[a]
SA01	2014R-258	Tribal allotment	High soil lead. Sarvisberry, kinnikinnick, black tree lichen, ponderosa pine, chokecherry, wild rose, camas and wild strawberry recorded during August 2017 field reconnaissance, as well as habitat for bitterroot, lomatium, morel, shaggy mane, spring beauty, and Indian carrot.	678	46.8
SA02	2014R-401	Tribal allotment	High soil lead. Sarvisberry, kinnikinnick, ponderosa pine, chokecherry, and hazelnut recorded during August 2017 field reconnaissance, as well as habitat for morels.	1120	80.8
SA03	2014R-441	Tribal allotment	High soil lead. Sarvisberry, kinnikinnick, ponderosa pine, chokecherry, wild rose, hazelnut, and wild strawberry recorded during August 2017 field reconnaissance, as well as habitat for morels.	624	43.6
SA04	2014R-402	Tribal allotment	Sarvisberry, kinnikinnick, ponderosa pine, chokecherry, wild rose, hazelnut, dwarf huckleberry, and wild strawberry recorded during August 2017 field reconnaissance, as well as habitat for bitterroot, lomatium, morel, shaggy mane, and Indian carrot.	542	34
SA05	2014R-410	Tribal allotment	Sarvisberry, kinnikinnick, black tree lichen, ponderosa pine, chokecherry, and wild rose recorded during August 2017 field reconnaissance, as well as habitat for bitterroot, lomatium, shaggy mane, and Indian carrot.	370	35
SA06	2014R-403	Tribal allotment	Sarvisberry, kinnikinnick, wild rose, and hazelnut recorded during August 2017 field reconnaissance, as well as habitat for shaggy mane.	394	26.1

Table A1. Proposed Sampling Areas

Sampling Area ID	Overlap with Prior UCR RI/FS Sampling Area[a]	Type of Property Ownership/ Management	Rationale for Inclusion	Average IC Soil Lead (mg/kg)[a]	Average IC Soil Arsenic (mg/kg)[a]
SA07	2014R-259	Tribal allotment	Sarvisberry, black tree lichen, ponderosa pine, chokecherry, and wild rose recorded during August 2017 field reconnaissance, as well as habitat for bitterroot, lomatium, shaggy mane, and Indian carrot.	226	19.7
SA08	2014U-ADA-023	Tribal allotment	Sarvisberry, black tree lichen, ponderosa pine, chokecherry, wild rose, and hazelnut recorded during August 2017 field reconnaissance, as well as habitat for bitterroot, lomatium, shaggy mane, and Indian carrot.	151	18.3
SA09	2014R-442	Tribal allotment	Sarvisberry, ponderosa pine, chokecherry, wild rose, red willow, hazelnut, and wild strawberry recorded during August 2017 field reconnaissance, as well as habitat for shaggy mane, spring beauty, and Indian carrot.	243	21.7
SA10	2016R-808-O2	Tribal allotment	Sarvisberry, kinnikinnick, ponderosa pine, puffball, and wild rose recorded during August 2017 field reconnaissance, as well as habitat for bitterroot, lomatium, shaggy mane, and Indian carrot.	42.6	6.98
SA11	2016R-804-O1	Tribal allotment	Sarvisberry, black tree lichen, ponderosa pine, wild rose, and hazelnut recorded during August 2017 field reconnaissance, as well as habitat for bitterroot, lomatium, shaggy mane, and Indian carrot.	121	7.74
SA12	2014R-440	Tribal allotment	Sarvisberry, black tree lichen, chokecherry, and hazelnut recorded during August 2017 field reconnaissance, as well as habitat for shaggy mane, spring beauty, and Indian carrot.	136	9.12
SA13	2016R-801-O3	Tribal allotment	Sarvisberry, chokecherry, and wild rose recorded during August 2017 field reconnaissance, as well as habitat for bitterroot, lomatium, shaggy mane, and Indian carrot.	37	15.1

Table A1. Proposed Sampling Areas

Sampling Area ID	Overlap with Prior UCR RI/FS Sampling Area[a]	Type of Property Ownership/ Management	Rationale for Inclusion	Average IC Soil Lead (mg/kg)[a]	Average IC Soil Arsenic (mg/kg)[a]
SA14	2016R-805-O2	Tribal allotment	Sarvisberry, black tree lichen, wild rose, hazelnut, wild mint, and tule recorded during August 2017 field reconnaissance, as well as habitat for bitterroot, lomatium, shaggy mane, and Indian carrot.	54.1	5.31
SA15[b]	RFA-001, RFA-002, RFA-003, RFA-004, RFA-005	Washington Department of Natural Resources	Possible presence of willows in a previously sampled area with moderately high concentrations of lead in relict floodplain soil.	389[c]	15.8[c]
SA16[b]	Barnaby Island Campground	National Park Service	Possible presence of willows in a previously sampled area with low concentrations of lead in beach sediment.	46.2[d]	1.99[d]

Notes:

[a] Based on the UCR 2010 Beach Sediment Study (Integral 2014), the UCR 2014 Residential Soil Study (CH2M Hill 2016), the UCR 2014 Upland Soil Study (Windward et al. 2015), or the UCR 2016 Residential Soil Study (Ramboll Environ 2017).

[b] Sampling areas were not visited during the August 2017 field reconnaissance event. Areas are publicly-accessible and are included for potential sampling of willows.

[c] These averages represent the average of pre-averaged replicates for each of the five decision units sampled.

[d] Averages are based on sample results for <63 µm, 63 to 125 µm, and 125 to 250 µm size fraction samples.

IC - incremental composite

Table A2. Target Plant Tissue and Soil Sample Mass Requirements

Field Event	Sample Type	Plant Scientific Name	Target Plant Tissue	Target Sample Mass[a]	Minimum Sample Mass	Alternate Target Sample Mass	Alternate Minimum Sample Mass
All	Soil	NA	NA	200 g	200 g		
	Black tree lichen	*Bryoria fremontii*	Lichen	2.3 g	1.2 g		
Mid April to Early May	Camas	*Camassia quamash*	Bulbs	4.5 g	2.3 g		
	Kinnikinnick[b]	*Arctostaphylos uva-ursi*	Leaves	5.3 g	2.7 g	2.5 g	1.3 g
	Wild rose (stems and leaves)[b]	*Rosa* spp.	Leaves	48.5 cm with leaves	24.5 cm with leaves	23 cm with leaves	11.5 cm with leaves
	Puffball[c]	*Calvatia gigantea*	Fruiting body	5.3 g	2.7 g		
	Bitterroot	*Lewisia rediviva*	Root	29 g	15 g		
	Lomatium	*Lomatium* spp.	Roots	8.1 g	4.1 g		
	Morel	*Morchella esculenta*	Fruiting body	5.3 g	2.7 g		
	Shaggy mane	*Coprinus comatus*	Fruiting body	20 g	10 g		
	Spring beauty/Indian potato	*Claytonia lanceolata*	Corm	3.8 g	1.9 g		
	Indian carrot[d]	*Perideridia gairdneri*	Roots	13.4 g	6.7 g		
	Green willow[b]	*Salix exigua*	Inner bark	189 cm	126 cm	90 cm	60 cm
	Red willow/red-osier dogwood[b]	*Cornus sericea*	Inner bark	189 cm	126 cm	90 cm	60 cm
Late June	Sarvisberry	*Amelanchier alnifolia*	Berries	31 g	16 g		
	Wild strawberry	*Fragaria vesca & F. virginiana*	Berries	31 g	16 g		
Late August	Chokecherry	*Prunus virginiana*	Cherries	62 g	32 g		
	Hazelnut	*Corylus cornuta var. californica*	Nuts	6 nuts	3 nuts		
	Ponderosa pine	*Pinus ponderosa*	Nuts	20 pine cones	10 pine cones		
	Wild rose (hips)	*Rosa* spp.	Rose hips	8.7 g	4.4 g		
	Huckleberry	*Vaccinium* spp.	Berries	31 g	16 g		
	Wild mint[b]	*Mentha arvensis*	Leaves	8.4 g	4.2 g	4.0 g	2.0 g
	Tule[b]	*Schoenoplectus acutus*	Culms	189 cm	126 cm	90 cm	60 cm

Notes:

[a] Based on consultation with ALS Environmental (ALS), the target sample mass is two times the minimum sample mass expected to result in a 1 g-dw sample for analysis.

[b] Plant target will be analyzed for mercury in addition to other TAL metals (except calcium, magnesium, potassium, and sodium). Where tissue mass is not sufficient for analysis of mercury in addition to TAL metals, allocation of available mass will be prioritized for analysis of TAL metals and sample collection will proceed based on the Alternate Target Sample Mass or Alternate Minimum Sample Mass.

[c] Sample mass estimates are assumed to be the same as morel estimates.

[d] Ideally gathered in May or June, before flowering.

NA - not applicable

ATTACHMENT A1

GENERAL SITE HEALTH AND SAFETY PLAN ADDENDUM
PLANT TISSUE STUDY

CONTENTS

LIST OF TABLES

ACRONYMS AND ABBREVIATIONS

CFR	Code of Federal Regulations
COPC	chemical of potential concern
GPS	global positioning system
HAZWOPER	hazardous waste operations and emergency response
OSHA	Occupational Safety and Health Administration
PFD	personal flotation device
PPE	personal protective equipment
RI/FS	remedial investigation and feasibility study
SHSP	site health and safety plan
Site	Upper Columbia River site
TAI	Teck American Incorporated
UCR	Upper Columbia River
Ramboll	Ramboll US Corporation
WISHA	Washington Industrial Safety and Health Act

UNITS OF MEASURE

ft	foot/feet
L	liter(s)
m	meter(s)

SITE HEALTH AND SAFETY PLAN ADDENDUM APPROVAL

This Addendum to the general site health and safety plan (SHSP) has been reviewed and approved by Teck American Incorporated's (TAI) lead technical consultant (Ramboll US Corporation [Ramboll]) for the Plant Tissue Study at the Upper Columbia River (UCR) site (Site) in support of the remedial investigation and feasibility study (RI/FS) for the Site.

Ramboll Task Manager Date

Ramboll Project Health and Safety Officer Date

SITE HEALTH AND SAFETY PLAN ADDENDUM ACKNOWLEDGEMENT

This Addendum to the general SHSP (TCAI 2009) is approved by TAI for use at the Site. The general SHSP and Addendum are the minimum health and safety standard for the Site and will be strictly enforced for all personnel conducting plant tissue and soil sampling activities at the Site. Subcontracted personnel may request to adopt a subcontractor-specific plan in lieu of this Addendum to the general SHSP, but must obtain prior written approval from TAI and provide written concurrence from the subcontractor that the subcontractor will assume direct responsibility and liability for administering the plan to its employees.

I have reviewed this Addendum to the general SHSP for the study. I have had an opportunity to ask any questions I may have and have been provided with satisfactory responses. I understand the purpose of the plan, and I consent to adhere to its policies, procedures, and guidelines.

Employee signature	Company	Date
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1 INTRODUCTION

This Addendum to the UCR RI/FS general SHSP provides specific Site information and health and safety provisions to protect workers from potential hazards during plant sampling at locations along the UCR.

Site background information and general health and safety provisions to protect workers from potential hazards during work at the Site are presented in the general SHSP (TCAI 2009).

Subcontractors that are contracted to perform field work associated with the RI/FS may adopt the general SHSP and this Addendum or develop and follow their own SHSPs. However, subcontractor SHSPs must be consistent with the provisions outlined in the Addendum and the general SHSP, and any discrepancies will follow the most protective practices.

It is Ramboll's policy to provide a safe and healthful work environment. No aspect of the work is more important than protecting the health and safety of all workers.

Ramboll cannot guarantee the health or safety of any person entering the Site. Because of the potentially hazardous nature of the Site and the activity occurring thereon, it is not possible to regulate personal diligence or to discover, evaluate, and provide protection for all possible hazards that may be encountered. Strict adherence to the health and safety guidelines set forth herein will reduce, but not eliminate, the potential for injury and illness at the Site. The health and safety guidelines in this plan were prepared specifically for the Site and should not be used on any other site without prior evaluation by trained health and safety personnel.

A copy of this Addendum and the general SHSP must be in the custody of the field crew during field activities. All individuals performing field work must read, understand, and comply with this plan before undertaking field activities. Once the information has been read and understood, the individual must sign the Site Health and Safety Acknowledgment Form provided with this Addendum to the general plan. Any changes to the plan will be written in the plan and initialed by all potentially affected field personnel. The signed form and any initialed changes will become part of Ramboll's project file. A copy of the form will be provided to TAI.

This Addendum may be modified at any time based on the judgment of the site safety officer in consultation with the corporate health and safety officer and project manager or designee. Any modification will be presented to the onsite team during a safety briefing and will be recorded in the field notebook.

1.1 ORGANIZATION

Task-specific safety procedures associated with plant sampling are presented in this Addendum to the general SHSP. In addition, this Addendum provides detailed field study area and hospital location maps, air monitoring requirements, specific requirements for personal protective equipment (PPE), work zone definitions, and key emergency contact information.

The general SHSP (TCAI 2009) provides background site information and general health and safety provisions to protect workers from potential hazards during field activities. The information includes general safety guidelines for physical hazards, a chemical hazard evaluation, health and safety training requirements, general PPE requirements, emergency planning, general decontamination procedures, vehicle safety, and spill containment.

1.2 SCOPE OF WORK

Plant tissue and soil sampling events will be conducted by a TAI field contractor during three separate mobilizations (spring 2018, late June 2018, and late August 2018). Plant samples will be collected by TAI's field contractor from up to 16 sampling areas located on Tribal allotments and other publicly accessible locations within the Site. It is anticipated that sampling will be carried out by one vehicle-based team (up to 4 to 6 people) and supported by an additional vehicle-based survey team. The coordinates of each sampling location will be surveyed using a global positioning system (GPS) unit.

1.3 DEFINITIONS

Contamination reduction zone	Area between the exclusion and support zones that provides a transition between contaminated and clean zones
Exclusion zone	Any area of the Site where hazardous substances are present, or are reasonably suspected to be present, and pose an exposure hazard to personnel
HAZWOPER	Hazardous Waste Operations and Emergency Response standard, as described in 29 Code of Federal Regulations (CFR) Part 1910.120
OSHA	Occupational Safety and Health Administration
Support zone	Any area of the Site, so designated, that is outside the exclusion and contamination reduction zones
WISHA	Washington Industrial Safety and Health Act, as described in Chapter 49.17 Revised Code of Washington

2 SAFETY GUIDELINES FOR PHYSICAL HAZARDS

2.1 GENERAL PROJECT HAZARDS

All work will be done using the buddy system. Depending upon the time of year and the location of work, biting insects, venomous snakes, and other wildlife may be an issue when accessing any of the sampling locations during the sampling events. Table 2-1 summarizes potential physical hazards posed by proposed Site activities. Table 2-2 presents potential physical hazards that are expected to be present during sampling activities.

Table 2-1. Summary of Activities and Potential Hazards

Activity	Potential Hazard
Tissue and soil sampling	Water hazards; uneven terrain/tripping, slippery walking surfaces, cold/hypothermia (depending on sampling event); heat stress (depending on sampling event); material handling; adverse weather; work in remote areas; wildlife; traversing rough terrain

Table 2-2. Potential Physical Hazards and Proposed Safety Procedures

Potential Hazard	Yes	No	Proposed Safety Procedure
Uneven terrain/tripping, slippery walking surfaces	X		Use caution; wear properly fitting shoes or boots with good gripping capacity and ankle support; keep work area orderly.
Cold/hypothermia	X		Keep warm and dry, bring changes of clothes; do not work in extreme conditions without proper equipment and training; follow cold stress information (Attachment A1-2); potential for cold/hypothermia will depend on season.
Heat stress	X		Drink water frequently in hot weather; take work breaks; follow the heat-related illness policy (Attachment A1-3); potential for heat stress will depend on season.
Material handling	X		Lift properly; seek assistance if necessary; do not overfill coolers or boxes.
Adverse weather	X		Seek shelter during storms; work in adverse weather conditions only with proper training, clothing, and equipment.
Drowning	X		All employees, when working in or near water (i.e., within 4 feet/1.2 meters) where there is a potential to voluntarily, or involuntarily, enter the water must wear a Type III personal flotation device (PFD), Type V work vest, or better. All water work (including work near water) must be performed during daylight hours. Maintain good housekeeping during all activities to prevent slips, trips, and falls. Inspect the PFDs prior to use and do not use defective PFDs. Keep sampling equipment on the shore organized at all times.

Table 2-2. Potential Physical Hazards and Proposed Safety Procedures (continued)

Potential Hazard	Yes	No	Proposed Safety Procedure
Work in remote areas	X		Use the buddy system; carry radio and/or cellular telephone; carry satellite telephone, bring sufficient equipment in case of accident or injury (first aid kit, shelter if appropriate). A satellite telephone is necessary due to the unpredictable cellular network.
Biting insects, ticks, and mosquitos	X		**Biting insects.** Use repellents, as needed. **Ticks.** Wear long-sleeved clothing and ankle length boots and try to avoid excessive contact with tall brush or grass. Personnel should change clothes and inspect their skin and scalps for ticks after every day of field work. If individuals discover a tick embedded in their skin, it should be removed as soon as possible. Grasp the tick with a blunt pair of tweezers as close to the skin as possible and remove it using slow even pressure. Do not break off the head or release fluids from the tick. Gently scrub the area with soap and water after removal. Note the date of the bite and watch for symptoms such as fever, chills, aches, and rashes for a month after the bite. If these symptoms occur, consult a doctor. **Mosquitos.** Use an insect repellent containing N, N-diethyl-m-toluamide (DEET). Wear long-sleeved shirts, pants, and hat; spray clothing with insect repellent containing DEET. Avoid handling dead animals. The risk of getting West Nile Virus is very low. Symptoms include fever, headache, neck stiffness, stupor, disorientation, tremors, convulsions, muscle weakness, paralysis, and body aches. If you develop any of these symptoms, contact your health care provider.
Stinging insects, bees/wasps (allergic reaction)			Avoiding wearing bright colors or scents. Use an appropriate insect repellent. Wear long-sleeved shirt, hat, and gloves. Employees must notify supervisor if they have allergies to bee/wasp stings prior to engaging in field activities. Employees with allergies may be required to carry an appropriate antidote kit.
Poisonous plants, poison ivy, poison sumac	X		Poison ivy generally has three green leaves on each stem. The color and appearance can vary throughout the year. Poison sumac generally occurs as a woody shrub or small tree with 7 to 13 leaflets as pairs along a central midrib and a single leaf at the end. The color and appearance can vary throughout the year. It has a smooth texture and are bright orange (spring) or glossy dark green with red midribs (summer). Avoid contact with all parts of the poison ivy or sumac plants. Contact with the oily resins on the plant may cause a skin rash. The rash usually appears within 24 to 48 hours and can last for weeks. If poison ivy or sumac is contacted, remove the affected clothing and wash the skin with soap and water to remove the oil resins as soon as possible.
Wildlife encounter	X		See individual animals listed below.

Table 2-2. Potential Physical Hazards and Proposed Safety Procedures (continued)

Potential Hazard	Yes	No	Proposed Safety Procedure
Poisonous snakes (rattlesnakes)	X		Wear appropriate PPE such as ankle-high leather boots, long pants, snake chaps, long sleeves when possible, a hat, and gloves if cutting brush or handling and moving vegetation. Do not reach into burrows or dens, under rocks, or logs. Walk heavily through brush. Back away if a snake is encountered. Take snake bite kit with a complete set of instructions. In case of a snake bite, seek prompt medical assistance. The injured employee should rest while awaiting (or being transported to) medical assistance. Workers should seek medical attention if bitten.
Black bear (potential attack)	X		If you come in contact with a black bear, stay calm and avoid eye contact. Try to stay upwind and identify yourself as a human being by standing up, talking, and waving your hands above your head. If you cannot safely move away from the bear and the animal does not flee, try to scare it away by clapping your hands or yelling. If the bear attacks, fight back aggressively. As a last resort if the attack continues, protect yourself by curling into a ball or lie on the ground on your stomach playing dead. Do not stand between mother and cub. Take bear mace with a complete set of instructions. All employees must be trained in the proper use of bear spray, which includes reading the manufacturer's instructions and discussion during project planning and daily health and safety meetings.
Grizzly bear/ brown bear (potential attack)	X		If you are attacked by a grizzly bear, play dead. Lie flat on your stomach or curl up in a ball with your hands behind your head. Remain motionless as long as possible. Do not run. Do not stand between mother and cub. Take bear mace with a complete set of instructions. All employees must be trained in the proper use of bear spray, which includes reading the manufacturer's instructions and discussion during project planning and daily health and safety meetings.
Cougar (potential attack)	X		If you come in contact with a cougar, stop, stand tall, and don't run. Try to appear larger than the cougar. Never take your eyes off the animal or turn your back. If the animal displays aggressive behavior, shout, wave your arms, and throw rocks. If the cougar attacks, fight back aggressively and stay on your feet.
Moose (between mother/calf)	X		If you come in contact with a moose, step back. Look for the nearest tree, fence, or building or other obstruction to hide behind. It's usually a good idea to run from a moose because it usually won't chase you far. If a moose knocks you down, curl up in a ball, protect your head with your arms and hands, and hold still. Don't move or try to get up until the moose moves a safe distance away.

2.2 PROJECT-SPECIFIC HAZARDS

Wildlife. As listed above, there is an abundance of wildlife in the study area. Based on previous sampling events in the study area during summer months, there have been encounters with snakes, evidence of bear foraging, and bee/wasp nests both in trees and in the grass. Employees should remain alert and aware of their surroundings during the field

event and follow proposed safety procedures above for wildlife known to inhabit the area. In the event of a wildlife encounter that causes a safety concern, use field vehicles for shelter if they can be reached safely. Use your best judgement while still following safety tips described above to determine if you can reach the vehicle.

Water work. This work will include sampling by TAI field contractors in or near water where there is a potential to voluntarily, or involuntarily, enter the water. Tule samples may be collected from shallow water within waterbodies (e.g., a seasonal pond) in the study area. All employees, when working **in** or **near** water (i.e., within 4 ft/1.2 m) where the danger of drowning exists, must wear a Type III personal flotation device (PFD), Type V work vest, or better. The PFDs must be inspected prior to use and not used if defective. It is recommended that employees wear a PFD during oversight of sampling activities in water (even if the employee is located outside of the 4-ft exclusion zone near the water) in the event that an emergency situation arises, which may require the employee to move into the 4-ft exclusion zone by the water. All water work (including work near water) must be performed during daylight hours. All employees must maintain good housekeeping during all sampling activities to prevent slips, trips, and falls.

Traversing through rough terrain. If traversing unpaved roads or rough terrain, always drive slowly and cautiously on site and between sites. Do not attempt to drive in areas such as steep, degraded, and/or undrivable roads. If rough terrain is encountered while driving, stop the car in a secure location, and if safe, attempt to assess the roadway condition on foot. If a roadway is blocked, stop the car, and if safe by foot, determine if there is a suitable and safe alternative route around the obstruction at that location. Consult local maps to determine if an alternative route is available. If a different route is unavailable, contact the Ramboll Principal Investigator or TAI Project Coordinator to discuss alternative options.

3 CHEMICAL HAZARD EVALUATION

A chemical hazard evaluation is presented in the general SHSP (TCAI 2009) and incorporated herein by reference.

4 PERSONAL PROTECTIVE EQUIPMENT AND SAFETY EQUIPMENT

The following sections address PPE and safety equipment required for completing the sampling activities.

4.1 PERSONAL PROTECTIVE EQUIPMENT

Based on chemical and physical hazards associated with the plant tissue and soil sampling activities, Tables 4-1 and 4-2 identify the PPE required for sampling.

Table 4-1. Level of Protection Required for Site Activities

	Level of Protection	
Site Activity	Initial[a]	Contingency[b]
Tissue and soil sampling	MD	Leave Site, reassess situation
Sample handling	D	Leave Site, reassess situation

[a] See Table 4-2 for definitions

[b] Based on unexpected change in Site conditions

Table 4-2. Levels of Protection and Personal Protective Equipment

Protection Level	Required	Personal Protective Equipment
Level D	X	Long pants and shirt or work coveralls, safety glasses or goggles (as appropriate), and nitrile, neoprene, or Barrier® 5-layer laminate gloves (as appropriate). Hard hat and hearing protection as needed.
Level MD	X	Same as Level D with modification (M) of adding rain gear and PFD, as needed.

Is there potential for a respirator to be donned during field work? Yes _____ No X

4.2 SAFETY EQUIPMENT

The following safety equipment will be on site during the proposed field activities.

Air Monitoring (check the items required for this project)

☐ Photoionization detector	☐ Air sampling pumps
☐ Lower Explosive Limit/Oxygen meter	☐ Miniram
☐ Hydrogen sulfide meter	☐ Radiation meter
☐ Detector pump and tubes	☐ Other _____

First Aid Kit (mandatory, including adhesive band-aids, gauze, tape, gloves, cardiopulmonary resuscitation shield, triangle bandage)

[X]	Emergency blanket	[X]	Sunscreen
[X]	Insect repellent	[ ]	Other _____

Other (check the items required for this project)

[X]	Eyewash	[ ]	Fit test supplies
[X]	Drinking water	[X]	Fire extinguisher
[ ]	Stop watch for monitoring heart	[ ]	Windsock
[ ]	Thermoscan® thermometer (or equivalent) for heat stress monitoring	[X]	Cellular telephone
		[X]	Radio sets (if no cellular or satellite telephone service)
[X]	Survival kit	[X]	Global positioning system
[X]	Personal flotation device	[X]	Other: Satellite telephone, hip/chest waders, snake chaps, bear spray

5 AIR MONITORING

The principal chemicals of potential concern (COPCs) at the Site are not volatile (i.e., metals). The chemical hazard evaluation presented in the general SHSP (TCAI 2009) concluded that, based on previous evaluations, none of the sediment or soil chemicals are expected to pose a threat to field personnel during sampling activities. If windblown dust becomes problematic to the field crew, operations may be suspended. Tables 5-1 and 5-2 provide air monitoring requirements and action levels to be used during sampling activities.

Table 5-1. Site-specific Air Monitoring Requirements

Monitoring Instrument	Calibration Frequency	Parameters of Interest	Monitoring Frequency
Visual	Not applicable	Dust	Continuous

Table 5-2. Action Levels Established to Determine the Appropriate Level of Personal Protection

Instrument	Reading	Action [a]	Comments
Visual	Visual Dust	Leave Site, if necessary	

6 EMERGENCY PLANNING

In case of any emergency affecting the Site, all affected personnel must immediately evacuate the work area and report to the Site safety officer at the following predetermined location:

DESIGNATED ASSEMBLY LOCATION—Field vehicle

In case of injury, field personnel should take precautions to protect the victim from further harm and notify local emergency services. In remote areas, it will be necessary to have first aid-trained personnel on the field team. The victim may require decontamination prior to treatment—requirements will vary based on Site conditions.

Emergency medical care will be provided by:

[X] Local emergency medical provider (i.e., fire department; see Table 6-1 for local contact information)

[X] First aid-trained field staff (for remote areas only).

Table 6-1. Local Emergency Telephone Numbers

Local Resources	Name	Telephone	Notified Prior to Work (Yes/No)?
Fire	Varies by location	911	Yes. Notify the E911 coordinator for Stevens County (Debby McCanna; 509-684-2555) of the schedule and location of work.
Police	Varies by location	911	Yes (see above)
Ambulance	Varies by location	911	Yes (see above)
Main Hospital	Mount Carmel Hospital, Colville, WA	509-684-2561	No
Directions to Mount Carmel Hospital	Begin traveling southeast on Highway 395. Highway 395 becomes Main Street in Colville. Turn LEFT on E. Columbia Avenue. Go 0.6 mile. Arrive at 982 E. Columbia Avenue. Hospital is on right. (See detailed hospital location maps in Attachment A1-1)		Not applicable
Alternative Hospitals	St. Joseph's Hospital, Cheweleh, WA	509-935-8211	No
	Ferry County Memorial Hospital, Republic, WA	509-775-3333	No
	Deer Park Hospital, Deer Park, WA	509-276-5061	No
	Coulee Community Hospital, Grand Coulee, WA	509-633-1753	No
	Holy Family Hospital, Spokane, WA	509-482-0111	No
	Veterans Affairs Medical Center, Spokane, WA	509-434-7032	No
	Sacred Heart Medical Center, Spokane, WA	509-474-3131	No

Table 6-1. Local Emergency Telephone Numbers (continued)

Local Resources	Name	Telephone	Notified Prior to Work (Yes/No)?
	Deaconess Medical Center-Spokane, Spokane, WA	509-473-7178	No
	Lincoln Hospital, Davenport, WA	509-725-7101	No
Field cellular telephone or satellite telephone	Cellular telephone coverage is spotty in the vicinity of the sampling areas. If cellular telephone coverage is lost due to a mountain or hill, drive a little farther to get coverage. If cellular telephone coverage is available, the 911 system will work. A satellite telephone may be necessary for areas with limited cellular telephone coverage.	Satellite telephone # TBD	Not applicable
Directions to Mount Carmel Hospital	Begin traveling southeast on Highway 395. Highway 395 becomes Main Street in Colville. Turn LEFT on E. Columbia Avenue. Go 0.6 mile. Arrive at 982 E. Columbia Avenue. Hospital is on right. (See detailed hospital location maps in Attachment A1-1)		Not applicable

In case of serious injuries, death, or other emergency, after local emergency services have been contacted, the TAI Project Coordinator and Ramboll Task Manager or Ramboll Principal Investigator must be notified immediately. Contact numbers are listed in Table 6-2.

Table 6-2. Corporate Emergency Telephone Numbers

Corporate Resources	Name	Work/Cellular Telephone
TAI Project Coordinator	Kris McCaig	Work: 509-623-4501 Cellular: 509-434-8542
TAI Assistant Project Coordinator	Denise Mills	Work: 509-623-4515 Cellular: 509-904-9375
Ramboll Task Manager	Lis Nelis	Work: 206-336-1659 Cellular: 773-209-9818
Ramboll Principal Investigator	Dina Johnson	Work: 206-336-1662 Cellular: 425-765-1218

Table 6-3 provides local hospital contact and location information. See Attachment A1-1 for a detailed hospital location map.

Table 6-3. Project Area Hospital Information

Facility Name	Open for Emergency Services	Telephone Number	Address	City
Mount Carmel Hospital	24 hours	509-684-2561	982 East Columbia Street	Colville
St Joseph's Hospital	24 hours	509-935-8211	500 East Webster Street	Chewelah
Ferry County Memorial Hospital	24 hours	509-775-3333	36 Klondike Road	Republic
Deer Park Hospital	24 hours	509-276-5061	East 1015 'D' Street	Deer Park
Coulee Community Hospital	24 hours	509-633-1753	411 Fortuyn Road	Grand Coulee
Holy Family Hospital	Dependent on case	509-482-0111	North 5633 Lidgerwood Avenue	Spokane
Veterans Affairs Medical Center	7:30 am to 4:00 pm	509-434-7032	North 4815 Assembly Street	Spokane
Sacred Heart Medical Center	24 hours	509-474-3131	West 101 Eighth Avenue	Spokane
Deaconess Medical Center-Spokane	24 hours	509-473-7178	West Fifth Avenue	Spokane
Lincoln Hospital	24 hours	509-725-7101	10 Nichols Street	Davenport

If any health or safety issue arises, after the victim receives appropriate medical treatment, the relevant field crew members will be interviewed to formally document the incident by, at a minimum, the Field Supervisor and Task Manager. All incidents will be documented in the field logbook. If applicable, a corrective action form will be filled out (see Field Sampling Plan Attachment A3) to ensure future health and safety issues are addressed.

7 WORK ZONES

The following work zones are defined for the tissue sampling activities:

Exclusion zone. The area immediately around the sampling activities will be designated as the exclusion zone. Traffic cones and/or caution tape will be used to delineate the specific areas.

Contamination reduction zone. Not applicable. All sampling activities will occur within the exclusion zone.

Support zone. Not applicable. All sampling activities will occur within the exclusion zone.

Controls to be used to prevent entry by unauthorized persons. The sampling staff will remain cognizant of people approaching the exclusion zone. All unauthorized persons will be instructed to remain outside of the sampling area.

8 DECONTAMINATION

The field team will decontaminate all sampling equipment that comes into contact with plant tissue or soil prior to the commencement of sampling at each location and upon completion of the study. This will include equipment such as plant trimmers, trowels, mixing bowls, and utensils. The decontamination will consist of thoroughly rinsing all of the equipment with potable water, then with soap (i.e., Alconox®), and rinsed with potable water after each use.

Clean gloves will be worn at each sampling location to avoid transfer of potential contaminants among samples. Otherwise, decontamination procedures will follow those presented in the general SHSP (TCAI 2009) and are incorporated herein.

9 VEHICLE SAFETY, SPILL CONTAINMENT, AND SHIPPING INSTRUCTIONS

Vehicle safety, spill containment, and shipping instructions are presented in the general SHSP (TCAI 2009) and are incorporated herein.

10 TASK-SPECIFIC SAFETY PROCEDURES

Slips, trips, and falls are anticipated to be the greatest hazards to field personnel during the plant tissue and soil sampling events, as well as unexpected contact with the sampling equipment. Field personnel should always move about the shore or upland area with caution, and wear properly fitting shoes or boots with non-slip soles and good ankle support.

The Site is located in a remote region with limited cellular telephone coverage. All field crews will have two-way radios or a satellite telephone to maintain communication with the Field Supervisor. The field crews will coordinate departure and expected return times for all field activities with the Field Supervisor. Field crews will provide the Field Supervisor and Principal Investigator with status updates at least every 4 hours while performing field collection activities.

The areas that will be sampled are accessible to the public. Field crew members should always be aware of their surroundings, use the buddy system, and/or keep in line-of-sight contact with other sampling personnel at all times. Samples or sampling equipment should not be left unattended. If a crew member feels threatened, or if the situation feels unpredictable, leave the area immediately.

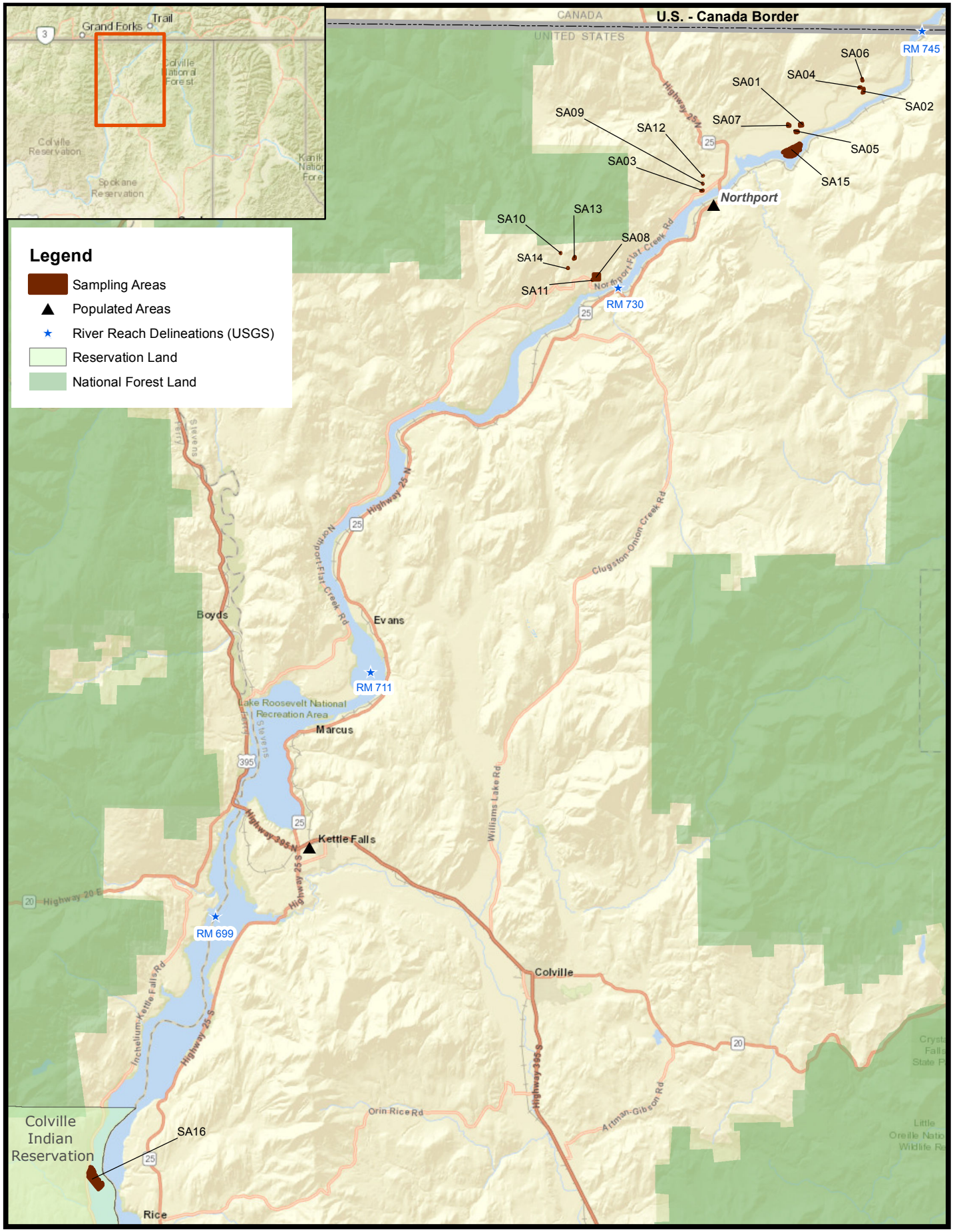
When handling sampling equipment or samples, or preservative chemicals (if required), nitrile gloves should always be worn, along with safety glasses or goggles when appropriate. Keep a 1-L eyewash bottle accessible during all field work. Avoid getting preservatives on the skin or clothes. If any preservatives are spilled or splashed on the skin or clothes, immediately rinse the affected area with potable water and get medical attention, if warranted. If any preservative is splashed in the eye, flush the eye with the eyewash solution, and get immediate medical attention.

11 REFERENCE

TCAI (Teck Cominco American Incorporated). 2009. Upper Columbia River general site health and safety plan for the remedial investigation and feasibility study. Prepared for Teck American Incorporated. Integral Consulting Inc., Mercer Island, Washington, and Parametrix, Seattle, WA.

ATTACHMENT A1-1

STUDY AREA MAP



RAMBOLL ENVIRON

0 1 2 4 Kilometers

0 1 2 4 Miles

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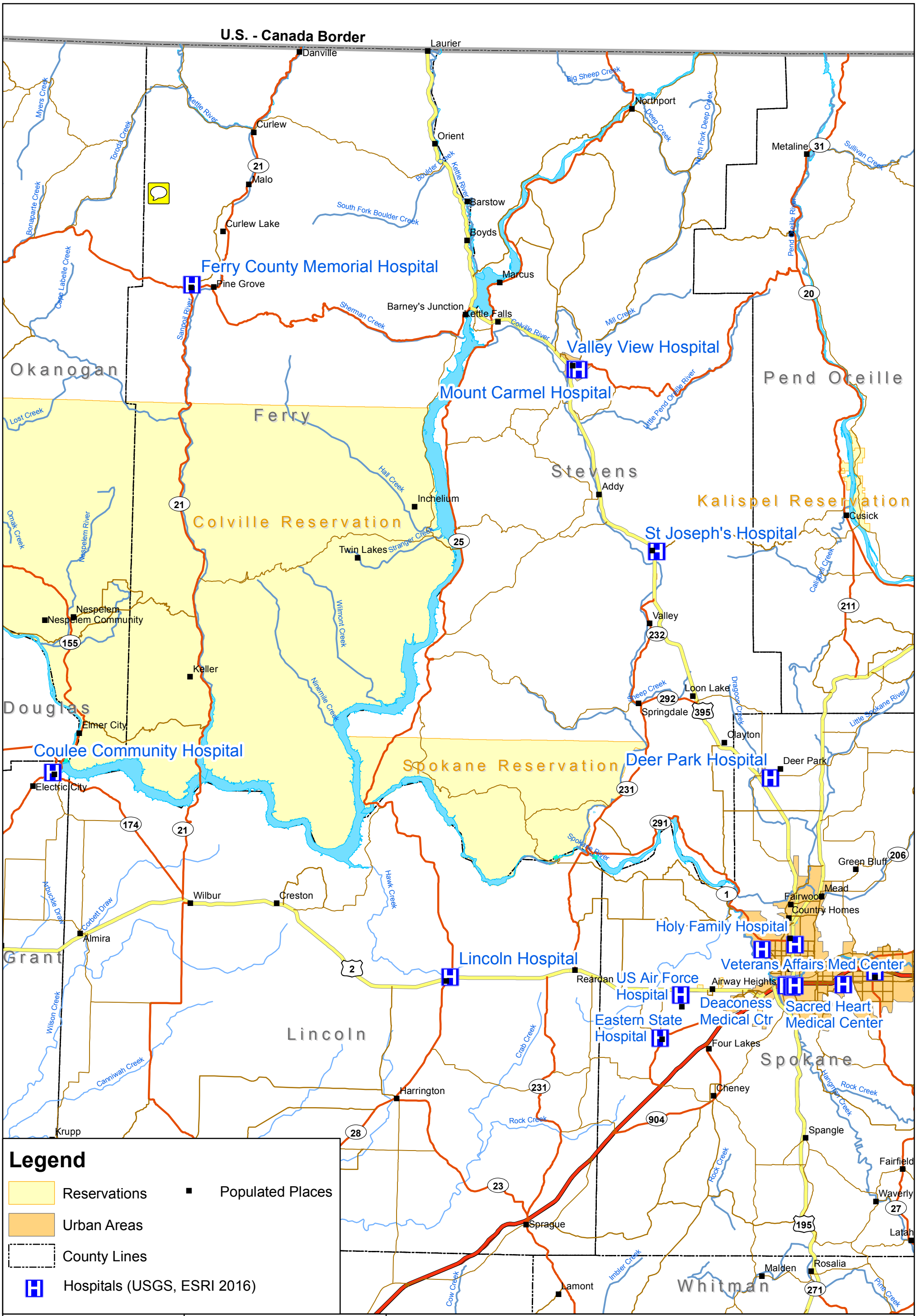
Attachment A1-1. Study Area Map

Upper Columbia River, WA


U.S. - Canada Border
Laurier
Danville
Myers Creek
Kettle River
Curlew
Orient
Big Sheep Creek
Northport
Deep Creek
North Fork Deep Creek
Metaline
31
Sullivan Creek
Toroda Creek
21
Malo
Boulder Creek
Kettle River
Barstow
Bonaparte Creek
South Fork Boulder Creek
Curlew Lake
Boyds
Pend Oreille River
Ferry County Memorial Hospital
Marcus
Cape Labelle Creek
Pine Grove
Sherman Creek
Barney's Junction
Kettle Falls
Colville River
Mill Creek
20
Sanpoil River
Valley View Hospital
Okanogan
Mount Carmel Hospital
Pend Oreille
Lost Creek
Ferry
Little Pend Oreille River
Stevens
Hall Creek
Inchelium
Addy
Kalispel Reservation
Onak Creek
Nespelem River
21
Colville Reservation
Cusick
St Joseph's Hospital
Twin Lakes
Stranger Creek
25
Calispell Creek
Wilmont Creek
211
Valley
Nespelem
Nespelem Community
232
155
Keller
Ninemile Creek
Sheep Creek
Loon Lake
Dragoon Creek
Douglas
292
Springdale
395
Little Spokane River
Elmer City
Clayton
Coulee Community Hospital
Spokane Reservation
Deer Park Hospital
Deer Park
Electric City
231
174
21
291
Spokane River
Green Bluff
206
Arbuckle Draw
Corbett Draw
Wilbur
Creston
Hawk Creek
1
Mead
Fairwood
Country Homes
Holy Family Hospital
Almira
Grant
Lincoln Hospital
2
Reardan
US Air Force Hospital
Airway Heights
Veterans Affairs Med Center
Deaconess Medical Ctr
Sacred Heart Medical Center
Wilson Creek
Eastern State Hospital
Lincoln
Crab Creek
Four Lakes
Spokane
Canniwah Creek
Harrington
231
Cheney
Hangman Creek
Rock Creek
904
Rock Creek
Spangle
Krupp
28
Fairfield
23
Waverly
27
Sprague
195
Latah
Malden
Rosalia
Imbler Creek
Lamont
Whitman
271
Cow Creek
Pine Creek
Legend
Reservations
Populated Places
Urban Areas
County Lines
Hospitals (USGS, ESRI 2016)
Ramboll Environ
Km
0 5.5 11
Miles
0 5 10
N
Hospital Location Map
Upper Columbia River, WA

ATTACHMENT A1-2

COLD-STRESS FACT SHEET

FROSTBITE

What happens to the body:

Freezing in deep layers of skin and tissue; pale, waxy-white skin color; skin becomes hard and numb; usually affects fingers, hands, toes, feet, ears, and nose.

What to do: (land temperatures)

- Move the person to a warm, dry area. Don't leave the person alone.
- Remove wet or tight clothing that may cut off blood flow to the affected area.
- **Do not** rub the affected area because rubbing damaged the skin and tissue.
- Gently place the affected area in a warm water bath (105°) and monitor the water temperature to **slowly** warm the tissue. Don't pour warm water directly on the affected area because it will warm the tissue too fast, causing tissue damage. Warming takes 25-40 minutes.
- After the affected area has been warmed, it may become puffy and blister. The affected area may have a burning feeling or numbness. When normal feeling, movement, and skin color have returned, the affected area should be dried and wrapped to keep it warm. **Note:** If there is a chance the affected area may get cold again, do not warm the skin. If the skin is warmed and then becomes cold again, it will cause severe tissue damage.
- Seek medical attention as soon as possible.

How to Protect Workers

- Recognize the environmental and workplace conditions that lead to potential cold-induced illnesses and injuries.
- Learn the signs and symptoms of cold-induced illnesses/injuries and what to do to help the worker.
- Train workers about cold-induced illnesses and injuries.
- Select proper clothing for cold, wet, and windy conditions. Layer clothing to adjust to changing environmental temperatures. Wear a hat and gloves, in addition to underwear that will keep water away from the skin (polypropylene.)
- Take frequent short breaks in warm, dry shelters to allow the body to warm up.
- Perform work during the warmest part of the day.
- Avoid exhaustion or fatigue because energy is needed to keep muscles warm.
- Use the buddy system (work in pairs.)
- Drink warm, sweet beverages (sugar water, sports-type drinks.) **Avoid drinks with caffeine** (coffee, tea, or hot chocolate) **or alcohol**.
- Eat warm, high-calorie foods like hot pasta dishes.

Workers are at increased risk when…

- They have predisposing health conditions such as cardiovascular disease, diabetes, and hypertension.
- They take certain medications. Check with your doctor, nurse, or pharmacy and ask if medicines you take affect you while working in cold environments.
- They are in poor physical condition, have a poor diet, or are older.

HYPOTHERMIA - (Medical Emergency)

What happens to the body:

Normal body temperature (98.6°F/37°C) drops to or below 95°F/35°C; fatigue or drowsiness; uncontrolled shivering; cool, bluish skin; slurred speech; clumsy movements; irritable, irrational, or confused behavior.

What to do: (land temperatures)

- Call for emergency help (i.e., ambulance or 911).
- Move the person to a warm, dry area. Don't leave the person alone.
- Remove wet clothing and replace with warm, dry clothing or wrap the person in blankets.
- Have the person drink warm, sweet drinks (sugar water or sports-type drinks) if he is alert. **Avoid drinks with caffeine** (coffee, tea, or hot chocolate) **or alcohol**.
- Have the person move his arms and legs to create muscle heat. If he is unable to do this, place warm bottles or hot packs in the armpits, groin, neck, and head areas. **Do not** rub the person's body or place him in a warm water bath. This may stop his heart.

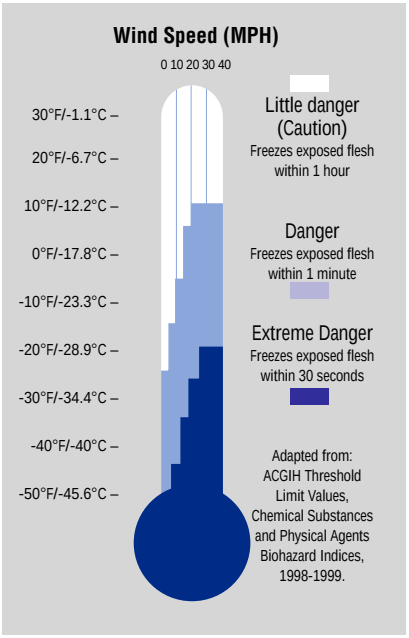
What to do: (water temperatures)

- Call for emergency help (i.e., ambulance or 911). Body heat is lost up to 25 times faster in water.
- **Do not** remove any clothing. Button, buckle, zip, and tighten any collars, cuffs, shoes, and hoods because the layer of trapped water closest to the body provides a layer of insulation that slows the loss of heat. Keep the head out of the water and put on a hat or hood.
- Get out of the water as quickly as possible or climb on anything floating. **Do not** attempt to swim unless a floating object or another person can be reached because swimming or other physical activity uses body heat and reduces survival time by about 50 percent.
- If getting out of the water is not possible, wait quietly and conserve body heat by folding arms across the chest, keeping thighs together, bending knees, and crossing ankles. If another person is in the water, huddle together with chests held closely.

THE COLD STRESS EQUATION

LOW TEMPERATURE + WIND SPEED + WETNESS = INJURIES & ILLNESS

When the body is unable to warm itself, serious cold-related illnesses and injuries may occur, and permanent tissue damage and death may result. **Hypothermia** can occur when *land temperatures* are **above** freezing or *water temperatures* are below 98.6°F/37°C. Cold-related illnesses can slowly overcome a person who has been chilled by low temperatures, brisk winds, or wet clothing.



Oregon Occupational Safety & Health Division
440-3336E-S (9/03)

ATTACHMENT A1-3

HEAT-RELATED ILLNESS PREVENTION POLICY

HEAT EXHAUSTION

What happens to the body:

Headaches, dizziness, or light-headedness, weakness, mood changes, irritability or confusion, feeling sick to your stomach, vomiting, fainting, decreased and dark-colored urine, and pale, clammy skin.

What should be done:

- Move the person to a cool shaded area. Don't leave the person alone. If the person is dizzy or light-headed, lay him on his back and raise his legs about 6-8 inches. If the person is sick to his stomach, lay him on his side.
- Loosen and remove heavy clothing.
- Have the person drink some cool water (a small cup every 15 minutes) if he is not feeling sick to his stomach.
- Try to cool the person by fanning him. Cool the skin with a cool spray mist of water or wet cloth.
- If the person does not feel better in a few minutes call for emergency help (ambulance or call 911.)

(If heat exhaustion is not treated, the illness may advance to heat stroke.)

HEAT STROKE - A Medical Emergency

What happens to the body:

Dry, pale skin (no sweating); hot red skin (looks like a sunburn); mood changes; irritability, confusion, and not making any sense; seizures or fits, and collapse (will not respond).

What should be done:

- Call for emergency help (i.e., ambulance or 911.)
- Move the person to a cool, shaded area. Don't leave the person alone. Lay him on his back and if the person is having seizures, remove objects close to him so he won't hit them. If the person is sick to his stomach, lay him on his side.
- Remove heavy and outer clothing.
- Have the person drink some cool water (a small cup every 15 minutes) if he is alert enough to drink anything and not feeling sick to his stomach.
- Try to cool the person by fanning him or her. Cool the skin with a cool spray mist of water, wet cloth, or wet sheet.
- If ice is available, place ice packs in armpits and groin area.

How to Protect Workers

- Learn the signs and symptoms of heat-induced illnesses and what to do to help the worker.
- Train workers about heat-induced illnesses.
- Perform the heaviest work during the coolest part of the day.
- Slowly build up tolerance to the heat and the work activity (usually takes up to 2 weeks.)
- Use the buddy system (work in pairs.)
- Drink plenty of cool water (one small cup every 15-20 minutes.)
- Wear light, loose-fitting, breathable (like cotton) clothing.
- Take frequent short breaks in cool, shaded areas (allow your body to cool down.)
- Avoid eating large meals before working in hot environments.
- Avoid caffeine and alcoholic beverages (these beverages make the body lose water and increase the risk of heat illnesses.)

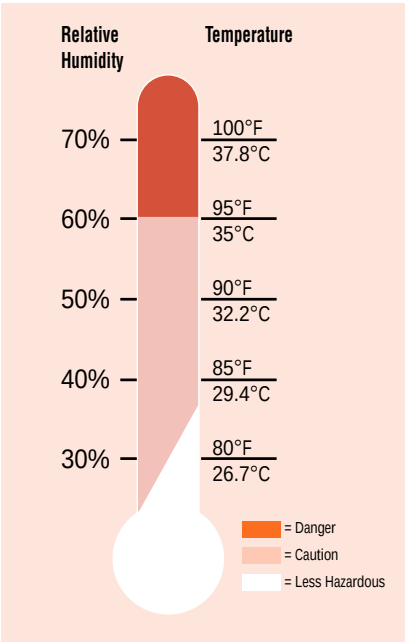
Workers are at increased risk when…

- They take certain medications. Check with your doctor, nurse, or pharmacy to see if medicines you take affect you when working in hot environments.
- They have had a heat-induced illness in the past.
- They wear personal protective equipment.

THE HEAT EQUATION

HIGH TEMPERATURE + HIGH HUMIDITY + PHYSICAL WORK = HEAT ILLNESS

When the body is unable to cool itself through sweating, **serious** heat illnesses may occur. The most severe heat-induced illnesses are **heat exhaustion** and **heat stroke**. If actions are not taken to treat heat exhaustion, the illness could progress to heat stroke and **death**.



Oregon Occupational Safety & Health Division
440-3333E-S(9/03)

ATTACHMENT A2

PLANT TISSUE SAMPLING STANDARD OPERATING PROCEDURES

STANDARD OPERATING PROCEDURE SOP-1

SAMPLE AREA SELECTION

Scope and Applicability

This standard operating procedure (SOP) is specific to the 2018 Plant Tissue Study (hereinafter "the study") being conducted for Teck American Incorporated (TAI) in the northern portion of the Upper Columbia River site in northeastern Washington. The purpose of this SOP is to describe procedures for selecting sample areas (SAs) during each of the three field sampling events. SAs that may be visited for plant tissue and soil/sediment sample collection were pre-selected based on information obtained during prior studies and field reconnaissance efforts. SAs include three high lead SAs and 13 lower lead SAs (SA01 to SA03 high lead, SA04 to SA16 lower lead; see Table A1 of the field sampling plan [FSP]).

All three high lead SAs will be visited at the beginning of each sampling event. Visitation of lower lead SAs for possible sample collection will be prioritized based on the likelihood that a potential target plant species will be found in sufficient abundance. Lower lead SAs that have the highest likelihood of providing plant biomass based on the results of the field reconnaissance effort from August 2017 will be visited first. Collection of each target species will be limited to one of three sampling events in 2018 (spring, late June, or late August), with the exception of wild rose and black tree lichen. Wild rose will be a target species for two field events: spring to collect leaves and stems, and August to collect rose hips. Black tree lichen will be targeted for sample collection during each field event or until mass requirements have been met or the collection threshold has been reached. Because each sampling event will target different species, different SA selection decision trees have been established for each event.

Procedures for Sample Area Selection—Spring

1. During spring, note the presence of strawberry plants.
2. Survey high lead SAs (SA01, SA02, and SA03) to determine if camas, puffballs, bitterroot, Lomatium, morel, shaggy mane, spring beauty, and wild caraway are present and appear sufficiently abundant to meet sample targets. Note the presence of wild strawberry to assist with planning for the late-June field sampling event.
3. Collect plant tissue and soil samples for spring plant list targets from the three high lead SAs.

4. Collect plant tissue and soil samples for the spring plant list targets from the following three lower lead SAs: SA04, SA05, and SA06.

5. Upon completion of sampling at SA01 through SA06, re-evaluate sampling needs based on the plant list targets obtained. If sample targets from the spring plant list have been met (excluding willow), no additional spring sampling is necessary. If sample targets for the spring plant list have not been met, proceed with sampling according to one of the four pathways shown in Figure 1 (use species in parentheses following each SA number to select which plot to visit first):

    a. **Insufficient Mass Pathway.** If puffballs and spring beauty were not sampled in high lead SAs and more mass is needed for other species, then proceed to SA07 (rose and lichen), SA08 (rose and lichen), and SA10 (rose and kinnikinnick) for collection of additional samples. Upon completion of sampling at SA07, SA08, and SA10, re-evaluate sampling needs based on the plant list targets obtained. If sample targets from the spring plant list have been met, no additional spring sampling is necessary. If sample targets for spring plant list have not been met, proceed with sampling at SA11 (rose and lichen). Upon completion of sampling at SA11, the level of effort for the sampling event will be met whether or not sample targets for all species on the spring list have been obtained.

    b. **Puffball Pathway.** If puffballs were sampled in high lead SAs, proceed to SA10 (puffballs, rose, kinnikinnick) for collection of additional samples. Upon completion of sampling at SA10, re-evaluate sampling needs based on the plant list targets obtained. If sample targets from the spring plant list have been met, no additional spring sampling is necessary. If sample targets for the spring plant list have not been met, proceed with sampling at SA07 (rose and lichen), SA08 (rose and lichen), and SA13 (rose). Upon completion of sampling at SA07, SA08, and SA13, the level of effort for the sampling event will be met whether or not sample targets for all species on the spring list have been obtained.

    c. **Spring Beauty Pathway.** If spring beauty were sampled in high lead SAs, proceed to SA09 (rose, habitat for spring beauty) and SA12 (lichen, habitat for spring beauty) for collection of additional targets. Upon completion of sampling at SA09 and SA12, re-evaluate sampling needs based on the plant list targets obtained. If sample targets from the spring plant list have been met, no additional spring sampling is necessary. If sample targets for the spring

plant list have not been met, proceed with sampling at SA08 (rose and lichen) and SA10 (rose and kinnikinnick). Upon completion of sampling at SA08 and SA10, the level of effort for the sampling event will be met whether or not sample targets for all species on the spring list have been obtained.

d. **Spring Beauty and Puffball Pathway.** If both spring beauty and puffballs were sampled in high lead SAs, proceed to SA09 (rose, habitat for spring beauty), SA10 (puffballs, rose, kinnikinnick), and SA12 (lichen, habitat for spring beauty) for collection of additional targets. Upon completion of sampling at SA09, SA10, and SA12, re-evaluate sampling needs based on the plant list targets obtained. If sample targets from the spring plant list have been met, no additional spring sampling is necessary. If sample targets for the spring plant list have not been met, proceed with sampling at SA08 (rose and lichen). Upon completion of sampling at SA08, the level of effort for the sampling event will be met whether or not sample targets for all species on the spring list have been obtained.

6. Sample SA15 and SA16 for willows.

7. If, over the course of the spring sampling event, the weather is warm and sunny, additional plants may bloom during the event. In this case and provided the sampling contingency day built into the schedule is still available, the field survey team will return to one high lead SA towards the end of the event; they will select the high lead SA that has the potential to yield the highest number of plant species. If the field survey team finds a target species or plant part that it was unable to find earlier, the collection team may return to the SA for additional collection.

8. At the end of the April field sampling event, the field team will make a list of plant species that were completely sampled and those where fewer than the 12 targeted samples were collected. For plant targets that are not completely sampled during the April field sampling event, supplementary samples will be collected during the normal course of the June sampling event if the target is found and sufficient mass is available to make one or more samples. Species that are fully collected in April will not be collected in subsequent events.

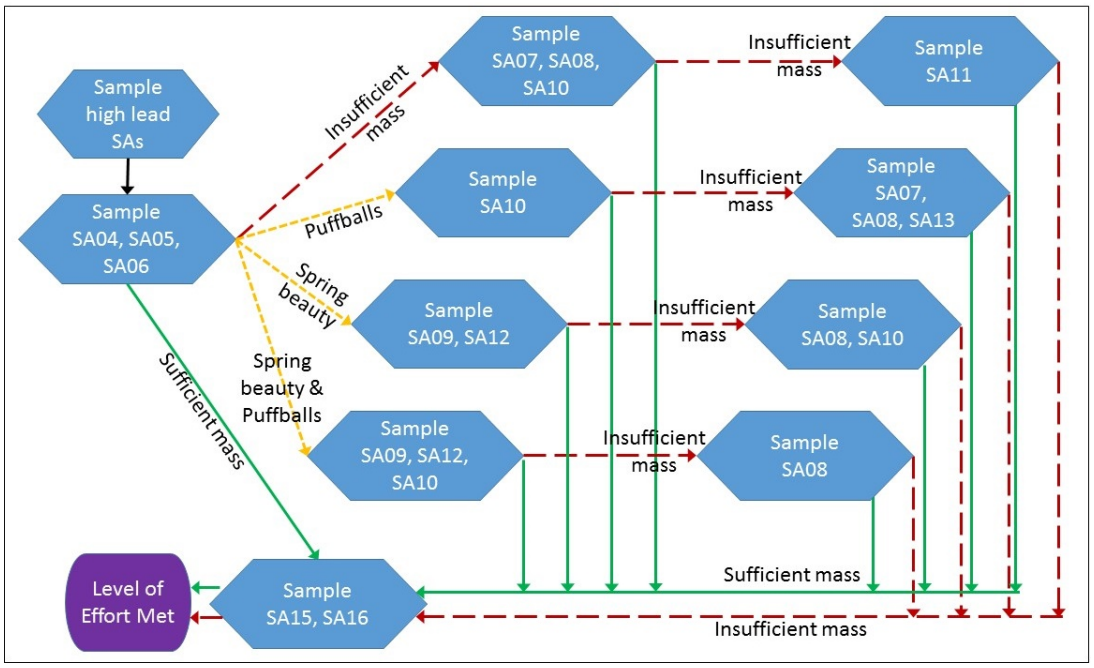


Figure 1. Sampling area (SA) selection flow chart for spring 2018 sampling effort.

Procedures for Sample Area Selection—Late June

High lead SAs (SA01, SA02, and SA03) will be surveyed to determine if sarvisberry and wild strawberry are present and appear sufficiently abundant to meet sample mass requirements for collection. If sufficient black tree lichen was collected in spring, it will not be collected in late June. If black tree lichen samples are still needed, they will be collected. During the normal course of the June sampling event, if missing samples from the spring sampling event are found they will be collected. Prior to sampling, plot selection flow charts will be updated based on identification of wild strawberry plants from spring surveys.

In addition to sampling, during the June event the survey or sampling team will do a trial run to see if using landscaping tree trimmers will allow them to collect pine cones directly from ponderosa pine trees. Pine cones that fall to the ground are often already damaged; hopefully, those collected directly from trees may contain more nuts. If sampling using landscaping tree trimmers is efficient, the sampling team will use the trimmers during the August sampling event when pine cones are ripe.

1. Collect plant tissue and soil samples for June plant list targets from the three high lead SAs (SA01, SA02, SA03) for sarvisberry, wild strawberry, and black tree lichen (as needed).
2. Collect plant tissue and soil samples in SA04, SA07, and SA09.
3. If more mass is needed, sample SA05 (sarvisberry and lichen), SA06 (sarvisberry), and SA08 (sarvisberry and lichen).
4. If more mass is needed, sample SA13 (sarvisberry). If mass is still insufficient after sampling SA13, then level of effort for the sampling event will still be met.
5. If sampling teams were unable to collect willow from SA15 and SA16 during the April sampling event, make another attempt during the June sampling event.
6. At the end of the June field sampling event, the field team will make a list of plant species that were completely sampled and those where fewer than the 12 targeted samples were collected. If supplementary samples are found for species that were not completely sampled during the normal course of the August sampling event, they will be collected. Species that were fully collected in prior sampling events will not be collected in subsequent events.

Figure 2 shows the SA flow chart for the late-June 2018 sampling effort.

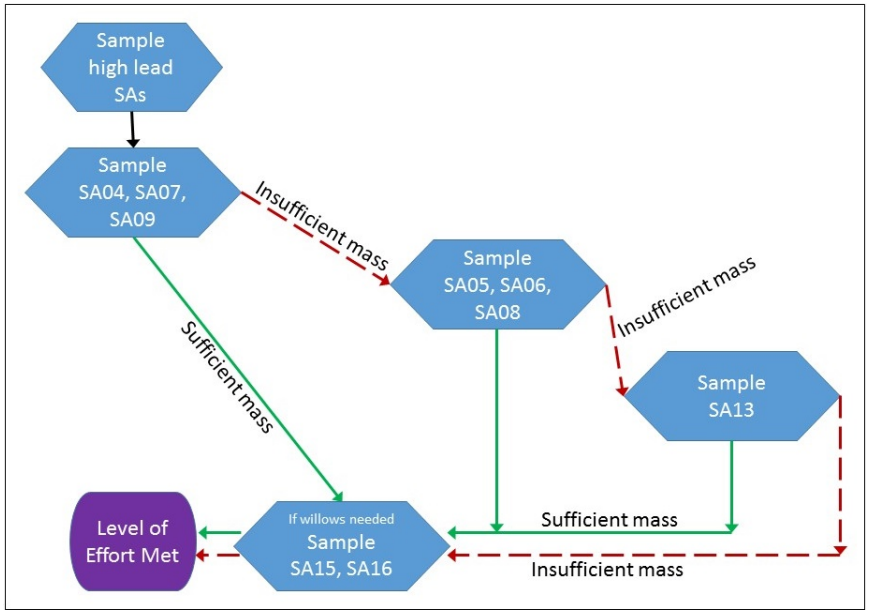


Figure 2. SA selection flow chart for late-June 2018 sampling effort.

Procedures for Sample Area Selection—Late August

High lead SAs (SA01, SA02, and SA03) will be surveyed to determine if hazelnut, wild rose hips, ponderosa pine, and chokecherry are present and appear sufficiently abundant to meet sample mass requirements for collection. If sufficient black tree lichen was collected in spring, it will not be collected in late June. If black tree lichen samples are still needed, they will be collected. During the normal course of the August sampling event, if supplementary samples are found for species that were not completely sampled during prior sampling events, they will be collected.

1. Collect plant tissue and soil samples in high lead SAs (SA01, SA02, and SA03).
2. Collect plant tissue and soil samples from lower lead SA05, SA06, and SA14.
3. If more mass is needed, sample SA04 (hazelnut, rose, pine, and chokecherry), SA07 (pine, chokecherry, rose, and lichen), and SA08 (hazelnut, rose, pine, chokecherry, and lichen).
4. If more mass is needed for hazelnut, sample SA09 (hazelnut, rose, pine, and chokecherry).
5. If more mass is needed for wild rose or chokecherry, sample SA13.
6. In addition to all other sampled SAs, sample two SAs for willows and co-located soil/sediment: SA15 and SA16. If tules are also found on those sites, sample the tules with co-located soil/sediment as well. Because SA15 and SA16 must be sampled, they may be sampled at any point during the late-August sampling event to make the event as efficient as possible.
7. If mass is still insufficient after sampling all these SAs, then level of effort for the sampling event will still be met.
8. If sampling teams were unable to collect willow from SA15 and SA16 during the prior sampling events, make another attempt during the August sampling event.

Figure 3 shows the SA flow chart for the late-August 2018 sampling effort.

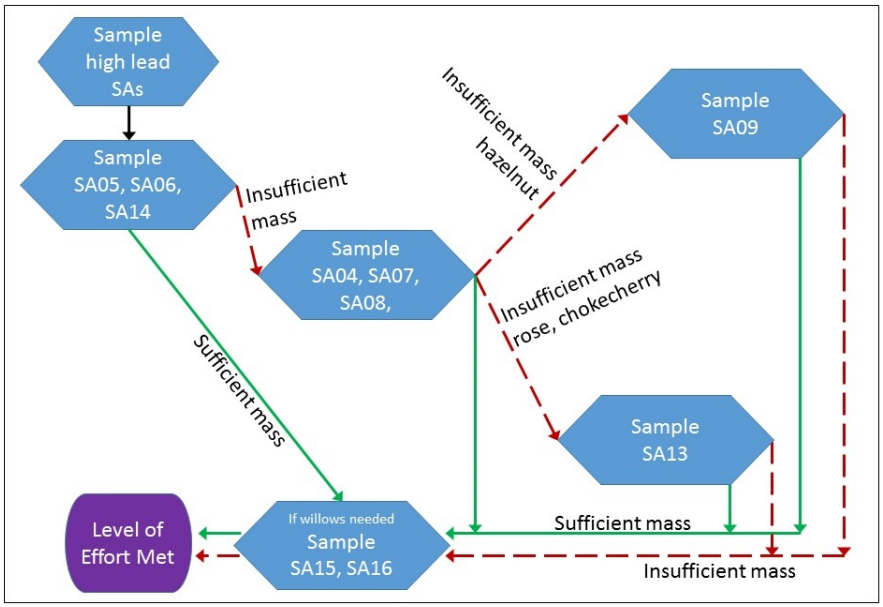


Figure 3. SA selection flow chart for late-August 2018 sampling effort.

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STANDARD OPERATING PROCEDURE SOP-2

RECORDING PLANT TISSUE AND SOIL/SEDIMENT COLLECTION LOCATIONS

Scope and Applicability

This standard operating procedure (SOP) is specific to the 2018 Plant Tissue Study (hereinafter "the study") being conducted for Teck American Incorporated (TAI) in the northern portion of the Upper Columbia River site in northeastern Washington. This SOP describes procedures used for recording plant tissue and soil/sediment sampling stations within the study area. Accurate station positioning is required to help ensure quality and consistency in collecting samples and in data interpretation and analysis. Station positioning must be both absolutely accurate in that it correctly defines a position by latitude and longitude, and relatively accurate in that the position must be repeatable. The methods described in this SOP should be usable for any hand-held global positioning system (GPS) unit; however, the owner's manual for any GPS unit used should be consulted and used to support this SOP.

Equipment and Materials

The following is a list of equipment and materials needed by the field sampling team:

- Hand-held GPS unit (e.g., Trimble GeoXH)
- Spare batteries
- Charging unit.

A GPS hardware system, such as a Trimble GeoXH GPS (or equivalent device), should be used for recording the location of each plant and co-located soil/sediment sample. The standard projection method to be used during field activities is the horizontal datum of the World Geodetic System of 1984 (WGS84).

Positioning System Verification

GPS requires no calibration because all signal propagation is controlled by the U.S. government (the Department of Defense for satellite signals, and the U.S. Coast Guard and U.S. Forest Service for differential corrections). Verification of the accuracy of the GPS requires that coordinates be known for one (or more) horizontal control points within the study area. The GPS position reading at any given station can then be compared to the known

control point. Each morning a survey monument[1] will be referenced and added to the maps for increased accuracy.

Station Location Procedures

Sampling area boundaries and other applicable geographic information system (GIS) data layers (e.g., aerial photographs, topography) will be uploaded into the hand-held GPS units prior to the sampling effort, if appropriate. A position will be recorded electronically at each location where plant tissues and soil/sediment are collected, unless individual plant collections form part of a composite. In the case of composite plant samples, only individual plants farther than 3 m apart will have their GPS location recorded. While each composite sample has only one final sample number, there may be multiple GPS locations recorded when plants are more than 3 m apart. In that case, GPS locations for composite samples farther than 3 m apart will be labeled with a number in sequence after the sample number (SA04-SP05-P01-1, SA04-SP05-P01-2, SA04-SP05-P01-3, etc.). Ancillary information will be recorded in the field logbook, and may include the personnel operating the GPS system, water depth of soil/sediment sample, and the time samples were collected.

A brief summary of procedures to locate a specific sampling location using a hand-held GPS unit are as follows:

- Turn on the unit, start the GPS data collection program.
- Keep the antenna stationary and wait for it to acquire the location of at least four satellites.
- With the antenna still stationary, initiate the data collection for the sample location. Ensure that the GPS unit receives at least 20 observations from the satellites before closing out the data collection.
- Save the location into the GPS memory according to the SOP-3 naming convention (site coordinates may also be noted on field forms [Attachment A3 of this FSP] or in the field logbook).
- Charge unit and batteries when not in use.

Upon completion of the sampling effort, all data points will be downloaded from the GPS unit and displayed on a GIS map. Any sampling locations outside of the originally defined sampling areas will be mapped and described with supporting documentation (photographs, species present, and distance from sampling area) in the field sampling report.

[1] A list of nearby monuments is found at: http://www.wsdot.wa.gov/monument/search.aspx.

STANDARD OPERATING PROCEDURE SOP-3

SAMPLE LABELING

Scope and Applicability

This standard operating procedure (SOP) is specific to the 2018 Plant Tissue Study (hereinafter "the study") being conducted for Teck American Incorporated (TAI) in the northern portion of the Upper Columbia River site in northeastern Washington. This SOP describes the general procedures for completing sample labels that will be used for plant tissue and soil/sediment sampling. The project-specific field sampling plan (FSP) should be consulted regarding the rationale behind the sample labeling protocol.

Equipment and Materials

Equipment and materials for this task include:

- Sample labels
- Indelible marker
- Copy of the FSP.

Sample Identifiers

Sample identifiers will be established before field sampling begins and assigned to each sample as it is collected. Sample identifiers consist of codes designed to fulfill three purposes: 1) to identify related samples (i.e., co-located plant and soil/sediment samples and field split samples) to ensure proper data analysis and interpretation; 2) to clearly connect sample results to sampling locations; and 3) to track individual sample containers to ensure that the laboratory receives all of the material associated with a single sample. The following subsections describe the location identification numbers (IDs), and sample IDs for individual and composite samples.

Location and Sample Event IDs

All plants will be collected within predetermined sample areas (SAs), and each SA will each be assigned a unique identifier. These location IDs will consist of the following parts:

- Four-digit location identification codes with sequential numbers (e.g., SA01, SA02, SA03). The first three SAs, SA01 to SA03, represent high lead SAs; SA04 to SA13 represent lower lead SAs.
- Two-digit sample event designation—SP for spring, JU for late June, and LA for late August.

Examples:

SA04-SP = lower lead SA number 4 sampled in the spring

SA01-LA = high lead SA number 1 sampled in late August

These location IDs will be used to document sampling locations and sampling events.

IDs for Individual and Composite Samples

Each individual plant tissue or soil/sediment sample will be assigned a unique identifier, whether that sample is from a single plant or is a composite. The sample ID will include the location and sample event ID (as described above), the individual number, and the soil/sediment or plant code as shown below.

- Six-digit code that combines the SA ID and the sampling event code (as described above)
- Two-digit sequential number to indicate location of sample
- One-digit code to designate a plant or co-located soil/sediment sample—P for plant, S for soil/sediment
- Two-digit number to indicate if more than one specimen was collected from that location.

Examples:

SA04-SP05-P01 = first plant tissue sample collected from lower lead SA number 4, sampled in the spring from location 5

SA01-LA03-S02 = second co-located soil sample collected in high lead SA number 1 sampled in late August from location 3

Sample Labels

Sample ID information will be entered onto the sample label with an indelible marker. Other information that will be entered onto the sample label includes:

- Samplers' initials
- Date
- Time.

If necessary, corrections will be made on the sample labels by drawing a single line through the error and entering the correct information with an indelible marker. All corrections will be initialed and dated by the person performing the correction (i.e., the individual who made the error).

The sample labels will be placed on each sample container. Sample packaging is discussed in SOP-7.

STANDARD OPERATING PROCEDURE SOP-4

PLANT TISSUE SURVEYING AND SAMPLE COLLECTION

Scope and Applicability

This standard operating procedure (SOP) is specific to the 2018 Plant Tissue Study (hereinafter "the study") being conducted for Teck American Incorporated (TAI) in the northern portion of the Upper Columbia River site in northeastern Washington. This SOP discusses collection of plants by use of hand collection and the procedures for processing collected tissue. Plant tissue for this study will be collected (when available) from the species listed in Table A2 of the field sampling plan (FSP). The final list of collected plants is directly dependent on plant abundance and fecundity. Plant species that are not present, or are present below threshold abundance limits set by the Confederated Tribes of the Colville Reservation (CCT), will not be collected. If plant species are present but have not produced target reproductive tissues (i.e., berries, seeds, or rose hips), those species may not be sampled. The prioritization of sample areas (SAs) to be visited during each field sampling event is described in the FSP and in SOP-1.

Equipment and Materials

Equipment and materials to be used for this SOP are:

- Disposable laboratory gloves (nitrile or latex); these gloves must always be worn when handling plant tissues or soil/sediment samples
- Analytical balance (and calibrated taring weight) or Pesola scale
- Resealable plastic bags
- Waterproof labels and tags
- Permanent marking pen
- Garden shears (collection of small branches and twigs)
- Small saw (collection of larger branches)
- Hand-held spade (root collection)
- Shovel or soil corer (soil sample collection)
- Waders or rubber boots (collection of tules)
- Dropcloth, tarp, or piece of fabric
- Bowl or cup and potable bottled water (float test for hazelnuts)
- Small whiteboards, dry erase markers, whiteboard eraser, or paper towels

- Flags or flagging tape to mark plants
- Tape measure (minimum 20 m)
- Spray bottle with deionized water
- Paper towels
- Measuring cup or other volumetric measurement device
- Coolers and wet ice for plant and soil samples
- Personal protective equipment
- Protective gloves
- Decontamination supplies
- Two-way radios
- Global positioning system (GPS) receivers
- Maps (Site)
- Digital camera
- Field forms and logbook
- Pens and pencils
- First aid kits and health and safety manuals.

Procedures

Ideally, each plant tissue sample will come from one individual plant with a co-located soil/sediment sample taken from directly below the plant. The GPS coordinates of each plant and soil/sediment sample will be recorded. The required tissue from the plant (e.g., leaves, stems, berries, etc.) will be collected and weighed in the field according to the required weight for laboratory analysis of the appropriate species and tissue (FSP Table A2). When possible, discrete samples should be taken from physically distant individuals of the same species. Physically distant plants are less likely to be closely genetically related, are less likely to be sharing nutrients through connected root tissues, and are more likely to uptake nutrients from soil/sediment with different contaminant of interest (COI) concentrations. If all individuals of a target species occur within a patch, individuals as far from one another as possible should be selected for sampling. The U.S. Environmental Protection Agency (EPA) splits and replicates will only be collected from robust individuals when there is sufficient mass for sampling without destructively sampling the plant, or when the plant will be destructively sampled anyway, and the target plant part is large enough to be divided among samples.

Some plant species may not have enough mass for the entire sample to come from one individual. In that case, samples will be composited in the field between nearby individuals with soil/sediment samples composited from below each plant sampled. When possible, adjacent individuals should be selected. Nearer plants are more likely to be genetically related, more likely to be sharing nutrients through connected root networks, and are more likely to uptake nutrients from soil/sediment with similar COI concentrations. Individual plants in a composite must all come from the same SA. GPS coordinates should be collected for each plant sampled as part of a composite sample when individual plants are farther than 3 m from one another (due to GPS accuracy closer plants may not have distinguishable coordinates). Individual plants that require compositing will not be used for EPA splits or replicates.

Sampling will be conducted with two teams—a two-person survey team that will flag potential samples in SAs ahead of the sampling team, which will collect samples. In each SA, the survey team will estimate whether there is enough mass for one or more samples from each target species and flag individual plants accordingly. To determine whether there is sufficient mass for samples, the survey team will make an educated guess based on the plant mass table (FSP Table A2), their prior experience with plant sampling, and in consultation with the sampling team (if needed). The survey team will identify potential limitations regarding the availability of sufficient plant material for kinnikinnick leaves, wild rose leaves and stems, wild mint, willows, and tules to support analysis of mercury, in addition to target analyte list (TAL) metals. If available plant material is not expected to be sufficient to support both types of analysis, allocation of available material for analysis of TAL metals will be prioritized and the decision documented by the field team in consultation with EPA, CCT, and TAI field representatives.

If the survey team does not discover enough plant mass for one sample of a given species, that plant species will not be sampled within that SA. If the survey team estimates that there is enough mass within the SA but the sampling team collects below the minimum sample mass (or below the alternative minimum sample mass[1]) for one sample after collecting all flagged plant specimens within the SA, the sampling team will do one of three actions depending on the type of tissue that was collected: 1) samples of roots and bulbs that are short on mass should be replanted in roughly the same place they were removed; 2) other plant parts (berries, stems, and leaves) will be recorded as a sample if they are within 10 percent of the minimum sample mass because there is a possibility that there will be sufficient mass once dried; 3) samples of non-root plant parts more than 10 percent below the minimum sample

[1] Applies to kinnikinnick leaves, wild rose leaves and stems, wild mint, willows, and tules only.

mass should be returned to the SA for natural decomposition because they are unlikely to have sufficient mass when dried.

For each target plant species, sampling teams will collect up to 12 samples per species (up to 6 in high lead SAs and 6 from lower lead SAs), except for species that form patches. For patch-forming species (kinnikinnick, mint, tule, wild strawberry), additional samples past the 12 may be collected if all 12 samples have been collected but multiple samples were taken from one patch. If after collecting 12 samples another patch is found in a subsequent SA, one additional sample may be collected per new patch to reach 6 samples from distinct high lead patches and 6 samples from distinct lower lead patches. In this case, the second sample collected from a single patch at a previously visited SA may be submitted for analysis as a field duplicate or archived.

The minimum sample mass for each species is designed to provide the laboratory with 0.9 g of dry-weight plant tissue to allow for sampling of TAL metals (except calcium, magnesium, potassium, and sodium), and an additional 1.2 g of dry tissue for species that will additionally be analyzed for mercury[2]. However, the minimum sample mass does not allow for any losses that may occur in the laboratory, such as a glass vessel cracking during testing. Therefore, the target sample mass is twice the minimum sample mass. The minimum sample mass for each species was determined by conducting drying experiments on specimens of the species collected in the field or on reasonable substitutes when field-collected specimens were not available (for example, store-bought mushrooms and tubers). All substituted tissues for drying experiments were selected based on similar mass and tissue density by the field team botanist in consultation with University of Washington colleagues or review of herbarium specimens. Once the wet-to-dry weight ratio was established for each species, the amount of wet weight needed to achieve the dry-weight minimum sample mass was calculated, and 15 percent additional mass was added to account for natural variation in plant material. The target sample weight is twice the minimum sample weight. Alternative target sample mass and the alternative minimum sample mass will only be collected if available plant material is not expected to be sufficient to support both TAL metal and mercury analyses.

Additional considerations were made for plant species needing preparation that will take place in the laboratory: hazelnut, ponderosa pine, and chokecherries. The laboratory will pit the chokecherries and shell the hazelnuts and pine nuts before processing. For hazelnuts, store-bought hazelnuts were dried and weighed without the shell to give better estimates of the number of hazelnuts needed per sample; additional hazelnuts were added to the sample

[2] Analysis of mercury will be limited to the following plant targets: kinnikinnick leaves, wild rose leaves and stems, wild mint, willows, and tules.

to account for field-grown hazelnuts being smaller than store-bought hazelnuts. To estimate field collection mass for chokecherries, the mass for strawberries (which were used in a drying trial) was doubled on the assumption that they have relatively similar density but that chokecherries also have a pit that needs to be removed. Ponderosa pine nuts were collected in the field but were weighed with the husk; to estimate the weight needed for sampling pine nuts without the husks, the sampling team used a literature value for the weight of one pine nut and the number of nuts in a pine cone in conjunction with feedback about how many nuts were found in each pine cone during the August 2017 reconnaissance study.

Splits and Replicates

The target level of splits and replicates is 5 percent of samples across all species. Assuming collection of 6 high lead and 6 lower lead samples for each of the 22 target plant tissues, 5 percent of 264 total samples would round up to 14 replicates and 14 splits. As sampling progresses, sampling teams should take split or replicate samples from each individual plant that has enough target tissue for both samples until the split and replicate targets are met. If more than 28 plants with sufficient mass for twice the sample mass are found, additional splits or replicates can be collected up to 40 splits and 40 replicates (15 percent); however, efforts should be made to collect replicates and splits over all three events rather than all during a single event.

Replicates are created in the field when there is twice the target sample mass on one plant. Two separate samples will be created from the same plant and each will have its own sample number. Collected tissue must be different parts of the same plant (different leaves, different berries, etc.); no roots or bulbs will be used for replicates.

Splits are created in the laboratory when there is twice the sample mass on one plant. In the field, one sample with one sample number is created with twice the target sample mass. Splits can be the same part of the plant (e.g., bulb, root, etc.) or different parts of the plant (berries, leaves, etc.).

If a plant has so much available mass that four times the target sample mass can be collected, both a split and a replicate can be made from the same plant. In that case, the field sampling team will collect three samples. Two samples will have the target sample mass, and will be recorded as the parent sample and the field replicate. The other sample will have twice the target sample mass and will be processed by the laboratory as a split.

For plant tissues that will be analyzed for mercury in addition to TAL metals (kinnikinnick leaves, wild rose leaves and stems, wild mint, willows, and tules), allocation of limited plant

material from an individual plant will be prioritized as follows: analysis for TAL metals, then analysis for mercury, then collection of a field replicate.

Survey and Sampling Procedures

1. The field sampling team will include a field survey team (two members) that identifies target species prior to collection, and a collection team (four members) that collects the physical samples. Additionally, there will be a field supervisor. A registered professional archaeologist will support the field team for the kick-off meeting to assist with tribal communication if needed, and one of the four-person collection team will be a field archaeologist who will stay with the team throughout sampling.
2. The survey team will proceed to each SA concurrently or in advance of the field sampling team. The survey team will notify the field sampling team of any access issues and the field team supervisor will consult with TAI and EPA, as needed, to determine an alternative SA.
3. For accessible SAs, the survey team will walk through each SA ahead of the field sampling team. Surveyor ribbon or pin flags will be used by the survey team to mark the location of plant list targets for potential sample collection by the field sampling team.
4. Selection of plants for flagging and later sampling will be made to the best ability of the survey team. Ideally, robust and physically dispersed individual plants will be selected. The field survey team can select all six samples from within one high or one lower lead SA for a given species; this is recommended to ensure adequate sample collection in the case of species that is more difficult to find. However, if there is *a priori* knowledge that the given species is present in other SAs, the field survey team can spread collection across SAs at their discretion. Additional protocols to select individuals of specific species are provided below in this SOP.
5. When the field survey team finishes flagging each SA, they will make a list in the logbook of which target species were found in each SA, and whether there was enough estimated mass to sample that species (this will generate a list of species that were identified but had insufficient mass to collect).
6. As sufficient samples are collected for each species, the survey team will stop flagging new individuals of that species.
7. The survey team will communicate with the sampling team throughout the day at least each time one team finishes in an SA to ensure that teams are coordinated in their search for required species.
8. The sampling team will document sample location, weather conditions, date and collection time for each sample in the field logbook, and take digital photographs of the area, as indicated in SOP-5 and SOP-6.

9. Samples for plants and patches flagged by the survey team will be collected, measured, and packaged using disposable laboratory gloves. Depending on the target part, the plant material will be removed with garden shears, a spade, or by hand. The plant sample will be weighed. Depending on the target plant, the sample may include multiple leaves, fruits, stems, or roots. Once the required weight is achieved, a sample will be placed in a resealable plastic bag as described in SOP-7 and labeled according to SOP-3. Protocols for collection of individual species are outlined below in this SOP.
10. The target sample mass listed on Table A2 from the FSP should be collected for each sample if possible; however, sample teams are not limited to the target sample mass and can add additional mass if it is present.
11. At each sample location, the plant material will be collected before the soil/sediment is collected. This will avoid damaging any target plant part (e.g., roots) by the soil/sediment sampling effort and will allow for the proper placement of the soil/sediment sample if it is in the center of a patch. In addition, for composite samples this will allow for the correct proportion of soil/sediment to be collected from each location (as described in Step 7 of SOP-9).
12. After plant collection, the sampling team will collect pin flags or surveyor ribbons for disposal or reuse.

Protocols for Sampling Individual Species

Black tree lichen

1. Target sample mass is 2.3 g; minimum sample mass is 1.15 g.
2. To collect lichen, a 20-m-diameter circle should be selected as a "plot" from which black tree lichen should be picked from trees for a composite sample.
3. Individuals should be added to the sample until the sample weighs a minimum of 1.15 g. If there is additional sample mass available within the circular plot, continue adding samples up to or surpassing 2.3 g.
4. One soil sample can be taken from the center of the plot.
5. The GPS location for the plot should be taken with the soil sample from the center of the plot.
6. If sufficient mass has not been collected after one plot has been sampled, select another 20-m-diameter circular plot and sample as above for both lichen and soil.
7. Continue as above until sufficient mass is reached for a 1.15 g sample. All plant tissue should be composited; likewise, all soil samples should be composited.
8. It is unlikely that sufficient mass will be found to create splits or replicates from this species.

Root species

Camas, bitterroot, Lomatium, spring beauty, and Indian carrot are all plants with roots, corms, or bulbs as the target plant tissue. To collect these plant parts, the plant will have to be destructively sampled. The amounts of plant material to be sampled from any one area will be determined in consultation with CCT representatives prior to sampling.

Camas

1. Target sample mass is 4.47 g; minimum sample mass is 2.24 g.
2. Lift out the turf covering the camas, if present, in small sections. Then proceed to dig up the bulbs.
3. Remove the largest bulb and weigh using a Pesola scale. Add additional bulbs until the total weight of the sample is over the minimum.
4. If all of the bulbs of the plant do not weigh at least the minimum sample mass, select the closest camas plant and add sufficient bulbs to create a composite sample.
5. Replant any remaining bulbs and replace the turf.
6. If one camas plant has enough bulbs for twice the target sample mass, create a split or replicate sample. However, do not attempt to create a replicate by cutting the bulb.
7. Wipe tissue sample to remove visible soil and debris, if necessary, using gloved hands or paper towels.

Bitterroot

1. Target sample mass is 28.2 g; minimum sample mass is 14.4 g.
2. Lift out the turf (if present) in small sections.
3. Dig up the root.
4. Remove the red “embryo” or “heart” near the top of the root and discard.
5. Weigh remaining root using a Pesola scale.
6. If the root weighs less than the minimum sample weight, select the next closest plant and add the additional root to the first sample to create a composite sample.
7. Do not create replicates of this species. If a root is twice the target sample mass, it can be used as a laboratory split.
8. Wipe the tissue sample to remove visible soil and debris, if necessary, using gloved hands or paper towels.
9. Rinse the tissue samples with deionized water from a spray bottle to remove soil or loose debris, dry with a paper towel, and place in a sample bag.

Lomatium

There are several species of Lomatium that occur in this area. All sampled species will be lumped into *Lomatium* spp. although the actual species name should be marked on field data sheets if identifiable.

1. Target sample mass is 8.05 g; minimum sample mass is 4.03 g.
2. Lift out the turf (if present) in small sections then dig up the root.
3. Weigh the root with a Pesola scale.
4. If the root weighs less than the minimum sample weight, select the next closest plant and add the root to the first sample to create a composite sample. If possible, composite samples should be composed of the same species.
5. Do not create replicates of this species. If a root is twice the target sample mass, it can be used as a laboratory split.
6. Wipe the tissue sample to remove visible soil and debris, if necessary, using gloved hands or paper towels.
7. Rinse the tissue samples with deionized water from a spray bottle to remove soil or loose debris, dry with a paper towel, and place in a sample bag.

Spring beauty/Indian potato

1. Target sample mass is 3.79 g; minimum sample mass is 1.89 g.
2. Lift out the turf (if present) in small sections.
3. Dig up the corm.
4. Remove the largest corm (about 1 cm long, 0.5 cm in diameter, and close to the surface) and weigh using a Pesola scale.
5. Add additional corms until the total weight of the sample is above the minimum sample mass. Replant any remaining corms and replace the turf.
6. If all of the corms of the plant do not weigh at least the minimum sample mass, select the closest spring beauty plant and add sufficient corms to create a composite sample.
7. If one spring beauty plant has enough corms for a split or replicate, create a split or replicate sample. If not, do not attempt to create a replicate by cutting the corm.
8. Wipe the tissue sample to remove visible soil and debris, if necessary, using gloved hands or paper towels.

Indian carrot

1. Target sample weight is Pending g; minimum sample weight is Pending g.
2. Lift out the turf (if present) in small sections.
3. Dig out the root.

4. Weigh using a Pesola scale.
5. If the root weighs less than the minimum sample weight, select the next closest plant and add the root to the first sample to create a composite sample.
6. Do not create splits or replicates of this species.
7. Wipe the tissue sample to remove visible soil and debris, if necessary, using gloved hands or paper towels.
8. Rinse the tissue samples with deionized water from a spray bottle to remove soil or loose debris, dry with a paper towel, and place in a sample bag.

Kinnikinnick

1. Kinnikinnick grows in large patches and forms roots from multiple branches, making it difficult to determine what constitutes an individual plant. If possible, samples should be taken from different patches.
2. Target sample mass is 5.3 g (around 120 leaves); minimum sample mass is 2.7 g.
3. If the site has only one patch of kinnikinnick, "individual" samples should be collected from at least 20 ft apart because an individual can spread up to 15 ft.
4. Pull leaves off the plant and weigh with a Pesola scale until the minimum sample mass or the target sample mass is met.
5. Only take up to one-third of an individual plant's leaves to avoid damaging the plant if the sampler can determine what constitutes an individual.
6. If an individual plant is large enough, take twice the sample and create a split or replicate sample.
7. If one kinnikinnick plant does not supply over the minimum sample mass, select the next closest plant and add the leaves to the first sample to create a composite sample. If the nearest plant is within the same patch it is probably the same individual; thus, it will not be considered a composite sample, and one soil sample can be taken in the middle of the sampled plants.
8. Wipe the tissue sample to remove visible soil and debris, if necessary, using gloved hands or paper towels.
9. If after compositing all available tissue within an SA there is not enough mass for a minimum sample, an alternative target sample (2.5 g) or an alternative minimum sample (1.3 g) should be collected for analysis of TAL metals without mercury.

Mushrooms

These directions apply for all puffballs, morels, and shaggy manes. These species may grow as an individual mushroom, in pairs, or in a group. If the mushroom is growing singly, the soil sample should be taken next to the stalk. If the mushroom is growing in a pair or group

(within 1 m), the soil sample can be taken from the middle of the group of individuals sampled because they are likely all the same genetic individuals.

1. Target mass for puffballs is 5.29 g; minimum sample mass is 2.65 g.
2. Target mass for morels is 5.29 g; minimum sample mass is 2.65 g.
3. Target mass for shaggy manes is 19.4 g; minimum sample mass is 9.68 g.
4. Pick an individual mushroom and weigh it using a Pesola scale.
5. If the mushroom is part of a group (within 1 m), add additional mushrooms until the sample is over the minimum sample mass.
6. If it does not weigh at least the minimum sample weight, select the next closest mushroom or group of mushrooms of the same species and add the mushroom to the first sample as a composite sample.
7. If a group of mushrooms is large enough, take a split or replicate sample. Individual mushrooms should not be cut to create replicate samples.
8. Wipe the tissue sample to remove visible soil and debris, if necessary, using gloved hands or paper towels.

Wild rose

The wild rose species group is composed of *Rosa gymnocarpa, R. nutkana,* and *R. woodsii*; individual species name should be marked on each sample if they are identifiable, otherwise mark samples as *Rosa* sp. To increase the odds that rose hips are available in August, the tops of large plants or branches with flower buds should not be snipped if sufficient mass can be collected without doing so. Smaller rose plants can have all aboveground vegetative material sampled; they do not produce many rose hips and they should grow back from the roots.

1. Target sample mass (length) is 48.5 cm; minimum sample mass (length) is 24.5 cm.
2. Samples should include young leaves and tender stems.
3. If one individual plant does not have enough stem length for sampling, select the next closest plant and add sufficient stems and leaves for a composite sample. If the plant is large enough, a split or replicate sample can be taken.
4. Wipe the tissue sample to remove visible soil and debris, if necessary, using gloved hands or paper towels.
5. If after compositing all available tissue within an SA there is still not enough mass for a minimum sample, an alternative target sample (23 cm) or an alternative minimum sample (11.5 cm) should be collected for analysis of TAL metals without mercury.

Willows

1. All of the following species are used by tribes and have various local names: *Salix exigua*, *S. scouleriana*, and *S. bebbiana*. Therefore, the most prevalent species found at the SA should be selected as a representative of the guild.
2. Target sample mass (length) is 189 cm; minimum sample mass (length) is 126 cm.
3. Select a branch that is no more than 0.5 in. in diameter and cut it off from an individual tree.
4. Continue to collect branches from an individual tree for each sample until the combined branch length is at least 60 cm.
5. Wipe the tissue sample to remove visible soil and debris, if necessary, using gloved hands or paper towels.
6. Take a soil/sediment sample from beneath the crown of the tree.
7. If after compositing all available tissue within an SA there is still not enough mass for a minimum sample, an alternative target sample (90 cm) or an alternative minimum sample (60 cm) should be collected for analysis of TAL metals without mercury.

Sarvisberry, wild strawberry, and huckleberry

The wild strawberry species group is composed of *Fragaria vesca* and *F. virginiana*; individual species name should be marked on each sample if they can be identified. Wild strawberries can grow from seeds or from rhizomes. If they are growing in a patch, it will be difficult to tell individual plants apart because they may be the same genetic individual. Within a patch, all strawberries can be collected as one discrete sample with one soil sample collected from the center of the patch.

1. Target sample mass is 30.6 g; minimum sample mass is 15.3 g.
2. Select an individual plant that is heavily berried, if possible.
3. Pick berries and add to sample until at least the minimum sample mass has been reached. If there are additional berries on the plant, add mass until the target sample mass has been achieved or all berries (both ripe and immature) have been picked.
4. The first time that a heavily berried plant is found for each species, also measure the volume of the sample. This will only be done one time per species to calculate the mass-to-volume ratio.
5. Sample soil from below the crown of the plant.
6. If an individual plant does not have enough berries, select the next closest plant and pick enough berries to exceed the minimum sample mass for one composite sample.
7. If a plant has twice the target sample mass, take a split or replicate sample.

Chokecherry

1. Target sample mass is 60 g; minimum sample mass is 30 g.
2. Select an individual plant that is heavily berried, if possible.
3. Pick berries and add to sample until at least the minimum sample mass has been reached. If there are additional berries on the plant, add mass until the target sample mass has been achieved or all berries (both ripe and immature) have been picked.
4. The first time that a heavily berried plant is found, also measure the volume of the sample. This will only be done one time per species to calculate the mass-to-volume ratio.
5. Sample soil from below the crown of the plant.
6. If an individual plant does not have enough berries, select the next closest plant and pick enough berries to exceed the minimum sample mass for one composite sample.
7. If a plant has twice the target sample mass, take a split or replicate sample.

Hazelnut

1. Target sample mass is 6 nuts; minimum sample mass is 3 nuts.
2. Spread a cloth along the ground under the plant of interest and gently shake the branches to collect ripe nuts.
3. Select up to 25 nuts and discard any that have obvious insect damage.
4. Put remaining nuts into a cup or bowl of deionized water and discard those that float (float test; nuts that float are empty or have insect damage that is not visible on the shell).
5. Dry off remaining nuts and put them into the sample bag. The float test and drying of hazelnuts takes the place of wiping and spraying to remove visible soil and debris because that is a side effect of the float test.
6. If there are not enough ripe nuts, pick immature nuts from the plant, check for insects, and do the float test.
7. If there are more nuts available, field teams can add more nuts per sample past the target sample mass up to 12 nuts per sample.
8. If there are still not enough nuts for one sample, select the next closest plant and repeat steps 2 through 6 until sufficient nuts have been collected for the composite sample.
9. If the plant has at least twice the target sample mass of hazelnuts that pass both visual inspection and the float test, take a split or replicate sample. Do not attempt to cut nuts to create replicate samples.

Ponderosa pine

1. Target sample mass is 20 undamaged pine cones or equivalent; minimum sample mass is 10 undamaged pine cones or equivalent. (Fifty-one pine nuts are necessary for one sample. Undamaged cones can have up to 70 nuts; however, that is rare.)
2. To collect from individual trees, it would be best to pick cones from the branches of one tree by hand or by using landscaping tree trimmers. If that is not possible, select an isolated tree and pick up cones from the ground directly beneath and surrounding it.
3. If the trees are in a dense stand, cones tend to spread no farther than 1.5 times the height of the tree. Pick up cones beneath an individual tree, then move at least 1.5 times the height of that tree before picking up cones below another tree.
4. If possible, the survey team should pick trees from multiple SAs because it is a common plant and individuals must be sampled at some distance from one another.
5. Field teams should attempt to select pine cones that do not show herbivore damage, mold, or rot.
6. Co-located soil samples should be taken from directly below the individual tree selected for pine cone collection.
7. If one tree does not have sufficient cones below it for a minimum sample, create a composite sample with the closest ponderosa pine. Cones can be picked up below each tree and from the ground between the two trees. Soil samples should be taken for each tree used in a composite sample.
8. If there are enough pine cones below one tree, take a split or replicate sample. Cones should not be cut to create replicate samples.

Wild mint

Wild mint grows in patches by spreading rhizomes, making it difficult to differentiate individual plants. If more than one patch is found in the same SA, distribute sample collection among patches.

1. The target sample mass is 8.4 g; the minimum sample mass is 4.2 g.
2. Because one plant usually does not have enough leaves for a whole sample, select the next closest plant and continue sampling. Nearby mint plants are probably the same individual; therefore, this will not be considered a composite sample.
3. One soil sample can be taken in the middle of the sampled plants. Mint is robust to foraging; thus, all of the leaves can be taken from several stems without harming the plant.
4. Within each patch select individual samples as far from one another as possible.
5. Note which samples come from the same patch on field data sheets.

6. If one patch has enough sample mass for twice the target sample mass, a split or replicate sample can be collected.
7. If after compositing all available tissue within an SA there is still not enough mass for a minimum sample, an alternative target sample (4 g) or an alternative minimum sample (2 g) should be collected for analysis of TAL metals without mercury.

Tule

Tule grow in large patches propagated by rhizomes. This means that it will be impossible to determine what constitutes a genetically distinct "individual" within our collection timeframe. If more than one patch is found in the same SA, distribute sample collection among patches.

1. Target sample mass (length) is 189 cm; minimum sample mass (length) is 126 cm.
2. Within each patch, select individual tule culms as far from one another as possible for different samples.
3. Select an individual culm that is no more than 0.5 in. in diameter and cut it close to the rhizome, below water level if water is present.
4. Remove the reproductive parts and discard them near the mature plants.
5. Shake as much water off as possible (if water is present) and measure the specimen.
6. If one culm does not exceed the target sample length, select the next closest culm and sample it as well. Because these culms are likely part of the same genetic individual, this is not considered a composite sample, and one soil/sediment sample can be taken from the middle of the sampled stems.
7. Continue to collect culms for each sample until the combined culm length is at least 60 cm.
8. Note which samples come from the same patch on field data sheets.
9. Collect soil/sediment samples from below the location in which tules were collected.
10. If a patch of tule has enough individual nearby culms to double the target sample length, a split or replicate sample can be collected. Replicate samples should consist of different culms.
11. Wipe the tissue sample to remove visible soil and debris, if necessary, using gloved hands or paper towels.
12. Rinse the tissue samples with deionized water from a spray bottle to remove soil, dry with paper towel, and place in a sample bag.
13. If after compositing all available tissue within an SA there is still not enough mass for a minimum sample, an alternative target sample (90 cm) or an alternative minimum sample (60 cm) should be collected for analysis of TAL metals without mercury.

STANDARD OPERATING PROCEDURE SOP-5

FIELD DOCUMENTATION

Scope and Applicability

This standard operating procedure (SOP) is specific to the 2018 Plant Tissue Study (hereinafter "the study") being conducted for Teck American Incorporated (TAI) in the northern portion of the Upper Columbia River site in northeastern Washington. This SOP presents the general information that will be recorded for all plant tissue and soil/sediment sampling activities conducted by TAI field personnel at sampling stations for the study. Proper record keeping will be conducted in the field to allow samples to be accurately traced from collection to final disposition. All information pertaining to field operations during sample collection must be properly documented to ensure transparency and reproducibility of methods and procedures. This information will be recorded by field personnel during each sampling event in field logbooks, on field forms, and using digital photography to achieve this purpose.

Equipment and Materials

Equipment and materials used for this SOP are:

- Field logbook (electronic or all weather paper)
- Field forms
- Black-ink pen
- Small whiteboards, dry erase markers, and whiteboard eraser or paper towels
- Digital camera.

Field Logbooks

During field sampling events, field logbooks, and field forms (Attachment A3 of this FSP) are used to record all daily field activities. The purpose of the field logbook is to thoroughly document the sampling event to ensure transparency and reproducibility. The field logbook will contain sampling-related information supplemental to the field forms. Any deviations from the project-specific field sampling plan (FSP) that occur during sampling (e.g., personnel, responsibilities, sample station locations) and the reasons for these changes must be documented in the field logbook. Other types of information that will be recorded in the logbook include the following:

- Project name
- Name of each person making entries and other field staff

- The names and affiliations of any onsite visitors
- Observations made during sample collection, including difficulties encountered during sample collection, visible debris, and other details not entered onto the field form
- A record of site health and safety meetings, updates, and related monitoring
- Presence of construction and maintenance activities or man-made features that may influence plant abundance or distribution.

The field supervisor is responsible for maintaining the field logbook and ensuring that the field logbook and all field data forms are correct. Requirements for logbook entries include the following:

- Entries will be made legibly with black (or dark) waterproof ink.
- Unbiased, accurate language will be used.
- Entries will be made while activities are in progress or as soon afterward as possible (the date and time that the notation is made should be documented, as well as the time of the observation itself).
- Each consecutive day's first entry will be made on a new, blank page.
- The field supervisor must sign and date the last page of each daily entry in the field logbook (either electronic or hardcopy).
- When field activity is complete, the logbook will be entered into the TAI project file.

All logbook entries must be completed at the time any observations are made. Logbook corrections will be made by drawing a single line through the original entry, allowing the original entry to be read. The corrected entry will be written alongside the original. Corrections will be initialed and dated and may require a footnote for explanation. Blank lines on a page or blank pages in the field logbook will be lined out to indicate that they were intentionally left blank.

In addition to the preceding requirements, if a paper logbook is used, the person recording the information must initial and date each page of the field logbook. If more than one individual makes entries on the same page, each recorder must initial and date each entry. The bottom of the page must be signed and dated by the individual who makes the last entry.

Upon completion of the field sampling event, the field supervisor will be responsible for submitting all field logbooks to be copied and distributed as described below.

Field Forms

Field data forms will be used to record the relevant sample information collected during a sampling event. These forms will be filled out completely by the sampling team during sample collection and will include the following information:

- Project name and date
- Field crew initials
- The time each sample was collected
- Sampling area identifier, as specified in the FSP
- The global positioning system (GPS) unit used to record the sampling location details
- The sample identifier and analyses to be performed, as specified in the FSP
- A list of numbered photographs associated with each sample
- Any additional collection comments.

Upon completion of the field sampling event, the field supervisor will be responsible for submitting all field data forms to be copied and distributed as described below.

Photographs

Reference SOP-6 of the FSP for procedures regarding the documentation requirements for digital photographs.

Distribution of Copies

Electronic scans of the field logbooks and field data forms will be made after completion of the field sampling event and stored electronically in the project files for use by project staff. The original field logbooks and forms will be placed in a locked file cabinet at the task manager's location.

Setup of Locking File Cabinet

Each field event will have its own dedicated section in a locking file cabinet. The section label will include the project name and TAI work order number. The following documents may be included in this cabinet for each field event:

- Original field logbooks
- Original field data forms
- Photograph CDs (see SOP-6)
- Original signed chain-of-custody (COC) forms.

STANDARD OPERATING PROCEDURE SOP-6

DIGITAL CAMERA USE AND DOCUMENTATION PROCEDURES

Scope and Applicability

This standard operating procedure (SOP) is specific to the 2018 Plant Tissue Study (hereinafter "the study") being conducted for Teck American Incorporated (TAI) in the northern portion of the Upper Columbia River site in northeastern Washington. This SOP applies to taking digital photographs and placing the digital data in a database. Digital photographs may be taken to document field activities, site conditions and features, and sampling locations.

Equipment and Materials

Equipment and materials for taking digital photographs are:

- Digital camera
- Digital storage card
- Spare batteries
- 12-V charger
- Digital camera-carrying case and manual
- Field form
- Small whiteboards, dry erase markers, and whiteboard eraser or paper towels
- Compass
- Personal computer.

Typical Camera Features

- Ability to save photographs (in standard mode) directly to a memory stick, digital storage card, or comparable data storage device
- Auto focus; manual focus available if required
- Zoom appropriate for medium distances (no micro or macro lenses should be necessary)
- Brightness control
- Playback of photographs on camera screen

- Display of photograph number, date, and time
- Flash
- Timer
- Display showing time remaining on battery and remaining storage capacity (memory stick, secure digital card, or other)
- Ability to protect and delete images that have been taken.

Camera Use

Digital cameras will be used by the field team to document field activities. Each field team will be directly responsible for the camera and ensure that it is not exposed to excessive heat, cold, or moisture. The field team leader will be responsible for digital photograph documentation or for assigning documentation duties to a team member.

Digital photographs will be taken to document field activities and sampling locations. Examples of field activities for which photo documentation will be useful include: 1) individual plants sampled; 2) soil/sediment sample location; and 3) field sampling techniques used, such as equipment use and operation.

Digital photographs will be collected at a high-pixel setting such that enlargements can be made with minimal degradation in picture quality.

Photograph Documentation

Field Team Responsibilities

Each field team member will keep a daily hardcopy log of all photographs taken during the day. The following digital photograph data will be collected:

- Date and time—as provided by the camera display
- Location ID
- Sample ID
- Photograph ID
- Photograph direction
- Photograph notes
- Comments (including camera number if more than one camera is used)

Digital Photograph File Name

At the end of each field day, the member of the field team who is responsible for the camera will transfer the electronic data from the camera to the field operations computer. The folder structure will be as follows:

\\DATA\PHOTOS\YYYYMMDD\SAMPLE AREA\file\[1, 2, 3, … N]

The notation YYYYMMDD represents the year, month, and day. The sample area (SA) is the sampling area name (e.g., SA01) as specified in the Field Sampling Plan (FSP). The individual files for the day (e.g., file 1, file 2, … file N) will be placed within this folder using the default file identifier provided by the camera.

Transfer of Information and Archive

After data from the photograph disks have been uploaded, the original hard copy of the photograph log will be initialed and dated by the team member who downloaded the photographs, then archived by the responsible field team leader.

Sample Processing Coordinator Responsibilities

The field team leader will be responsible for: 1) reviewing electronic photographs and the associated logs as they are made available to ensure consistency and completeness of annotations; 2) collecting and archiving hard copies of the photograph logs; and 3) notifying the sampling team leader of apparent inconsistencies, and making recommendations for corrective action when they are discovered.

Key Checks and Items

Important checks for digital camera management are:

- Make sure the camera's battery and spare batteries are fully charged on a daily basis
- Keep extra memory sticks or digital storage cards available
- Use flash only when necessary to save battery life
- Make sure the camera quality level is set at "best" or equivalent (high pixel)
- Review photograph records periodically to ensure that the electronic photographs and the data log agree
- Allow enough time at the end of the field day to transfer the data.

STANDARD OPERATING PROCEDURE SOP-7

SAMPLE PACKAGING AND SHIPPING

Scope and Applicability

This standard operating procedure (SOP) is specific to the 2018 Plant Tissue Study (hereinafter "the study") being conducted for Teck American Incorporated (TAI) in the northern portion of the Upper Columbia River site in northeastern Washington. This SOP presents the requirements and procedures to be used when packaging plant tissue and soil/sediment samples collected from sampling stations across the study area for hand-delivery or shipping by commercial carrier to the analytical laboratory. Specific requirements for sample packaging and shipping must be followed to ensure the proper transfer and documentation of environmental samples collected during field operations. Procedures for the careful and consistent transfer of samples from the field to the laboratory are outlined herein.

Equipment and Materials

Specific equipment or supplies necessary to properly pack and ship environmental samples include the following:

- Field sampling plan (FSP)
- Project-specific field logbook
- Resealable airtight bags (assorted sizes)
- Food-grade heavy duty aluminum foil
- Laboratory-supplied hard-sided plastic coolers
- Thermometer
- Drum liners or sturdy trash bags for securing samples within coolers
- Large plastic garbage bags (preferably 3 mil [0.003 in.] thick) for cooler lining
- Fiber-reinforced packing tape and duct tape
- Clear plastic packing tape
- Scissors or knife
- Bubble wrap
- Chain-of-custody (COC) forms; these may be produced in an electronic format using a database program (e.g., FORMS II Lite), in which case a computer and printer is also required

- COC seals
- Paper towels
- "Fragile," "This End Up" or "Handle With Care" labels, and "Perishable Goods" labels
- Address labels for processing laboratory
- Airbills for overnight shipping to the laboratory.

Procedure

After completing each day of sampling, all plant tissue samples will be transferred from the coolers with wet ice into a freezer or cooler with dry ice, and held there until preparation for shipment to the laboratory. Samples will be frozen within 20 hours of collection, typically as soon as the field crew returns to the location where samples are being held until shipment to the laboratory. The temperature of the cooler or freezer will be recorded in the field logbook twice daily (once in the morning and once in the evening).

In the field, soil/sediment samples will be sealed into wide-mouthed jars and stored in a cooler with wet ice at 4°C ±2°C (if being analyzed for mercury) until being shipped overnight to the laboratory. Samples not being analyzed for mercury do not have to be stored on ice. The temperature of the cooler will be recorded in the field logbook twice daily (once in the morning and once in the evening).

Depending on the logistics of the operation, field personnel may transport samples to the laboratory themselves or use a commercial courier or shipping service. If a courier service is used, then field personnel should be aware of certain factors that may limit the capability for timely shipping (e.g., availability of overnight service and weekend deliveries to specific areas of the country, and shipping regulations regarding "restricted articles") prior to shipping the samples.

Sample Storage Prior to Shipment

Samples will be placed in secure storage (i.e., locked room or vehicle) or remain in the possession of sampling personnel before shipment. Sample storage areas will be locked and secured to maintain sample integrity and COC requirements. The storage temperature will be maintained for plant tissue samples in a secure area while they are awaiting shipping.

Sample Preparation for Plant Tissue Samples

The following steps should be followed to ensure the proper transfer of samples from the field to the laboratory.

At the Sample Collection Site and Following the Completion of the Sampling Day

1. Document all samples appropriately using the proper field logbooks, field forms, and required sample container identification (i.e., sample labels with unique identification numbers [IDs]) by following the sample labeling procedures described in SOP-3.
2. Clean the outside of all dirty sample containers to remove any residual material that may lead to cross contamination.
3. Place labeled and bagged plant tissue samples in a second resealable plastic bag such that the sample label can be read, and place double-bagged sample into a cooler.
4. Because the samples have a required storage temperature, place a sufficient amount of wet ice in the sample cooler to maintain the temperature inside the cooler (e.g., 4°C) throughout the sampling day.
5. At the end of each sampling day, plant tissue samples will be transferred from the coolers with wet ice into a freezer or cooler with dry ice, and held there until preparation for shipment to the laboratory.

To Prepare Plant Tissue Samples and Coolers for Shipping

1. Choose the appropriately sized laboratory-supplied hard-sided plastic cooler for dry ice, making sure that the outside and inside of the cooler is clean of gross contamination.
2. Line the cooler with bubble wrap.
3. Concurrently with placing samples in the shipping cooler, the field supervisor will fill out electronic COC forms with sample IDs and laboratory analyses to be performed (see example blank and filled out COC forms in Attachment A3 to the FSP).
4. Make sure any applicable laboratory quality control sample designations have been made on the COC forms.
5. Check sample IDs for all samples against the COC form to ensure all samples intended for shipment to the laboratory are included.
6. Samples will be placed inside a large plastic bag (e.g., sturdy garbage bag or drum liner); the bag will be tied closed and sealed at the tied area with a custody seal to ensure that custody is maintained if the cooler is opened for inspection during shipment.
7. Because the samples have a required storage temperature, add enough dry ice to keep the samples frozen during overnight shipping or driving (i.e., <0°C). The amount of dry ice that may be required should always be overestimated to ensure

the samples are kept frozen. Ice should be enclosed in a resealable plastic bag and then placed in a second sealable plastic bag to prevent leakage. Avoid separating the samples from the ice with excess bubble wrap because it will insulate the containers from the ice.

8. Sufficient plant tissue samples will be placed in each cooler to occupy approximately 60 to 70 percent of the cooler volume, and the remaining space in the cooler will be filled with dry ice.

9. After all samples and ice have been added to the cooler, use bubble wrap (or other available clean packing material) to fill any empty space to keep the samples from shifting during transport.

10. The field supervisor will sign and date the completed COC form and retain a copy for the project files. Place the signed COC form in a resealable bag and tape the bag containing the form to the inside of the cooler lid. Each cooler should contain an individual COC form, or multiple forms as appropriate, for the samples contained in that particular cooler.

11. After the cooler is sufficiently packed to prevent shifting of the containers, close the lid and seal it shut with fiber-reinforced packing tape. The cooler should be taped shut using nylon strapping tape around the opening between the lid and the bottom of the cooler and around the circumference of the cooler at both hinges.

12. As security against unauthorized handling of the samples, apply two COC seals across the opening of the cooler lid—one on the front of the cooler and one on the side. Be sure the seals are properly affixed to the cooler so they are not removed during shipment. Additional clear packing tape placed across the seal may be necessary if the outside of the cooler is wet.

13. Affix appropriate shipping labels indicating the use of dry ice to the outside of the coolers.

14. Also attach the address label for the processing laboratory, overnight shipping bill, a "Perishable Goods" label, and at least one of the following labels: "This End Up," "Fragile," or "Handle With Care."

15. Notify the laboratory project manager and quality assurance manager that samples will be shipped and the estimated arrival time. Upon completion of field activities, the field supervisor will provide copies of all COC forms to the task manager and task analytical chemistry quality assurance and quality control (QA/QC) coordinator.

Sample Preparation for Soil/Sediment Samples

The following steps should be followed to ensure the proper transfer of samples from the field to the laboratory.

At the Sample Collection Site and Following the Completion of the Sampling Day

1. Document all samples appropriately using the proper field logbooks, field forms, and required sample container identification (i.e., sample labels with unique IDs) by following the sample labeling procedures described in SOP-3.
2. Clean the outside of all dirty sample containers to remove any residual material that may lead to cross-contamination.
3. Store each sample container in an individual sealable plastic bag that allows the sample label to be read.
4. Place a sufficient amount of wet ice in the sample cooler to maintain the temperature inside the cooler (i.e., 4°C ±2°C) throughout the sampling day if mercury is being analyzed in the samples. Samples not being analyzed for mercury do not have to be stored on ice.
5. Place labeled soil/sediment sample jars into a box or cooler such that they are not at risk for breaking or coming into contact with ice melt water during transport from the field.
6. Store all sample containers under custody until ready for shipping.

To Prepare Soil/Sediment Samples and Coolers for Shipping

1. Choose the appropriately sized laboratory-supplied hard-sided plastic cooler and make sure that the outside and inside of the cooler is clean of gross contamination. If the cooler has an external drain, the drain must be capped and thoroughly taped shut with duct tape.
2. Use bubble wrap to line the cooler and place an opened large plastic bag (preferably a bag with a thickness of 3 mil) inside the cooler and inside of the bubble wrap.
3. Check that the bag protecting the sample container is zipped completely and does not appear to have leaks. If the resealable bag is damaged, exchange the bag for a new one (this will protect the sample from contact with and potential contamination from iced water). Individually wrap each sample container in bubble wrap, wrapping around the resealable bag, using either packing tape or a rubber band to secure and hold the bubble wrap in place. Place the bubble-wrapped sample into the large plastic bag in the cooler.

4. For samples being analyzed for mercury, add enough ice to keep the samples refrigerated during shipping (i.e., 4°C ±2°C). Always overestimate the amount of ice that may be required. Place the ice in sealable plastic bags and then place each bag into a second sealable plastic bag to prevent leakage. Avoid separating the samples from the ice with excess bubble wrap because it will insulate the containers from the ice.
5. Samples designated for mercury analysis will be placed in a cooler to occupy approximately 60 to 70 percent of the interior space, and the remaining space inside the cooler will be filled with wet ice. Samples not designated for mercury analysis do not need to be cooled or stored on ice following collection or during transport. Separate coolers will be used for soil samples that require refrigeration (those designated for mercury analysis) and those that do not require refrigeration (no mercury analysis).
6. After all samples and ice (if applicable) have been added to the cooler, use bubble wrap (or other available clean packing material) to fill any empty space to keep the samples from shifting during transport.
7. While the samples are being placed in the shipping cooler, the field supervisor will fill out the COC form and include the sample IDs and laboratory analyses to be performed (see example blank and filled out COC forms in Attachment A3 to the FSP).
8. Make sure all applicable laboratory quality control sample designations have been made on the COC forms.
9. Check sample containers against the COC form to ensure all samples intended for shipment to the laboratory are included. Information on the COC form shall only include sample information for the samples within the individual cooler.
10. The field supervisor will sign and date the completed COC form and retain a copy for the project files. Place the signed COC form in a resealable bag and tape the bag containing the form to the inside of the cooler lid. Each cooler must contain an individual COC form (or multiple COC forms) for the samples contained in that particular cooler.
11. After the cooler is sufficiently packed to prevent shifting of the containers, close the lid and seal it shut with fiber-reinforced packing tape. The cooler must be taped shut around the opening between the lid and the bottom of the cooler and around the circumference of the cooler at both hinges.
12. As security against unauthorized handling of the samples, apply two COC seals across the opening of the cooler lid—one on the front of the cooler and one on the

side. Be sure the seals are properly affixed to the cooler so they are not removed during shipment. Additional clear packing tape placed across the seal may be necessary if the outside of the cooler is wet.

13. Also attach the address label for the processing laboratory, overnight shipping bill, and at least one of the following labels: "This End Up," "Fragile," or "Handle With Care."

14. Notify the laboratory project manager and quality assurance manager that samples will be shipped and the estimated arrival time. Upon completion of field activities, the field supervisor will provide copies of all COC forms to the task manager and task analytical chemistry QA/QC coordinator.

Sample Shipping

Hand Delivery to the Analytical Laboratory

1. The field supervisor will notify the analytical laboratory contact and the team project QA/QC coordinator that samples will be hand-delivered to the laboratory by a field team member, and will provide the estimated arrival time.
2. All environmental samples that are hand-delivered to the analytical laboratory will be received by the laboratory on the same day that they were packed in the coolers.
3. Copies of all COC forms will be provided to the task manager.

Shipped by Commercial Carrier to the Laboratory

1. Use an address label and label the cooler with destination and return addresses, and add other appropriate stickers, such as "This End Up," "Fragile," and/or "Handle With Care," and "Perishable Goods" labels for coolers packed with plant tissue samples. For shipments containing two or more coolers, indicate on the address label the total number of coolers that the analytical laboratory should expect to receive (e.g., 1 of 2; 2 of 2). Place clear tape over the mailing label to firmly affix it to the outside of the cooler and to protect it from the weather. This is a secondary label in case the airbill is lost during shipment.
2. Fill out the airbill as required and fasten it to handle tags provided by the shipper (or the top of the cooler if handle tags are not available).

STANDARD OPERATING PROCEDURE SOP-8

DECONTAMINATION OF SAMPLING EQUIPMENT

Scope and Applicability

This standard operating procedure (SOP) is specific to the 2018 Plant Tissue Study (hereinafter "the study") being conducted for Teck American Incorporated (TAI) in the northern portion of the Upper Columbia River site in northeastern Washington. This SOP describes procedures for decontaminating sampling and processing equipment contaminated by inorganic materials.

In general, plant tissue and soil/sediment sampling will not require equipment decontamination. Plastic bags will be used as a barrier between weighing equipment. For example, a resealable plastic bag may be used to hold plant tissue during weighing instead of placing the specimen directly on the scale.

To prevent potential cross-contamination of samples, all reusable sampling and processing equipment will be decontaminated before each use. Decontaminated equipment will be stored away from areas that may cause recontamination. When handling decontamination chemicals, field personnel will follow all relevant procedures and will wear protective clothing as stipulated in the site health and safety plan (SHSP).

Equipment and Materials

Equipment required for decontamination includes the following:

- Plastic buckets (e.g., 5-gallon bucket)
- Properly labeled squirt bottles (or large spray bottles if needed)
- Long-handled, hard-bristle brushes
- Plastic sheeting, garbage bags, and aluminum foil
- Tap water or potable site water
- Personal protective equipment as specified in the SHSP.

Decontamination Procedures

When necessary, reusable sampling equipment should be decontaminated before and after the sampling effort and between sampling stations. The specific procedures for decontaminating reusable sampling equipment are as follows:

1. Rinse the equipment thoroughly with tap or potable site water to remove any visible sediment or debris.

2. Pour a small amount of concentrated laboratory detergent (e.g., Alconox, Liquinox) into a bucket (e.g., about 1/2 tablespoon per 5-gallon bucket) and fill it halfway with tap or potable site water. If the detergent is in crystal form, all crystals should be completely dissolved prior to use.
3. Scrub the equipment in the detergent solution using a long-handled brush with rigid bristles, using a back-and-forth motion. Be sure to clean the outside of samplers, bowls, and other tools that may be covered with sediment or tissue. Remove all particulate matter and surface films.
4. Rinse with tap or potable site water. Equipment does not need to be dried before use.
5. If the decontaminated sampling equipment will not be used immediately, wrap small items in aluminum foil for storage with the dull side facing the cleaned surfaces until the next use. Large items such as shovels will be wrapped in clean plastic bags.
6. If the sampling collection or processing equipment is cleaned at the field laboratory and transported to the site, wrap the decontaminated equipment in aluminum foil, with the dull side facing the cleaned surfaces, and store and transport the cleaned equipment in a clean plastic bag (e.g., a trash bag) until ready for use, unless the FSP specifies that special handling procedures should be followed.

STANDARD OPERATING PROCEDURE SOP-9A

DISCRETE SOIL SAMPLE COLLECTION

This procedure describes the project-specific requirements to ensure the collection of accurate, representative soil samples within the plant tissue sampling areas identified in the associated Field Sampling Plan (FSP), but requires vigilant care and precision by each sampling team member. Discrete soil samples will be co-located with and collected adjacent to corresponding discrete plant tissue sampling locations.

Scope and Applicability

This standard operating procedure (SOP) is specific to the 2018 Plant Tissue Study (hereinafter "the study") being conducted for Teck American Incorporated (TAI) in the northern portion of the Upper Columbia River site in northeastern Washington. This SOP describes the procedures used for collecting discrete soil samples during the study.

The following procedures may be modified in the field by the field supervisor (in consultation with TAI and the U.S. Environmental Protection Agency [EPA]) based on conditions encountered in the field. Such changes should be noted in the field logbook and on field change forms along with the names of the TAI and EPA representatives who have authorized the change.

Equipment and Materials

The equipment and materials needed by the sampling team includes the following items:

- Project-specific FSP and site health and safety plan (SHSP)
- Maps for each sample area (SA) (see the FSP)
- Hand-held global positioning system (GPS) device
- Auger or coring device (e.g., stainless-steel hand auger or equivalent equipment)
- Spade (for high gravel and cobble content soils)
- Stainless-steel spoons or single-use disposable sampling scoops
- Tape measure or stainless-steel ruler
- Survey stakes or pin flags
- Camera and digital storage card
- Field logbook
- Small whiteboards, dry erase markers, and whiteboard eraser
- Indelible ink markers

- Laboratory-prepared sample jars
- Chain-of-custody (COC) forms and custody seals
- Field data sheets
- Sample labels
- Resealable plastic bags (one quart size)
- Stiff-bristle brush
- Paper towels
- Coolers
- Wet ice for samples designated for mercury analysis
- Canvas or plastic sheet on which to work with collected samples
- Disposable nitrile gloves for handling samples
- 5-gallon buckets
- Concentrated low-phosphate laboratory detergent (e.g., Liquinox or Alconox) and potable water
- Two-way radios.

Procedures for Discrete Soil Sample Collection

The steps below detail the discrete soil sample collection procedures for this sampling effort. Soil samples are to be co-located with the locations of individual plants where plant parts are collected for the study, and will generally be collected concurrently with the plant tissue sampling effort (see SOP-4).

1. Transport field personnel and sampling equipment to the planned plant tissue sampling location.
2. Determine the discrete soil sample location in consultation with EPA's onsite field personnel, and mark the location with a pin flag.
3. Ensure that a cultural resources monitor inspects and approves each discrete soil sample location prior to any soil disturbance. If the monitor's observations result in the sample location being shifted away from the selected location to avoid disturbance, the change will be recorded in the field logbook.
4. Move sampling equipment and personnel to the location, and measure and record the location coordinates (latitude and longitude) using a hand-held GPS.
5. In the field logbook, document the vegetation and any visual evidence of anthropogenic disturbance in the vicinity of the marked sample location (see SOP-5 for more information on documenting vegetation information). Take digital photographs of the sample location and record each in the photograph log (see SOP-6).

6. Clear large surface debris (e.g., woody debris, duff, vegetation, rocks) from the location. Retain residual organic matter overlying mineral soil.
7. Collect soil 0 to 3 in. below the ground surface using a decontaminated auger, coring, or spade tool. Soil samples should be collected as close as possible to the marked location. Use a stainless-steel ruler to measure the sample depth to verify that the sample is collected from the target interval.
    a. The volume of the collected soil sample will be visually estimated to exceed the volume of the laboratory-supplied sample jar, with a goal of collecting the minimum sample mass of 200 g required for laboratory preparation and analysis.
8. Manually or using a sieve, remove any sticks, twigs, rocks, and other bulky material from the sample.
9. Place the discrete soil sample into a quart-sized resealable plastic bag.
10. Allow the cultural resources monitor to inspect the sample.
    a. If the sample passes cultural resources review, continue with the next sample collection steps.
    b. If the sample does not pass cultural resources review, STOP SAMPLE COLLECTION, and identify an alternative discrete soil sample location in consultation with EPA oversight personnel.
        i. Return rejected sample to the sample location.
        ii. Remove residual soil material from the coring device using a stiff-bristle brush or clean paper towel.
        iii. Repeat steps 3 through 9.
11. Hand mix the soil sample inside the resealable plastic bag until the sample is uniform in size and texture.
12. Transfer the sample into laboratory-supplied sample jars using a clean stainless-steel spoon or single-use disposable sampling scoop, and label the jars in accordance with the procedures described in SOP-3. Return any residual material to the sample location.
13. Store each sample container in an individual sealable plastic bag that allows the sample label to be read. For samples designated for mercury analysis, store the bagged sample in a cooler with wet ice maintained at a temperature of 4°C ±2°C. Samples not designated for mercury analysis do not need to be stored on ice.
14. Complete field documentation for this sample in accordance with the procedures described in SOP-5.
15. Decontaminate sampling equipment between soil sample locations following the procedures described in Section 2.2.11 of the FSP and in SOP-8.

16. After collecting each soil sample, discard single-use sampling supplies such as nitrile gloves and resealable plastic bags for handling and disposal in accordance with Section 2.6 of the FSP.
17. Package and ship sample-filled coolers to the analytical laboratory along with all appropriate documentation in accordance with the procedures described in SOP-7.

STANDARD OPERATING PROCEDURE SOP-9B

COMPOSITE SOIL SAMPLE COLLECTION

This procedure describes the project-specific requirements to ensure the collection of accurate, representative soil samples within the plant tissue sampling areas identified in the associated Field Sampling Plan (FSP), but requires vigilant care and precision by each sampling team member. Composite soil samples will be co-located with and collected adjacent to corresponding composite plant tissue sampling locations.

Scope and Applicability

This standard operating procedure (SOP) is specific to the 2018 Plant Tissue Study being conducted for Teck American Incorporated (TAI) in the northern portion of the Upper Columbia River site in northeastern Washington. This SOP describes the procedures used for collecting composite soil samples during the Plant Tissue Study. This SOP applies only to those locations where mass limitations at an individual plant necessitate collection of a composite plant tissue sample. Correspondingly, the co-located soil sample will also be collected as a composite of co-located soil samples for each individual plant that is used to make up the composite plant tissue sample.

The following procedures may be modified in the field by the field supervisor (in consultation with TAI and the U.S. Environmental Protection Agency [EPA]) based on conditions encountered in the field. Such changes should be noted in the field logbook and on field change forms along with the names of the TAI and EPA representatives authorizing the change.

Equipment and Materials

The following is a list of equipment and materials needed by the sampling team:

- Project-specific FSP and site health and safety plan (SHSP)
- Maps for each SA (see the FSP)
- Hand-held global positioning system (GPS) device
- Auger or coring device (e.g., stainless-steel hand auger or equivalent equipment)
- Spade (for high gravel-and-cobble content soils)
- Stainless-steel mixing bowl
- Stainless-steel spoons or single-use disposable sampling scoops
- Tape measure or stainless-steel ruler
- Survey stakes or pin flags

- Camera and digital storage card
- Field logbook
- Small whiteboards, dry erase markers, and whiteboard eraser
- Indelible ink markers
- Laboratory-prepared sample jars
- Glass or stainless-steel 2-cup measure
- Chain-of-custody records and custody seals
- Field data sheets
- Sample labels
- Resealable plastic bags
- Stiff-bristle brush
- Paper towels
- Coolers
- Wet ice for samples designated for mercury analysis
- Canvas or plastic sheet on which to work with collected samples
- Disposable nitrile gloves for handling samples
- 5-gallon buckets
- Soft-bristle brush
- Concentrated low-phosphate laboratory detergent (e.g., Liquinox or Alconox) and potable water
- Two-way radios.

Procedures for Composite Soil Sample Collection

The steps below detail the composite soil sample collection procedures for this sampling effort.

1. Transport field personnel and sampling equipment to the planned plant tissue sampling location.
2. Determine the component locations where soil will be collected for the composite soil sample in consultation with EPA oversight personnel and mark those locations with pin flags.
3. Convey sampling equipment and personnel to those locations and record coordinates for each component location using a hand-held GPS.

4. Ensure that a cultural resources monitor inspects and approves each component location selected for the composite sample prior to any soil disturbance.
5. Document the vegetation and any anthropogenic changes in the vicinity of the component sampling locations in the field notebook. Take digital photographs of the locations (record in the photograph log).
6. Clear large surface debris (e.g., woody debris, duff, vegetation, rocks) from the component locations. Retain any organic matter overlying mineral soil.
7. Collect soil to a depth of 0 to 3 in. below the ground surface using a decontaminated auger, coring, or spade tool . A stainless-steel ruler will be used to ensure that the sample is collected from the correct depth interval.
    a. All component soil samples should be collected as close as possible to the selected location in consultation with EPA oversight personnel.
    b. The target minimum sample mass to be collected for any composite soil sample is 200 g, or the volume of the laboratory-supplied sample jar.
8. Remove manually sticks, twigs, rocks, and other large material from the sample.
9. Place each component sample that comprises the composite into individual quart-sized resealable plastic bag.
10. Remove residual soil material from the coring device between the component sample locations using a stiff-bristle brush or paper towel.
11. Allow the cultural resources monitor to inspect each bagged sample.
    a. If an individual bagged component sample passes cultural resources review continue sample collection.
    b. If an individual bagged component sample does not pass cultural resources review, STOP SAMPLE COLLECTION, and set this material aside and determine an alternative component soil sample location in consultation with EPA oversight personnel.
        i. Return rejected sample to the sample location.
        ii. Repeat steps 3 through 10.
12. Hand mix each individual component of the sample within its resealable plastic bag using a decontaminated stainless-steel spoon or a single-use disposable sample scoop until the individual soil sample is uniform in size and texture.
13. Estimate the proportions of each plant that makes up the associated composite plant material sample (e.g., two-thirds plant A, one-third plant B).
14. Transfer soil from individual component samples into a stainless-steel bowl in equivalent proportions to those from the associated plants (continuing example from above: two-thirds soil associated with plant A, one-third soil associated with plant B).
15. Hand mix the component soil sample portions in the stainless-steel bowl using a decontaminated stainless-steel spoon or single-use disposable sample scoop until the composite soil sample is uniform in size and texture.

16. Transfer the sample into laboratory-provided sample jars using a clean stainless-steel spoon or single-use disposal sampling scoop and label the jars according to procedures described in SOP-3. Return any residual material left in sample bags to the sample locations and return unused mixed soil to the general area under any of the sampled plants.
17. Store each sample container in an individual sealable plastic bag that allows the sample label to be read.
18. For samples designated for mercury analysis, store the bagged sample in a cooler with wet ice maintained at a temperature of 4°C ±2°C. Samples not designated for mercury analysis do not need to be stored on ice.
19. Complete field documentation for this composite soil sample according to procedures described in SOP-5.
20. Fully decontaminate sampling equipment between composite soil sampling locations as described in Section 2.2.11 of the FSP and SOP-8.
21. Discard dedicated sampling equipment such as nitrile gloves, quart-sized resealable bags, and single-use sample scoops.
22. Package and ship sample-filled coolers to the analytical laboratory along with all appropriate documentation in accordance with the procedures described in SOP-7.

STANDARD OPERATING PROCEDURE SOP-9C

SEDIMENT SAMPLE COLLECTION

This procedure describes the project-specific requirements to ensure the collection of accurate, representative sediment samples within the plant tissue sampling areas identified in the associated Field Sampling Plan (FSP). Sediment samples will be co-located with and collected adjacent to corresponding discrete plant tissue sampling locations.

Scope and Applicability

This standard operating procedure (SOP) is specific to the 2018 Plant Tissue Study being conducted for Teck American Incorporated (TAI) in the northern portion of the Upper Columbia River site in northeastern Washington. This SOP describes the procedures used for collecting sediment samples during the Plant Tissue Study. This SOP applies only to sampling areas in which the plants sampled are growing in beach sediment along the Columbia River shoreline, and to other wetland areas where tules are collected.

The following procedures may be modified in the field by the field supervisor (in consultation with TAI and the U.S. Environmental Protection Agency [EPA]) based on conditions encountered in the field. Such changes should be noted in the field logbook and on field change forms along with the names of the TAI and EPA representatives authorizing the change.

Equipment and Materials

The following is a list of equipment and materials needed by the sampling team:

- Project-specific FSP and site health and safety plan (SHSP)
- Maps for each SA (see the FSP)
- Hip or chest waders
- Hand-held global positioning system (GPS) device
- Auger or coring device (e.g., stainless-steel hand auger or equivalent equipment)
- Spade (for high gravel-and-cobble content soils)
- Portable grab sampler (Ponar or equivalent) may be required depending on water levels
- Peristaltic pump, portable battery, and associated tubing may be required depending on water levels (used to extract overlying water from sample)
- Stainless-steel mixing bowl

- Stainless-steel spoons or single-use disposable sampling scoops
- Tape measure or stainless-steel ruler
- Survey stakes or pin flags
- Camera and digital storage card
- Field logbook
- Small whiteboards, dry erase markers, and whiteboard eraser
- Indelible ink markers
- Laboratory-supplied sample jars
- Chain-of-custody records and custody seals
- Field data sheets
- Sample labels
- Resealable plastic bags
- Stiff-bristle brush
- Paper towels
- Coolers
- Wet ice for samples designated for mercury analysis
- Canvas or plastic sheet on which to work with collected samples
- Disposable nitrile gloves for handling samples
- 5-gallon buckets
- Soft-bristle brush
- Concentrated low-phosphate laboratory detergent (e.g., Liquinox or Alconox) and potable water
- Two-way radios.

Procedures for Sediment Sample Collection

The steps below detail the sediment sample collection procedures for this sampling effort.

1. Transport field personnel and sampling equipment to the planned plant tissue sampling location.
2. Determine the sediment sample location in consultation with EPA oversight personnel and mark the location with a survey stake.
3. Convey sampling equipment and personnel to the location, and record coordinates for the location using a hand-held GPS.

4. Allow the cultural resources monitor to inspect and approve each sediment sample location prior to any substrate disturbance.
5. Document the vegetation and any anthropogenic changes in the vicinity of the location in the field notebook. Take digital photographs of the location (record in the photograph log).
6. Clear large surface debris (e.g., woody debris, duff, vegetation, rocks) from the location. Retain any organic matter overlying mineral sediment. If overlying water is present, determine appropriate sampling technique based on depth of water. If less than 1 to 2 feet, use a 55-gallon bucket with the bottom removed to isolate sample area and remove overlying water with the peristaltic pump. If water depth is greater than 2 feet, plan to use a grab sampler or auger.
7. Collect sediment to a depth of 0 to 3 in. below the ground surface using a decontaminated auger, coring tool, spade tool, or grab sampler. A stainless-steel ruler will be used to ensure that the sample is collected from the correct depth interval.
    a. All sediment samples should be collected as close as possible to the selected location in consultation with EPA oversight personnel.
    b. The target minimum sample mass to be collected for any sediment sample is 200 g, or the volume of the laboratory-supplied sample jar.
8. Remove manually sticks, twigs, rocks, and other large material from the sample.
9. Place the sediment sample into a decontaminated stainless-steel mixing bowl.
10. Allow the cultural resources monitor to inspect the sample.
    a. If the sample passes cultural resources review, continue sample collection.
    b. If the sample does not pass cultural resources review, STOP SAMPLE COLLECTION, and determine an alternative sediment sample location in consultation with EPA oversight personnel.
        i. Return rejected sample to the sample location.
        ii. Rinse the coring device and stainless-steel bowl with site water.
        iii. Repeat steps 3 through 10.
11. Hand mix the sample inside the stainless-steel bowl until the sediment sample is uniform in size and texture.
12. Transfer the sample into laboratory-provided sample jars using a clean stainless-steel spoon or single-use disposal sampling scoop, and label the jars according to procedures described in SOP-3. Return any residual material to the sample location.
13. Store each sample container in an individual sealable plastic bag that allows the sample label to be read.

14. For samples designated for mercury analysis, store the bagged sample in a cooler with wet ice maintained at a temperature of 4°C ±2°C. Samples not designated for mercury analysis do not need to be stored on ice.
15. Complete field documentation for this sediment sample according to procedures described in SOP-5.
16. Fully decontaminate sampling equipment between sediment sampling locations as described in Section 2.2.11 of the FSP and SOP-8.
17. Discard dedicated sampling equipment such as nitrile gloves, quart-sized resealable bags, and single-use sample scoops.
18. Package and ship sample-filled coolers to the analytical laboratory along with all appropriate documentation according to procedures described in SOP-7.

STANDARD OPERATING PROCEDURE SOP-10

HANDLING AND REPORTING OF CULTURAL RESOURCES

Scope and Applicability

This standard operating procedure (SOP) is specific to the 2018 Plant Tissue Study (hereinafter "the study") being conducted for Teck American Incorporated (TAI) in the northern portion of the Upper Columbia River site in northeastern Washington. This SOP describes the procedures to be followed by all TAI field personnel, including subcontractors, should potential discoveries, including inadvertent discoveries of cultural materials and deposits, and/or Indian burials and human remains occur during execution of the plant tissue, soil, or sediment sampling efforts. Cultural materials and deposits (including sacred objects, funerary objects, and objects of cultural patrimony) as well as Indian burials and human remains are defined in the Native American Graves Protection and Repatriation Act (NAGPRA).

The procedures detailed below were developed to ensure compliance with the National Historic Preservation Act (NHPA) and the applicable requirements, procedures, and standards of the National Park Service (NPS), U.S. Bureau of Reclamation (USBR), Confederated Tribes of the Colville Reservation (CCT), and the Spokane Tribe of Indians (STI). Detailed information regarding existing discovery protocols for these entities, as well as implementing regulations, notification requirements, archaeological monitoring requirements, and other cultural resource coordination activities for the Upper Columbia River remedial investigation and feasibility study (RI/FS) are provided in the cultural resources coordination plan (CRCP) in Appendix C of the QAPP.

Discoveries When a Cultural Resources Monitor is Present

At the discretion of the cultural resources monitor or tribal representative, ground-disturbing sampling or associated activity may be slowed or halted at any time that a suspected archaeological object or archaeological resource is encountered. The objective of slowing or halting ground-disturbing activity is to allow the cultural resources monitor or tribal representative to confirm and/or make a preliminary assessment of the discovery. At the discretion of the cultural resources monitor or tribal representative, the discovery and the material in which it is contained may be returned to a location distinct from, but nearby, the original location of discovery. Any such relocation will be coordinated with the field supervisor.

At the request of the cultural resources monitor or tribal representative, the sampling personnel will either:

- Assist in securing access to the location of the discovery and take appropriate measures to protect the location of the discovery from rainfall, stormwater, and other possible disturbances; or
- Assist in moving the found artifacts to a protected and secure area away from the immediate sampling area. Removal of artifacts from the discovery location will be undertaken only if leaving the artifacts in place could jeopardize their integrity due to erosion or collection by unauthorized individuals.

The cultural resources monitor, tribal representative, or a member of the TAI field team will remain on site to ensure the security of the find until more extensive efforts can be made to secure the site from further disturbance, or until a more extensive evaluation and documentation of the discovery can be made.

Notification of any cultural resources that have the potential to delay or halt sampling activities (i.e., human remains or the items covered under NAGPRA) must be provided as soon as possible to the U.S. Environmental Protection Agency (EPA) for further coordination with the consulting parties.

Discovery of Human Remains

Native peoples in the study area consider the graves of their ancestors to be important in both their cultural identity and in defining their relationship with the land. These graves are therefore considered sacred and should be left undisturbed. Should inadvertent disturbance occur, the remains and associated materials (funerary objects) must be treated with respect and honor. All appropriate federal, tribal, and state laws, regulations, and procedures regarding burials should be rigorously enforced.

In the event that likely or confirmed human remains are encountered, all further sampling or other ground-disturbing activity must cease immediately. The protocol and notification procedures to be followed for any potential discoveries of human remains are provided in protocols of the NPS, USBR, CCT, and STI (Attachment C1 to the CRCP). Any discoveries within the boundaries of the Colville Indian Reservation or the Spokane Indian Reservation must also be reported immediately to the respective tribe.

The TAI field team will assist the cultural resources monitor and tribal representative in securing the location of the discovery.

Other conditions for responses to discoveries of archaeological materials may be defined in the Archeological Resources Protection Act permit issued for the sampling program. As detailed in the CRCP, responses to any discoveries of burials must also comply with provisions of NAGPRA and its implementing regulations, as well as the existing protocols of the NPS, USBR, CCT, and STI (Attachment C1 to the CRCP).

Discoveries When a Cultural Resources Monitor is not Present

As previously stated, a cultural resources monitor and/or tribal representative will be present during all sampling activities. In the event, however, that suspected or evident artifacts or other archaeological deposits are encountered when a cultural resources monitor or tribal representative is not present, the immediate vicinity of the discovery will be secured. The discovery will be mapped and photographed in place, but the discovery will be otherwise left as found (other than appropriate measures to secure the find and maintain this security).

In consultation with the land-managing agency or appropriate tribe, as well as other interested parties, TAI will arrange for the location of the discovery to be examined by a professional archaeologist and tribal representative in a timely manner. If the archaeologist confirms the presence of artifacts or other archaeological deposits, the procedures defined above for discoveries made during ground-disturbing activity monitored by an archaeologist will be implemented. The archaeologist will prepare appropriate State of Washington archaeological forms to document the find.

To ensure proper recognition of artifacts and other cultural items or deposits, all TAI field personnel will be trained by a professional archaeologist to recognize these materials prior to the initiation of any soil or sediment sampling.

Confidentiality

In accordance with state and federal law, all field personnel are required to keep the discovery of any found or suspected human remains, other cultural items, and potential historic properties confidential. Personnel are prohibited from contacting the media or any third party or otherwise sharing information regarding the discovery with any member of the public, and that they should immediately notify the field supervisor of any inquiry from the media or public. The field supervisor will then notify TAI of any such inquiries. To the extent permitted by law, prior to any release of information, TAI in coordination with EPA and other consulting parties shall concur on the amount of information, if any, to be released to the public, any third party, and the media, and the procedures for such a release.

STANDARD OPERATING PROCEDURE SOP-11

SAMPLE CUSTODY

Scope and Applicability

This standard operating procedure (SOP) is specific to the 2018 Plant Tissue Study (hereinafter "the study") being conducted for Teck American Incorporated (TAI) in the northern portion of the Upper Columbia River site in northeastern Washington. This SOP describes procedures for maintaining custody of environmental samples collected during all plant tissue, soil, and sediment sampling activities conducted by TAI field personnel for the study. The procedure outlined herein will be used in conjunction with SOP-3, which covers sample labeling; SOP-5, which covers field documentation; and SOP-7, which covers sample packaging and shipping.

Chain-of-custody (COC) forms (Attachment A3 of the field sampling plan [FSP]) ensure that samples are traceable from the time of collection through processing and analysis until final disposition. A sample is considered to be in a person's custody if any of the following criteria are met:

1. The sample is in the person's possession.
2. The sample is in the person's view after being in possession.
3. The sample is in the person's possession and is being transferred to a designated secure area.
4. The sample has been locked up to prevent tampering after it was in the person's possession.

It is unacceptable for samples to be outside of designated personnel's custody at any time unless the samples have been transferred to a secure area (i.e., locked up and custody sealed) or transferred to the laboratory. If the samples cannot be placed in a secure area, then a field team member must physically remain with the samples at all times (e.g., at meal times).

Materials and Methods

The following materials are required:

- COC forms (if COCs will be produced in an electronic format using a database program [e.g., FORMS II Lite], a computer and printer also need to be available)
- Custody seals
- Shipping airbills (if samples will be sent by air).

Chain-of-Custody Forms

The COC form is a critical document that records sample possession from the time of collection through the final disposition of the sample. The form also provides information to the laboratory regarding the analyses to be performed on the samples received by the laboratory. Therefore, COCs must include information only on the samples within the shipping container sent to the subject laboratory, and samples shall not be shipped without an associated and properly completed COC inside the container.

The COC form will be completed after each field collection activity and before the samples are shipped to the laboratory. Project-assigned sample identification (ID) numbers will be recorded on the COC form (see SOP-3). The COC form will also identify the sample collection date and time, the type of sample (e.g., plant tissue, soil, sediment), the project name, the sampling personnel, and the total number of coolers included in the accompanying shipment. The completed COC form, or multiple forms, will be placed into a plastic resealable bag and secured with tape to the inside of the lid of each sample-filled cooler. A copy of the COC forms will be retained by the field supervisor and provided to TAI for filing at the completion of the study.

Sampling personnel are responsible for the care and custody of the samples until they are shipped. When transferring possession of the samples, the individuals relinquishing and receiving the samples must sign the COC form and record the time and date that the transfer occurs where indicated on the COC form.

Procedures

The following guidelines will be followed to ensure the integrity of the samples:

1. Prior to sample shipping or storage, COC entries will be made for all samples electronically on a secure computer or hard copy. Information on the COCs will be checked against field logbook entries to verify the accuracy of the records.
2. At the bottom of each COC form is a space for the signatures of the persons relinquishing and receiving the samples, and to note the time and date that the transfer occurred. The time that the samples were relinquished should exactly match the time they were received by another party. Under no circumstances should there be any time when custody of the samples is undocumented.
3. The COC form should not be signed until the information has been checked for inaccuracies by the field supervisor. All changes should be made by drawing a single line through the incorrect entry, and initialing and dating the revision. Revised entries should be made in the space below the entries. Any blank lines remaining on the COC form after corrections are made should be marked out with single lines that are initialed and dated. This procedure will preclude any unauthorized additions.

4. If samples are sent by a commercial carrier not affiliated with the laboratory, such as Federal Express (FedEx) or United Parcel Service (UPS), the name of the carrier should be recorded on the COC form. Any tracking numbers supplied by the carrier should be also entered on the COC form. The time of transfer should be as close to the actual drop-off time as possible. After the COC forms are signed, they should be sealed inside the transfer container. A signed copy will be retained by the field supervisor.
5. If errors are found after the shipment has left the custody of sampling personnel, a corrected version of the forms must be made and sent to all relevant parties. Minor errors can be rectified by making the change on a copy of the original with a brief explanation and signature. Errors in the signature block may require a letter of explanation.
6. Upon completion of the field sampling event, the field supervisor will be responsible for submitting all project-related COC forms to TAI.

Custody Seal

To prevent unauthorized handling of the samples during shipping, two custody seals will be affixed to the outside of each sample cooler. Custody seals will be placed across the front and across one side of the cooler lid prior to shipping. Field personnel will ensure that the seals are securely affixed to the cooler so that they cannot be accidently removed during shipping. Additional tape may be placed across the seal to secure it in place.

Shipping Airbills

When samples are shipped from the field to the testing laboratory via a commercial carrier (e.g., FedEx, UPS), an airbill or receipt is provided by the shipper. Upon completion of the field sampling event, the field supervisor will be responsible for submitting the sender's copy of all shipping airbills to TAI. The airbill number (or tracking number) should be noted on the applicable COC forms before they are sealed inside the cooler.

Acknowledgement of Sample Receipt

In most cases, the laboratory will confirm the sample receipt with the analytical chemistry laboratory coordinator on the day samples are received by the testing laboratory. This confirmation may be via e-mail or an official laboratory 'Acknowledgment of Sample Receipt' form that confirms the sample ID numbers and analysis to be performed. If an error is detected by the TAI laboratory coordinator, the laboratory will be immediately contacted. Decisions made during any telephone conversation should be documented in writing and archived in the project file by the task manager. If necessary, corrections should be made to the COC form and the corrected version of the COC form should be sent to the laboratory (either via e-mail or facsimile) by the TAI laboratory coordinator.

ATTACHMENT A3

EXAMPLES OF VARIOUS FIELD FORMS

CHANGE REQUEST FORM
Upper Columbia River Plant Tissue Study, 2018

Page: _____ of _____ Change No: _____

CHANGE REQUEST

Applicable Reference:

Description of Change:

Reason for Change:

Impact on Present and Completed Work:

Requested By: _____ Date: _____
(AECOM Project Manager)

Acknowledged By: _____ Date: _____
(Teck Project Manager)

APPROVAL

Senior Technical Advisor _____ Date: _____

Teck Project Manager: _____ Date: _____

EPA Project Manager: _____ Date: _____

PROTOCOL MODIFCATION FORM
Upper Columbia River Plant Tissue Study, 2018

Page: _____ of _____ Field Modification No: _____

Material to be Sampled:

Standard Procedure for Field Collection and Laboratory Analysis (cite reference):

Reason for Change in Field Procedure or Analysis Variation:

Variation from Field or Analytical Procedure:

Special Equipment, Materials or Personnel Required:

APPROVAL

Initiated by _____ Date: _____

Project Manager: _____ Date: _____

QA Project Manager: _____ Date: _____

LOCATION COLLECTION FORM

Project Name: Upper Columbia River Plant Tissue Study, 2018 ***Field Crew Initials:***

Comments:

Collection Date	Collection Time	Sampling Area	Location ID	Collection Method	GPS Unit Used to Record Sampling Location	Photo ID	Comments

Notes:

FIELD RECONAISSANCE FORM

Project Name: Upper Columbia River Plant Tissue Study, 2018

Field Crew Initials: ***Parcel Idenfier:***

Date:

Time Begin/End:

Sampling Area	Species Common Name	*Scientific Name*	Plant Part	Species Present on Parcel	Specific Plant Part Observed	Species bundance across site [low=1-2, med=3-5, hi=6-10]	Not present, but site has correct habitat	Comments, Including Health	Spatial Data Collected	Photos
	Camas	*Camassia quamash*	Bulbs							
	Kinnikinnick	*Arctostaphylos uva-ursi*	Leaves							
	Bitterroot	*Lewisia rediviva*	Root embryo							
	Chokecherry	*Prunus virginiana*	fruit							
	Green Willow (coyote willow)	*Salix exigua*	leaves							
			stem, bark, inner cambium							
	Hazelnut	*Corylus cornuta*	nut							
	Huckleberry	*Vaccinium ovatum or V. membranaceum*	fruit							
	Indian Potato	*Orogenia fusiformis, Lomatium geyeri*	root							
	Morels	*Morchella esculenta*	fruiting body							
	Puffballs	*Lycoperdon peralatum*	fruiting body							
	Red Willow	*Cornus sericea*	leaves							
			stem, bark, inner cambium							
	Shaggy Manes	*Coprinus comatus*	fruiting body							

Sampling Area	Species Common Name	*Scientific Name*	Plant Part	Species Present on Parcel	Specific Plant Part Observed	Species bundance across site [low=1-2, med=3-5, hi=6-10]	Not present, but site has correct habitat	Comments, Including Health	Spatial Data Collected	Photos
	Spring Beauty	*Claytonia lanceolata, C. cordifolia*	tuber							
	Tule	*Schoenoplectus acutus*	stalk							
	Lomatiums	*Lomatium* spp.	Roots							
	Wild Rose	*Rosa nutkana*	rose hip							
			stem, root							
	Wild mint	*Mentha arvensis*	leaves							
	Black tree lichen	*Bryoria fremontii*	Lichen							
	Wild Caraway/Indian Carrot[b]	*Perideridia gairdneri*	Roots							
	Sarvisberry	*Amelanchier* spp.	Berries							
	Ponderosa Pine	*Pinus ponderosa*	Nuts							
	Wild Strawberry	*Fragaria virginiana, F. vesca*	fruit							
ADDITIONAL SPECIES (Write In)										

SAMPLE COLLECTION FORM

Project Name: Upper Columbia River Plant Tissue Study, 2018

Comments: ***Field crew initials:***

Date:

Sampling Area:

Collection Time	Location ID	Sample ID	Collection Method	Taxonomic Identification	Sample Weight (g)	Photo ID	Composite (Y/N)	Composite ID	Sample IDs of other samples sharing patch

SOIL/SEDIMENT COLLECTION FORM

Project Name: Upper Columbia River Plant Tissue Study, 2018 ***Field crew initials:***

Comments:

Date:

Sampling Area:

Collection Time	Location ID	Sample ID	Co-Located Plant Species	Sample Weight (G)	Photo ID	Composite (Y/N)	Composite ID

PHOTO LOG

Project Name: Upper Columbia River Plant Tissue Study, 2018

Comments: ***Field crew initials:***

Collection Date	Collection Time	Location ID	Sample ID	Photo ID	Photo Direction	Photo Notes	Comments

CHAIN-OF-CUSTODY RECORD

SR # / LAB USE ONLY

LABORATORY CLIENT:			CLIENT PROJECT NAME / NUMBER:		P.O. NO.:
ADDRESS:			PROJECT CONTACT:		Bill To.:
TEL:	Cell:	E-MAIL:			
AECOM CONTACT:			SHIPPING CARRIER & TRACKING NUMBER		
ADDRESS:			REQUESTED ANALYSES		TEMPERATURE UPON RECEIPT: - °C
TEL:	Fax:	E-MAIL:			
TURNAROUND TIME: SAME DAY 24 HR 48 HR 72 HR Standard					SAMPLER(S): (PRINT and SIGNATURE)
SPECIAL INSTRUCTIONS:					

	CLIENT SAMPLE ID	ALS LAB ID (Lab Use Only)	SAMPLING DATE	SAMPLING TIME	MATRIX* TYPE	NO. OF CONTAINERS	Comments
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							

Additional Comments:

REPORT REQUIREMENTS:
I. Routine Report: Method Blank, Surrogate, as required
II. Report Dup., MS, MSD as required
III. CLP Like Summary (no raw data)
X IV. Data Validation Report
X V. EDD

Relinquished by: (Print/Signature/Affiliation)	Date	Time	Received by: (Print/Signature/Affiliation)	Date	Time
Relinquished by: (Print/Signature/Affiliation)	Date	Time	Received by: (Print/Signature/Affiliation)	Date	Time
Relinquished by: (Print/Signature/Affiliation)	Date	Time	Received by: (Print/Signature/Affiliation)	Date	Time

EQUIPMENT CALIBRATION LOG

Project Name: Upper Columbia River Plant Tissue Study, 2018

Project No.:

Field Representative:

Circle or Write in Type/ Equipment Model	Serial No.	Calibrated By	Date	Time	Parameter Calibrated and Calibration Standard	Calibration/ check Pass Y/N?
scale					zeroing 0 grams of weight _____ initial reading: _____ adjustment: _____ Final reading: check 1 gram of weight: _____ 50 grams of weight: _____ 100 grams of weight: _____	
freezer					condition of samples (frozen/thawed)? _____ freezer setting? _____ freezer temperature? _____ Adjustment made to temperature sertting?	
refrigerator					condition of samples (frozen/thawed)? _____ refrigerator setting? _____ refrigerator temperature? _____ Adjustment made to temperature sertting?	
other equipment					zeroing units _____ initial reading: _____ adjustment: _____ Final reading: _____ check	

SHIPMENT TRACKING FORM

Project Name: Upper Columbia River Plant Tissue Study, 2018

Comments: ***Field crew initials:***

Decision Unit (s)	Location ID/ Sample ID	Sample Collection Date	Date Shipped	Tracking Number (s)	Date Received at Lab	Comments

SAMPLE TRACKING FORM

Project Name: Upper Columbia River Plant Tissue Study, 2018

Comments: ***Field crew initials:***

Collection Date	Collection Time	Location ID	Sample ID	Risk Assessment	Analyses Required	Comments

Cultural Resource Daily Monitoring Form
Upper Columbia River Remedial Investigation and Feasibility Study (UCR RI/FS)
2018 Plant Tissue Study

Monitor Name(s): _____ Monitor Organization:_____

Date of Sampling: _____ AECOM Sampling Team Representative: _____

Sampling Area (s) (e.g., "*Sampling Area 1*")	**Location of Cultural Materials**	**Observations of Cultural Materials**	**Notes**
	UTM for Cultural Materials: _____ *mE* _____ *mN* Landowner(s):	☐ No ☐ Yes* **If "yes" please fill out the following:* Describe what actions were taken on reverse. ☐ Done Was a site/isolate form completed (please justify if "no")? ☐ No ☐ Yes Temp #_____ Is this possibly associated with a previously documented site? ☐ No ☐ Yes Site #_____ ☐ Not Sure Confirm Photographs collected? ☐ Yes Camera/Digital File#_____ Confirm UTM data collected? ☐ Yes Confirm photographs, site/iso form, etc. have been transmitted to AECOM for use in monitoring report (please explain if "no"): ☐ No ☐ Yes _____	

Cultural Resource Daily Monitoring Form
Upper Columbia River Remedial Investigation and Feasibility Study (UCR RI/FS)
2018 Plant Tissue Study

Additional Notes: _____

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

Per the Cultural Resource Coordination Plan (CRCP):

- At the discretion of the monitor, sampling may be slowed or halted at any time a resource is suspected or encountered; the location of the sample probe shall be moved as necessary to avoid cultural or archaeological resources.
- Documentation and recordation of daily observations, including field notes and photographs, will be completed.
- If archaeological resources are found, sampling will cease and the discovery will be documented and redeposited at the location of discovery. EPA shall be notified within 24 hours of an archaeological discovery via telephone and email (Monica Tonel: 206-553-0323; monica.tonel@epa.gov).
- If human remains and/or funerary objects are encountered, all work will cease at the sampling location, the remains will be protected, and notification procedures outlined in the CRCP will be followed immediately.

CONTACT INFORMATION:

Sarah McDaniel, Principal Investigator Phone: (360) 624-4285 Email: sarah.mcdaniel@aecom.com
Mike S. Kelly, Alternate Phone: (503) 475-2426 Email: mike.s.kelly@aecom.com
AECOM, 111 SW Columbia St., Portland, OR 97201

ATTACHMENT A4

ARCHAEOLOGICAL MONITORING PROTOCOL

ARCHAEOLOGICAL MONITORING PROTOCOL

UPPER COLUMBIA RIVER RI/FS

Prepared for

Teck American Incorporated
P.O. Box 3087
Spokane, WA 99220-3087

Prepared by

RAMBOLL

901 Fifth Avenue, Suite 2820
Seattle, WA 98164

April 2018

CONTENTS

ACRONYMS AND ABBREVIATIONS

CCT	Confederated Tribes of the Colville Reservation
CRCP	cultural resources coordination plan
EPA	U.S. Environmental Protection Agency
NAGPRA	Native American Graves Protection and Repatriation Act
NHPA	National Historic Preservation Act
NPS	National Park Service
RI/FS	remedial investigation and feasibility study
STI	Spokane Tribe of Indians
TAI	Teck American Incorporated
UCR	Upper Columbia River
USBR	U.S. Bureau of Reclamation

INTRODUCTION

This protocol provides a summary of procedures to be followed by all Teck American Incorporated (TAI) technical team field personnel, including subcontractors, should potential discoveries, of cultural materials and deposits, and/or Indian burials and human remains occur during execution of field sampling programs and other activities associated with the Upper Columbia River (UCR) Site remedial investigation and feasibility study (RI/FS). Cultural materials and deposits (including sacred objects, funerary objects, and objects of cultural patrimony) as well as Indian burials and human remains are defined in the Native American Graves Protection and Repatriation Act (NAGPRA).

The procedures detailed below were developed to ensure compliance with the National Historic Preservation Act (NHPA) and the applicable requirements, procedures, and standards of the National Park Service (NPS), U.S. Bureau of Reclamation (USBR), Confederated Tribes of the Colville Reservation (CCT), and the Spokane Tribe of Indians (STI). Detailed information regarding existing discovery protocols for these entities, as well as implementing regulations, notification requirements, archaeological monitoring requirements, and other cultural resource coordination activities for the RI/FS are provided in the cultural resources coordination plan (CRCP) in Appendix C of the QAPP.

DISCOVERIES WHEN AN ARCHAEOLOGICAL MONITOR IS PRESENT

At the discretion of the archaeological monitor or Tribal representative, ground-disturbing sampling or associated activity may be slowed or halted at any time that a suspected archaeological object or archaeological resource is encountered. The objective of this slowing or halting of ground-disturbing cleanup activity is to allow the archaeological monitor or tribal representative to confirm and/or make a preliminary assessment of the discovery. At the discretion of the archaeological monitor or tribal representative, a specific sample may be relocated from the location of the discovery but still be within the sampling location. Such relocation will be coordinated with the field supervisor.

At the request of the archaeological monitor or tribal representative, the sampling personnel will either:

- Assist in securing access to the location of the discovery and take appropriate measures to protect the location of the discovery from rain, stormwater, and other possible disturbances, or
- Assist in moving the artifacts to a protected and secure area of the site away from the immediate sampling area. Removal of artifacts from the discovery location will be undertaken only if leaving the artifacts in place would jeopardize their integrity due to erosion or collection by unauthorized individuals.

The archaeological monitor, tribal representative, or a member of the TAI technical team will remain on site to ensure the security of the find until more extensive efforts can be made to secure the site discovery from further disturbance or a more extensive evaluation and documentation of the discovery can be made.

Notification of any cultural resources that have the potential to delay or halt sampling activities (i.e., human remains or those items covered under NAGPRA) must be provided as soon as possible to the U.S. Environmental Protection Agency (EPA) for further coordination with the consulting parties.

DISCOVERY OF HUMAN REMAINS

Native peoples in the study area consider the graves of their ancestors to be important in both their cultural identity and in defining their relationship with the land. These graves are therefore considered sacred and should be left undisturbed. Should inadvertent disturbance occur, the remains and associated materials (funerary objects) must be treated with respect and honor. All appropriate federal, tribal, and state laws, regulations, and procedures regarding burials should be rigorously enforced.

In the event that likely or confirmed human remains are encountered, all further sampling or other ground-disturbing activity will cease immediately. The protocol and notification procedures to be followed for any potential discoveries of human remains are provided in protocols of the NPS, USBR, CCT, and STI (Attachment C1 to the CRCP). Any discoveries within the boundaries of the Colville or the Spokane reservations must be reported immediately to the respective tribe.

The TAI technical team will assist the archaeological monitor and tribal representative in securing the location of the discovery.

Other conditions for responses to discoveries of archaeological materials may be defined in the Archeological Resources Protection Act permit issued for the sampling program. As detailed in the CRCP, responses to any discoveries of burials must also comply with provisions of NAGPRA and its implementing regulations, as well as the existing protocols of the NPS, USBR, CCT, and STI (Attachment C1 to the CRCP).

DISCOVERIES WHEN AN ARCHAEOLOGICAL MONITOR IS NOT PRESENT

As previously stated, an archaeological monitor and/or tribal representative will be present during all sampling activities. In the event, however, that suspected or evident artifacts or other archaeological deposits are encountered when an archaeological monitor or tribal representative is not present, the immediate vicinity of the discovery will be secured. The discovery will be mapped and photographed in place but will be otherwise left as found (other than appropriate measures to secure the find and maintain security). In consultation

with the land-managing agency or appropriate tribe, as well as other interested parties, TAI will arrange for the location of the discovery to be examined by a professional archaeologist and/or tribal representative in a timely manner. If the archaeologist confirms the presence of artifacts or other archaeological deposits, the procedures defined above for discoveries made during ground-disturbing activity monitored by an archaeologist will be implemented. The archaeologist will prepare appropriate State of Washington archaeological forms to document the find.

To ensure proper recognition of artifacts and other cultural items or deposits, all TAI field personnel will be provided with training in recognizing these materials by a professional archaeologist prior to the initiation of any plant tissue sampling.

CONFIDENTIALITY

In accordance with state and federal law, all field personnel are required to keep the discovery of any found or suspected human remains, other cultural items, and potential historic properties confidential. Personnel are instructed that they are prohibited from contacting the media or any third party or otherwise sharing information regarding the discovery with any member of the public, and that they should immediately notify the field supervisor of any inquiry from the media or public. The field supervisor will then notify TAI of any such inquiries. To the extent permitted by law prior to any release of information, TAI, in coordination with EPA and other consulting parties, shall concur on the amount of information, if any, to be released to the public, any third party, and the media and the procedures for such a release.

APPENDIX B

ALS QUALITY ASSURANCE MANUAL, SOPS, SAMPLE BENCHSHEET, AND SRMS

ALS Environmental

QUALITY ASSURANCE MANUAL

ALS Environmental – Kelso Facility
1317 South 13th Avenue
Kelso, WA 98626
360-577-7222
360-636-1068
www.alsglobal.com

QUALITY ASSURANCE MANUAL

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Approved By: _____ Date: 10/28/16
Laboratory Director – Jeff Grindstaff

Approved By: _____ Date: 10/28/16
QA Manager – Carl Degner

Approved By: _____ Date: 10/28/16
Technical Director, Metals – Jeff Coronado

Approved By: _____ Date: 10/28/16
Technical Director, General Chemistry – Harvey Jacky

Approved By: _____ Date: 10/28/16
Technical Director, Organics – Loren Portwood

Archival Date:	_____	Doc Control ID#:	_____	Editor:	_____

PERRY JOHNSON LABORATORY ACCREDITATION, INC.

Certificate of Accreditation

Perry Johnson Laboratory Accreditation, Inc. has assessed the Laboratory of:

ALS Environmental-Kelso

1317 South 13th Avenue, Kelso, WA 98626

(Hereinafter called the Organization) and hereby declares that Organization has met the requirements of ISO/IEC 17025:2005 "General Requirements for the competence of Testing and Calibration Laboratories" and the DoD Quality Systems Manual for Environmental Laboratories Version 5.0 July 2013 and is accredited is accordance with the:

United States Department of Defense
Environmental Laboratory Accreditation Program
(DoD-ELAP)

This accreditation demonstrates technical competence for the defined scope:

Chemical and Environmental Testing

(As detailed in the supplement)

Accreditation claims for such testing and/or calibration services shall only be made from addresses referenced within this certificate. This Accreditation is granted subject to the system rules governing the Accreditation referred to above, and the Organization hereby covenants with the Accreditation body's duty to observe and comply with the said rules.

For PJLA:

Tracy Szerszen
President/Operations Manager

Initial Accreditation Date:	*Issue Date:*	*Expiration Date:*
July 19, 2011	February 13, 2016	April 28, 2018
Revision Date:	*Accreditation No.:*	*Certificate No.:*
February 7, 2018	65188	L16-58-R4

Perry Johnson Laboratory Accreditation, Inc. (PJLA)
755 W. Big Beaver, Suite 1325
Troy, Michigan 48084

The validity of this certificate is maintained through ongoing assessments based on a continuous accreditation cycle. The validity of this certificate should be confirmed through the PJLA website: www.pjlabs.com

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental-Kelso

1317 South 13th Avenue, Kelso, WA 98626

Contact Name: Carl Degner Phone: 360-577-7222

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard/Method	Technology	Analyte
Aqueous	EPA 1631E	CVAFS	Mercury (Low level)
Aqueous	EPA 1664A	Gravimetry	Hexane Extractable Material (HEM)
Aqueous	EPA 1664A	Gravimetry	Total Petroleum Hydrocarbons (TPH)
Aqueous	EPA 180.1	Turbidimetry	Turbidity
Aqueous	EPA 2340B	Calculation by 6010	Hardness as CaCO3)
Aqueous	EPA 245.1	CVAA	Mercury
Aqueous	EPA 300.0	IC	Bromide
Aqueous	EPA 300.0	IC	Chloride
Aqueous	EPA 300.0	IC	Fluoride
Aqueous	EPA 300.0	IC	Nitrate as N
Aqueous	EPA 300.0	IC	Nitrite as N
Aqueous	EPA 300.0	IC	Sulfate
Aqueous	EPA 353.2	Colorimetry	Nitrate + Nitrite as N
Aqueous	EPA 537 MOD	HPLC/MS/MS	6:2 Fluorotelomersulfonate
Aqueous	EPA 537 MOD	HPLC/MS/MS	8:2 Fluorotelomersulfonate
Aqueous	EPA 537 MOD	HPLC/MS/MS	N-Ethylperfluorooctanesulfonamide
Aqueous	EPA 537 MOD	HPLC/MS/MS	N-Ethylperfluorooctanesulfonamidoethanol
Aqueous	EPA 537 MOD	HPLC/MS/MS	N-Methylperfluorooctanesulfonamide
Aqueous	EPA 537 MOD	HPLC/MS/MS	N-Methylperfluorooctanesulfonamidoethanol
Aqueous	EPA 537 MOD	HPLC/MS/MS	Perfluoroheptanesulfonate
Aqueous	EPA 537 MOD	HPLC/MS/MS	Perfluorooctane Sulfonamide
Aqueous	EPA 537 MOD	HPLC/MS/MS	Perfluorotetradecanoic acid
Aqueous	EPA 537 MOD	HPLC/MS/MS	Perfluorotridecanoic acid
Aqueous	EPA 7196A	Colorimetry	Chromium VI
Aqueous	EPA 7470A	CVAA	Mercury
Aqueous	EPA 8260C SIM	GC-MS	1,1,2,2-Tetrachloroethane
Aqueous	EPA 8260C SIM	GC-MS	1,1,2-Trichloroethane
Aqueous	EPA 8260C SIM	GC-MS	1,1-Dichloroethene
Aqueous	EPA 8260C SIM	GC-MS	1,2-Dibromoethane
Aqueous	EPA 8260C SIM	GC-MS	1,2-Dichloroethane
Aqueous	EPA 8260C SIM	GC-MS	1,3 Butadine
Aqueous	EPA 8260C SIM	GC-MS	1,4-Dichlorobenzene
Aqueous	EPA 8260C SIM	GC-MS	Bromodichloromethane
Aqueous	EPA 8260C SIM	GC-MS	Carbon Tetrachloride
Aqueous	EPA 8260C SIM	GC-MS	Chlorodibromomethane

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ISO/IEC 17025:2005 and DoD-ELAP

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Accreditation is granted to the facility to perform the following testing:

Matrix	Standard/Method	Technology	Analyte
Aqueous	EPA 8260C SIM	GC-MS	Chloroform
Aqueous	EPA 8260C SIM	GC-MS	Chloromethane
Aqueous	EPA 8260C SIM	GC-MS	cis-1,2-Dichloroethene
Aqueous	EPA 8260C SIM	GC-MS	Dichloromethane (Methylene Chloride)
Aqueous	EPA 8260C SIM	GC-MS	Tetrachloroethene
Aqueous	EPA 8260C SIM	GC-MS	trans-1,2-Dichloroethene
Aqueous	EPA 8260C SIM	GC-MS	Trichloroethene
Aqueous	EPA 8260C SIM	GC-MS	Vinyl chloride
Aqueous	EPA 9020B	Titrimetry	Total Organic Halides (TOX)
Aqueous	EPA 9040C	Potentiometry	pH
Aqueous	EPA 9060A	UV-VIS Spectrophotometry	Total Organic Carbons (TOC)
Aqueous	SM 2130B	Turbidimetry	Turbidity
Aqueous	SM 2320B	Titrimetry	Total Alkalinity (as CaCO3)
Aqueous	SM 2510B	Potentiometry	Specific Conductance
Aqueous	SM 2540B	Gravimetry	Solids, Total
Aqueous	SM 2540C	Gravimetry	Solids, Total Dissolved
Aqueous	SM 2540D	Gravimetry	Solids, Total Suspended
Aqueous	SM 4500-CN- G	Colorimetry	Cyanide, Amenable
Aqueous	SM 4500-P-E	Colorimetry	ortho-phosphorous
Aqueous	SM 4500-S2 D	Titrimetry	Sulfide
Aqueous	SM 4500-CN E	Colorimetry	Total Cyanide
Aqueous	SM4500-NH3 G	Colorimetry	Ammonia
Aqueous	SM5220C	Titrimetry	Chemical Oxygen Demand (COD)
Aqueous	SM5310C	UV-VIS Spectrophotometry	Total Organic Carbons (TOC)
Aqueous	SOP-LCP-PFC	HPLC/MS/MS	6:2 Fluorotelomersulfonate
Aqueous	SOP-LCP-PFC	HPLC/MS/MS	8:2 Fluorotelomersulfonate
Aqueous	SOP-LCP-PFC	HPLC/MS/MS	N-Ethylperfluorooctanesulfonamide
Aqueous	SOP-LCP-PFC	HPLC/MS/MS	N-Ethylperfluorooctanesulfonamidoethanol
Aqueous	SOP-LCP-PFC	HPLC/MS/MS	N-Methylperfluorooctanesulfonamide
Aqueous	SOP-LCP-PFC	HPLC/MS/MS	N-Methylperfluorooctanesulfonamidoethanol
Aqueous	SOP-LCP-PFC	HPLC/MS/MS	Perfluoroheptanesulfonate
Aqueous	SOP-LCP-PFC	HPLC/MS/MS	Perfluorooctane Sulfonamide
Aqueous	SOP-LCP-PFC	HPLC/MS/MS	Perfluorotetradecanoic acid

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Accreditation is granted to the facility to perform the following testing:

Matrix	Standard/Method	Technology	Analyte
Aqueous	SOP-LCP-PFC	HPLC/MS/MS	Perfluorotridecanoic acid
Drinking Water	EPA 504.1	GC-ECD	1,2-Dibromo-3-chloropropane (DBCP)
Drinking Water	EPA 504.1	GC-ECD	1,2-Dibromoethane (EDB)
Drinking Water	EPA 524.2	GC-MS	1,1,1,2-Tetrachloroethane
Drinking Water	EPA 524.2	GC-MS	1,1,1-Trichloroethane
Drinking Water	EPA 524.2	GC-MS	1,1,2,2-Tetrachloroethane
Drinking Water	EPA 524.2	GC-MS	1,1-Dichloroethane
Drinking Water	EPA 524.2	GC-MS	1,1-Dichloroethene
Drinking Water	EPA 524.2	GC-MS	1,1-Dichloropropene
Drinking Water	EPA 524.2	GC-MS	1,2,3-Trichlorobenzene
Drinking Water	EPA 524.2	GC-MS	1,2,3-Trichloropropane
Drinking Water	EPA 524.2	GC-MS	1,2,4-Trichlorobenzene
Drinking Water	EPA 524.2	GC-MS	1,2,4-Trimethylbenzene
Drinking Water	EPA 524.2	GC-MS	1,2-Dibromoethane (EDB)
Drinking Water	EPA 524.2	GC-MS	1,2-Dichlorobenzene
Drinking Water	EPA 524.2	GC-MS	1,2-Dichloroethane
Drinking Water	EPA 524.2	GC-MS	1,2-Dichloropropane
Drinking Water	EPA 524.2	GC-MS	1,3,5-Trimethylbenzene
Drinking Water	EPA 524.2	GC-MS	1,3-Dichlorobenzene
Drinking Water	EPA 524.2	GC-MS	1,3-Dichloropropane
Drinking Water	EPA 524.2	GC-MS	1,4-Dichlorobenzene
Drinking Water	EPA 524.2	GC-MS	2,2-Dichloropropane
Drinking Water	EPA 524.2	GC-MS	2-Chlorotoluene
Drinking Water	EPA 524.2	GC-MS	4-Chlorotoluene
Drinking Water	EPA 524.2	GC-MS	4-Isopropyltoluene
Drinking Water	EPA 524.2	GC-MS	Benzene
Drinking Water	EPA 524.2	GC-MS	Bromobenzene
Drinking Water	EPA 524.2	GC-MS	Bromochloromethane
Drinking Water	EPA 524.2	GC-MS	Bromodichloromethane
Drinking Water	EPA 524.2	GC-MS	Bromoform
Drinking Water	EPA 524.2	GC-MS	Bromomethane
Drinking Water	EPA 524.2	GC-MS	Carbon Tetrachloride
Drinking Water	EPA 524.2	GC-MS	Chlorobenzene
Drinking Water	EPA 524.2	GC-MS	Chlorodibromomethane
Drinking Water	EPA 524.2	GC-MS	Chloroethane

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Matrix	Standard/Method	Technology	Analyte
Drinking Water	EPA 524.2	GC-MS	Chloroform
Drinking Water	EPA 524.2	GC-MS	Chloromethane
Drinking Water	EPA 524.2	GC-MS	cis-1,2-Dichloroethene
Drinking Water	EPA 524.2	GC-MS	cis-1,3-Dichloropropene
Drinking Water	EPA 524.2	GC-MS	Dibromomethane
Drinking Water	EPA 524.2	GC-MS	Dichlorodifluoromethane
Drinking Water	EPA 524.2	GC-MS	Dichloromethane (Methylene Chloride)
Drinking Water	EPA 524.2	GC-MS	Ethylbenzene
Drinking Water	EPA 524.2	GC-MS	Hexachlorobutadiene
Drinking Water	EPA 524.2	GC-MS	Isopropylbenzene
Drinking Water	EPA 524.2	GC-MS	m+p-Xylene
Drinking Water	EPA 524.2	GC-MS	Naphthalene
Drinking Water	EPA 524.2	GC-MS	n-Butylbenzene
Drinking Water	EPA 524.2	GC-MS	n-Propylbenzene
Drinking Water	EPA 524.2	GC-MS	o-Xylene
Drinking Water	EPA 524.2	GC-MS	sec-Butylbenzene
Drinking Water	EPA 524.2	GC-MS	Styrene
Drinking Water	EPA 524.2	GC-MS	tert-butylbenzene
Drinking Water	EPA 524.2	GC-MS	Tetrachloroethene
Drinking Water	EPA 524.2	GC-MS	Toluene
Drinking Water	EPA 524.2	GC-MS	trans-1,2-Dichloroethene
Drinking Water	EPA 524.2	GC-MS	trans-1,3-Dichloropropene
Drinking Water	EPA 524.2	GC-MS	Trichloroethene
Drinking Water	EPA 524.2	GC-MS	Trichlorofluoromethane (Freon 11)
Drinking Water	EPA 524.2	GC-MS	Vinyl chloride
Drinking Water	EPA 524.2	GC-MS	Xylenes, total
Solid	ASTM D4129-92M, Lloyd Kahn	UV-VIS Spectrophotometry	Total Organic Carbons (TOC)
Solid	EPA 160.3M	Gravimetry	Solids, Total
Solid	EPA 1631E	CVFAS	Mercury (low level)
Solid	EPA 7471A, B	CVAA	Mercury
Solid	EPA 9045D	Potentiometry	pH
Solid	EPA 9056A	IC	Nitrate as N
Solid	EPA 9071A	Gravimetry	Hexane Extractable Material (HEM)
Solid	PSEP	Gravimetry	Particle Size

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Accreditation is granted to the facility to perform the following testing:

Matrix	Standard/Method	Technology	Analyte
Aqueous/Drinking Water	EPA 537	HPLC/MS/MS	Perfluorobutanesulfonic Acid
Aqueous/Drinking Water	EPA 537	HPLC/MS/MS	Perfluoroheptanoic Acid
Aqueous/Drinking Water	EPA 537	HPLC/MS/MS	Perfluorohexanesulfonic Acid
Aqueous/Drinking Water	EPA 537	HPLC/MS/MS	Perfluorononanoic Acid
Aqueous/Drinking Water	EPA 537	HPLC/MS/MS	Perfluorooctanesulfonic Acid
Aqueous/Drinking Water	EPA 537	HPLC/MS/MS	Perfluorooctanoic Acid
Aqueous/Solid	ASTM D 1426-93B	Potentiometry	Nitrogen, Total Kjeldahl (TKN)
Aqueous/Solid	EPA 1020A	Physical Property	Ignitability
Aqueous/Solid	EPA 350.1	Colorimetry	Ammonia
Aqueous/Solid	EPA 365.3	Colorimetry	Total Phosphorus
Aqueous/Solid	EPA 537 MOD	HPLC/MS/MS	Perfluorobutane sulfonate
Aqueous/Solid	EPA 537 MOD	HPLC/MS/MS	Perfluorobutanoic acid
Aqueous/Solid	EPA 537 MOD	HPLC/MS/MS	Perfluorodecane Sulfonate
Aqueous/Solid	EPA 537 MOD	HPLC/MS/MS	Perfluorodecanoic acid
Aqueous/Solid	EPA 537 MOD	HPLC/MS/MS	Perfluorododecanoic acid
Aqueous/Solid	EPA 537 MOD	HPLC/MS/MS	Perfluoroheptanoic acid
Aqueous/Solid	EPA 537 MOD	HPLC/MS/MS	Perfluorohexane sulfonate
Aqueous/Solid	EPA 537 MOD	HPLC/MS/MS	Perfluorohexanoic acid
Aqueous/Solid	EPA 537 MOD	HPLC/MS/MS	Perfluorononanoic acid
Aqueous/Solid	EPA 537 MOD	HPLC/MS/MS	Perfluorooctane sulfonate
Aqueous/Solid	EPA 537 MOD	HPLC/MS/MS	Perfluorooctanoic acid
Aqueous/Solid	EPA 537 MOD	HPLC/MS/MS	Perfluoropentanoic acid
Aqueous/Solid	EPA 537 MOD	HPLC/MS/MS	Perfluoroundecanoic acid
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Aluminum
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Antimony
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Arsenic
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Barium
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Beryllium

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Matrix	Standard/Method	Technology	Analyte
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Boron
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Cadmium
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Calcium
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Chromium, total
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Cobalt
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Copper
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Iron
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Lead
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Magnesium
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Manganese
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Molybdenum
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Nickel
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Potassium
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Selenium
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Silver
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Sodium
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Strontium
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Thallium
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Tin
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Titanium
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Vanadium
Aqueous/Solid	EPA 6010B, C/200.7	ICP	Zinc
Aqueous/Solid	EPA 6020, A/200.8	ICP-MS	Aluminum
Aqueous/Solid	EPA 6020, A/200.8	ICP-MS	Antimony
Aqueous/Solid	EPA 6020, A/200.8	ICP-MS	Arsenic
Aqueous/Solid	EPA 6020, A/200.8	ICP-MS	Barium
Aqueous/Solid	EPA 6020, A/200.8	ICP-MS	Beryllium
Aqueous/Solid	EPA 6020, A/200.8	ICP-MS	Boron
Aqueous/Solid	EPA 6020, A/200.8	ICP-MS	Cadmium
Aqueous/Solid	EPA 6020, A/200.8	ICP-MS	Chromium, total
Aqueous/Solid	EPA 6020, A/200.8	ICP-MS	Cobalt
Aqueous/Solid	EPA 6020, A/200.8	ICP-MS	Copper
Aqueous/Solid	EPA 6020, A/200.8	ICP-MS	Iron
Aqueous/Solid	EPA 6020, A/200.8	ICP-MS	Lead
Aqueous/Solid	EPA 6020, A/200.8	ICP-MS	Manganese

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental-Kelso
1317 South 13th Avenue, Kelso, WA 98626
Contact Name: Carl Degner Phone: 360-577-7222

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard/Method	Technology	Analyte
Aqueous/Solid	EPA 6020, A/200.8	ICP-MS	Molybdenum
Aqueous/Solid	EPA 6020, A/200.8	ICP-MS	Nickel
Aqueous/Solid	EPA 6020, A/200.8	ICP-MS	Selenium
Aqueous/Solid	EPA 6020, A/200.8	ICP-MS	Silver
Aqueous/Solid	EPA 6020, A/200.8	ICP-MS	Strontium
Aqueous/Solid	EPA 6020, A/200.8	ICP-MS	Thallium
Aqueous/Solid	EPA 6020, A/200.8	ICP-MS	Tin
Aqueous/Solid	EPA 6020, A/200.8	ICP-MS	Titanium
Aqueous/Solid	EPA 6020, A/200.8	ICP-MS	Vanadium
Aqueous/Solid	EPA 6020, A/200.8	ICP-MS	Zinc
Aqueous/Solid	EPA 6850	HPLC/MS/MS	Perchlorate
Aqueous/Solid	EPA 7742	AA, Borohydride Reduction; GFAA	Selenium
Aqueous/Solid	EPA 8011	GC-ECD	Ethylene Dibromide
Aqueous/Solid	EPA 8011	GC-ECD	1,2-Dibrom-3-chloropropane
Aqueous/Solid	EPA 8015C/AK103-RRO	GC-FID	Residual Range Organics (RRO)
Aqueous/Solid	EPA 8015C; AK101-GRO; NWTPH-Gx	GC-FID	Gasoline Range Organics (GRO)
Aqueous/Solid	EPA 8015C; AK102-DRO; NWTPH-Dx	GC-FID	Diesel Range Organics (DRO)
Aqueous/Solid	EPA 8081A, B	GC-ECD	Aldrin
Aqueous/Solid	EPA 8081A, B	GC-ECD	Alpha-BHC
Aqueous/Solid	EPA 8081A, B	GC-ECD	alpha-Chlordane
Aqueous/Solid	EPA 8081A, B	GC-ECD	Chlordane (total)
Aqueous/Solid	EPA 8081A, B	GC-ECD	DDD (4,4)
Aqueous/Solid	EPA 8081A, B	GC-ECD	DDE (4,4)
Aqueous/Solid	EPA 8081A, B	GC-ECD	DDT (4,4)
Aqueous/Solid	EPA 8081A, B	GC-ECD	delta-BHC
Aqueous/Solid	EPA 8081A, B	GC-ECD	Dieldrin
Aqueous/Solid	EPA 8081A, B	GC-ECD	Endosulfan I
Aqueous/Solid	EPA 8081A, B	GC-ECD	Endosulfan II
Aqueous/Solid	EPA 8081A, B	GC-ECD	Endosulfan sulfate
Aqueous/Solid	EPA 8081A, B	GC-ECD	Endrin
Aqueous/Solid	EPA 8081A, B	GC-ECD	Endrin aldehyde
Aqueous/Solid	EPA 8081A, B	GC-ECD	Endrin ketone

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ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental-Kelso

1317 South 13th Avenue, Kelso, WA 98626

Contact Name: Carl Degner Phone: 360-577-7222

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard/Method	Technology	Analyte
Aqueous/Solid	EPA 8081A, B	GC-ECD	gamma-BHC
Aqueous/Solid	EPA 8081A, B	GC-ECD	gamma-Chlordane
Aqueous/Solid	EPA 8081A, B	GC-ECD	Heptachlor
Aqueous/Solid	EPA 8081A, B	GC-ECD	Heptachlor Epoxide (beta)
Aqueous/Solid	EPA 8081A, B	GC-ECD	Methoxychlor
Aqueous/Solid	EPA 8081A, B	GC-ECD	Toxaphene (total)
Aqueous/Solid	EPA 8081B	GC-ECD	2,4-DDD
Aqueous/Solid	EPA 8081B	GC-ECD	2,4-DDE
Aqueous/Solid	EPA 8081B	GC-ECD	2,4-DDT
Aqueous/Solid	EPA 8081B	GC-ECD	Chlorpyrifos
Aqueous/Solid	EPA 8081B	GC-ECD	cis-Nonachlor
Aqueous/Solid	EPA 8081B	GC-ECD	Hexachlorobenzene
Aqueous/Solid	EPA 8081B	GC-ECD	Hexachlorobutadiene
Aqueous/Solid	EPA 8081B	GC-ECD	Hexachloroethane
Aqueous/Solid	EPA 8081B	GC-ECD	Isodrin
Aqueous/Solid	EPA 8081B	GC-ECD	Mirex
Aqueous/Solid	EPA 8081B	GC-ECD	Oxychlordane
Aqueous/Solid	EPA 8081B	GC-ECD	trans-Nonachlor
Aqueous/Solid	EPA 8082A	GC-ECD	2,2’,3,3’,4,4’,5,5’,6-Nonachlorobiphenyl (PCB 206)
Aqueous/Solid	EPA 8082A	GC-ECD	2,2’,3,3’,4,4’,5,6-Octachlorobiphenyl (PCB 195)
Aqueous/Solid	EPA 8082A	GC-ECD	2,2',3,3',4,4',5,5',6,6' Decachlorobiphenyl (PCB 209)
Aqueous/Solid	EPA 8082A	GC-ECD	2,2’,3,3’,4,4’,5-Heptachlorobiphenyl (PCB 170)
Aqueous/Solid	EPA 8082A	GC-ECD	2,2’,3,3’,4,4’-Hexachlorobiphenyl (PCB 128)
Aqueous/Solid	EPA 8082A	GC-ECD	2,2’,3,4,4’,5,5’-Heptachlorobiphenyl (PCB 180)
Aqueous/Solid	EPA 8082A	GC-ECD	2,2’,3,4,4’,5’,6-Heptachlorobiphenyl (PCB 183)
Aqueous/Solid	EPA 8082A	GC-ECD	2,2’,3,4,4’,5’-Hexachlorobiphenyl (PCB 138)
Aqueous/Solid	EPA 8082A	GC-ECD	2,2’,3,4,4’,6,6’-Heptachlorobiphenyl (PCB 184)
Aqueous/Solid	EPA 8082A	GC-ECD	2,2’,3,4’,5,5’,6-Heptachlorobiphenyl (PCB 187)
Aqueous/Solid	EPA 8082A	GC-ECD	2,2’,3,4,5’-Pentachlorobiphenyl (PCB 87)
Aqueous/Solid	EPA 8082A	GC-ECD	2,2’,3,4’,5-Pentachlorobiphenyl (PCB 90)

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Accreditation is granted to the facility to perform the following testing:

Matrix	Standard/Method	Technology	Analyte
Aqueous/Solid	EPA 8082A	GC-ECD	2,2',3,5'-Tetrachlorobiphenyl (PCB 44)
Aqueous/Solid	EPA 8082A	GC-ECD	2,2',4,4',5,5'-Hexachlorobiphenyl (PCB 153)
Aqueous/Solid	EPA 8082A	GC-ECD	2,2',4,5,5'-Pentachlorobiphenyl (PCB 101)
Aqueous/Solid	EPA 8082A	GC-ECD	2,2',5,6'-Tetrachlorbiphenyl (PCB 53)
Aqueous/Solid	EPA 8082A	GC-ECD	2,2',5-Trichlorobiphenyl (PCB 18)
Aqueous/Solid	EPA 8082A	GC-ECD	2,3,3',4,4',5,5'-Heptachlorobiphenyl (PCB 189)
Aqueous/Solid	EPA 8082A	GC-ECD	2,3,3',4,4',5-Hexachlorobiphenyl (PCB 156)
Aqueous/Solid	EPA 8082A	GC-ECD	2,3,3',4,4',5'-Hexachlorobiphenyl (PCB 157)
Aqueous/Solid	EPA 8082A	GC-ECD	2,3,3',4,4',6-Hexachlorobiphenyl (PCB 158)
Aqueous/Solid	EPA 8082A	GC-ECD	2,3,3',4,4'-Pentachlorobiphenyl (PCB 105)
Aqueous/Solid	EPA 8082A	GC-ECD	2,3',4,4',5,5' Hexachlorobiphenyl (PCB 167)
Aqueous/Solid	EPA 8082A	GC-ECD	2,3',4,4',5',6-Hexachlorobiphenyl (PCB 168)
Aqueous/Solid	EPA 8082A	GC-ECD	2,3,4,4',5-Pentachlorobiphenyl (PCB 114)
Aqueous/Solid	EPA 8082A	GC-ECD	2,3',4,4',5-Pentachlorobiphenyl (PCB 118)
Aqueous/Solid	EPA 8082A	GC-ECD	2,3',4,4',5-Pentachlorobiphenyl (PCB 123)
Aqueous/Solid	EPA 8082A	GC-ECD	2,3,4,4'-Tetrachlorobiphenyl (PCB 60)
Aqueous/Solid	EPA 8082A	GC-ECD	2,3',4,4'-Tetrachlorobiphenyl (PCB 66)
Aqueous/Solid	EPA 8082A	GC-ECD	2,4,4'-Trichlorobiphenyl (PCB 28)
Aqueous/Solid	EPA 8082A	GC-ECD	2,4'-Dichlorobiphenyl (PCB 8)
Aqueous/Solid	EPA 8082A	GC-ECD	3,3',4,4',5,5'-Hexachlorobiphenyl (PCB 169)
Aqueous/Solid	EPA 8082A	GC-ECD	3,3',4,4',5-Pentachlorobiphenyl (PCB 126)
Aqueous/Solid	EPA 8082A	GC-ECD	3,3',4,4'-Tetrachlorobiphenyl (PCB 77)
Aqueous/Solid	EPA 8082A	GC-ECD	3,4,4',5-Tetrachlorobiphenyl (PCB 81)
Aqueous/Solid	EPA 8082A	GC-ECD	Aroclor 1016
Aqueous/Solid	EPA 8082A	GC-ECD	Aroclor 1221
Aqueous/Solid	EPA 8082A	GC-ECD	Aroclor 1232
Aqueous/Solid	EPA 8082A	GC-ECD	Aroclor 1242
Aqueous/Solid	EPA 8082A	GC-ECD	Aroclor 1248
Aqueous/Solid	EPA 8082A	GC-ECD	Aroclor 1254
Aqueous/Solid	EPA 8082A	GC-ECD	Aroclor 1260
Aqueous/Solid	EPA 8082A	GC-ECD	Aroclor 1262
Aqueous/Solid	EPA 8082A	GC-ECD	Aroclor 1268
Aqueous/Solid	EPA 8260B, C	GC-MS	1,1,1,2-Tetrachloroethane
Aqueous/Solid	EPA 8260B, C	GC-MS	1,1,1-Trichloroethane

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Matrix	Standard/Method	Technology	Analyte
Aqueous/Solid	EPA 8260B, C	GC-MS	1,1,2,2-Tetrachloroethane
Aqueous/Solid	EPA 8260B, C	GC-MS	1,1,2-Trichloroethane
Aqueous/Solid	EPA 8260B, C	GC-MS	1,1-Dichloroethane
Aqueous/Solid	EPA 8260B, C	GC-MS	1,2-Dibromoethane
Aqueous/Solid	EPA 8260B, C	GC-MS	1,2-Dichlorobenzene
Aqueous/Solid	EPA 8260B, C	GC-MS	1,2-Dichloroethane
Aqueous/Solid	EPA 8260B, C	GC-MS	1,2-Dichloropropane
Aqueous/Solid	EPA 8260B, C	GC-MS	1,3,5-Trimethylbenzene
Aqueous/Solid	EPA 8260B, C	GC-MS	1,3-Dichlorobenzene
Aqueous/Solid	EPA 8260B, C	GC-MS	1,3-Dichloropropane
Aqueous/Solid	EPA 8260B, C	GC-MS	1,4-Dichlorobenzene
Aqueous/Solid	EPA 8260B, C	GC-MS	1-phenylpropane
Aqueous/Solid	EPA 8260B, C	GC-MS	2,2-Dichloropropane
Aqueous/Solid	EPA 8260B, C	GC-MS	2-Butanone (MEK)
Aqueous/Solid	EPA 8260B, C	GC-MS	2-Chloroethylvinylether
Aqueous/Solid	EPA 8260B, C	GC-MS	2-Chlorotoluene
Aqueous/Solid	EPA 8260B, C	GC-MS	2-Hexanone
Aqueous/Solid	EPA 8260B, C	GC-MS	4-Chlorotoluene
Aqueous/Solid	EPA 8260B, C	GC-MS	4-Isopropyltoluene
Aqueous/Solid	EPA 8260B, C	GC-MS	4-Methyl-2-pentanone (MIBK)
Aqueous/Solid	EPA 8260B, C	GC-MS	Acetone
Aqueous/Solid	EPA 8260B, C	GC-MS	Acetonitrile
Aqueous/Solid	EPA 8260B, C	GC-MS	Acrolein
Aqueous/Solid	EPA 8260B, C	GC-MS	Acrylonitrile
Aqueous/Solid	EPA 8260B, C	GC-MS	Benzene
Aqueous/Solid	EPA 8260B, C	GC-MS	Bromobenzene
Aqueous/Solid	EPA 8260B, C	GC-MS	Bromochloromethane
Aqueous/Solid	EPA 8260B, C	GC-MS	Bromodichloromethane
Aqueous/Solid	EPA 8260B, C	GC-MS	Bromoform
Aqueous/Solid	EPA 8260B, C	GC-MS	Bromomethane
Aqueous/Solid	EPA 8260B, C	GC-MS	Carbon disulfide
Aqueous/Solid	EPA 8260B, C	GC-MS	Carbon Tetrachloride
Aqueous/Solid	EPA 8260B, C	GC-MS	Chlorobenzene
Aqueous/Solid	EPA 8260B, C	GC-MS	Chlorodibromomethane
Aqueous/Solid	EPA 8260B, C	GC-MS	Chloroethane

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ALS Environmental-Kelso

1317 South 13th Avenue, Kelso, WA 98626

Contact Name: Carl Degner Phone: 360-577-7222

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard/Method	Technology	Analyte
Aqueous/Solid	EPA 8260B, C	GC-MS	Chloroform
Aqueous/Solid	EPA 8260B, C	GC-MS	Chloromethane
Aqueous/Solid	EPA 8260B, C	GC-MS	cis-1,2-Dichloroethene
Aqueous/Solid	EPA 8260B, C	GC-MS	cis-1,3-Dichloropropene
Aqueous/Solid	EPA 8260B, C	GC-MS	Dibromomethane
Aqueous/Solid	EPA 8260B, C	GC-MS	Dichlorodifluoromethane
Aqueous/Solid	EPA 8260B, C	GC-MS	Dichloromethane (Methylene Chloride)
Aqueous/Solid	EPA 8260B, C	GC-MS	Di-isopropylether (DIPE)
Aqueous/Solid	EPA 8260B, C	GC-MS	DIPE
Aqueous/Solid	EPA 8260B, C	GC-MS	ETBE
Aqueous/Solid	EPA 8260B, C	GC-MS	Ethyl Benzene
Aqueous/Solid	EPA 8260B, C	GC-MS	Ethylbenzene
Aqueous/Solid	EPA 8260B, C	GC-MS	Freon 11
Aqueous/Solid	EPA 8260B, C	GC-MS	Freon 113
Aqueous/Solid	EPA 8260B, C	GC-MS	Hexachlorobutadiene
Aqueous/Solid	EPA 8260B, C	GC-MS	Isopropylbenzene
Aqueous/Solid	EPA 8260B, C	GC-MS	Methyl-tert-butylether (MTBE)
Aqueous/Solid	EPA 8260B, C	GC-MS	Naphthalene
Aqueous/Solid	EPA 8260B, C	GC-MS	n-Butylbenzene
Aqueous/Solid	EPA 8260B, C	GC-MS	n-Propylbenzene
Aqueous/Solid	EPA 8260B, C	GC-MS	sec-Butylbenzene
Aqueous/Solid	EPA 8260B, C	GC-MS	Styrene
Aqueous/Solid	EPA 8260B, C	GC-MS	tert-amylmethylether (TAME)
Aqueous/Solid	EPA 8260B, C	GC-MS	tert-Butyl alcohol
Aqueous/Solid	EPA 8260B, C	GC-MS	tert-butylbenzene
Aqueous/Solid	EPA 8260B, C	GC-MS	Tetrachloroethene
Aqueous/Solid	EPA 8260B, C	GC-MS	Toluene
Aqueous/Solid	EPA 8260B, C	GC-MS	trans-1,2-Dichloroethene
Aqueous/Solid	EPA 8260B, C	GC-MS	trans-1,3-Dichloropropene
Aqueous/Solid	EPA 8260B, C	GC-MS	Trichloroethene
Aqueous/Solid	EPA 8260B, C	GC-MS	Trichlorofluoromethane (Freon 11)
Aqueous/Solid	EPA 8260B, C	GC-MS	Vinyl acetate
Aqueous/Solid	EPA 8260B, C	GC-MS	Vinyl chloride
Aqueous/Solid	EPA 8260B, C	GC-MS	Xylene, total
Aqueous/Solid	EPA 8260B, C	GC-MS	1,1-Dichloroethene

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Accreditation is granted to the facility to perform the following testing:

Matrix	Standard/Method	Technology	Analyte
Aqueous/Solid	EPA 8260B, C	GC-MS	1,1-Dichloropropene
Aqueous/Solid	EPA 8260B, C	GC-MS	1,2,3-Trichlorobenzene
Aqueous/Solid	EPA 8260B, C	GC-MS	1,2,3-Trichloropropane
Aqueous/Solid	EPA 8260B, C	GC-MS	1,2,4-Trichlorobenzene
Aqueous/Solid	EPA 8260B, C	GC-MS	1,2,4-Trimethylbenzene
Aqueous/Solid	EPA 8270C, D	GC-MS	1,2,4-Trichlorobenzene
Aqueous/Solid	EPA 8270C, D	GC-MS	1,2-Dichlorobenzene
Aqueous/Solid	EPA 8270C, D	GC-MS	1,3-Dichlorobenzene
Aqueous/Solid	EPA 8270C, D	GC-MS	1,4-Dichlorobenzene
Aqueous/Solid	EPA 8270C, D	GC-MS	2,4,5-Trichlorophenol
Aqueous/Solid	EPA 8270C, D	GC-MS	2,4,6-Trichlorophenol
Aqueous/Solid	EPA 8270C, D	GC-MS	2,4-Dichlorophenol
Aqueous/Solid	EPA 8270C, D	GC-MS	2,4-Dimethylphenol
Aqueous/Solid	EPA 8270C, D	GC-MS	2,4-Dinitrophenol
Aqueous/Solid	EPA 8270C, D	GC-MS	2,4-Dinitrotoluene
Aqueous/Solid	EPA 8270C, D	GC-MS	2,6-Dinitrotoluene
Aqueous/Solid	EPA 8270C, D	GC-MS	2-Chloronaphthalene
Aqueous/Solid	EPA 8270C, D	GC-MS	2-Chlorophenol
Aqueous/Solid	EPA 8270C, D	GC-MS	2-Methyl-4,6-Dinitrophenol
Aqueous/Solid	EPA 8270C, D	GC-MS	2-Methylnaphthalene
Aqueous/Solid	EPA 8270C, D	GC-MS	2-Methylphenol
Aqueous/Solid	EPA 8270C, D	GC-MS	2-Nitroaniline
Aqueous/Solid	EPA 8270C, D	GC-MS	2-Nitrophenol
Aqueous/Solid	EPA 8270C, D	GC-MS	3,3-Dichlorobenzidine
Aqueous/Solid	EPA 8270C, D	GC-MS	3-Nitroaniline
Aqueous/Solid	EPA 8270C, D	GC-MS	4-Bromophenyl-phenylether
Aqueous/Solid	EPA 8270C, D	GC-MS	4-Chloro-3-methylphenol
Aqueous/Solid	EPA 8270C, D	GC-MS	4-Chloroaniline
Aqueous/Solid	EPA 8270C, D	GC-MS	4-Chlorophenyl-phenylether
Aqueous/Solid	EPA 8270C, D	GC-MS	4-Methylphenol (and/or 3-Methylphenol)
Aqueous/Solid	EPA 8270C, D	GC-MS	4-Nitroaniline
Aqueous/Solid	EPA 8270C, D	GC-MS	4-Nitrophenol
Aqueous/Solid	EPA 8270C, D	GC-MS	Acenaphthene
Aqueous/Solid	EPA 8270C, D	GC-MS	Acenaphthylene
Aqueous/Solid	EPA 8270C, D	GC-MS	Aniline

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ALS Environmental-Kelso

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Accreditation is granted to the facility to perform the following testing:

Matrix	Standard/Method	Technology	Analyte
Aqueous/Solid	EPA 8270C, D	GC-MS	Anthracene
Aqueous/Solid	EPA 8270C, D	GC-MS	Benzidine
Aqueous/Solid	EPA 8270C, D	GC-MS	Benzo(a)anthracene
Aqueous/Solid	EPA 8270C, D	GC-MS	Benzo(a)pyrene
Aqueous/Solid	EPA 8270C, D	GC-MS	Benzo(b)fluoranthene
Aqueous/Solid	EPA 8270C, D	GC-MS	Benzo(g,h,i)perylene
Aqueous/Solid	EPA 8270C, D	GC-MS	Benzo(k)fluoranthene
Aqueous/Solid	EPA 8270C, D	GC-MS	Benzoic acid
Aqueous/Solid	EPA 8270C, D	GC-MS	Benzyl alcohol
Aqueous/Solid	EPA 8270C, D	GC-MS	bis(2-Chloroethoxy)methane
Aqueous/Solid	EPA 8270C, D	GC-MS	bis(2-Chloroethyl)ether
Aqueous/Solid	EPA 8270C, D	GC-MS	bis(2-Chloroisopropyl)ether
Aqueous/Solid	EPA 8270C, D	GC-MS	bis(2-ethylhexy)phthalate
Aqueous/Solid	EPA 8270C, D	GC-MS	Butyl benzyl phthalate
Aqueous/Solid	EPA 8270C, D	GC-MS	Carbazole
Aqueous/Solid	EPA 8270C, D	GC-MS	Chrysene
Aqueous/Solid	EPA 8270C, D	GC-MS	Dibenzo(a,h)anthracene
Aqueous/Solid	EPA 8270C, D	GC-MS	Dibenzofuran
Aqueous/Solid	EPA 8270C, D	GC-MS	Diethyl phthalate
Aqueous/Solid	EPA 8270C, D	GC-MS	Dimethylphthalate
Aqueous/Solid	EPA 8270C, D	GC-MS	di-n-butylphthalate
Aqueous/Solid	EPA 8270C, D	GC-MS	Di-n-octylphthalate
Aqueous/Solid	EPA 8270C, D	GC-MS	Fluoranthene
Aqueous/Solid	EPA 8270C, D	GC-MS	Fluorene
Aqueous/Solid	EPA 8270C, D	GC-MS	Hexachlorobenzene
Aqueous/Solid	EPA 8270C, D	GC-MS	Hexachlorobutadiene
Aqueous/Solid	EPA 8270C, D	GC-MS	Hexachlorocyclopentadiene
Aqueous/Solid	EPA 8270C, D	GC-MS	Hexachloroethane
Aqueous/Solid	EPA 8270C, D	GC-MS	Indeno(1,2,3, cd)pyrene
Aqueous/Solid	EPA 8270C, D	GC-MS	Isophorone
Aqueous/Solid	EPA 8270C, D	GC-MS	Naphthalene
Aqueous/Solid	EPA 8270C, D	GC-MS	Nitrobenzene
Aqueous/Solid	EPA 8270C, D	GC-MS	N-Nitrosodimethylamine
Aqueous/Solid	EPA 8270C, D	GC-MS	N-Nitroso-di-n-propylamine
Aqueous/Solid	EPA 8270C, D	GC-MS	N-Nitrosodiphenylamine

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Matrix	Standard/Method	Technology	Analyte
Aqueous/Solid	EPA 8270C, D	GC-MS	Pentachlorobenzene
Aqueous/Solid	EPA 8270C, D	GC-MS	Pentachlorophenol
Aqueous/Solid	EPA 8270C, D	GC-MS	Phenanthrene
Aqueous/Solid	EPA 8270C, D	GC-MS	Phenol
Aqueous/Solid	EPA 8270C, D	GC-MS	Pyrene
Aqueous/Solid	EPA 8270C, D	GC-MS	Pyridine
Aqueous/Solid	EPA 8270C, D	GC-MS	2,3,4,6-Tetrachlorophenol
Aqueous/Solid	EPA 8270C,D	GC-MS	1,2,4,5-Tetrachlorobenzene
Aqueous/Solid	EPA 8270D	GC-MS	1- Methylnaphthalene
Aqueous/Solid	EPA 8270 SIM	GC-MS	2-Methylnaphthalene
Aqueous/Solid	EPA 8270 SIM	GC-MS	Acenaphthene
Aqueous/Solid	EPA 8270 SIM	GC-MS	Acenaphthylene
Aqueous/Solid	EPA 8270 SIM	GC-MS	Anthracene
Aqueous/Solid	EPA 8270 SIM	GC-MS	Benzo(a)anthracene
Aqueous/Solid	EPA 8270 SIM	GC-MS	Benzo(a)pyrene
Aqueous/Solid	EPA 8270 SIM	GC-MS	Benzo(b)fluoranthene
Aqueous/Solid	EPA 8270 SIM	GC-MS	Benzo(g,h,i)perylene
Aqueous/Solid	EPA 8270 SIM	GC-MS	Benzo(k)fluoranthene
Aqueous/Solid	EPA 8270 SIM	GC-MS	Chrysene
Aqueous/Solid	EPA 8270 SIM	GC-MS	Dibenzo(a,h)anthracene
Aqueous/Solid	EPA 8270 SIM	GC-MS	Fluoranthene
Aqueous/Solid	EPA 8270 SIM	GC-MS	Fluorene
Aqueous/Solid	EPA 8270 SIM	GC-MS	Indeno(1,2,3, cd)pyrene
Aqueous/Solid	EPA 8270 SIM	GC-MS	Naphthalene
Aqueous/Solid	EPA 8270 SIM	GC-MS	p-Dioxane
Aqueous/Solid	EPA 8270 SIM	GC-MS	Phenanthrene
Aqueous/Solid	EPA 8270 SIM	GC-MS	Pyrene
Aqueous/Solid	EPA 9012B	Colorimetry	Total Cyanide
Aqueous/Solid	EPA 9030B	Distillation	Sulfide
Aqueous/Solid	EPA 9056A	IC	Bromide
Aqueous/Solid	EPA 9056A	IC	Chloride
Aqueous/Solid	EPA 9056A	IC	Fluoride
Aqueous/Solid	EPA 9056A	IC	Sulfate
Aqueous/Solid	NWTPH-Dx	GC-FID	Residual Range Organics
Aqueous/Solid	SOC-Butyl	GC-FPD	Di-n-butyltin

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Matrix	Standard/Method	Technology	Analyte
Aqueous/Solid	SOC-Butyl	GC-FPD	n-Butyltin
Aqueous/Solid	SOC-Butyl	GC-FPD	Tetra-n-butyltin
Aqueous/Solid	SOC-Butyl	GC-FPD	Tri-n-butyltin
Aqueous/Solid	SOP-LCP-PFC	HPLC/MS/MS	Perfluorobutane sulfonate
Aqueous/Solid	SOP-LCP-PFC	HPLC/MS/MS	Perfluorobutanoic acid
Aqueous/Solid	SOP-LCP-PFC	HPLC/MS/MS	Perfluorodecane Sulfonate
Aqueous/Solid	SOP-LCP-PFC	HPLC/MS/MS	Perfluorodecanoic acid
Aqueous/Solid	SOP-LCP-PFC	HPLC/MS/MS	Perfluorododecanoic acid
Aqueous/Solid	SOP-LCP-PFC	HPLC/MS/MS	Perfluoroheptanoic acid
Aqueous/Solid	SOP-LCP-PFC	HPLC/MS/MS	Perfluorohexane sulfonate
Aqueous/Solid	SOP-LCP-PFC	HPLC/MS/MS	Perfluorohexanoic acid
Aqueous/Solid	SOP-LCP-PFC	HPLC/MS/MS	Perfluorononanoic acid
Aqueous/Solid	SOP-LCP-PFC	HPLC/MS/MS	Perfluorooctane sulfonate
Aqueous/Solid	SOP-LCP-PFC	HPLC/MS/MS	Perfluorooctanoic acid
Aqueous/Solid	SOP-LCP-PFC	HPLC/MS/MS	Perfluoropentanoic acid
Aqueous/Solid	SOP-LCP-PFC	HPLC/MS/MS	Perfluoroundecanoic acid

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Matrix	Standard/Method	Technology	Analyte
Aqueous	EPA 1640	Reductive Metals Precipitation	Prep Method
Aqueous	EPA 3010A	Acid Digestion	Metals Digestion
Aqueous	EPA 3020A	Acid Digestion	Metals Digestion
Aqueous	EPA 3511	Microextraction	Extractable Prep
Aqueous	EPA 3520C	Continuous Liquid-Liquid Extraction	Extractable Prep
Aqueous	EPA 3535A	Solid Phase Extraction	Prep Method
Aqueous	EPA 5030B	Purge and Trap	Volatile Prep
Aqueous	SOP-MET-DIG	Acid Digestion	Metals Digestion
Solid	EPA 3050B	Acid Digestion	Metals Digestion
Solid	EPA 3060	Alkaline Digestion	Alkaline Digestion for Cr(VI) only
Solid	EPA 3541	Automated Soxhlet Extraction	Extractable Prep
Solid	EPA 3546	Microwave Extraction	Extractable Prep
Solid	EPA 3550B	Ultrasonic Extraction	Extractable Prep
Solid	EPA 5035A	Purge and Trap	Voc Organics
Solid	EPA 5050	Bomb Digestion	Prep Method
Solid	EPA 9013	Midi-Distillation	Cyanides
Solid	SOP-GEN-AVS	Acid Digestion	Simultaneously Extracted Metals
Aqueous/Solids	ASTM D3590-89	Digestion	TKN
Aqueous/Solids	EPA 1311	TCLP Extraction	Physical Extraction
Aqueous/Solids	EPA 3620C	Florisil Clean Up	Extractable Cleanup
Aqueous/Solids	EPA 3630C	Silica Gel Clean Up	Extractable Prep
Aqueous/Solids	EPA 3640A	Gel-Permeation Clean Up	Extractable Cleanup
Aqueous/Solids	EPA 3660	Sulfur Clean Up	Extractable Prep
Aqueous/Solids	EPA 3665A	Acid Clean Up	Extractable Cleanup

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Current Data Quality Objectives (DQOs) may be requested from the laboratory for specified methods or projects.

QA MANUAL CROSS REFERENCE TABLE

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1) Introduction and Scope

ALS Environmental, Kelso is a professional analytical services laboratory which performs chemical and microbiological analyses on a wide variety of sample matrices, including drinking water, groundwater, surface water, wastewater, soil, sludge, sediment, tissue, industrial and hazardous waste, air, and other material.

We recognize that quality assurance requires a commitment to quality by everyone in the organization – individually, within each operating unit, and throughout the entire laboratory. Laboratory management is committed to ensuring the effectiveness of its quality systems and to ensure that all tests are carried out in accordance to customer requirements. Key elements of this commitment are set forth in the SOP *Laboratory Ethics and Data Integrity* (CE-GEN001) and in this Quality Assurance Manual. ALS – Kelso is committed to operate in accordance with these requirements and those of regulatory agencies, accrediting authorities, and certifying organizations. The laboratory also strives for improvement through varying continuous improvement initiatives and projects.

Quality Management Systems are established, implemented and maintained by management. Policies and procedures are established in order to meet requirements of accreditation bodies and applicable programs, such as the Department of Defense (DOD) Environmental Laboratory Accreditation Program, as well as client's quality objectives. Systems are designed so that there will be sufficient Quality Assurance (QA) activities conducted in the laboratory to ensure that all analytical data generated and processed will be scientifically sound, legally defensible, of known and documented quality, and will accurately reflect the material being tested. Quality Systems are applicable to all fields of testing in which the laboratory in involved.

Quality Control (QC) procedures are used to continually assess performance of the laboratory and quality systems. The laboratory maintains control of analytical results by adhering to written standard operating procedures (SOPs), using analytical control parameters with all analyses, and by observing sample custody requirements. All analytical results are calculated and reported in units consistent with project specifications to allow comparability of data.

This QAM is applicable to the facility listed on the title page. The information in this manual has been organized according to requirements found in the National Environmental Laboratory Accreditation Program (NELAP) Quality Systems Standards (2003 and 2009), the EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5, USEPA, 2001; and General Requirements for the Competence of Testing and Calibration Laboratories, ISO/IEC 17025:2005. A glossary of pertinent terms and acronyms is included in Appendix A.

2) Organization

The ALS Environmental, Kelso staff, consisting of approximately 110 employees, includes chemists, technicians and support personnel. They represent diverse educational backgrounds and experience, and provide the comprehensive skills that the laboratory requires. During seasonal workload increases, additional temporary employees may be hired to perform specific tasks. All employees share the responsibility for maintaining and improving the quality of our analytical services.

ALS – Kelso is legally identifiable as ALS Group USA, Corp., dba ALS Environmental. ALS Group USA, Corp. is a component of ALS Limited, a publicly held Australian company. The ALS global website may be referred to for corporate ownership information (www.alsglobal.com/Our-Company). The laboratory is divided into operational and managerial units based upon specific disciplines. Each department is responsible for establishing, maintaining and documenting QA and QC practices meeting laboratory needs. Organizational charts of the laboratory, as well as the resumes of these key personnel, can be found in Appendix B. This laboratory organization is designed so that potential conflict of interest is avoided, and such

that an adequate amount of supervisory personnel are in place to provide oversight and supervision of day to day operations.

3) Management

The purpose of the QA program at ALS Environmental, Kelso is to ensure that our clients are provided with analytical data that is scientifically sound, legally defensible, and of known and documented quality. The concept of Quality Assurance can be extended, and is expressed in the mission statement:

> "The mission of ALS Environmental, Kelso is to provide high quality, cost-effective, and timely professional testing services to our customers. We recognize that our success as a company is based on our ability to maintain customer satisfaction. To do this requires constant attention to customer needs, maintenance of state-of-the-art testing capabilities and successful management of our most important asset - our people - in a way that encourages professional growth, personal development and company commitment."

3.1 Quality Management Systems

In support of this mission, the laboratory has developed a Quality Management System to ensure all products and services meet our client's needs. The system is implemented and maintained by the Quality Assurance Manager with corporate oversight by the Manager of Quality Assurance, USA. These systems are based upon ISO 17025:2005 standards, upon which fundamental programs (NELAC 2003, 2009 and DoD QSM) are based. Implementation and documentation against these standards are communicated in corporate policy statements, this QAM, and SOPs. Actual procedures, actions and documentation are defined in both administrative and technical SOPs. Quality systems include:

- Accreditation and certification program compliance
- Standard Operating Procedures
- Sample management and Chain of Custody procedures
- Document control
- Demonstration of Capability
- Analytical traceability
- Ethics training and data integrity processes
- Corrective action procedures
- Statistical control charting
- Management reviews

The effectiveness of the quality system is assessed in several ways, including:

- Internal and external audits
- Periodic reports to management
- Analysis of customer feedback
- Proficiency testing

The responsibilities of key positions within the laboratory are described below. Table 3-1 lists the ALS - Kelso personnel assigned to these key positions. Managerial staff members are

provided the authority and resources needed to perform their duties. In the event that work is stopped in response to quality problems, as described below, only the Laboratory Director or Quality Assurance Manager has the authority to resume work.

Laboratory Director – The role of the Laboratory Director is to provide technical, operational, and administrative leadership through planning, allocation and management of personnel and equipment resources. The Laboratory Director provides leadership and support for the QA program and is responsible for overall laboratory efficiency and financial performance. The Laboratory Director has the authority to stop work in response to quality problems. The Laboratory Director also provides resources for implementation of the QA program, reviews and approves this QA Manual, reviews and approves standard operating procedures (SOPs), and provides support for business development by identifying and developing new markets through continuing support of the management of existing client activities.

Quality Assurance Manager (QAM) – The Quality Assurance Manager has the authority and responsibility for implementing, maintaining, and improving the quality system. This includes coordination of QA activities in the laboratory, ensuring that personnel understand the quality system, ensuring communication takes place in the laboratory regarding implementation of the quality system, ensuring adequate staff training, and monitoring overall quality system compliance. The QAM continually evaluates potential improvements in the quality system. Audit and surveillance results, control charts, proficiency testing results, data analysis, corrective and preventive actions, customer feedback, and management reviews are used to support quality system implementation. The QAM is responsible for ensuring compliance with all applicable regulatory compliance quality standards (i.e. NELAP/TNI, ISO, DoD QSM, etc.). The QAM works with laboratory staff to establish effective quality control and assessment processes and has the authority to stop work in response to quality problems. The QAM is responsible for maintaining the laboratory's certifications and approvals, for maintaining the QA Manual and performing an annual review of it, reviewing and approving SOPs and ensuring the annual review of technical SOPs, maintaining QA records (metrological records, archived logbooks, PT results, etc.), document control, conducting proficiency testing studies, approving nonconformity and corrective action reports, and performing internal QA audits.

The QAM reports directly to the Laboratory Director and reports indirectly to the ALS Manager of Quality Assurance, USA. It is important to note that when evaluating data, the QAM does so in an objective manner and free of outside, or managerial, influence.

The Manager of Quality Assurance, USA is responsible for the overall QA program at all the ALS Environmental Group laboratories. The Manager of Quality Assurance, USA is responsible for oversight of QAM's regulatory compliance efforts (NELAP, ISO, DOD, etc.) and may perform internal audits to evaluate compliance. The Manager of Quality Assurance, USA approves company-wide SOPs and provides assistance to the laboratory QA staff and laboratory managers as necessary.

Deputy Laboratory Director and QA Manager – In the case of absence of the Laboratory Director or QAM, deputies are assigned to act in that role. Default deputies for these positions are the Client Services Manager or Metals Department Manager (for the Laboratory Director) and the Laboratory Director (for the QAM).

Environmental Health and Safety (EH&S) Officer – The EH&S officer is responsible for the administration of the laboratory health and safety policies. This includes the formulation and implementation of safety policies, the supervision of new-employee safety training, the review of accidents, incidents and prevention plans, the monitoring of hazardous waste disposal and the conducting of departmental safety inspections.

The EH&S officer is also designated as the Chemical Hygiene Officer. The EH&S Officer has a dotted-line reporting responsibility to the ALS North America EH&S Manager.

Client Services Manager (CSM) – The CSM is responsible for the Client Services Department defined for the laboratory. This includes management and oversight of Project Managers, electronic deliverables, and support functions. The Client Services Department provides a complete interface with clients from initial project specification to final deliverables. The Client Services Manager has the responsibility and authority to stop work in response to accreditation/certification or quality problems, or in response to similar subcontractor quality problems.

Department Managers and Supervisors – Each manager or supervisor has the responsibility to ensure that QA and QC functions are carried out as specified when executing the analyses and related tasks and to ensure the production of high quality data. Managers and bench-level supervisors monitor the day-to-day operations to ensure that productivity and data quality objectives are met. A department manager has the authority to stop work in response to quality problems in their area. Managers and supervisors are responsible for ensuring that analysts perform testing according to applied methods, SOPs, and QC guidelines particular to the laboratory department.

Sample Management Office (SMO) – The Sample Management Office plays a key role in the laboratory QA program by handling all activities associated with receiving, storage, and disposal of samples, and maintaining documentation for all samples received. SMO staff is also responsible for the proper disposal of samples after analysis. The Support Services Manager oversees SMO and bottle preparation functions.

Information Technology (IT) – IT staff is responsible for the administration of the Laboratory Information Management System (LIMS) and other necessary support services. Other functions of the IT staff include laboratory network maintenance, IT systems development and implementation, education of analytical staff in the use of scientific software, Electronic Data Deliverable (EDD) support, and data back-up, archival and integrity operations.

3.2 Ethics, Professional Conduct and Data Integrity

One of the most important aspects of the success of ALS – Kelso is the emphasis placed on the integrity of the data provided and the services rendered. This success is reliant on both the professional conduct of all employees within ALS – Kelso as well as established laboratory practices. All personnel involved with environmental testing and calibration activities must familiarize themselves with the quality documentation and implement the policies and procedures in their work.

All employees are required to sign and adhere to the requirements set forth in the *ALS Code of Conduct Policy* and agree to the *Confidentiality Agreement (*Appendix C).

3.2.1 Professional Conduct

To promote quality ALS – Kelso requires certain standards of conduct and ethical performance among employees. The following examples of documented ALS policy are representative of these standards, and are not intended to be limiting or all-inclusive:

- Under no circumstances is the willful act of fraudulent manipulation of analytical data condoned. Such acts are to be reported immediately to senior management for appropriate corrective action.
- Unless specifically required in writing by a client, alteration, deviation or omission of written contractual requirements is not permitted. Such changes must be in writing and approved by senior management.

- Falsification of data in any form will not be tolerated. While much analytical data is subject to professional judgment and interpretation, outright falsification, whenever observed or discovered, will be documented, and appropriate remedies and punitive measures will be taken toward those individuals responsible.

3.2.2 Confidentiality

It is the responsibility of all laboratory employees to safeguard sensitive company information, client data, records, and information; and matters of national security concern should they arise. The nature of our business and the well-being of our company and of our clients is dependent upon protecting and maintaining confidential and/or proprietary company and client information. All information, data, and reports (except that in the public domain) collected or assembled on behalf of a client is treated as confidential.

Information may not be given to third parties without the consent of the client. Unauthorized release of confidential information about the company or its clients is taken seriously and is subject to formal disciplinary action. All employees sign a confidentiality agreement upon hire to protect the company and client's confidentiality and proprietary rights.

3.2.3 Prevention and Detection of Improper, Unethical or Illegal Actions

It is the intention of ALS – Kelso to proactively prevent and/or detect any improper, unethical or illegal action conducted within the laboratory. This is performed by the implementation of a program designed for not only the detection but also prevention. Prevention consists of educating all laboratory personnel in their roles and duties as employees, company policies, inappropriate practices, and their corresponding implications as described here.

In addition to education, appropriate and inappropriate practices are included in SOPs such as manual integration, data review and specific method procedures. Electronic and hardcopy data audits are performed regularly, including periodic audits of chromatographic electronic data. Requirements for internal QA audits are described in the SOP *Internal Audits* (CE-QA001). All aspects of this program are documented and retained on file according to the company policy on record retention.

The ALS Employee Handbook also contains information on the ALS ethics and data integrity program, including mechanisms for reporting and seeking advice on ethical decisions.

3.2.4 Laboratory Data Integrity, Ethics, and Computer Security Training

Each employee receives data integrity and ethics training on an annual basis. The topics covered and training participation are documented. It is the responsibility of the QAM to ensure that the training is conducted as described. Additionally, new employees are given a QA and data integrity/ethics orientation within the first month of hire, followed by the routine annual training.

Key topics covered are the organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting, record keeping, and reporting data integrity issues. Training includes discussion regarding all data integrity procedures, data integrity training documentation, in-depth data monitoring and data integrity procedures. Training topics also cover examples of improper actions, legal and liability implications (company

and personal), causes, prevention, awareness, and reporting options. Computer security is also included, covering ALS computing security awareness, passwords and access, and related topics.

Trainees are required to understand that any infraction of the laboratory data integrity procedures will result in an investigation that could lead to serious consequences including immediate termination, or civil/criminal prosecution.

3.2.5 Management and Employee Commitment

ALS – Kelso makes every attempt to ensure that employees are free from any commercial, financial, or other undue pressures that might affect their quality of work. Related policies are described in the ALS Employee Handbook. This includes:

- ALS Open Door Policy (ALS Employee Handbook) – Employees are encouraged to bring any work related problems or concerns to the attention of local management or their Human Resources representative. However, depending on the extent or sensitivity of the concern, employees are encouraged to directly contact any member of upper management.
- ALS Integrity Hotline – An anonymous and confidential reporting system available to all employees that is used to communicate misconduct and other concerns. The program shall help minimize negative morale, promote a positive work place, and encourage reporting suspected misconduct without retribution. Associated upper management is notified and the investigations are documented.
- Use of flexible work hours. Within reason and as approved by supervisors, employees are allowed flexible work hours in order to help ease schedule pressures which could impact decision-making and work quality.
- Operational and project scheduling assessments are continually made to ensure that project planning is performed and that adequate resources are available during anticipated periods of increased workloads. Procedures for subcontracting work are established, and within the ALS Environmental laboratory network additional capacity is typically available for subcontracting, if necessary.
- Gifts and Favors (ALS Employee Handbook) – To avoid possible conflict of interest implications, employees do not give unusual gifts or favors to, nor accept such gifts or favors from, persons outside the Company who are, or may be, in any way concerned with the projects on which the Company is professionally engaged.

Table 3-1
Summary of Technical Experience and Qualifications - Key Personnel

Personnel	Years of Experience	Project Role
Jeff Grindstaff, B.S.	26	Laboratory Director
Carl Degner, M.S.	31	Quality Assurance Manager
Kurt Clarkson	8	Client Services Manager
Jeff Coronado, B.S.	25	Metals Department Manager
Harvey Jacky, B.S.	26	General Chemistry Department Manager
Loren Portwood, B.S.	26	Organics Department Manager
Les Kennedy, B.A.	24	Support Services Manager
Eileen Arnold, B.A.	33	Environmental Health and Safety Officer
Joe Caulfield	16	Information Technology

4) Document Control

Procedures for control and maintenance of documents are described in SOP *Document Control* (CE-CEN005). The requirements of the SOP apply to all laboratory logbooks (standards, maintenance, run logbooks, etc.), certificates of analysis, SOPs, QAMs, quality assurance project plans (QAPPs), Environmental Health & Safety (EHS) manuals, and other controlled ALS Environmental documents.

Each controlled copy of a controlled document is released after a document control number is assigned and the recipient is recorded on a document distribution list. Filing and distribution is performed by the QAM, or designee, and ensure that only the most current version of the document is distributed and in use. A document control number is assigned to logbooks. Completed logbooks that are no longer in use are archived in a master logbook file. Logbook entries are standardized following SOP *Making Entries onto Analytical Records* (CE-QA007). The logbook entries are reviewed and approved at a regular interval (quarterly).

A records system is used which ensures all laboratory records (including raw data, reports, and supporting records) are retained and available. The archiving system is described in SOP *Data Archiving* (ADM-ARCH).

External documents relative to the management system are managed by the QAM. To prevent the use of invalid and/or outdated external documents, the laboratory maintains a master list of current documents and their availability. The list is reviewed before making the documents available. External documents are not issued to personnel.

5) Review of Requests, Tenders and Contracts

Requests for new work are reviewed prior to signing any contracts or otherwise agreeing to perform the work. The specific methods to be used are agreed upon between the laboratory and the client. A capability review is performed to determine if the laboratory has or needs to obtain certification to perform the work, to determine if the laboratory has the resources (personnel, equipment, materials, capacity, skills, expertise) to perform the work, and if the laboratory is able to meet the client's required reporting and QC limits. The results of this review are communicated to the client and any potential conflict, deficiency, lack of appropriate accreditation status, or concerns of the ability to complete the client's work are resolved. Any differences between the request or tender and the contract shall be resolved before any work commences. The client should be notified at this time if work is expected to be subcontracted. Each contract shall be acceptable both to the laboratory and the client. Records are maintained of pertinent discussions with a client relating to the client's requirements or the results of the work. If a contract needs to be amended after work has commenced, the contract review process is repeated and any amendments are communicated to all affected personnel. Changes in accreditation status affecting ongoing projects must be reported to the client.

6) Subcontracting of Tests

Analytical services are subcontracted when the laboratory needs to balance workload or when the requested analyses are not performed by the laboratory. Subcontracting is only done with the knowledge and approval of the client and to qualified laboratories. Subcontracting to another ALS Environmental Group laboratory is preferred over external-laboratory subcontracting. Further, subcontracting is done using capable and qualified laboratories. Established procedures are used to qualify external subcontract laboratories. These procedures are described in SOP *Qualification of Subcontract Laboratories* (CE-QA004). The Quality Assurance staff is responsible for maintaining a list of qualified subcontract laboratories.

7) Purchasing Services and Supplies

The quality level of reagents and materials (grade, traceability, etc.) required is specified in analytical SOPs. Department supervisors ensure that the proper materials are purchased. Inspection and verification of material ordered is performed at the time of receipt by receiving personnel. The receiving staff labels the material with the date received. Expiration dates are assigned as appropriate for the material. Storage conditions and expiration dates are specified in the analytical SOP. *Quality of Reagents and Standards* (CE-QA012) and Reagent and *Standards Login and Tracking* (ADM-RLT) provides default expiration requirements. Supplies and services that are critical in maintaining the quality of laboratory testing are procured from pre-approved vendors. The policy and procedure for purchasing and procurement are described in SOP *Procurement and Control of Laboratory Services and Supplies* (CE-CEN007).

Receipt procedures include technical review of the purchase order/request to verify that what was received is identical to the item ordered. The laboratory checks new lots of reagents for unacceptable levels of contamination prior to use in sample preservation, sample preparation, and sample analysis by following SOP *Reagent and Standards Login and Tracking* (ADM-RLT).

8) Service to the Client

ALS - Kelso utilizes a number of processes to ensure that adequate resources exist to meet service demands. Senior staff meetings, tracking of outstanding proposals, and a current synopsis of incoming work all assist the senior staff in properly allocating sufficient resources. Status/production meetings are conducted regularly with the laboratory and Project Managers to inform the staff of the status of incoming work, future projects, or project requirements.

The Project Manager is a scientist assigned to each client to act as a technical liaison between the client and the laboratory. The Project Manager is responsible for ensuring that the analyses performed by the laboratory meet all project and contract requirements. This entails coordinating with the laboratory staff to ensure that client-specific needs are understood and that the services provided are properly executed and satisfy the requirements of the client.

Laboratory management also monitors a number of other indicators to assess the overall ability of the laboratory to successfully perform analyses for its clients. This includes on-time performance, customer complaints, training reports and non-conformity reports. A frequent assessment is made of the laboratory's facilities and resources in anticipation of accepting an additional or increased workload.

All Requests for Proposal (RFP) documents are reviewed by the Project Manager and appropriate managerial staff to identify any project specific requirements that differ from the standard practices of the laboratory. Any requirements that potentially cannot be met are noted and communicated to the client, as well as requesting the client to provide any applicable project specific Quality Assurance Project Plans (QAPPs).

When a client requests a modification to an SOP, policy, or standard specification the Project Manager will discuss the proposed deviation with the Client Services Manager, Laboratory Director, and department manager to obtain approval for the deviation. The QAM may also be involved. All project-specific requirements must be on-file and with the service request upon logging in the samples. The modification or deviation must be documented. A Project-Specific Communication Form, Form V, or similar, may be used to document such deviations.

The laboratory affords clients cooperation to clarify the client's request and to monitor the laboratory's performance in relation to the work performed, provided that the laboratory ensures confidentiality to other clients. The laboratory maintains and documents timely communication with the client for the purposes of seeking feedback and clarifying customer requests. Feedback is used and analyzed to improve the quality of services. The SOP *Handling Customer Feedback* (CE-CEN010) is in place for these events.

9) Complaints

In addition to project communication and internal communication of data issues, the laboratory also maintains a system for dealing with customer complaints. The procedure is described in SOP *Handling Customer Feedback* (CE-GEN010). The person who initially receives feedback in the form of a complaint (typically the Project Manager) is responsible for documenting the complaint. If the Project Manager is unable to satisfy the customer, the complaint is brought to the attention of the Client Services Manager, Laboratory Director, or QAM for final resolution. The complaint and resolution are documented.

10) Facilities and Equipment

The ALS Environmental Kelso laboratory features over 45,000 square feet of laboratory and administrative workspace. The laboratory has been designed and constructed to provide safeguards against cross-contamination of samples and is arranged according to work function, which enhances the efficiency of analytical operations. The ventilation system has been specially designed to meet the needs of the analyses performed in each work space. Also, ALS - Kelso minimizes laboratory contamination sources by employing janitorial and maintenance staff to ensure that good housekeeping and facilities maintenance are performed. In addition, the segregated laboratory areas are designed for safe and efficient handling of a variety of sample types. These specialized areas (and access restrictions) include:

- Shipping and Receiving/Purchasing
- Sample Management Office, including controlled-access sample storage areas
- Inorganic/Metals Sample Preparation Laboratories (2)
- Inorganic/Metals "clean room" sample preparation laboratory
- ICP-AES Laboratory
- ICP-MS Laboratory
- Low-level Mercury Laboratory
- Water Chemistry & General Chemistry Laboratories (3)
- Semi-volatile Organics Sample Preparation Laboratory
- Gas Chromatography and High Performance Liquid Chromatography Laboratories
- Gas Chromatography/Mass Spectrometry Laboratories (2)
- Semi-volatile Organics Drinking Water Laboratory
- Volatile Organics Laboratory
    - Separate sample preparation laboratory
    - Access by semi-volatile sample preparation staff only after removing lab coat and solvent-contaminated gloves, etc.
- Microbiology Laboratory
- Laboratory Deionized Water Systems (2)
- Laboratory Management, Client Service, Report Generation and Administration
- Data Archival, Data Review and support functions areas

In addition, the designated areas for sample receiving, refrigerated sample storage and dedicated sample container preparation and shipping areas provide for the efficient and safe

handling of a variety of sample types. The laboratory is equipped with state-of-the-art analytical and administrative support equipment. The equipment and instrumentation are appropriate for the procedures in use. Refer to Appendix D for a Laboratory Floor Plan and Appendix E for a list of major equipment, illustrating the laboratory's overall capabilities and depth.

11) Sample Management

11.1 Sampling and Sample Preservation

The quality of analytical results is highly dependent upon the quality of the procedures used to collect, preserve and store samples. ALS - Kelso recommends that clients follow sampling guidelines described in 40 CFR 136, 40 CFR 141, USEPA SW 846, and state-specific sampling guidelines, if applicable. Sampling factors that must be taken into account to insure accurate, defensible analytical results include:

- Amount of sample taken
- Type of container used
- Type of sample preservation
- Sample storage time
- Proper custodial documentation

The laboratory uses the sample preservation, container, and holding-time recommendations published in a number of documents. The primary documents of reference are: USEPA SW-846, Third Edition and Updates I, II, IIA, IIB, III, IV for hazardous waste samples; USEPA 600/4-79-020, 600/4-91-010, 600/4-82-057, 600/R-93/100, 600/4-88-039, 600/R-94-111, and Supplements; EPA 40CFR parts 136 and 141 and associated Method Update Rules; and Standard Methods for the Examination of Water and Wastewater for water and wastewater samples (see Section 23 for complete references). The container, preservation and holding time information for these references is summarized in Appendix F for soil, water, and drinking water. The current EPA CLP Statement of Work should be referred to for CLP procedures. Where allowed by project sampling and analysis protocols (such as Puget Sound Protocols) the holding time for sediment, soil, and tissue samples may be extended for a defined period when stored frozen at -20°C.

ALS - Kelso provides clients with sample containers with applicable preservatives. Containers are purchased as pre-cleaned to a level 1 status, and conform to the requirements for samples established by the USEPA. Certificates of analysis for sample containers are available upon request. Reagent water used for sampling blanks (trip blanks, etc.) and chemical preservation reagents are tested by the laboratory to ensure that they are free of interferences and documented. Our sample kits typically consist of pre-cleaned, rinsed, and air-dried shipping coolers with foam liners, specially prepared and labeled sample containers individually wrapped in protective material (VOC vials are placed in a specially made foam holder), chain-of-custody (COC) forms, and custody seals. Container labels and custody seals are provided for each container. Figure 11-1 shows the chain-of-custody form routinely used at ALS - Kelso and included with sample kits. Dry ice or gel ice is the only temperature preservative used. For large sample container shipments the containers may be shipped in their original boxes. Such shipments will consist of labeled and preserved sample containers and sufficient materials (bubble wrap, COC forms, custody seals, shipping coolers, etc.) for return to ALS, unless otherwise instructed by the client.

ALS – Kelso also provides courier service that makes regularly scheduled trips on the I-5 corridor between the Greater Portland, Oregon area and the Great Seattle/Tacoma area, and nearby communities and facilities.

Returning shipping coolers are cleaned and decontaminated. If any such cooler exhibits an odor or other abnormality after receipt and cleaning, a more vigorous decontamination process is employed. Containers which cannot be decontaminated are discarded. ALS – Kelso keeps client-specific shipping requirements on file and utilizes major transportation carriers to necessary to meet sample shipping requirements (same-day, overnight, etc.).

When ALS – Kelso ships samples to other laboratories for analysis, similar sample integrity processes are used to ensure preservation and proper sample handling, and to avoid any possible breakage, cross-contamination of samples, or identification problems. Alternatively, the receiving laboratory's procedures may be specified. Chain of custody is maintained during the process.

11.2 Sample Receipt and Handling

Standard procedures are established for the receiving of samples into the laboratory and are found in SOP SMO-CEN, *Sample Receiving*. These procedures ensure that samples are received and properly logged into the laboratory, and that all associated documentation, including chain of custody forms, is complete and consistent with the samples received.

Once samples are received or delivered to the laboratory the sample management office uses a Cooler Receipt and Preservation Check Form (CRF – Figure 11-2) is used to assess the shipping cooler and its contents as received by the laboratory. Any anomalies or discrepancies observed during the initial assessment are recorded on the CRF and COC documents. Verification of sample integrity includes the following activities:

- Assessment of custody seal presence/absence, location and signature;
- Temperature of sample containers upon receipt;
- Chain of custody documents properly used (entries in ink, signature present, etc.);
- Sample containers checked for integrity (broken, leaking, etc.);
- Sample is clearly marked and dated (bottle labels complete with required information);
- Appropriate containers (size, type) are received for the requested analyses;
- The minimum amount of sample material is provided for the analysis.
- Sample container labels and/or tags agree with chain of custody entries (identification, required analyses, etc.);
- Assessment of proper sample preservation (if inadequate, corrective action is employed); and
- VOC containers are inspected for the presence/absence of bubbles. (Assessment of proper preservation of VOC containers is performed by lab personnel).

Samples are logged into a Laboratory Information Management System (LIMS). Potential problems with a sample shipment are addressed by contacting the client and discussing the pertinent issues. When the Project Manager and client have reached a satisfactory resolution, the login process may continue and analysis may begin. During

the login process each sample container is given a unique laboratory code and a Service Request form is generated which contains client information, sample descriptions, sample matrix information, required analyses, sample collection dates, analysis due dates and other pertinent information. The service request is reviewed by the applicable Project Manager for accuracy and completeness.

Samples are stored as per method requirements until analysis, unless otherwise specified, using various refrigerators, freezers, or designated secure areas. ALS - Kelso has multiple walk-in and refrigerator cold storage units which house the majority of samples, including dedicated refrigerated storage of VOC samples. The VOC storage units are monitored using storage blanks as described in SOP VOC-BLAN, *VOA Storage Blanks*. ALS - Kelso also has multiple sub-zero freezers capable of storing samples at -10 to -30°C primarily used for tissue and sediment samples. The temperature of each sample storage unit is monitored real time with an electronic temperature monitoring system.

ALS - Kelso adheres to the method-prescribed or project-specified holding times for all analyses. Analysts monitor holding times by obtaining analysis-specific reports from the LIMS. These reports provide holding time information on all samples for the analysis, calculated from the sampling date and the holding time requirement. To document holding time compliance, the date and time analyzed is printed or written on the analytical raw data. Unless other arrangements have been made in advance, upon completion of all analyses and submittal of the final report, aqueous samples are retained at ambient temperature for 30 days, soil samples are retained at ambient temperature for 60 days, and tissue samples are retained frozen for 3 months. Upon expiration of these time limits, the samples are either returned to the client or disposed of according to approved disposal practices. Sample extracts are retained as specified in analytical SOPs. All samples are characterized according to hazardous/non-hazardous waste criteria and are segregated accordingly. All hazardous waste samples are disposed of according to formal procedures outlined in the ALS Environmental Health and Safety Manual and in accordance with applicable laws. Documentation is maintained for each sample from initial receipt through final disposal to ensure that an accurate history of the sample from "cradle to grave" is available.

11.3 Sample Custody

Sample custody transfer at the time of sample receipt is documented using chain-of-custody (COC) forms accompanying the samples. During sample receipt, it is also noted if custody seals were present.

Facility security and access is important in maintaining the integrity of samples received at ALS - Kelso. Access to the laboratory facility is limited by use of locked exterior doors with a coded/card entry, except for the reception area and sample receiving doors, which are staffed during business hours and locked at all other times. In addition, the sample storage area within the laboratory is a controlled access area with locked doors with a coded entry. The facility is equipped with an alarm system and the laboratory employs a private security firm to provide nighttime and weekend security.

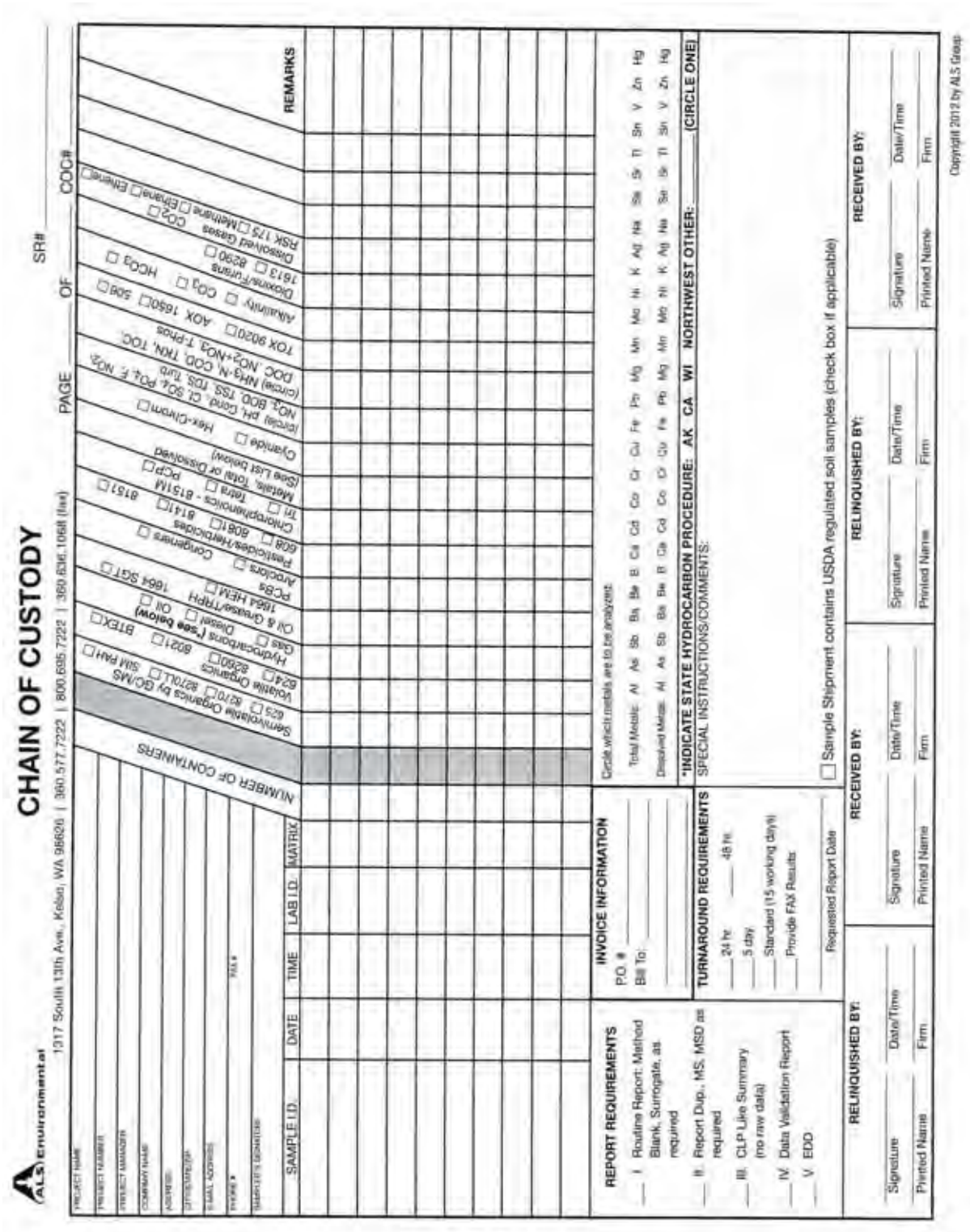
A barcoding system is used to document internal sample custody. Each person removing or returning samples from/to sample storage is required to document this custody transfer (via custodian or directly). The system uniquely identifies sample containers and provides an electronic record of the sample custody. Procedures are also defined for sample extracts, digestates, and leachates. The procedures are described in the SOP SMO-SCOC, *Sample Tracking and Internal Chain of Custody*.

11.4 Project Setup

The analytical method(s) used for sample analysis are chosen based on the client's requirements. LIMS codes are chosen to identify the analysis method used for analysis. The Project Manager ensures that the correct methods are selected for analysis, deliverable requirements are identified, and due dates are specified on the Service Request. For SW-846 methods, some projects may require the most recent promulgated version, and some projects may require the most recent published version. The Project Manager will ensure that the correct method version is used. Functionality incorporated in the LIMS is used to communicate and specify project-specific requirements and demographics, including the use of attachments to LIMS delivery group (SDG or SR) such as specification forms, analyte lists, deliverable requirements, and other pertinent information.

Figure 11-1

ALS Environmental Standard Chain of Custody Form



Cooler Receipt and Preservation Form

Client _____ Service Request *K16* _____

Thermometer ID	Corr. Factor	@20 min. Raw Blank	@20 min. Corr. Blank	@40 min. Raw Blank	@40 min. Corr. Blank	@60 min. Raw Blank	@60 min Corr. Blank

Sample ID on Bottle	Sample ID on COC	Identified by:

Sample ID	Bottle Count Bottle Type	Out of Temp	Head-space	Broke	pH	Reagent	Volume added	Reagent Lot Number	Initials	Time

Notes, Discrepancies & Resolutions:

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Cooler Receipt and Preservation Form

Client _____ Service Request *K16* _____

Notes, Discrepancies & Resolutions:

7/25/16

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12) Analytical Procedures

ALS - Kelso employs methods and analytical procedures from a variety of external sources. The primary method references are: USEPA SW-846, Third Edition and Updates I, II, IIA, IIB, III, IVA, IVB, and online updates for hazardous waste samples, and USEPA 600/4-79-020, 600/4-91-010, 600/4-82-057, 600/R-93/100, 600/4-88-039, 600/R-94-111, EPA 40CFR parts 136 and 141 and associated Method Update Rules and Supplements; Standard Methods for the Examination of Water and Wastewater for water and wastewater samples, and American Society for Testing and Materials (ASTM). Complete citations for these references can be found in Section 23. Other published procedures, such as state-specific methods, program-specific methods (such as Puget Sound Protocols), or in-house methods may be used. Several factors are involved with the selection of analytical methods to be used in the laboratory. These include the method detection/reporting limit, the expected concentration of the analyte(s) being measured, method selectivity, accuracy and precision of the method, the type of sample being analyzed, and the regulatory compliance objectives. The implementation of methods by ALS - Kelso is described in SOPs specific to each method. A list of NELAP-accredited methods is given in Appendix J.

12.1 Standard Operating Procedures (SOPs) and Laboratory Notebooks.

ALS Environmental, Kelso maintains SOPs for use in both technical and administrative functions. SOPs are written following standardized format and content requirements as described in SOP *Preparation of Standard Operating Procedures* (CE-GEN009). Each SOP is reviewed and approved by a minimum of two managers (the Laboratory Director and/or Department Manager and the Quality Assurance Manager). All SOPs undergo a documented review to make sure current practices are described. The QAM maintains a comprehensive list of current SOPs. The document control process ensures that only the most currently approved version of an SOP is being used. The procedures for document control are described in SOP *Document Control* (CE-GEN005). In addition to SOPs, each laboratory department maintains the current methods used to perform analyses accessible to all laboratory staff. Laboratory notebook entries are standardized using the procedure in SOP *Making Entries onto Analytical Records* (CE-QA007). Laboratory notebook entries are reviewed and approved by the appropriate supervisor at a regular interval. A list of current SOPs is given in Appendix G.

12.2 Deviation from Standard Operating Procedures

When a client requests a modification to an SOP (such as a change in reporting limit, addition or deletion of target analyte(s), etc.), the Project Manager handling that project must discuss the proposed deviation with the department manager in charge of the analysis and obtain their approval to accept the project. The Project Manager is responsible for documenting the approved or allowed deviation from the SOP by placing a description of the deviation attached with the project documents and also providing an instructional comment with the Service Request.

For circumstances when a deviation or departure from company policies or procedures involving any non-technical function is found necessary, approval must be obtained from the appropriate supervisor, manager, the Laboratory Director, or other level of authority. Frequent departure from policy is not encouraged. However, if frequent departure from any policy is noted, the laboratory director will address the possible need for a change in policy.

12.3 Modified Procedures

ALS - Kelso strives to perform published methods as described in the referenced documents. If there is a material deviation from the published method, the method is cited as a "Modified" method in the analytical report. Modifications to the published methods are listed in the standard operating procedure. Standard operating

procedures are available to analysts and are also available to our clients for review. Client approval is obtained for the use of "Modified" methods prior to the performance of the analysis.

12.4 Analytical Batch

The basic unit for analytical quality control is the analytical batch. The definition that ALS – Kelso has adopted for the analytical batch is listed below. The overriding principle for describing an analytical batch is that all the samples in a batch, both field samples and quality control samples are to be handled exactly the same way, and all of the data from each analysis is to be manipulated in exactly the same manner. The minimum requirements of an analytical batch are:

1) The number of (field) samples in a batch is not to exceed 20.

2) All (field) samples in a batch are of the same matrix.

3) The QC samples to be processed with the (field) samples include:

- Method Blank (a.k.a. Laboratory Reagent Blank)
- Laboratory Control Sample
- Matrix Spiked (field) Sample (a.k.a. Laboratory Fortified Sample Matrix)*
- Duplicate Matrix Spiked (field) Sample or Duplicate (field) Sample (a.k.a. Laboratory Duplicate)*

* A sample identified as a field blank, an equipment blank, or a trip blank is not to be matrix spiked or duplicated.

4) A single lot of reagents is used to process the batch of samples.

5) Each operation within the analysis is performed by a single analyst, technician, chemist, or by a team of analysts/technicians/chemists.

6) Samples are analyzed in a continuous manner over a timeframe not to exceed 24-hours between the start of processing of the first and last sample of the batch.

7) Samples are analyzed in a continuous manner over a timeframe not to exceed 24-hours.

8) Field samples are assigned to batches commencing at the time that sample processing begins.

9) The QC samples are to be analyzed in conjunction with the associated field samples prepared with them. However, for tests which have a separate sample preparation step that defines a batch (digestion, extraction, etc.), the QC samples in the batch do not require analysis each time a field sample within the preparation batch is analyzed (multiple instrument sequences to analyze all field samples in the batch need not include re-analyses of the QC samples).

10) The batch is to be assigned a unique identification number that can be used to correlate the QC samples with the field samples.

11) Batch QC refers to the QC samples that are analyzed in a batch of (field) samples.

12) Project-specific requirements may be exceptions. If project, program, or method requirements are more stringent than these laboratory minimum requirements, then the project, program, or method requirements will take precedence. However, if the project, program, or method requirements are less

stringent than these laboratory minimum requirements, these laboratory minimum requirements will take precedence.

12.5 Specialized Procedures

ALS – Kelso not only strives to provide results that are scientifically sound, legally defensible, and of known and documented quality; but also strives to provide the best solution to analytical challenges. Procedures using specialized instrumentation and methodology have been developed to improve sensitivity (provide lower detection limits), selectivity (minimize interferences while maintaining sensitivity), and overall data quality for low concentration applications. Examples are trace-level Mercury and Methyl Mercury analyses, reductive precipitation metals analysis, leaching procedures, incremental sampling protocols, specialized GC/MS analyses, LC/MS analyses, and ultra-low level organics analyses (including PAHs, pesticides and PCBs); including those for emerging contaminants of concern.

12.6 Sample Cleanup

The laboratory commonly employs several cleanup procedures to minimize known common interferences prior to analysis. EPA methods (3620, 3630, 3640, 3660, and 3665) for cleanup of sample extracts for organics analysis are routinely used to minimize or eliminate interferences that may adversely affect sample results and data usability.

13) Measurement Traceability and Calibration

All equipment and instruments used at ALS – Kelso are operated, maintained and calibrated according to the manufacturer's recommendations and criteria set forth in the analytical methods. All analytical measurements generated are performed using materials that are traceable to a reference material, unless unavailable. Documentation of calibration information is maintained in appropriate reference files. Brief descriptions of the calibration procedures for our major laboratory equipment are described below. Calibration verification is performed according to the analytical methods and SOPs, and criteria are listed in the SOPs. Documentation of calibration verification is maintained in appropriate reference files. Records are maintained to provide traceability of reference materials and reference equipment.

Laboratory support equipment (thermometers, balances, and weights) are routinely verified on an annual basis by a vendor accredited to ISO/IEC 17025:2005, or more frequently if program-specified. Metrology equipment (analytical balances, thermometers, etc.) is calibrated using reference materials traceable to the National Institute of Standards and Technology (NIST). These primary reference materials are themselves recertified on an annual basis. Vendors used for metrology support are required to verify compliance to International Standards by supplying the laboratory with a copy of their scope of accreditation.

Equipment shown by verification to be malfunctioning or defective is taken out of service until it is repaired. When an instrument is taken out of service, an Out of Service sign is placed by the laboratory on the instrument. The equipment is placed back in service only after verifying, by calibration, that the equipment performs satisfactorily.

13.1 Temperature Control Devices

Temperatures are monitored and recorded each day for all of the temperature-regulating support equipment such as sample refrigerators, freezers, and standards refrigerators/freezers. Temperatures are recorded in either laboratory logbook or through Check Point® Wireless Monitoring System. During weekends and holidays a min/max thermometer may be used.

Laboratory records contain the recorded temperature, identification and location of equipment, acceptance criteria and the initials of the technician who performed the

checks. The procedure for performing these measurements is provided in the SOP *Support Equipment Monitoring and Calibration* (ADM-SEMC).

Where the operating temperature is specified as a test condition (such as ovens, incubators, evaporators) the temperature is recorded on the raw data. All thermometers are identified according to serial number, and the calibration is checked annually against a National Institute of Standards and Technology (NIST) certified thermometer. The NIST thermometer is recertified by a vendor accredited to ISO/IEC 17025:2005 on an annual basis.

13.2 Analytical Balances

The calibration of each analytical balance is checked by the user each day of use with three Class S or S-1 weights, which assess the accuracy of the balance at low, mid-level and high levels bracketing the working range. Records are kept which contain the recorded measurements, identification of the balance, acceptance criteria, and the initials of user who performed the check. The procedure for performing these measurements and use of acceptance criteria is described in the SOP *Support Equipment Monitoring and Calibration* (ADM-SEMC). The weights are recertified using NIST traceable standards by an accredited metrology organization on an annual basis. As needed, the balances are recalibrated using the manufacturers recommended operating procedures. Analytical balances are serviced on a semi-annual basis by an accredited metrology organization.

13.3 Water Purification Systems

ALS - Kelso uses two independent water purification systems is designed to produce deionized water meeting method specifications. One system consists of a series of pumps, filters, and resin beds designed to yield deionized water meeting the specifications of ASTM Type II water, and Standard Methods for the Examination of Water and Wastewater (SM1080, 20th Ed.) High Quality water. Activated carbon filters are also in series with the demineralizers to produce "organic-free" water. A second system consists of pumps, filters, and treatment components designed to yield deionized water meeting the specifications of ASTM Type I water, and Standard Methods for the Examination of Water and Wastewater (SM1080, 20th Ed.) High Quality water. The status of each system is monitored continuously for conductivity and resistivity with an on-line meter and indicator light, and readings recorded daily. The meter accuracy is verified annually. Deionizers are rotated and replaced on a regular schedule. Microbiology water is checked on a daily basis at a point downstream of the purification system at a tap in the laboratory.

13.4 Standards and Reference Materials

Consumable reference materials routinely purchased by the laboratories (e.g., analytical standards) are purchased from nationally recognized, reputable vendors. All vendors where possible have fulfilled the requirements for 9001 certification and/or are ISO 17025 accredited. ALS - Kelso relies on a primary vendor for the majority of its analytical supplies. Consumable primary stock standards are obtained from certified commercial sources or from sources referenced in a specific method. Supelco, Ultra Scientific, AccuStandard, Chem Services, Inc., Aldrich Chemical Co., Baker, Spex, etc. are examples of the vendors used. Reference material information is recorded in the appropriate logbook(s) and materials are stored under conditions that provide maximum protection against deterioration and contamination. The logbook entry includes such information as an assigned logbook identification code, the source of the material (i.e. vendor identification), solvent (if applicable) and concentration of analyte(s), reference to the certificate of analysis and an assigned expiration date. The date that the standard is received in the laboratory is marked on the container. When

the reference material is used for the first time, the date of usage and the initials of the analyst are also recorded on the container.

Stock solutions and calibration standard solutions are prepared fresh as often as necessary according to their stability. All standard solutions are properly labeled as to analyte concentration, solvent, date, preparer, and expiration date; these entries are also recorded in the appropriate notebook(s) following the SOP for *Reagent Login and Tracking* (ADM-RTL). Prior to sample analysis, all calibration reference materials are verified with a second, independent source of the material.

13.5 Inductively Coupled Plasma-Atomic Emission Spectrograph (ICP-AES)

Each emission line on the ICP is calibrated daily against a blank and against standards whose concentrations fall within the instruments linear range. Analyses of calibration standards, initial and continuing calibration verification standards, and inter-element interference check samples are carried out as specified in the applicable method SOP and analytical method (i.e. EPA 200.7, 6010B, 6010C, CLP SOW, etc.).

13.6 Inductively Coupled Plasma-Mass Spectrometer (ICP-MS)

Each element of interest is calibrated for using a blank and a single standard. Prior to calibration, a short-term stability check is performed on the system. Following calibration, an independent check standard is analyzed, and a continuing calibration verification standard (CCV) is analyzed with every ten samples.

13.7 Atomic Absorption Spectrophotometers (AAS)

These instruments are calibrated daily using a minimum of four standards and a blank. Calibration is validated using reference standards, and is verified at a minimum frequency of once every ten samples. Initial calibration points cannot be "dropped" from the resulting calibration curve.

13.8 GC/MS Systems

All GC/MS instruments are calibrated at multiple concentration levels for the analytes of interest (unless specified otherwise) using procedures outlined in Standard Operating Procedures and/or appropriate USEPA method citations. All reference materials used for this function are vendor-certified standards. Calibration verification is performed at method-specified intervals following the procedures in the SOP. For internal standard and isotope dilution procedures, the internal standard response and/or labeled compound recovery must meet method criteria. Method-specific instrument tuning is regularly checked the method-specified compounds. Mass spectra for the tuning compounds must meet method/SOP criteria before analyses can proceed. Calibration policies for organics chromatographic analyses are described in the SOP *Calibration of Instruments for Organics Chromatographic Analyses* (SOC-CAL).

13.9 Gas Chromatographs and High Performance Liquid Chromatographs

Calibration and standardization follow SOP guidelines and/or appropriate USEPA method citations. All GC and HPLC instruments are calibrated at a minimum of five different concentration levels for the analytes of interest (unless specified otherwise). The lowest standard is equivalent to the method reporting limit; additional standards define the working range of the GC or LC detector. Results are used to establish response factors (or calibration curves) and retention-time windows for each analyte. Calibration is verified at a minimum frequency of once every ten samples, unless otherwise specified by the reference method. Calibration policies for organics chromatographic analyses are described in the SOP *Calibration of Instruments for Organics Chromatographic Analyses* (SOC-CAL).

LC/MS Systems:

Calibration and tuning procedures are included in analytical SOPs written specifically for these tests. In general, multiple concentration levels for the analytes of interest are used to generate calibration curves. All reference materials used for this function are vendor-certified standards. Calibration and tuning verification is performed at SOP-defined intervals. Any other system performance checks are described in the applicable SOP. Calibration policies for organics chromatographic analyses are described in the SOP *Calibration of Instruments for Organics Chromatographic Analyses* (SOC-CAL).

13.10 UV-Visible Spectrophotometer (manual colorimetric analyses)

Routine calibrations for colorimetric and turbidimetric analyses involve generating a 5 point calibration curve including a blank. Initial calibration points cannot be "dropped" from the resulting calibration curve. Correlation coefficients must meet method or SOP specifications before analysis can proceed. Independent calibration verification standards (ICVs) are analyzed with each batch of samples. Continuing calibration is verified at a minimum frequency of once every ten samples. Typical UV-Visible spectrophotometric methods at ALS Environmental, Kelso include total phenolics, phosphates, surfactants and tannin-lignin.

13.11 Flow Injection Analyzer (automated colorimetric analysis)

A minimum of six standards and a blank are used to calibrate the instrument for cyanide analysis. A blank and (minimum of) five standards are used to calibrate the instrument for all other automated chemistries. Initial calibration points cannot be "dropped" from the resulting calibration curve. Standard ALS Environmental, Kelso acceptance limits are used to evaluate the calibration curve prior to sample analysis.

13.12 Discrete Auto-Analyzer (automated absorbance analysis)

A minimum of five standards and a blank are used to calibrate the instrument. Initial calibration points cannot be "dropped" from the resulting calibration curve. Method specific acceptance limits are used to evaluate the calibration curve prior to sample analysis.

13.13 Ion Chromatographs

Calibration of the ion chromatograph (IC) involves generating a calibration curve with the method-specified number of points (or more). Initial calibration points cannot be "dropped" from the resulting calibration curve. A correlation coefficient of > 0.995 for the curve is required before analysis can proceed. Quality Control (QC) samples that are routinely analyzed include blanks and laboratory control samples. The target analytes typically determined by the IC include nitrate, nitrite, chloride, fluoride, sulfate and drinking water inorganic disinfection byproducts. Calibration verification is performed at method-specified intervals following the procedures in the SOP and reference method.

13.14 Turbidimeter

Calibration of the turbidimeter requires analysis of three Nephelometric Turbidity Unit (NTU) formazin standards. Quality Control samples that are routinely analyzed include blanks, Environmental Resource Associates QC samples (or equivalent) and duplicates.

13.15 Ion-selective electrode

The method-prescribed numbers of standards are used to calibrate the electrodes before analysis. The slope of the curve must be within acceptance limits before analysis can proceed. Quality Control samples that are routinely analyzed include blanks, LCSs and duplicates.

13.16 Pipets

The calibration of pipets and autopipettors used to make critical-volume measurements is verified following SOP *Checking Volumetric Labware* (ADM-VOLWARE). Both accuracy and precision verifications are performed, at intervals applicable to the pipet and use. The results of all calibration verifications are recorded in bound logbooks.

13.17 Other Instruments

Calibration for the total organic carbon (TOC), total organic halogen (TOX), and other instruments is performed following manufacturer's recommendations and applicable SOPs.

14) Assuring the Quality of Results

A primary focus of ALS - Kelso's QA Program is to ensure the accuracy, precision and comparability of all analytical results. Prior to using a procedure for the analysis on field samples, acceptable method performance is established by performing demonstration of capability analyses. Performance characteristics are established by performing method detection limit studies and assessing accuracy and precision according to the reference method. ALS - Kelso has established Quality Control (QC) objectives for precision and accuracy that are used to determine the acceptability of the data that is generated. These QC limits are either specified in the test methodology or are statistically derived based on the laboratory's historical data. Quality Control objectives are defined below.

14.1 Quality Control Objectives

14.1.1 Demonstration of Capability - A demonstration of capability (DOC) is made prior to using any new test method or when a technician is new to the method. This demonstration is made following regulatory, accreditation, or method specified procedures. In general, this demonstration does not test the performance of the method in real world samples, but in the applicable clean matrix free of target analytes and interferences.

A quality control sample material may be obtained from an outside source or may be prepared in the laboratory. The analyte(s) is (are) diluted in a volume of clean matrix (for analytes which do not lend themselves to spiking, e.g., TSS, the demonstration of capability may be performed using quality control samples). Where specified, the method-required concentration levels are used. Four aliquots are prepared and analyzed according to the test procedure. The mean recovery and standard deviations are calculated and compared to the corresponding acceptance criteria for precision and accuracy in the test method or laboratory-generated acceptance criteria (if there are not established mandatory criteria). All parameters must meet the acceptance criteria. Where spike levels are not specified, actual Laboratory Control Sample results may be used to meet this requirement, provided acceptance criteria is met.

14.1.2 Accuracy - A measure of the closeness of an individual measurement (or an average of multiple measurements) to a true or expected value and expressed as percent recovery (% REC.) of the measured value, relative to the true or expected value. If a measurement process produces results whose mean is not the true or expected value, the process is said to be biased. Bias is the systematic error either inherent in a method of analysis or caused by an artifact of the measurement system (e.g., contamination). Ongoing accuracy is determined by calculating the mean value of results from ongoing analyses of laboratory control sample, standard reference materials, or standard

solutions. In addition, matrix-spiked samples are also measured and recovery indicates the accuracy or bias in the actual sample matrix.

ALS - Kelso utilizes several quality control measures to eliminate analytical bias, including systematic analysis of method blanks, laboratory control samples and independent calibration verification standards. Because bias can be positive or negative, and because several types of bias can occur simultaneously, only the net, or total, bias can be evaluated in a measurement.

14.1.3 Precision - Precision is the ability of an analytical method or instrument to reproduce its own measurement. It is a measure of the variability, or random error, in sampling, sample handling and in laboratory analysis. The American Society of Testing and Materials (ASTM) recognizes two levels of precision: repeatability - the random error associated with measurements made by a single test operator on identical aliquots of test material in a given laboratory, with the same apparatus, under constant operating conditions, and reproducibility - the random error associated with measurements made by different test operators, in different laboratories, using the same method but different equipment to analyze identical samples of test material.

"Within-batch" precision is measured using replicate sample or QC analyses and is expressed as the relative percent difference (RPD) between the measurements. The "batch-to-batch" precision is determined from the variance observed in the analysis of standard solutions or laboratory control samples from multiple analytical batches.

14.1.4 Control Limits - The control limits for accuracy and precision originate from two different sources. For analyses having enough QC data, control limits are calculated at the 99% confidence limits. For analyses not having enough QC data, or where the method is prescriptive, control limits are taken from the method on which the procedure is based. If the method does not have stated control limits, then control limits are assigned method-default or reasonable values based on similar methods. Control limits are reviewed each year and may be updated if new statistical limits are generated for the appropriate surrogate, laboratory control sample, and matrix spike compounds (typically once a year) or when method prescribed limits change. The updated limits are reviewed by the QAM. The new control limits replace the previous limits and data is assessed using the new values. Current *Data Quality Objectives*, including acceptance limits for accuracy and precision are available from the laboratory. For inorganics, the precision limit values listed are for laboratory duplicates. For organics, the precision limit values listed are for duplicate laboratory control samples or duplicate matrix spike analyses. Procedures for establishing control limits are found in SOP *Control Limits* (CE-QA009).

14.1.5 Representativeness - The degree to which the field sample, being properly preserved, free of contamination, and properly analyzed, represents the overall sample site or material. This can be extended to the sample itself, in that representativeness is the degree to which the subsample that is analyzed represents the entire field sample submitted for analysis. ALS - Kelso has sample handling procedures to ensure that the sample used for analysis is representative of the entire sample. These include the SOP for *Subsampling and Compositing of Samples* (GEN-SUBS) and the SOP for *Tissue Sample Preparation* (MET-TISP). Further, analytical SOPs specify sample handling and sample sizes to further ensure the sample aliquot that is analyzed is representative in entire sample.

14.1.6 Comparability – Comparability expresses the confidence with which one data set can be compared to another and is directly affected by data quality (accuracy and precision) and sample handling (sampling, preservation, etc.). Only data of known quality can be compared. The objective is to generate data of known quality with the highest level of comparability, completeness, and usability. This is achieved by employing the quality controls listed below and standard operating procedures for the handling and analysis of all samples. Data is reported in units specified by the client and using ALS Environmental, Kelso or project-specified data qualifiers.

14.2 Method Detection Limits, Method Reporting Limits, Limits of Detection, and Limits of Quantitation

Method Detection Limits (MDL) for methods performed at ALS – Kelso are determined during initial method set up and when significant changes are made. If an MDL study is not performed annually, the established MDL is verified by performing a Limit of Detection (LOD) verification on every instrument used in the analysis. The MDLs are determined by following the SOP *Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation* (CE-QA011), which is based on the procedure in 40 CFR Part 136, Appendix B. As required by NELAP and DoD protocols, the validity of MDLs is verified using LOD verification samples.

The Method Reporting Limit (MRL) is the lowest amount of an analyte in a sample that can be quantitatively determined with stated, acceptable precision and accuracy under stated analytical conditions (i.e. Limit of Quantitation- LOQ). LOQ are analyzed at the frequency specified in the SOP *Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation* (CE-QA011) and at specified concentrations (not lower than the lowest calibration standard). Current MDL/LOD and MRL/LOQ values are available from the laboratory.

14.3 Quality Control Procedures

The specific types, frequencies, and processes for quality control sample analysis are described in detail in method-specific standard operating procedures and listed below. These sample types and frequencies have been adopted for each method and a definition of each type of QC sample is provided below. Unique test-specific requirements may also exist and are found in the laboratory SOP.

14.3.1 Method Blank (a.k.a. Laboratory Reagent Blank)

The method blank is an analyte-free matrix (water, soil, etc.) subjected to the entire analytical process. When analyte-free soil is not available, anhydrous sodium sulfate, organic-free sand, or an acceptable substitute is used. The method blank is analyzed to demonstrate that the analytical system itself does not introduce contamination. The method blank results should be below the Method Reporting Limit (MRL) or, if required for DoD projects, < ½ MRL for the analyte(s) being tested. Otherwise, corrective action must be taken. A method blank is included with the analysis of every sample preparation batch, every 20 samples, or as stated in the method, whichever is more frequent.

14.3.2 Calibration Blank

For some methods, calibration blanks are prepared along with calibration standards in order to create a calibration curve. Calibration blanks are free of the analyte of interest and, where applicable, provide the zero point of the calibration curve. Additional project-specific requirements may also apply to calibration blanks.

14.3.3 Continuing Calibration Blank

Continuing calibration blanks (CCBs) are solutions of analyte-free water, reagent, or solvent that are analyzed in order to verify the system is contamination-free when CCV standards are analyzed. The frequency of CCB analysis is either once every ten samples or as indicated in the method, whichever is greater. Additional project-specific requirements may also apply to continuing calibration blanks.

14.3.4 Calibration Standards

Calibration standards are solutions of known concentration prepared from primary standard or stock standard materials. Calibration standards are used to calibrate the instrument response with respect to analyte concentration. Standards are analyzed in accordance with the requirements stated in the particular method being used.

14.3.5 Initial (or Independent) Calibration Verification Standard (ICV)

The ICV standard is prepared from materials obtained from a source independent of that used for preparing the calibration standards ("second-source"). The ICV is analyzed after calibration but prior to sample analysis in order to verify the validity and accuracy of the standards used in calibration. Once it is determined that there is no defect or error in the calibration standard(s), the standards are considered valid and may be used for subsequent calibrations and quantitative determinations (as expiration dates and methods allow). ICVs are also analyzed in accordance with method-specific requirements.

14.3.6 Continuing Calibration Verification Standard

Continuing calibration verification (CCV) standards are midrange standards that are analyzed in order to verify that the calibration of the analytical system is still acceptable. The frequency of CCV analysis is either once every ten samples, or as indicated in the method.

14.3.7 Internal Standards

Internal standards are known amounts of specific compounds that are added to each sample prior to instrument analysis. Internal standards are generally used for GC/MS and ICP/MS procedures to correct sample results that have been affected by changes in instrument conditions or changes caused by matrix effects. The requirements for evaluation of internal standards are specified in each method and SOP.

14.3.8 Surrogates

Surrogates are organic compounds which are similar in chemical composition and analytical behavior to the analytes of interest, but which are not normally found in environmental samples. Depending on the analytical method, one or more of these compounds is added to method blanks, calibration and check standards, and samples (including duplicates, matrix spike samples, duplicate matrix spike samples and laboratory control samples) prior to extraction and analysis in order to monitor the method performance on each sample. The percent recovery is calculated for each surrogate, and the recovery is a measurement of the overall method performance.

$$\text{Recovery (\%)} = (M/T) \times 100$$

Where: M = The measured concentration of analyte,
T = The known concentration of analyte added.

14.3.9 Laboratory Control Samples (a.k.a Laboratory Fortified Blank - LFB)

The laboratory control sample (LCS) is an aliquot of analyte-free water or analyte-free solid (or anhydrous sodium sulfate or equivalent) to which known amounts of the method analyte(s) is (are) added. A reference material of known matrix type, containing certified amounts of target analytes, may also be used as an LCS. An LCS is prepared and analyzed at a minimum frequency of one LCS per 20 samples, with every analytical batch or as stated in the method, whichever is more frequent. The LCS sample is prepared and analyzed in exactly the same manner as the field samples.

The percent recovery of the target analytes in the LCS is compared to established control limits and assists in determining whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements at the required reporting limit. Comparison of batch-to-batch LCS analyses enables the laboratory to evaluate batch-to-batch precision and accuracy.

$$\text{Recovery (\%)} = (M/T) \times 100$$

Where: M = The measured analyte concentration,
T = The known analyte concentration added.

14.3.10 Laboratory Fortified Blank - MRL Level

A laboratory blank fortified at the MRL used to verify that the method reporting limit can be achieved. This LFB is carried through the entire extraction and analytical procedure. A MRL LFB is required with every batch of drinking water samples.

14.3.11 Matrix Spikes (MS)

Matrix spiked samples are aliquots of samples to which a known amount of the target analyte (or analytes) is (are) added. The samples are then prepared and analyzed in the same analytical batch, and in exactly the same manner as are routine samples. For the appropriate methods, matrix spiked samples are prepared and analyzed and at a minimum frequency of one spiked sample (and one duplicate spiked sample, if appropriate) per twenty samples. The spike recovery measures the effects of interferences caused by the sample matrix and reflects the accuracy of the method for the particular matrix in question. Spike recoveries are calculated as follows:

$$\text{Recovery (\%)} = (S - A)/T \times 100$$

Where: S = The measured analyte concentration in the spiked sample,
A = The measured analyte concentration in the parent sample,
T = The known analyte concentration added to the spiked sample.

14.3.12 Laboratory Duplicates and Duplicate Matrix Spikes

Duplicates are additional replicates of samples that are subjected to the same preparation and analytical scheme as the original sample. Depending on the method of analysis, either a duplicate analysis (and/or a matrix spiked sample) or a matrix spiked sample and duplicate matrix spiked sample (MS/DMS) are analyzed. The relative percent difference between duplicate

analyses or between an MS and DMS is a measure of the precision for a given method and analytical batch. The relative percent difference (RPD) for these analyses is calculated as follows:

$$\text{Relative Percent Difference (RPD)} = (S1 - S2) \times 100 \div S_{ave}$$

Where:

S1 and S2 = The analyte concentrations in the sample and its duplicate, or in the matrix spike and its duplicate matrix spike, and,

S_{ave} = The average of analyte concentrations in the sample and its duplicate, or in the matrix spike and its duplicate matrix spike.

Depending on the method of analysis, either duplicates (and/or matrix spikes) or MS/DMS analyses are performed at a minimum frequency of one set per 20 samples. If an insufficient quantity of sample is available to perform a laboratory duplicate or duplicate matrix spikes, duplicate LCSs will be prepared and analyzed.

14.3.13 Interference Check Samples (ICS)

An ICS is a solution containing both interfering and analyte elements of known concentration that can be analyzed to verify background and interelement correction factors in metals analyses. The ICS is prepared to contain known concentrations (method or program specific) of elements that will provide an adequate test of the correction factors. The ICS is analyzed at the beginning and end of an analytical run or at a method-specified frequency. Results must meet method criteria and any project-specific criteria.

14.3.14 Post Digestion Spikes

Post digestion spikes are samples prepared for metals analyses that have an analyte spike added to determine if matrix effects may be a factor in the results. The spike addition should produce a method-specified minimum concentration above the method reporting limit. A post digestion spike is analyzed with each batch of samples and recovery criteria are specified for each method.

14.3.15 Control Charting

The generation of control charts is routinely performed at ALS. Surrogate, Matrix Spike and LCS recoveries are all monitored and charted. In addition, the laboratory also monitors the Relative Percent Difference (RPD) measurement of precision. Control charts are available to each individual laboratory unit to monitor the data generated in its facility using control charts that have been programmed to identify various trends in the analytical results. If trends in the data are perceived, various means of corrective action may then be employed in order to prevent future problems with the analytical system(s). Finally, data quality reports using control charts are generated for specific clients and projects pursuant to contract requirements. The control charting procedure is described in SOP *Control Limits* (CE-QA009).

14.3.16 Glassware Washing

Glassware washing and maintenance play a crucial role in the daily operation of a laboratory. The glassware used at ALS - Kelso undergoes a rigorous

cleansing procedure prior to every usage. A number of SOPs have been generated that outline the various procedures used at ALS; each is specific to the end-use of the equipment as well as to the overall analytical requirements of the project. In addition, other equipment that may be routinely used at the laboratory is also cleaned following instructions in the appropriate SOP.

14.3.17 Uncertainty

Measurement uncertainty is associated with most of the results obtained in laboratory testing. It may be meaningful to estimate the extent of the uncertainty associated with each result generated by the laboratory. It is also useful to recognize that this measurement uncertainty is likely to be much less than that associated with sample collection activities. The uncertainty associated with the analytical measurement processes can be estimated from quality control data. When requested, the laboratory provides uncertainty information as described in the SOP *Estimate of Uncertainty of Analytical Measurements* (CE-QA010). The estimation of uncertainty relates only to measurements conducted in the laboratory.

14.4 When data quality objectives or quality control measures are not met, due to the sample matrix or anomalies, incompatibility of the methodology and sample type, statistical outliers, random error, or other factors, it may be necessary to apply data qualifiers to reported data. A list of standard data qualifiers is given in Appendix H.

15) Control of Non-Conforming Environmental Testing Work

The laboratory takes all appropriate steps necessary to ensure all sample results are reported with acceptable quality control results. When sample results do not conform to established quality control procedures, responsible management will evaluate the significance of the nonconforming work and take corrective action to address the nonconformance.

Nonconforming events such as errors, deficiencies, deviations from SOP, proficiency (PT) failure or results that fall outside of established QC limits are documented using the NCAR database. The procedure and responsibilities for addressing nonconforming work is defined in SOP *Nonconformance and Corrective Action* (CE-QA008). Nonconformances are reported to the client using various means (voice, email, narrative, etc.). When a nonconformance occurs that casts doubt on the validity of the test results or additional client instructions are needed, the Project Manager notifies the client the same business day that the nonconformance is confirmed and reported. The QAM reviews each problem, ensuring that appropriate corrective action has been taken by the appropriate personnel. The QAM periodically reviews all NCARs looking for chronic, systematic problems that need more in-depth investigation and alternative corrective action consideration. In addition, the appropriate Project Manager is promptly notified of any problems in order to inform the client and proceed with any action the client may want to initiate.

Results from non-conforming environmental testing work generally require the need for qualified data on analytical reports. A list of standard data qualifiers is given in Appendix H. Additionally, the report narrative will provide an explanation of the nonconformance and potential impact on results.

16) Corrective Action, Preventive Action, and Improvement

If a quality control measure is found to be out of control, and the data is to be reported, all samples associated with the failed quality control measure shall be reported with the appropriate data qualifier(s). Failure to meet established analytical controls, such as the quality control objectives, prompts corrective action. Corrective action may take several forms and may involve a review of the calculations, a check of the instrument maintenance and

operation, a review of analytical technique and methodology, and reanalysis of quality control and field samples. If a potential problem develops that cannot be solved directly by the responsible analyst, the supervisor, team leader, the department manager, and/or the QAM may examine and pursue alternative solutions. In addition, the appropriate Project Manager is notified in order to ascertain if the client needs to be notified.

Part of the corrective action process involves determining the root cause. Identifying the root cause of a nonconformance can be difficult, but important for implementing effective corrective action. Root cause principles are used to determine assignable causes, which leads to corrective action taken to prevent recurrence. Various preventive action processes are used for eliminating a potential problem or averting a problem before it occurs. This is explained in SOP *Nonconformance and Corrective Action* (CE-QA008).

Preventive action is focused on using existing information or experiences to anticipate potential problems and eliminating the likely causes of them. Preventive action is a pro-active process and tied to results from corrective action as well as opportunities for improvement. ALS - Kelso used preventive action processes to avoid errors and implement improvements. The SOP *Preventive Action* (CE-GEN004) describes procedures used. Examples of preventive action are given in the SOP. The laboratory also uses ideas from staff, client feedback, and other input mechanisms to identify potential improvements. The monthly lab-wide meeting regularly includes reports on improvements made or underway.

16.1 Preventive maintenance

Preventive maintenance is a crucial element of the QA program. Equipment and instruments at ALS - Kelso are regularly maintained by qualified laboratory staff or under commercial service contracts. All instruments are operated and maintained according to the instrument operating manuals. All routine and special maintenance activities pertaining to the instruments are recorded in instrument maintenance logbooks. The maintenance logbooks used at ALS Environmental, Kelso contain extensive information about the instruments used at the laboratory, including:

- The equipment's serial number;
- Date the equipment was received;
- Date the equipment was placed into service;
- Condition of equipment when received (new, used, reconditioned, etc.); and
- Prior history of damage, malfunction, modification or repair (if known).

Preventive maintenance procedures, frequencies, etc. are available for each instrument used at ALS. They may be found in the various SOPs for routine methods performed on an instrument and may also be found in the operating or maintenance manuals provided with the equipment at the time of purchase.

Responsibility for ensuring that routine maintenance is performed lies with the section supervisor. In the case of non-routine repair of capital equipment, the section supervisor is responsible for providing the repair, either by performing the repair themselves with manufacturer guidance or by acquiring on-site manufacturer repair. Each laboratory section maintains a critical parts inventory. This inventory or "parts list" also includes the items needed to perform any other routine maintenance and certain in-house non-routine repairs such as gas chromatography/mass spectrometry jet separators and electron multipliers and ICP/MS nebulizer. When performing maintenance on an instrument (whether preventive or corrective), additional information about the problem, attempted repairs, etc. is also recorded in the notebook. Typical logbook entries include the following information:

- Details and symptoms of the problem;

- Repairs and/or maintenance performed;
- Description and/or part number of replaced parts;
- Source(s) of the replaced parts;
- Analyst's signature and date; and
- Demonstration of return to analytical control.

See the Appendix E for a list of equipment and whether primarily maintained by laboratory of service providers.

17) Control of Records

ALS - Kelso maintains a records system which ensures that all laboratory records of analysis data retained and available. Analysis data is retained for 5 years from the report date unless contractual terms or regulations specify a longer retention time. The archiving system is described in the SOP for *Data Archiving* (ADM-ARCH).

17.1 Documentation and Archiving of Sample Analysis Data

The archiving system includes the following items for each set of analyses performed:

- Benchsheets describing sample preparation (if appropriate) and analysis;
- Instrument parameters (or reference to the data acquisition method);
- Sample analysis sequence;
- Instrument printouts, including chromatograms and peak integration reports for all samples, standards, blanks, spikes and reruns;
- Logbook ID number for the appropriate standards;
- Copies of report sheets submitted to the work request file; and
- Copies of Nonconformity and Corrective Action Reports, if necessary.

Individual sets of analyses are identified by analysis date and service request number. Since many analyses are performed with computer-based data systems, the final sample concentrations can be automatically calculated. If additional calculations are needed, they are written on the integration report or securely stapled to the chromatogram, if done on a separate sheet.

For organics analysis, data applicable to all analyses within the batch, such as GCMS tunes, CCVs, batch QC, and analysis sequences; are kept using a separate documentation system. This system is used to archive data on a batch-specific basis and is segregated according to the date of analysis. This system also includes results for the most recent calibration curves, as well as method validation results.

18) Audits

Quality audits are an essential part of the Quality Assurance program. There are two types of audits used at the facility: System Audits are conducted to qualitatively evaluate the operational details of the QA program, while Performance Audits are conducted by analyzing proficiency testing samples in order to quantitatively evaluate the outputs of the various measurement systems.

18.1 System Audits

The system audit examines the presence and appropriateness of laboratory systems. External system audits of ALS/Kelso are conducted regularly by various regulatory

agencies and clients. Appendix J lists the certification and accreditation programs in which ALS/Kelso participates. Programs and certifications are added as required.

Internal system audits of ALS/Kelso are conducted regularly under the direction of the Quality Assurance Manager. The internal audit procedures are described in SOP *Internal Audits* (CE-QA001). The internal audits are performed as follows:

- System audit – this is an annual audit of the implementation of the quality system in the laboratory.
- Process audit – this is an audit of all operational areas in the laboratory to evaluate compliance with operational and technical procedures. Focus is on sample handling, preparation and analysis and technically sound practices. Three primary concepts are 1) is the procedure in use the same as that described in the SOP, 2) the use of sound analytical techniques and practices, and 3) sample handling/preparation. Topics as calibration, sample/analytical batching, standards traceability, QC criteria, instrument operation and maintenance, data interpretation, and reporting results are included. Hardcopy data and/or report audits may be included.

  Process audits may be one larger audit event or a series of audits such that all areas of the laboratory are audited over a one year period. Audits conducted over the four calendar quarters will follow the schedules listed in an audit plan.
- Electronic data audits focus on organic chromatographic data and include an examination of audit trails, peak integrations, calibration practices, GCMS tuning data, use of appropriate files, and other components of the analysis. Each applicable instrument is periodically audited using audit software and randomly selected data files.

All audit findings and corrective actions are documented. The results of each audit are reported to the Laboratory Director and Department Managers for review. Any deficiencies identified are summarized in the audit report. Managers must respond with corrective actions correcting the deficiency within a defined timeframe. Should problems impacting data quality be found during an internal audit, any client whose data is adversely impacted will be given written notification within the corrective action period (if not already provided).

Additional internal audits or data evaluations may be performed as needed to address any potential data integrity issues that may arise.

18.2 Performance Audits

ALS – Kelso participates in the analysis of interlaboratory proficiency testing (PT) samples. Participation in PT studies is performed on a regular basis and is designed to evaluate all analytical areas of the laboratory. General procedures for these analyses are described in SOP *Proficiency Sample Testing Analysis* (CE-QA006). ALS – Kelso routinely participates in the following studies:

- Water Pollution (WP) and additional water parameters, 2 per year.
- Water Supply (WS) PT studies, 2 per year.
- Hazardous Waste/Soil/UST PT studies, 2 per year.
- Microbiology (WS and WP) PT studies, 2 per year.
- State-specific Underground Storage Tank PT studies, 1 per year, or as specified for accreditation.
- Other studies as required for certifications, accreditations, or validations.

PT samples are processed by entering them into the LIMS system as samples and are processed the same as field samples (following the PT provider instructions). The laboratory sections handle samples the same as field samples, performing the analyses following method requirements and performing data review. The laboratory sections submit results to the QA Manager for subsequent reporting to the appropriate agencies or study provider. Results of the performance evaluation samples and audits are received by the QAM and distributed to Laboratory Director and department managers for review. For any results outside acceptance criteria, the analysis data is reviewed to identify a root cause for the deficiency, and corrective action is taken and documented through nonconformance (NCAR) procedures.

19) Management Review

An annual Review of the laboratory's quality system and testing activities is conducted by the laboratory's management team to ensure the continuing suitability and effectiveness of the quality system and testing activities and to introduce any necessary changes or improvements. The review ensures that the quality system of the laboratory continues to conform to the requirements of the ISO 17025:2005 and various accrediting authorities, including NELAP/TNI.

General procedures for the Review are described in *Laboratory Management Review* (SOP CE-QA005). When conducting the review a standard list of items and categories is evaluated. The quality policies and their relation to testing activities are reviewed and any changes that are necessary are identified. The review also notes significant changes that have taken place or need to take place in the quality system; and the organization, facilities, equipment, procedures, and activities of the laboratory.

The Review is documented by the laboratory QA Manager. Action items, including preventive actions and improvements, should be identified. Results should feed into the laboratory's planning process planning.

20) Personnel

20.1 Personnel Training

Job descriptions, including technical position descriptions, are used for all employees, regardless of position or level of seniority. These documents are maintained by the Human Resources personnel and are available for review. In order to assess the technical capabilities and qualifications of a potential employee, all candidates for employment are evaluated, in part, against the appropriate technical description.

Training begins the first day of employment at ALS - Kelso when the company policies are presented and discussed. Safety and Quality System requirements are integral parts of initial and ongoing training processes at the laboratory. Safety training begins with the reading of the ALS Environmental Health and Safety Manual. Employees are also required to attend periodic safety meetings where additional safety training may be performed by the Environmental, Health and Safety Officer.

Quality Systems training begins with QA orientation for new employees which includes reading the Quality Assurance Manual and ethics/data integrity introductory training. Additional training on laboratory quality systems as they relate to job functions is incorporated into training plans. Employees are responsible for complying with the requirements of the QA Manual and QA/QC requirements associated with their function(s).

ALS - Kelso also encourages its personnel to continue to learn and develop new skills that will enhance their performance and value to the company. Ongoing training occurs for all employees through a variety of mechanisms. The corporate, company-wide training and development program, external and internal technical seminars and

training courses, and laboratory-specific training exercises are all used to provide employees with professional growth opportunities.

All technical training is documented and records are maintained in the QA department. Training requirements and its documentation are described in SOP *ALS-Kelso Training Procedure* (ADM-TRAIN). A training plan is developed whenever an employee starts a new procedure to new position. The training plan includes a description of the step-by-step process for training an employee and for initial demonstration of capability. Where the analyst performs the entire procedure, a generic training plan may be used.

20.2 Initial Demonstration of Capability (IDOC)

Training in analytical procedures typically begins with the reading of the SOP for the method. Hands-on training begins with the observation of an experienced analyst performing the method, followed by the trainee performing the method under close supervision, and culminating with independent performance of the method on quality control samples. Successful completion of the applicable Demonstration of Capability analysis qualifies the analyst to perform the method independently. Demonstration of Capability is performed by one of the following:

- Successful completion of an Initial Precision and Recovery (IPR) study (required where mandated by the method).
- Analysis of 4 consecutive Laboratory Control Samples, with acceptable accuracy and precision.
- Where spiking is not possible but QC standards are used ("non-spiked" LCS), analysis of 4 consecutive LCS analyses with acceptable accuracy and precision.
- Where one of the three above is not possible, special requirements are as follows:
    - Total Settleable Solids: Successful single-blind PT sample analysis and duplicate results with RPD<10%.
    - Color: Four consecutive prepared LCSs with acceptable accuracy and precision of <10% RSD.
    - Physical Tests (Grain size, Corrosivity to Steel, etc.): Supervisor acknowledgement of training and approval.

A flowchart identifying the Demonstration of Proficiency requirements is given in Figure 20-1. The flowchart identifies allowed approaches to assessing Demonstration of Capability when a 4-replicate study is not mandated by the method, when spiking is not an option, or when QC samples are not readily available.

20.3 Continuing Demonstration of Proficiency

A periodic demonstration of proficiency is required to maintain continuing qualification. Continuing Demonstration of Proficiency is required each year, and may be performed one of the following ways:

- Successful performance on external (independent) single-blind sample analyses using the test method, or a similar test method using the same technology. I.e. PT sample or QC sample blind to the analyst.
- Performing Initial Demonstration of Capability as described above, with acceptable levels of precision and accuracy.
- Analysis of at least 4 consecutive LCSs with acceptable levels of accuracy and precision from in-control analytical batches.

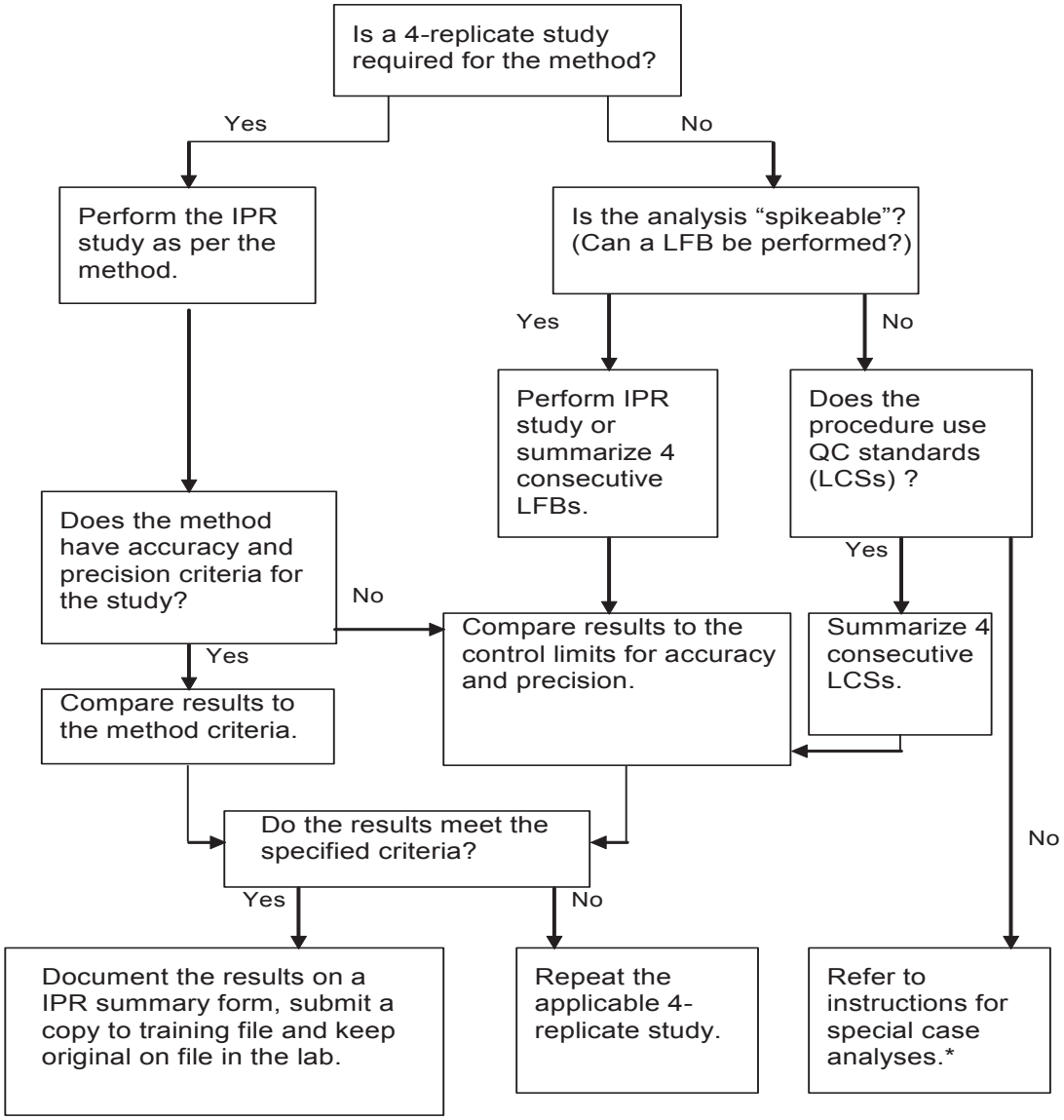
- If the above cannot be performed, analysis of authentic samples with results statistically indistinguishable from those obtained by another trained analyst.
- For methods for which PT samples are not available and a spiked analysis (LFB, MDL, etc.) is not possible, analysis of field samples that have been analyzed by another analyst with statistically indistinguishable results.

20.4 Documentation of Training

Records are maintained to indicate the employee has the necessary training, education, and experience to perform their functions. Information of previously acquired skills and abilities for a new employee is maintained in Human Resources personnel files and internal resumes. The QA department maintains a record of the various technical skills and training acquired while employed by ALS. Information includes the employee's name, a description of the skill including the appropriate method and SOP reference, the mechanism used to document proficiency, and the date the training was completed. General procedures for documenting technical training are described in SOP *ALS-Kelso Training Procedure* (ADM-TRAIN).

Figure 20-1

Demonstration of Proficiency Flowchart



21) Reporting of Results

ALS - Kelso reports the analytical data produced in its laboratories to the client via the Analytical Report. This report includes a transmittal letter, a case narrative, client project information, sample receipt and chain of custody information, specific test results, quality control data (as requested), and any other project-specific support documentation. The following procedures describe the procedures used for data reduction, validation and reporting.

21.1 Data Reduction and Review

Results are generated by the analyst who performs the analysis and works up the raw data. All data is initially reviewed and processed by analysts using appropriate methods (e.g., chromatographic software, instrument printouts, hand calculation, etc.). Equations used for calculation of results are found in the applicable analytical SOPs. Policies and procedures for manual editing of data are established. The analyst making the change must initial and date the edited data entry, without obliteration of the original entry. The policies and procedures are described in the SOP *Making Entries onto Analytical Records* (CE-QA007).

The resulting data set is either manually entered (e.g., titrimetric or microbiological data) into an electronic report form or is electronically transferred into the report. Once the complete data set has been transferred into the proper electronic report form(s), it is then printed. The resulting hardcopy version of the electronic report is then reviewed by the analyst for accuracy. Once the primary analyst has checked the data for accuracy and acceptability, the data and report hardcopy is forwarded to the supervisor or second qualified analyst who reviews the data. Where calculations are not performed using a validated software system, the reviewer rechecks a minimum of 10% of the calculations. Analysts performing routine testing are responsible for generating a data quality narrative or data review document with every analytical batch processed. This report also allows the analyst to provide appropriate notes and/or a narrative if problems were encountered with the analyses. A Nonconformance and Corrective Action Report (NCAR) may also be attached to the data prior to review. Supervisors or qualified analysts review all of the completed analytical batches to ensure that all QC criteria have been examined and any deficiencies noted and addressed. Data review procedures are described in the SOP for *Laboratory Data Review Process* (ADM-DREV).

Policies and procedures for electronic manual integration of chromatographic data are established. The analyst performing the integration must document the integration change by printing both the "before" and "after" integrations and including them in the raw data records. The policies and procedures are described in SOP *Manual Integration Policy* (CE-QA002) and SOP *Manual Integration of Chromatographic Peaks* (ADM-MI).

21.1.1 Validation of Results

The validity of the data generated is assessed through the evaluation of the sample results, calibrations, and QC samples (method blanks, laboratory control samples, sample duplicates, matrix spikes, trip blanks, etc.). A brief description of the evaluation of these analyses is described below, with details listed in applicable SOPs. The criteria for evaluation of QC samples are listed within each method-specific SOP. Other data evaluation measures may include (as necessary) a check of the accuracy check of the QC standards and a check of the system sensitivity. Data transcriptions and calculations are also reviewed.

Note: Within the scope of this document, all possible data assessment requirements for various project protocols cannot be included in the listing below. This listing gives a general description of data evaluation practices used in the laboratory in compliance with NELAP Quality Systems requirements. Additional requirements exist for certain programs, such as projects under the DoD QSM protocols, and project-specific QAPPs.

- Initial Calibration – Following the analysis of calibration standards according to the applicable SOP the data is fit to an applicable and allowed calibration model (correlation coefficient, linear, average response factor, quadratic, etc.) and the resulting calibration is compared to specified criteria. If the calibration meets criteria analysis may continue. If the calibration fails, any problems are isolated and corrected and the calibration standards reanalyzed. Following calibration and analysis of the independent calibration verification standard(s) the percent difference for the ICV is calculated. If the percent difference is within the specified limits the calibration is complete. If not, the problem associated with the calibration and/or ICV are isolated and corrected and verification and/or calibration is repeated.
- Continuing Calibration Verification (CCV) – Following the analysis of the CCV standard the percent difference is calculated and compared to specified criteria. If the CCV meets the criteria analysis may continue. If the CCV fails, routine corrective action is performed and documented and a 2nd CCV is analyzed. If this CCV meets criteria, analysis may continue, including any reanalysis of samples that were associated with a failing CCV. If the routine corrective action failed to produce an immediate CCV within criteria, then either acceptable performance is demonstrated (after additional corrective action) with two consecutive calibration verifications or a new initial calibration is performed.
- Method Blank – Results for the method blank are calculated as performed for samples. If results are less than the MRL (<½ MRL for DoD projects), the blank may be reported. If not, associated sample results are evaluated to determine the impact of the blank result. If possible, the source of the contamination is determined. If the contamination has affected sample results the blank and samples are reanalyzed. If positive blank results are reported, the blank (and sample) results are flagged with an appropriate flag, qualifier, or footnote.
- Sample Results (Inorganic) – Following sample analysis and calculations (including any dilutions made due to the sample matrix) the result is verified to fall within the calibration range. If not, the sample is diluted and analyzed to bring the result into calibration range. When sample and sample duplicates are analyzed for precision, the calculated RPD is compared to the specified limits. The sample and duplicate are reanalyzed if the criteria are exceeded. The samples may require re-preparation and reanalysis. For metals, additional measures as described in the applicable SOP may be taken to further evaluate results (dilution tests and/or post-digestion spikes). Results are reported when within the calibration range, or as estimates when outside the calibration range. When dilutions are performed the MRL is elevated accordingly and qualified. Efforts are made to meet the project MRL's including alternative analysis.

- Sample Results (Organic) – For GC/MS analyses, it is verified that the analysis was within the prescribed tune window. If not, the sample is reanalyzed. Following sample analysis and calculations (including any dilutions made due to the sample matrix) peak integrations, retention times, and spectra are evaluated to confirm qualitative identification. Internal standard responses and surrogate recoveries are evaluated against specified criteria. If internal standard response does not meet criteria, the sample is diluted and reanalyzed. Results outside of the calibration range are diluted to within the calibration range. For GC and HPLC tests, results from confirmation analysis are evaluated to confirm positive results and to determine the reported value. The procedure to determine which result to report is described in the SOP for *Confirmation Procedure for GC and HPLC Analysis* (SOC-CONF). If obvious matrix interferences are present, additional cleanup of the sample using appropriate procedures may be necessary and the sample is reanalyzed. When dilutions are performed the MRL is elevated accordingly and qualified. Efforts are made to meet the project MRL's including additional cleanup.
- Surrogate Results (Organic) – The percent recovery of each surrogate is compared to specified control limits. If recoveries are acceptable, the results are reported. If recoveries do not fall within control limits, the sample matrix is evaluated. When matrix interferences are present or documented, the results are reported with a qualifier that matrix interferences are present. If no matrix interferences are present and there is no cause for the outlier, the sample is reprepared and reanalyzed. However, if the recovery is above the upper control limit with non-detected target analytes, the sample may be reported. All surrogate recovery outliers are appropriately qualified on the report.
- Duplicate Sample and/or Duplicate Matrix Spike Results – The RPD is calculated and compared to the specified control limits. If the RPD is within the control limits the result is reported. If not, an evaluation of the sample is made to verify that a homogenous sample was used. Despite the use of homogenizing procedures prior to sample preparation or analysis, the sample may not be homogenous or duplicate sample containers may not have been sample consistently. If non-homogenous, the result is reported with a qualifier about the homogeneity of the sample. Also, the results are compared to the MRL. If the results are less than five times the MRL, the results are reported with a qualifier that the high RPD is due to the results being near the MRL. If the sample is homogenous and results above five times the MRL, the samples and duplicates are reanalyzed. If re-analysis also produces out-of-control results, the results are reported with an appropriate qualifier.
- Laboratory Control Sample Results – The LCS percent recovery is calculated and compared to specified control limits. If the recovery is within control limits, the analysis is in control and results may be reported. If not, this indicates that the analysis is not in control. Samples associated with the 'out of control' LCS, shall be considered suspect and the samples re-extracted or re-analyzed or the data reported with the appropriate qualifiers. For analysis where a large number of analytes are in the LCS, it becomes more likely that some analytes (marginal exceedences) will be outside the control limits. The procedure described in the 2009 NELAC standards, V1M4 Section

1.7.4.2 are used to determine if the LCS is effective in validating the analytical system and the associated samples.

- Matrix Spike Results – The MS percent recovery is calculated and compared to specified control limits. If the recovery is within control limits the results are reported. If not, and the LCS is within control limits, this indicates that the matrix potentially biases analyte recovery. It is verified that the spike level is at least five times the background level. If not, the results are reported with a qualifier that the background level is too high for accurate recovery determination. If matrix interferences are present or results indicate a potential problem with sample preparation, steps may be taken to improve results; such as performing any additional cleanups, dilution and reanalysis, or re-preparation and reanalysis. Results that do not meet acceptance limits are reported with an appropriate qualifier.

21.1.2 Qualitative Data Evaluation

All sample results and QC results are reviewed to ensure correct identification of target analytes, when not inherent to the test method. Details particular to each analysis are given in the analytical SOP.

Identification criteria for GC, LC or GC/MS methods are summarized below:

- GC and LC Methods
  - The analyte must fall within the retention time window specified in the applicable SOP. The retention time window is established prior to analysis and documented.
  - For analyses all positive results are confirmed by a second column, a second detector, a second wavelength (HPLC/UV), or by GC/MS analysis. Details for confirmation analysis are described in the SOP *Confirmation Procedures for GC and HPLC Analyses* (SOC-CONF). Confirmation data will be provided as specified in the method.
  - When sample results are confirmed by two dissimilar columns or detectors, the agreement between quantitative results must be evaluated. The relative percent difference between the two results is calculated and evaluated against SOP and/or method criteria.
- GC/MS and LC/MS Methods – Two criteria are used to verify identification:
  - Elution of the analyte is at the same relative retention time (as defined by the method) as demonstrated in the standard.
  - The mass spectrum of the analyte in the sample must, in the opinion of a qualified analyst or the department manager, correspond to the spectrum of the analyte in the standard or the current GC/MS reference library.
  - When Tentatively Identified Compounds are to be reported for GC/MS, the spectrum for non-target peaks is compared to the current GC/MS reference library.

21.2 Data Reporting

It is the responsibility of each laboratory unit to provide the Project Manager with a final report of the data for each analysis, accompanied by signature approval. When the entire data set has been found to be acceptable, a final copy of the report is generated and approved by the laboratory supervisor, departmental manager or designated laboratory staff. The entire data package for the analysis is then placed into the service request file, and an electronic copy of the final data package is forwarded to the appropriate personnel for archival. Footnotes and/or narrative notes must accompany any data package if problems were encountered that require further explanation to the client. Each data package is submitted to the appropriate Project Manager.

When all analyses and departmental reports are completed the Project Manager reviews the entire collection of analytical data for completeness and to ensure that any and all client-specified objectives were successfully achieved. A report narrative is written by the Project Manager to explain any unusual problems with a specific analysis or sample, etc. Prior to release of the report to the client, the Project Manager reviews and approves the entire report for completeness and to ensure that any and all client-specified objectives were successfully achieved. The original raw data, along with a copy of the final report, is scanned and archived by service request number.

To the extent possible, samples shall be reported only if all QC measures are acceptable. If a QC measure is found to be out of control, and the data is to be reported, all samples associated with the failed quality control measure shall be reported with the appropriate data qualifier(s). The SOP for *Data Reporting and Report Generation* (ADM-RG) addresses the flagging and qualification of data. The ALS-defined data qualifiers, state-specific data qualifiers, or project-defined data qualifiers are used depending on project requirements. A case narrative may be written by the Project Manager to explain problems with a specific analysis or sample, etc.

When requested by the client or relevant to the validity of reported results, the estimation of measurement uncertainty will be provided to a client or regulatory agency. How the uncertainty will be reported may be dictated by the client's reporting specifications. Procedures for determining and reporting uncertainty are given in SOP *Estimation of Uncertainty of Analytical Measurements* (CE-QA010).

For subcontracted analyses, the Project Manager verifies that the report received from the subcontractor is complete. This includes checking that the correct analyses were performed, the analyses were performed for each sample as requested, a report is provided for each analysis, and the report is signed. The Project Manager accepts the report if all verification items are complete. Acceptance is demonstrated by forwarding the report to the client.

21.3 Deliverables

In order to meet individual project needs, ALS - Kelso provides several levels of analytical reports. Standard specifications for each level of deliverable are described in Table 21-1. Variations may be provided based on client or project specifications. This includes (but is not limited to) deliverables for DoD QSM projects and state-specific drinking water formats.

When requested, ALS - Kelso provides Electronic Data Deliverables (EDDs) in the format specified by client need or project specification. ALS - Kelso is capable of generating EDDs with many different formats and specifications. The EDD is prepared by report production staff using the electronic version of the laboratory report to minimize transcription errors. User guides and EDD specification outlines are used in

preparing the EDD. The EDD is reviewed and compared to the hard-copy report for accuracy.

Table 21-1
Descriptions of ALS Environmental – Kelso Standard Data Deliverables*
Tier I. Routine Analytical Report includes the following:
• Transmittal letter
• Chain of custody documents and sample/cooler receipt documentation
• Sample analytical results
• Method blank results
• Surrogate recovery results and acceptance criteria for applicable organic methods
• Dates of sample preparation and analysis for all tests
• Case narrative – optional
Tier II. In addition to the Tier I Deliverables, this Analytical Report includes the following:
• Laboratory Control Sample results with calculated recovery and associated acceptance criteria
• Matrix spike results with calculated recovery and associated acceptance criteria
• Duplicate or duplicate matrix spike result(s) (as appropriate to method), with calculated relative percent difference
• Case narrative – optional
Tier III. Data Validation Package. In addition to the Tier II Deliverables, this CAR includes the following:
• Case narrative – required
• Summary forms for all associated QC and Calibration parameters, with associated control criteria/acceptance limits
• Other summary forms specified in QAPPs or project/program protocols, or those related to specialized analyses such as HRGC/MS are included.
Tier IV. Full Data Validation Package.
• All raw data associated with the sample analysis, including but not limited to: • Preparation and analysis bench sheets and instrument printouts, • For organics analyses, all applicable chromatograms, spectral, confirmation, and manual integration raw data. For GC/MS this includes tuning results, mass spectra of all positive results, and the results and spectra of TIC compounds when requested.
• QC data
• Calibration data (initial, verification, continuing, etc.),
• Calibration blanks or instrument blanks (as appropriate to method).

* If a project QAPP or program reporting protocol applies the report will be presented as required for the project.

22) Summary of Changes and Document History

Revision Number	Effective Date	Document Editor	Description of Changes
25	11/12/2016	C. Degner	Minor changes and updates to text sections 1-23, updated key personnel, organization charts, and equipment. Updated appendices.

23) References for Quality System Standards, External Documents, Manuals, and Test Procedures

The analytical methods used at ALS Environmental, Kelso generally depend upon the end-use of the data. Since most of our work involves the analysis of environmental samples for regulatory purposes, specified federal and/or state testing methodologies are used and followed closely. Typical methods used at ALS Environmental, Kelso are taken from the following references:

- National Environmental Laboratory Accreditation Program (NELAP), 2009 Quality Standards.
- TNI Standard – Environmental Laboratory Sector, Volume 1, *Management and Technical Requirements for Laboratories Performing Environmental Analysis,* EL-V1-2009.
- Quality Standards. American National Standard *General requirements for the competence of testing and calibration laboratories,* ANSI/ISO/IEC 17025:2005(E)
- *DoD Quality Systems Manual for Environmental Laboratories,* Versions 4.2 and 5.0
- *Good Automated Laboratory Practices, Principles and Guidance to Regulations For Ensuring Data Integrity In Automated Laboratory Operations*, EPA 2185 (August 1995).
- *Manual for the Certification of Laboratories Analyzing Drinking Water*, 5th Edition, EPA 815-B-97-001 (January 2005).
- *Procedure Manual for the Environmental Laboratory Accreditation Program,* Washington Department of Ecology, 10-03-048, September 2010.
- *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, SW-846, Third Edition, (September 1986) and Updates I (July 1992), II (September 1994), IIA (August 1993), IIB (January 1995), III (December 1996), Final Update IV (February 2007), and updates posted online at http://www.epa.gov/epaoswer/hazwaste/test/sw846.htm. See Chapters 1, 2, 3, and 4.
- *Methods for Chemical Analysis of Water and Wastes*, EPA-600/4-79-020, (Revised March 1983).
- *Methods for the Determination of Inorganic Substances in Environmental Samples*, EPA/600/R-93/100 (August 1993).
- *Methods for the Determination of Metals in Environmental Samples*, EPA/600/4-91/010 (June 1991) and Supplements.
- *Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater*, EPA 600/4-82-057 (July 1982) and 40 CFR Part 136, Appendix A.
- *Methods for the Determination of Organic Compounds in Drinking Water*, EPA/600/4-88/039 (December 1988) and Supplements.

- Standard Methods for the Examination of Water and Wastewater, 20th Edition (1998) and SM On-Line. See Introduction in Part 1000.
- 40 CFR Part 136, Guidelines for Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, and EPA Method Update Rule 2007 and 2012.
- 40 CFR Part 141, National Primary Drinking Water Regulations and EPA Method Update Rule 2007.
- Analytical Methods for Petroleum Hydrocarbons, ECY 97-602, Washington State Department of Ecology, June 1997.
- State-specific total petroleum hydrocarbon methods for the analysis of samples for gasoline, diesel, and other petroleum hydrocarbon products (Alaska, Arizona, California, Oregon, Washington, Wisconsin, etc.).
- Annual Book of ASTM Standards, Part 31, Water.
- U. S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review, EPA-540/R-94/012 (February 1993).
- U. S. EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review, EPA-540/R-94/013 (February 1994).
- Recommended Protocols for Measuring Selected Environmental Variables in Puget Sound, for USEPA and USACE (March 1986), with revisions through April 1997.
- WDOE 83-13, Chemical Testing Methods for Complying with the State of Washington Dangerous Waste Regulations (March 1982) and as Revised (July 1983 and April 1991).
- Identification and Listing of Hazardous Waste, California Code of Regulations, Title 22, Division 4.5, Chapter 11.
- Analytical Methods for the Determination of Pollutants in Pulp and Paper Industry Wastewater, EPA 821-R-93-017 (October 1993).
- Analytical Methods for the Determination of Pollutants in Pharmaceutical Manufacturing Industry Wastewaters, EPA 821-B-98-016 (July 1998).
- National Council of the Pulp and Paper Industry for Air and Stream Improvement (NCASI).

Internal program-level QA documents are listed in Appendix I.

APPENDIX A – Glossary

Acceptance Criteria: Specified limits placed on characteristics of an item, process, or service defined in requirement documents.

Accreditation: The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory.

Accreditation Body: The territorial, state or federal agency having responsibility and accountability for environmental laboratory accreditation and which grants accreditation.

Accreditation Standard: The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of standard setting and meets the approval requirements of standard adoption organizations procedures and policies.

Accuracy: The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components that are due to sampling and analytical operations; a data quality indicator.

Analysis Date: The calendar date of analysis associated with the analytical result reported for an accreditation or experimental field of proficiency testing.

Analyst: The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.

Analytical Uncertainty: A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis.

Assessment: The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of laboratory accreditation).

Audit: A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives.

Bias: The systematic distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value).

Calibration: A set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards.

Calibration Standard: A substance or reference material used for calibration.

Certified Reference Material (CRM): Reference material accompanied by a certificate, having a value, measurement uncertainty, and stated metrological traceability to a national metrology institute.

Chain of Custody: Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and types of containers; the mode of collection; the collector; time of collection; preservation; and requested analyses.

Confirmation: Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to: second column confirmation, alternate wavelength, derivatization, mass spectral interpretation, alternative detectors, or additional cleanup procedures.

Data Reduction: The process of transforming the number of data items by arithmetic or statistical calculation, standard curves, and concentration factors, and collating them into a more useful form.

Demonstration of Capability: A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision.

Field of Accreditation: Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.

Field of Proficiency Testing (FoPT): Analytes for which a laboratory is required to successfully analyze a PT sample in order to obtain or maintain accreditation, collectively defined as: matrix, technology/method, analyte.

Finding: An assessment conclusion referenced to a laboratory accreditation standard and supported by objective evidence that identifies a deviation from a laboratory accreditation standard requirement.

Holding Time: The specified maximum time that can elapse between two specified sampling and/or analytical activities.

Internal Standard: A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical method.

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes and taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a reference method. It is generally used to establish evaluate accuracy and bias for associated sample analyses.

Legal Chain of Custody Protocols: Procedures employed to record the possession of samples from the time of sampling through the retention time specified by the client or program. These procedures are performed at the special request of the client and include the use of a Chain of Custody Form that documents the collection, transport, and receipt of compliance samples by the laboratory. In addition, these protocols document all handling of the samples within the laboratory.

Limit of Detection (LOD): A laboratory's estimate of the minimum amount of an analyte in a given matrix that an analytical process can reliably detect.

Limit of Quantitation (LOQ): The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence.

Matrix: The substrate of a test sample.

Matrix Duplicate: A replicate matrix prepared in the laboratory and analyzed to obtain a measure of precision.

Matrix Spike (spiked sample or fortified sample): A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of sample for which an independent test result of target analyte concentration is available. Matrix spikes are used to determine the effect of the matrix on a method's recovery efficiency.

Matrix Spike Duplicate (spiked sample or fortified sample duplicate): A replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

Measurement System: A method, as implemented at a particular laboratory, and which includes the equipment used to perform the test and the operator(s).

Method: A body of procedures and techniques for performing an activity (e.g., sampling, chemical analysis, quantification), systematically presented in the order in which they are to be executed.

National Institute of Standards and Technology (NIST): A federal agency of the US Department of Commerce's Technology Administration that is designed as the United States National Metrology Institute (NMI).

Precision: The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator.

Preservation: Any conditions under which a sample must be kept in order to maintain chemical and/or biological integrity prior to analysis.

Primary Accreditation Body (Primary AB): The TNI-NELAP accreditation body responsible for assessing a laboratory's total quality system, on-site assessment, and PT performance tracking for fields of accreditation.

Procedure: A specified way to carry out an activity or process. Procedures can be documented or not.

Proficiency Testing (PT): A means to evaluate a laboratory's performance under controlled conditions relative to a given set of criteria, through analysis of unknown samples provided by an external source.

Proficiency Testing Provider (PTP): A person or organization accredited by the TNI-approved Proficiency Testing Provider Accreditor to operate a TNI-compliant PT program.

Proficiency Testing Sample (PT Sample): A sample, the composition of which is unknown to the laboratory and is provided to test whether the laboratory can produce analytical results within the specified acceptance criteria.

Proficiency Testing Study (PT Study): A single complete sequence of circulation of proficiency testing samples to all participants in a proficiency test program.

Quality Assurance: An integrated system of management activities involving planning, implementation, assessment, reporting, and quality improvement to ensure that a process, item, or service is of the type and quality needed and expected by the client.

Quality Control: The overall system of technical activities that continually measures the performance of a process, item, or service against defined standards to verify that they meet the stated requirements. Also, the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against "out of control" conditions and ensuring that the results are of acceptable quality.

Quality Control Sample: A sample used to assess the performance of all or a portion of the measurement system.

Quality Manual: A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users.

Quality System: A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required quality assurance (QA) and quality control (QC) activities.

Quality System Matrix: These matrix definitions be used for purposes of batch and quality control requirements:

Air and Emissions: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbent tube, impinger solution, filter, or other device.

Aqueous: Any aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine. Includes surface water, ground water effluents, and TCLP or other extracts.

Biological Tissue: Any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples are grouped according to type of tissue (i.e. marine vs. plant).

Chemical Waste: A product or by-product of an industrial process that results in a matrix not otherwise defined.

Drinking Water: Any aqueous sample that has been designated a potable or potential potable water source.

Non-Aqueous Liquid: Any organic liquid, product, or solvent not miscible in water and with <15% settleable solids.

Saline/Estuarine: Any aqueous sample from an ocean or estuary, or other salt water source.

Solids: Includes soils, sediments, sludges and other matrices with >15% settleable solids.

Raw Data: The documentation generated during sampling and analysis that records the original work steps, observations, and measurements, whether performed by an analyst or instrument. This documentation includes, but is not limited to field notes, electronic data, analysis bench sheets, run/injection logs, printouts, chromatograms, instrument outputs, and handwritten records for calibration, sample preparation, and sample analysis for field samples and QC samples.

Reference Material: Material or substance one or more of whose property values are sufficiently homogeneous and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials.

Reference Standard: Standard used for the calibration of working measurement standards in a given organization or at a given location.

Sampling: Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.

Secondary Accreditation Body (Primary AB): A TNI-NELAP accreditation body responsible that accredits the laboratory based on the Primary AB accreditation and procedures.

Selectivity: The ability to analyze, distinguish, and determine a specific analyte or parameter from another component that may be a potential interferent or that may behave similarly to the target analyte or parameter within the measurement system.

Sensitivity: The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest.

Standard Operating Procedure (SOP): A written document that details the process for an operation, analysis, or action, with thoroughly prescribed techniques and steps. SOPs are officially approved as the procedures for performing certain routine or repetitive tasks.

Technology: A specific arrangement of analytical instruments, detection systems, and/or preparation techniques.

Traceability: The ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical constants or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project.

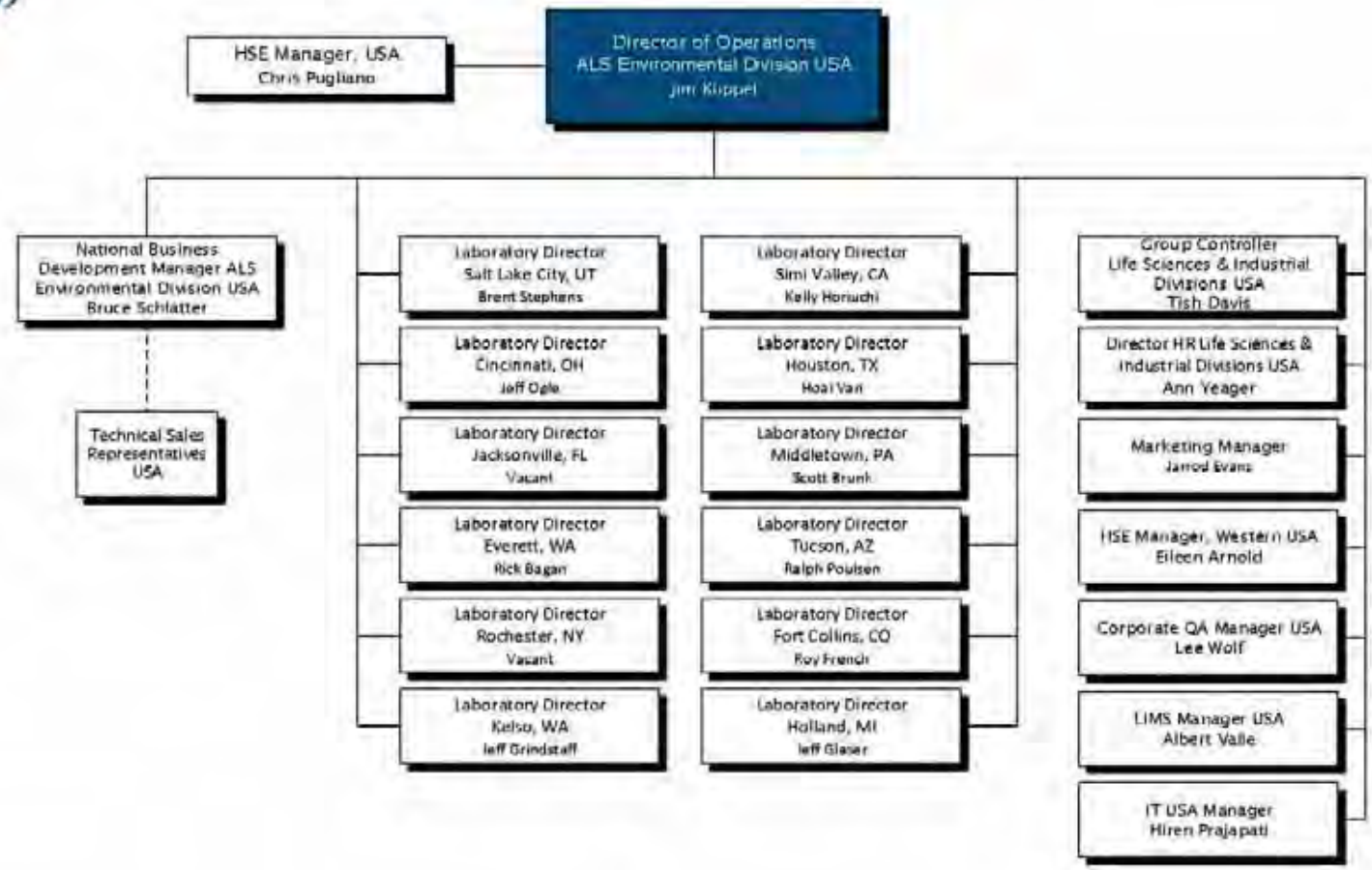
Verification: Confirmation by examination and objective evidence that specified requirements have been met.

Acronyms

ASTM – American Society for Testing and Materials
A2LA – American Association for Laboratory Accreditation
CARB – California Air Resources Board
CAS – Number Chemical Abstract Service registry Number
CFC – Chlorofluorocarbon
CFU – Colony-Forming Unit
DEC – Department of Environmental Conservation
DEQ – Department of Environmental Quality
DHS – Department of Health Services
DOE – Department of Ecology
DOH – Department of Health
EPA – U. S. Environmental Protection Agency
ELAP – Environmental Laboratory Accreditation Program
GC – Gas Chromatography
GC/MS – Gas Chromatography/Mass Spectrometry
LOD – Limit of Detection
LOQ – Limit of Quantitation
LUFT – Leaking Underground Fuel Tank
M – Modified
MCL – Maximum Contaminant Level is the highest permissible concentration of a substance allowed in drinking water as established by the USEPA.
MDL – Method Detection Limit
MPN – Most Probable Number
MRL – Method Reporting Limit
NA – Not Applicable
NC – Not Calculated
NCASI – National Council of the Paper Industry for Air and Stream Improvement
ND Not Detected
NIOSH – National Institute for Occupational Safety and Health
PQL – Practical Quantitation Limit
RCRA – Resource Conservation and Recovery Act
SIM – Selected Ion Monitoring
TNI – The NELAC Institute
TPH – Total Petroleum Hydrocarbons

APPENDIX B – Organization Charts, Key Personnel, and Report Signatories

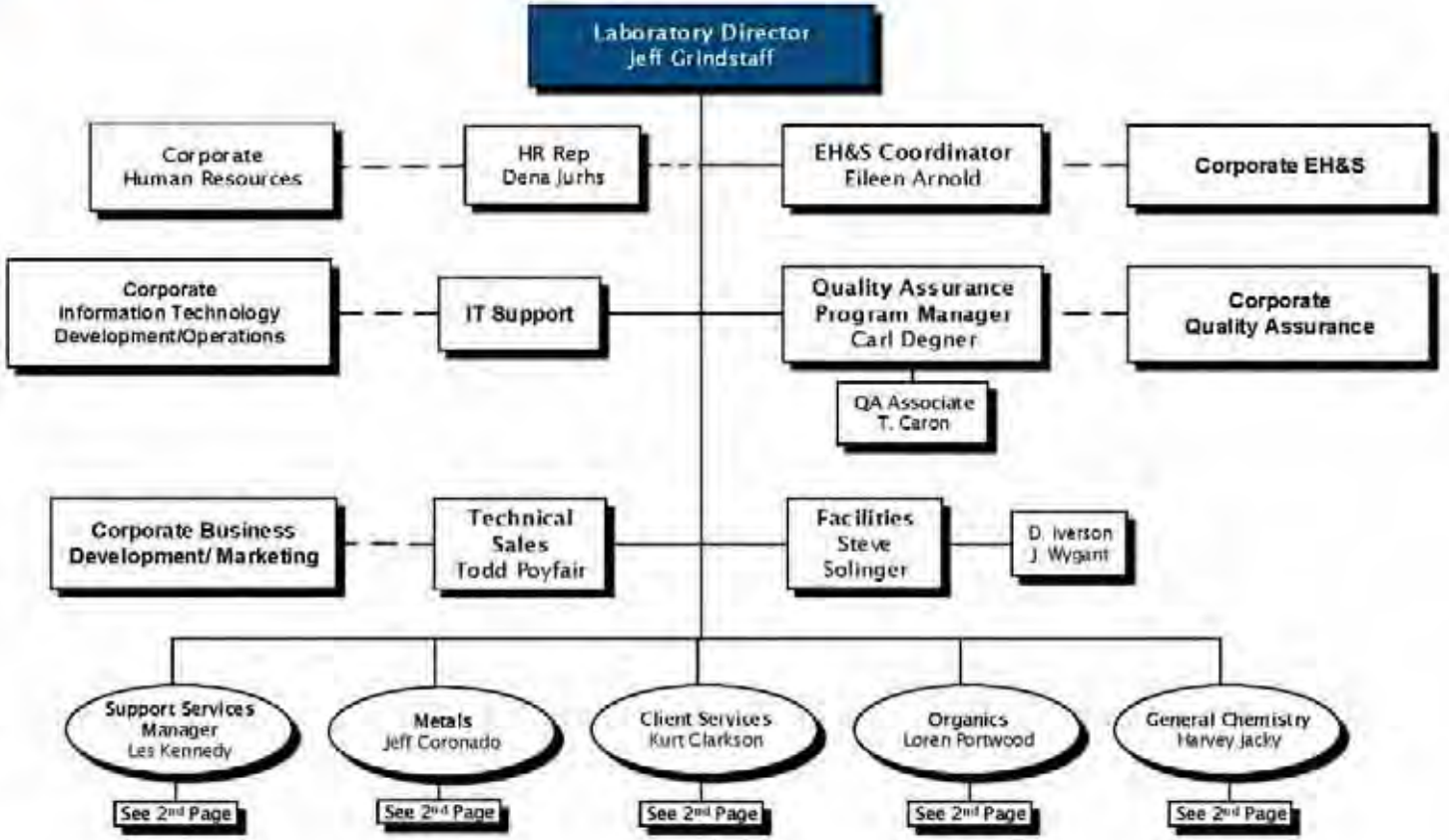
ALS Environmental USA



Revised 7/28/2016

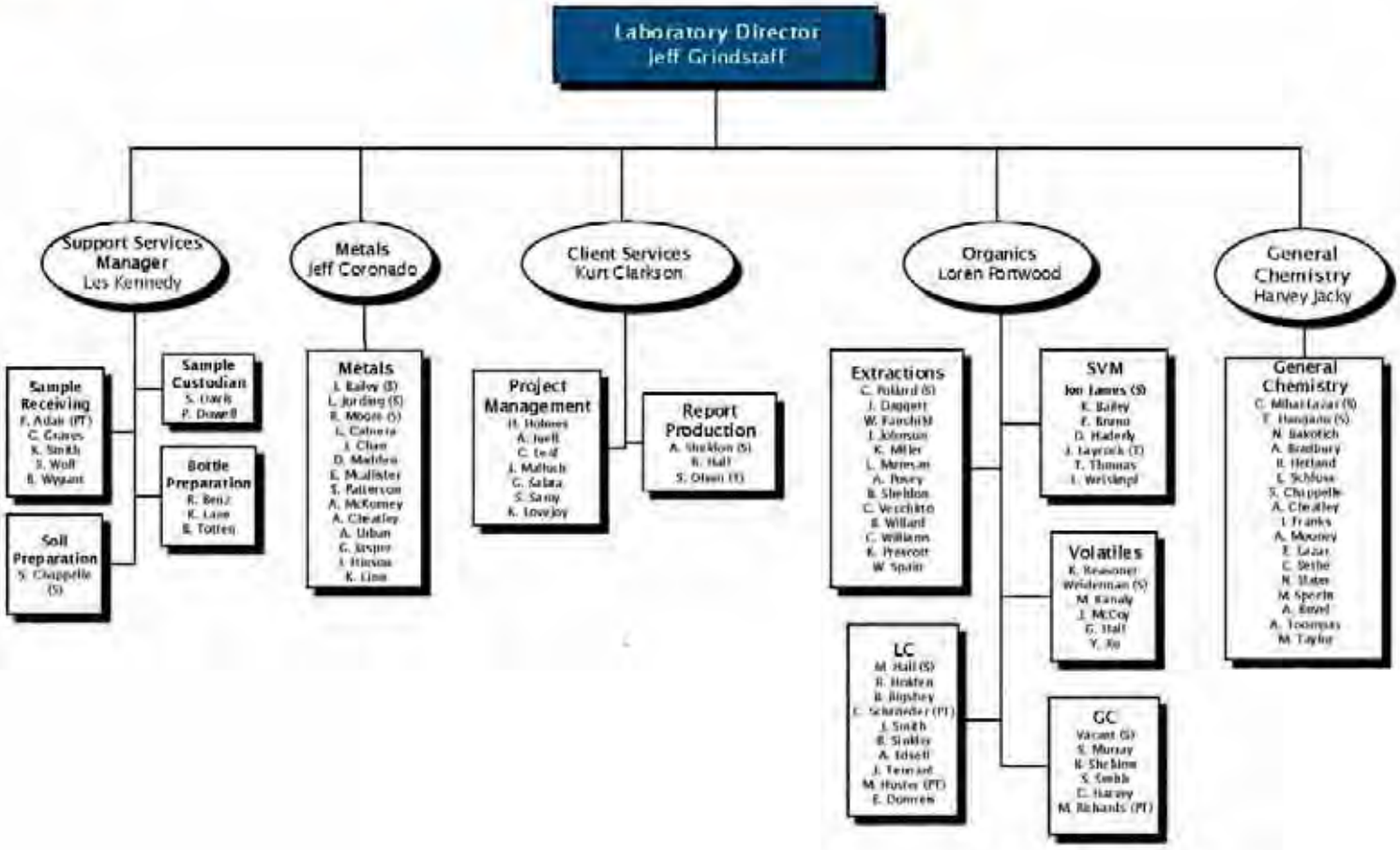
Kelso, Washington Laboratory

October 5, 2016



Kelso, Washington Laboratory

October 5, 2016
Operations



ALS Group USA, Corp.
1317 S. 13th Avenue
Kelso, WA 98626
T +1 360 577 7222 F +1 360 636 1068

JEFF GRINDSTAFF

Laboratory Director, 2010 – Present
Kelso Laboratory

Responsible for all phases of laboratory operations at the Kelso Laboratory, including project planning, budgeting and quality assurance. Primary duties include the direct management and operational oversight of the Kelso laboratory and all department managers.

PREVIOUS EXPERIENCE

Technical Manager III, Pharmaceutical GC/MS, VOC and SVOC Laboratories, 1997–2010
Columbia Analytical Services, Inc.
Kelso, WA

Primary responsibilities include leadership of the Pharmaceutical GC/MS, VOC and SVOC staff, management of method development, training, data review, tracking department workload, scheduling analyses. Responsible for ensuring data quality and timeliness. Also responsible for project management and coordination for pharmaceutical clients.

Manager, GC/MS VOA Laboratory, 1994–1997
Columbia Analytical Services, Inc.
Kelso, WA

Responsible for supervision of GC/MS VOA staff development, method development, training, data review, tracking department workload, scheduling analyses, and general maintenance and troubleshooting of GC/MS systems.

Scientist III, GC/MS VOA Laboratory, 1991–1994
Columbia Analytical Services, Inc.
Kelso, WA

Responsible included scheduling workload, data review, instrument maintenance and troubleshooting and personnel training and evaluation.

EDUCATION

Allan Hancock College –
Santa Maria, CA
AA, Liberal Arts
1986

California Polytechnic State University –
San Luis Obispo, CA
BS, Chemistry
1989

Hewlett-Packard Analytical Education Center
Mass Selective Detector Maintenance 1993

Richard Rogers Group
Leadership Training, 1996

PTI International
Sampling and Testing of Raw Materials, 2004

American Chemical Society,
1989

Mr. Grindstaff has a number of publications and presentations. For a complete list, contact ALS.

ALS Group USA, Corp.
1317 S. 13th Avenue
Kelso, WA 98626
T +1 360 577 7222 F +1 360 636 1068

KURT CLARKSON

Client Services Manager, 2016 – Present
Kelso Laboratory

Management of the Client Services Departments: Project Management, Electronic Data Deliverables and Report Generation, and Sample Management. Oversee the client services. Personally responsible for project management, technical and regulatory interpretation assistance, as well as project organization of work received by the laboratory.

PREVIOUS EXPERIENCE

Project Manager, 2015-2016
ALS Group USA, Corp.
Kelso, WA

Responsible for technical project management, ensuring overall data quality and compliance with customer requirements. Provide technical support to clients regarding laboratory application to projects. Additionally, acts as a consultant to clients regarding industrial/environmental compliance issues; serving as liaison between clients and regulatory agencies. Responsible for direct technical project management providing technical and regulatory interpretation assistance, as well as project organization of work received and reported by the laboratory.

Client Services Manager, 2013-2015
Western Environmental Testing Laboratory
Sparks, NV

Management of the Client Services Departments: Project Management, direct employees, continuous improvement initiatives, and laboratory revenue. Worked directly with clients to develop strong customer relationships. Met deadlines and quick turnaround times.

Customer Service Department Manager/Sr. Project Manager, 2012-2013
STAT Analysis Corporation
Chicago, IL

Management of the Client Services Departments: Project Management, direct employees, continuous improvement initiatives, and laboratory revenue. Worked directly with clients to develop strong customer relationships. Met deadlines and quick turnaround times.

EDUCATION

University of Alaska, Anchorage
Anchorage, AK
MBA Leadership and Operations Management
2011

University of Reno, Nevada -Reno, NV
BS Biology and Business Management
2007

ALS Group USA, Corp.
1317 S. 13th Avenue
Kelso, WA 98626
T +1 360 577 7222 F +1 360 636 1068

CARL DEGNER

Quality Manager, 2015 – Present
Kelso Laboratory

Directing the quality systems and ethics programs for the Kelso, WA laboratory facility. Responsible for ensuring that ALS quality systems and data integrity standards are implemented. Act as liaison with government entities involving quality, technical and operational issues. This includes maintaining accreditations and certifications, and maintaining all necessary documents (QA Manual, SOPs, and QA records). Act as primary point of contact during laboratory audits and provide audit responses and corrective actions. Coordinate performance audits (PE/PT testing) and conduct internal audits.

PREVIOUS EXPERIENCE

Technical Manager, SVM, 2011–2014
ALS Group USA, Corp.
Kelso, WA

Responsible for daily operation of Semi-volatiles GC/MS laboratory. This includes scheduling workloads of 3 analyst, data review, reporting and long-range planning for SVM laboratory. Work with PMs on client specific project requirements.

Technical Manager, SVM, 2001–2011
Columbia Analytical Services, Inc.
Kelso, WA

Same as above.

Scientist IV, SVM, 1998–2001
Columbia Analytical Services, Inc.
Kelso, WA

Responsible for all phases of operation of GC/MS systems, utilizing SIM and 8270C methodologies, including preparation of standards, QC verifications, data review, and reporting.

Project Chemist/Principal Organic Scientist, 1993–1998
Environ Express Laboratory
LaPorte, TX

Responsible for SV Extractions and GC/MS laboratories. Set up, operated, and maintained three HP GC/MS systems and worked with clients on technical issues.

EDUCATION

University of Houston – Houston, TX
MS Environmental Management
1998

University of Houston – Houston, TX
BS Biochemistry/Biophysical Science
1984

ALS Group USA, Corp.
1317 S. 13th Avenue
Kelso, WA 98626
T +1 360 577 7222 F +1 360 636 1068

EILEEN ARNOLD

Health, Safety and Environmental Manager, Western USA, 2015 – Present

Responsibilities include development, support and implementation of Environmental, Health and Safety policies for lab locations in the Western US, including national corporate policies for respiratory protection and hazardous waste generation. At the Kelso facility, also responsible for incident reporting and investigation, maintenance of all safety related equipment, review of monthly safety audits, and completion of all Federal and State mandated EH&S reports.

PREVIOUS EXPERIENCE

Scientist IV Metals Laboratory/Kelso Health and Safety Officer, 2012–2015
ALS Group USA, Corp.
Kelso, WA

Supervisor of the Metals reporting group responsible for ensuring timely, accurate reporting of all metals reports. Responsible for updating instrument specific data, such as MDL and control limits. Analyst for the Inductively Coupled Argon Plasma (ICAP) Emission Spectrometer. This involves digestion, instrumental analysis, and report generation for environmental samples using approved EPA techniques. Also, Environmental, Health and Safety Officer.

Scientist IV Metals Laboratory/Kelso Health and Safety Officer, 1994–2012
Columbia Analytical Services, Inc.
Kelso, WA

Same as above.

Project Chemist, 1992–1994
Columbia Analytical Services, Inc.
Kelso, WA

Duties included technical project management and customer service. Responsible for meeting the clients' needs of timely and appropriate analyses, and to act as liaison for all client-related activities within Columbia Analytical Services, Inc.

EDUCATION

Immaculata College –
Immaculata, PA
BS Chemistry
1977

ALS Group USA, Corp.
1317 S. 13th Avenue
Kelso, WA 98626
T +1 360 577 7222 F +1 360 636 1068

JEFF CORONADO

Technical Manager IV, Metals Department Manager, 1992 - Present
Kelso Laboratory

Management of the Kelso Metals Department staff and annual revenues approaching $4 million. Responsible for data quality and timeliness, annual budgeting, revenues, expenses, workload coordination, method development efforts, and resource allocation. Participation in multiple LIMS development teams responsible for defining the ALS product. Team leader for defining specifications of the Sample Preparation Module to capture preparation information across all laboratory departments.

PREVIOUS EXPERIENCE

Supervisor, GFAA Laboratory, 1989-1992
Columbia Analytical Services, Inc.
Kelso, WA

Responsibilities included supervision of metals analysis by graphite furnace atomic absorption following SW 846 and EPA CLP methodologies. Duties include workload scheduling, data review, instrument maintenance, personnel training and evaluation.

EDUCATION

Western Washington University - Bellingham, WA
BS Chemistry
1988

Western Washington University - Bellingham, WA
BA Business Administration
1985

Winter Conference on Plasma Spectrochemistry - Tucson, AZ, 2012

LC/ICP-MS Training Course - PerkinElmer, 2008

Field Immunoassay Training Course - EnSys Inc., 1995

Winter Conference on Plasma Spectrochemistry - San Diego, CA, 1994

ICP-MS Training Course - VG-Elemental, 1992

ALS Group USA, Corp.
1317 S. 13th Avenue
Kelso, WA 98626
T +1 360 577 7222 F +1 360 636 1068

HARVEY JACKY

General Chemistry Department Manager, 2008 – Present
Kelso Laboratory

Oversee the operation of the General Chemistry and Microbiology groups. Responsible for the quality and timeliness of the inorganic laboratories analytical reports, departmental budgets, workload coordination, method development efforts, cost-effectiveness, and resource allocation.

PREVIOUS EXPERIENCE

Project Manager III, 1999–2008
Columbia Analytical Services, Inc.
Kelso, WA

Responsible for technical project management, ensuring overall data quality and compliance with customer requirements, and providing technical support to clients regarding laboratory application to projects. Additionally, acts as a consultant to clients regarding industrial/environmental compliance issues; serving as liaison between clients and regulatory agencies.

Director of Project Management, 1997–1999
Coffey Laboratories
Portland, OR

Responsible for technical project management. Communicated with clients to determine needs and expectations. Monitored laboratory production and ensured the timely completion of analytical projects. Technical consultant for clients regarding environmental compliance. Supervised and managed other members of the project management team. Served as a member of the senior management team for oversight of general operations, strategic planning, finances, and policy.

Project Manager/Chemist, 1997–1999
Coffey Laboratories
Portland, OR

Responsibilities: Served as primary liaison between Coffey Laboratories and major clients. Ensured that work was completed in a timely manner and done to client specifications. Served as technical consultant regarding environmental chemistry, soil remediation, and waste water industrial compliance.

EDUCATION

Oregon State University – Corvallis, OR
BS Zoology, 1988

Oregon State University – Corvallis, OR
BS General Science, 1988

Linfield College – McMinnville, OR **General Studies**, 1981 – 1982

40-Hour Hazmat Certification, PBS Environmental, 1996

Industrial Emergency Response, SFSP Seminar, 1991

American Chemical Society, Member since 1988

Biochemical and Physical Factors Involved in the Application and Measurement of a Soil Bioremediation System, Biogeochemistry, Portland State University, 1996

ALS Group USA, Corp.
1317 S. 13th Avenue
Kelso, WA 98626
T +1 360 577 7222 F +1 360 636 1068

LOREN PORTWOOD

Organics Manager, 2016 – Present
Kelso Laboratory

Oversee the operation of the Volatiles GC/MS, Semi-volatile GC/MS and HPLC laboratories. Responsibilities include organizing and prioritizing workload, training and development of staff, working with PMs on client-specific project requirements, workload coordination, method development efforts and resource allocation. Responsible for the quality and timeliness of analytical reports. Other responsibilities include ensuring compliance with ALS QA protocols, and assisting staff with troubleshooting equipment and procedural problems.

PREVIOUS EXPERIENCE

Technical Manager, Drinking Water Laboratory/SVG Supervisor, 2008–2016
ALS Group USA, Corp.
Kelso, WA

Responsible for management of the Semi-volatile Organics Gas chromatograph and drinking water department. Also responsible for implementation and oversight of UCMR2 analyses. Perform method development. Project management of drinking water accounts. Develop SOPs for Drinking Water methods, EPA 600 methods and SW-846 methods. Operation of Varian GC/MS ion trap, Thermo GC/MS ion trap, Agilent GC/ECD, Agilent GC/FPD, Agilent GC/FID. Responsible for data interpretation, quality control and data reporting. Additional responsibilities include SOP generation; handling routine and advanced maintenance and troubleshooting of instrumentation; and assisting in the training of staff department analysts. Assists the department manager and/or other senior scientists in setting up more complex procedures. Serves as senior technical advisor for teams and projects.

Lead Analyst, 2002–2008
Columbia Analytical Services
Kelso, WA

Primary responsibilities include management of the petroleum hydrocarbon team, initiating new methods and process improvements, and staff development and training. Other duties include department wide compliance with CAS quality assurance guidelines, routine system checks, assist and encourage staff in troubleshooting equipment and procedural problems in a manner consistent with company, state and federal guidelines.

EDUCATION

Whitworth College –
Spokane, WA
BS in Chemistry, Emphasis in Biochemistry
1990

HP 5890 GC Maintenance and Troubleshooting,
Hewlett Packard, 1993

Capilliary Chromatography
Restek, 1993

HP6890 – Fast GC
Hewlett Packard, 1996

HP5890-C-GC Advanced Operations
Hewlett Packard, 1996

Purge and Trap Theory and Troubleshooting, Full Spectrum Analytics, 2001

Comprehensive HPLC.
Restek, 2002

ALS Group USA, Corp.
1317 S. 13th Avenue
Kelso, WA 98626
T +1 360 577 7222 F +1 360 636 1068

LES KENNEDY

Sample Custodian/Sample Management Manager, 2010 - Present
Kelso Laboratory

Responsible for the operation of the Sample Management, Sample Control, Bottle preparation departments, including sample receiving, courier service, sample control, storage and disposal, bottle preparation and shipping, and general freight receiving. Responsible for employee supervision, personnel evaluations, workload coordination, and adherence to all standard operating procedures within said departments. Additional duties include oversight of quarantined soil importation for laboratory testing. Is the designated Sample Custodian for the laboratory.

PREVIOUS EXPERIENCE

Project Manager/SMO Supervisor, 1999-2011
Columbia Analytical Services, Inc.
Kelso, WA

Responsible for technical project management, ensuring overall data quality and compliance with customer requirements, and serving as liaison to clients and regulatory agencies. Oversight of the daily activities in sample management department including receipt, login, storage, and proper disposal of all samples received in the laboratory.

Organic Extractions Supervisor, 1997-1999
Columbia Analytical Services, Inc.
Kelso, WA

Responsible for managing work load; directing efficiency; and ensuring that all critical holding times and QC are met each day. This involves GC/MS prep work, including extracting and GPC clean up; and subsequent sample screening of the GC/MS prep work. Additional responsibilities include data processing of GC/MS analytical runs including all steps of the data review and reporting process.

Senior Analyst, GC/MS Laboratory, 1996-1997
Columbia Analytical Services, Inc.
Kelso, WA

Primary duties were performing analyses by EPA Method 8270, SIM TCL, SIM PAH, including all steps in the data review and reporting process.

EDUCATION

Lower Columbia College
Longview, WA
Coursework, General Studies
1988-1990

Portland Bible College
Portland, OR
Bachelor of Theology,
2009

APPROVED SIGNATORIES FOR FINAL ANALYTICAL REPORTS

ALS Environmental, Kelso, WA

CLARKDON, KURT
CORONADO, JEFFREY
DEGNER, CARL
GRINDSTAFF, JEFF
HOLMES, HOWARD
JACKY, HARVEY
JUELL, AMANDA
LEAF, CHRIS
LOVEJOY, KELLEY
MALLOCH, JANET
SALATA, GREGORY
SAMY, SHAR

Update: October, 2016 Approved by: Kurt Clarkson, Client Services Manager

APPENDIX C

ALS Environmental Confidentiality Agreement

Confidentiality Agreement

The Confidentiality Agreement (the "Agreement") is entered into by and between ALS Group (hereinafter referred to as the "Company") and _____ (hereinafter referred to as "Employee").

WHEREAS, employee is presently employed by the Company in a position in which Employee will receive and have access to confidential business information and other secrets of the Company, and shall, to the best of Employee's ability, assist the Company in improving and developing the products and services of the Company; and

WHEREAS, employee is desirous of continuing such employment and receiving such disclosures of confidential business information, and assisting the Company in improving and developing its products and services.

NOW, this Agreement being a condition therefore and ancillary thereto, and in further consideration of the benefits to Employee pursuant to the employment by the Company, the receipt and sufficiency of all such consideration being hereby acknowledged by Employee, it is agreed between the Company and Employee as follows:

1. **Confidential Business Information.** Employee recognizes and agrees that the Company has certain confidential business information, including, but not limited to, compilations of information, customer lists, customer data, records, specifications, and trade secrets, and related business methods and techniques, which confidential business information are used by the Company to obtain a competitive advantage over the Company's competitors who do not know or use this information. Employee further recognizes and agrees that the protection of such confidential business information against unauthorized disclosure and use is of critical importance to the company to maintain its competitive position and Employee therefore agrees that use of, or disclose to any other person or entity, except as authorized by the Company in writing, any of the confidential business information of the Company. Employee also agrees not to disclose to the Company or utilize on the Company's behalf, any of the trade secrets or other confidential information of any of the Employee's former employers.

2. **Return of Confidential Business Information.** Upon termination of his employment for any reason, employee shall promptly deliver to the Company all drawings, manuals, letters, photographs, tapes or video recordings, records of any kind, and all copies thereof, that may be in the possession of, or under the control of, Employee pertaining to the Company's employers.

3. **Assignment of Rights to Company.** Employee agrees to assist the Company in all possible ways in the discovery, perfection, and development of new ideas, inventions, discoveries, devices, and methods in processes, all for the benefit of the Company and as its exclusive property. Employee agrees to and does hereby assign, transfer, and convey to the Company, or at the written direction of the Company and which are made, developed or conceived by Employee, either solely or jointly with others, during Employee's employment with the Company, whether prior or subsequent to the signing of this Agreement, whether made, developed or conceived by Employee during or outside of regular working hours or on or away from the

Company's premises or at Employee's expense, the expense of the Company or some other person or persons. At any time, the Employee shall execute such documents requested by the Company to confirm the rights of the Company in the ideas, inventions, discoveries, and devices, methods and processes referenced in this Section 3.

4. **Reasonableness of Covenants**. Employee specifically acknowledges and agrees as follow: (I) the covenants set forth in this Agreement are reasonable and necessary to protect the goodwill and the operations and business of the Company; (ii) the time duration of the covenants set forth in this Agreement and are reasonable and necessary to protect the goodwill and the operations and business of the Company; (iii) the geographical area limitations of the covenants set forth in this Agreement are reasonable and necessary to protect the goodwill and the operations and business of the Company; (iv) the covenants set forth in this Agreement are not oppressive to Employee and do not impose a greater restraint on Employee than is necessary to protect the goodwill and the operations and business of the Company.

5. **Remedies**. Employee recognizes that irreparable injury or damage will result to the business of the company in the event to the breach of any covenant contained in this Agreement and Employee therefore agrees that in the event of such breach on the part of the Employee, the Company shall be entitled, in addition to any legal or equitable remedies and damages available, to an injunction to restrain the violation thereof by Employee and all other persons action for or on behalf of Employee. Any claim of Employee against the Company shall not prevent the Company from enforcing any provision of this agreement. Further, in the event legal action is necessary to enforce any of Employee's obligations hereunder and the Company prevails in such legal action, the Company shall be entitled to a recovery of its attorney's fees expended in such action.

6. **Reformation**. Whenever possible, each provision of this agreement shall be interpreted in such manner as to be effective and valid under applicable law; provided, however, incase any on or more of the provisions contained in this Agreement shall, for any reason, be held to be invalid, illegal, or unenforceable in any respect, such invalidity, illegality, or unenforceability shall no affect any other provision of this agreement, and this Agreement shall be construed as if such invalid, illegal, or unenforceable provision had never been contained herein. Should a court of competent jurisdiction declare any of the provisions of this Agreement unenforceable due to any restriction of duration, territorial coverage, scene of activity, or otherwise, in lieu of declaring such provisions unenforceable, the parties hereto expressly authorize the court, to the extent permissible by law, to revise or reconstruct such provisions in a manner sufficient to cause them to be enforceable.

7. **Affiliates**. This agreement, and Employee's obligations hereunder, shall apply to any confidential business information, formulas, recipes, patterns, devices, secret inventions, processes, compilations of information, materials, ingredients, customer lists, records, specifications and trade secrets of any affiliate of the Company. For the purpose of this Agreement, the "affiliate" means any person that, directly or indirectly, controls, or controlled by, or is under common control with, another person'; "person" means any individual, corporation, partnership, joint venture, limited liability company, association, joint stock company, trust, unincorporated

organization or any other form of entity; and "control" means the power to direct or cause the direction of the management and policies of a person, directly or indirectly, whether through the ownership of voting securities by contract, or otherwise.

8. **Compelled Disclosure**. In the event that Employee is requested or required (by oral questions, interrogatories, requested for information or documents, subpoenas, civil investigative demand or similar process) to disclose any of the confidential business information of the Company, it is agreed that Employee will provide the Company with immediate notice of such request(s), so that the Company may seek an appropriate protective order or, if appropriate, waive Employee's compliance with this agreement. Employee agreed that, if in the absence of a protective order or the receipt of a waive hereunder, Employee is nonetheless, in the reasonable opinion of Employee's counsel, legally compelled to disclose the confidential business information of the Company or else stand liable for contempt or suffer other censure or penalty, Employee may, after prior notice to the Company, disclose such the confidential business information of the Company to the extent legally required.

9. **Indemnity**. Employee agrees to indemnify and hold harmless the Company, and its directors, officers, employees, agents, and attorneys, from and after the date hereof, against any and all actions, causes of action, claims, suites, proceedings, demands, assessments, demands, settlement, judgment, damages, loses, costs, and legal and other expenses arising out of or resulting from the breach or failure of Employee to Company with any covenant or agreement made herein.

10. **Choice of Law: Waiver of Trial by Jury**. This Agreement shall be construed in accordance with, and governed for all purposes by the laws of the State of Texas and obligations and undertakings of each of the parties to this contract shall be performable at Houston, Harris County. TO THE EXTENT NOT PROHIBITED BY APPLICABLE LAW, THE PARTIES HEREBY KNOWLINGLY, VOLUNTARILY, AND INTENTIONALLY WAIVE ANY RIGHT TO TRIAL BY JURY THAT THE COMPANY OR EMPLOYEE MAY HAVE IN ACTION OR PROCEEDING, IN LAW OR IN EQUITY, IN CONNECTION WITH THIS AGREEMENT, EACH PARTY REPRESENTS AND WARRANTS THAT NEITHER PARTY HAS REPRESENTED, EXPRESSLY, OR OTHERWISE THAT IT WILL NOT, IN THE EVENT OF LITIGATION, SEEK TO ENFORCE THIS RIGHT TO JURY TRIAL WAIVER. EACH PARTY ACKNOWLEDGES THAT THE OTHER PARTY HAS BEEN INCLUDED TO ENTER INTO THIS AGREEMENT BY, AMONG OTHER THINGS, THE PROVISIONS OF THE WAIVER.

11. **Waiver**. No waiver of any provision of this Agreement shall constitute a waiver of any other provision of this agreement, nor such waiver constitute a waiver of any subsequent breach of such provision.

12. **Acknowledgement of Receipt.** Employee acknowledges a receipt of a copy of this Agreement, which has been executed in multiple copies, all executed copies of that shall be deemed originals.

13. **No Promise of Employment.** It is expressly agreed that this Agreement is not a promise of future employment.

14. **Assignment**: **Survival**. This agreement shall not be assignable by Employee. This agreement and the obligations of Employee hereunder, shall survive the termination of Employee's employment with the Company.

15. **Entire Agreement**. This Agreement entered into by the Company and Employee, embodies the entire agreement and understanding between the Company and the Employee relating to the subject matter hereof, and supersedes all prior agreements and understandings relating to the employment and compensation of the Employee and may only be amended by a written agreement signed by all parties hereto.

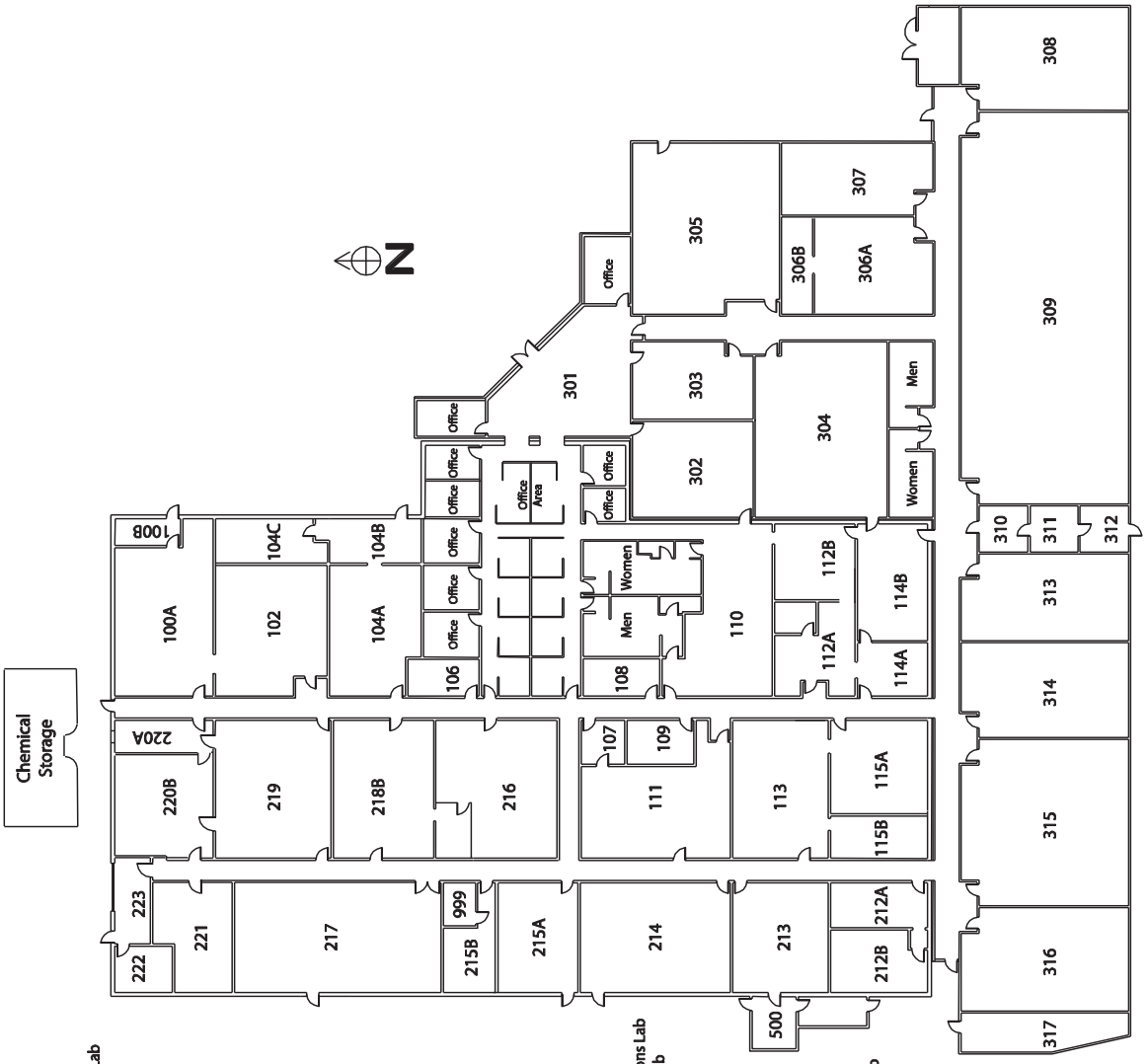
Employee Signature: _____ Date: _____

Employee Printed Name: _____

Witness: _____ Date: _____

Witness Printed Name: _____

APPENDIX D – Laboratory Floor Plan



Room #	
100A	Semivolatile Organics GC Instrument Lab
100B	Electrical Room
102	Semivolatile Organics GC Office
104 A - C	Organics LC Labs and Offices
106	Deionized Water System Room
107	Oven Room
108	Zero Headspace Extraction Lab
109	Storeroom
110	Lunch Room
111	General Chemistry Lab
112A	Microbiology Lab
112B	Copy Center
113	ICP MS Lab
114A	Grain Size
114B	Total Solids/Oven Room
115A	ICP Lab
115B	Metals Reporting
212A	Mercury and Flame AA Lab
212B	Low Level Mercury Lab
213	General Chemistry Lab
214	General Chemistry Lab
215A	Drinking Water Lab
215B	Information Technology
216	Data Review & Storage
217	Sample Storage
218	Volatiles Organics Lab
219	Semivolatile Organics GC/MS Lab
220A	Semivolatile Organics GC/MS Office
220B	Semivolatile Organics GC/MS Extractions Lab
221	Drinking Water Sample Preparation Lab
222	Purchasing Office
223	General Receiving
301	Reception Lobby
302	Lunch Room
303	Conference Room
304	Sample Storage
305	Sample Receiving
306A	Pharmaceutical Metals
306B	Pharmaceutical Microbiology
307	Pharmaceutical Instrument Lab
308	Pharmaceutical General Chemistry Lab
309	Organics Sample Preparation Lab
310	Communication Room
311	Electrical Room
312	Fire Room/Water System
313	Glass Wash Room
314	Clean Room
315	Metals Digestion Room
316	Tissue Preparation
317	Mechanical
500	Janitorial Room
999	Computer Room

Revised 9/21/10

APPENDIX E – Analytical Equipment

GENERAL CHEMISTRY/WATER CHEMISTRY LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balances (14):			
Precisa, Mettler, Ohaus, Adams models	1990-2011	LM	13
Autoclave - Market Forge Sterilmatic	1988	LM	5
Autoclave – Heidolph Brinkman 3870EP	2010	LM	3
Autotitrator – Thermo Orion 500	2007	LM	3
Calorimeters (2):			
Parr 1241 EA Adiabatic	1987	LM	4
Parr 6300 Isoparabolic	2005	LM	4
Centrifuge - Damon/IEC Model K	1992	LM	13
Colony Counter - Quebec Darkfield	1988	LM	2
Conductivity Meter (1):			
YSI Model 3200	2004	LM	4
Digestion Systems (4):			
COD (2)	1989	LM	4
Kjeldahl, Lachat 46-place (1)	1999	LM	3
Skalar Micro Digester, 120 place (1)	2016	LM	2
Dissolved Oxygen Meter - YSI Model 58 (2)	1988, 1991	LM	4
Distillation apparatus (Midi) - Easy Still (1)	2000	LM	5
Drying Ovens (12):			
Shel-Lab and VWR models	1990-2010	LM	13
Flash Point Tester (1):			
Petroleum Systems Services	2005	LM	3
Flow-Injection Analyzers (2):			
Bran-Leubbe	2002	LM	2
Lachat 8500	2007	LM	2
Ion Chromatographs (4)			
Thermo/Dionex ICS-2500	2002	LM	3
Thermo/Dionex ICS-2000	2006	LM	3
Thermo/Dionex ICS-1600	2009	LM	3
Thermo/Dionex ICS-1600	2015	LM	3
Meters (ISE and pH) (4)			
Fisher Scientific Accument Model 50	1997	LM	4
Fisher Scientific Accument Model 25	1993	LM	4
Fisher Scientific Accument Model 20	2000	LM	4
Fisher Scientific Accument Model AR25	1992	LM	4
Microscope - Olympus	1988	LM	1
Muffle Furnace- Sybron Thermolyne Model F-A1730	1991	LM	13

Shatter Box (2):			
GP 1000	1989	LM	5
SPEX 8530	2011	LM	5
Sieve Shakers (2):			
CE Tyler - Portable RX 24	1990	LM	5
WS Tyler - RX 86	1991	LM	5
Thomas-Wiley Laboratory Mill, Model 4	1989	LM	5
Total Organic Carbon (TOC) Analyzers (3)			
Coulemetrics Model 5012	1997	LM	3
Teledyne Tekmar Fusion 1	2009	LM	3
Analytik Jena 2500	2013	LM	3
Total Organic Halogen (TOX) Analyzers (2):			
Mitsubishi TOX-100	2001	LM	2
Mitsubishi TOX-200	2015	LM	2
Turbidimeter - Hach Model 2100N	1996	LM	5
UV-Visible Spectrophotometers (3):			
Beckman-Coulter DU520	2005	LM	4
Perkin Elmer Lambda 25	2008	LM	4
Abrazix	2011	LM	2
Discrete Autoanlayzer –Westco SmartChem AD20-1	2011	LM	2
Vacuum Pumps (3):			
Welch Duo-Seal Model 1376	1990	LM	13
Busch R-5 Series Single Stage	1991		
Chem Star 1402N-01	2011		
Water Baths/Incubators (5):	1986 - 2009	LM	13
Various Fisher Scientific and VWR Models			
Drill Press – Craftsman	2012	-	4

METALS LABORATORY

Equipment Description	Year Acquired	Manufacturer or Laboratory Maintained (MM/LM)	# of Trained Operators
Analytical Balance (8)			
Mettler AE 200 analytical balance	1988-2010	MM	12
Various Mettler, Sartorius, and Ohaus models			
Atomic Absorption Spectrophotometers (4):			
Perkin Elmer AAnalyst 200 Flame AA	2005	MM	3
CETAC Mercury Analyzer M-6100	2010	MM	3
Buck AA Spectrophotometer Model 205	2008	LM	3
Atomic Fluorescence Spectrophotometer (2)			
Brooks-Rand Model III	2005	LM	3
Brooks-Rand Merx	2014	LM	3
Centrifuge - IEC Model Clinical Centrifuge	1990	LM	12
Drying Oven - VWR Model 1370F	1990	LM	12

Freeze Dryers (1) - Labconco	2006	LM	5
Inductively Coupled Plasma Atomic Emission Spectrometer (ICP-AES) (2)			
Thermo Scientific Model iCAP 6500	2007	MM	3
Thermo Scientific Model iCAP 6500	2012	MM	3
Inductively Coupled Plasma Mass Spectrometers (ICP-MS) (4):			
Agilent 7700	2014	MM	2
Agilent 7800	2016	MM	2
Thermo X-Series	2006	MM	2
Nexion Model 300D	2011	MM	2
Muffle Furnace (2) - Thermolyne Furnatrol - 53600	1991, 2005	LM	5
Shaker - Burrell Wrist Action Model 75	1990	LM	12
TCLP Extractors (3)	1989, 2002	LM	5
Turbidimeter – Hach			
SEMIVOLATILE ORGANICS SAMPLE PREPARATION LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balance (3)			
Mettler PM480, AG204, AE240	1999 - 2015	MM	6
Sartorius LP3200D	2016	MM	
Centrifuge – Sorvall GLC-1 (2)	2014	LM	3
Drying Ovens (2)			
Fisher Model 655G	1991	LM	3
VWR Model 1305U	1999	LM	3
Evaporators/concentrators			
Organomation N-Evap (7)	1990-2010	LM	4
Organomation S-Evap (7)	1990-2010	LM	7
Biotage Turbovap (3)	2013 - 2016	LM	2
Extractor Heaters: Lab-Line Multi-Unit for Soxhlet and Continuous Liquid-Liquid Extractions (90)	1987-2007	LM	4
Solids Extractors:			
Sonic Bath VWR	1994	LM	3
Sonic Horn (4)	1994	LM	3
Soxtherm		LM	
Gerhardt (4)	2000	LM	2
OI Analytical (5)	2008	LM	2

Extractors, TCLP (8):			
Millipore TCLP Zero Headspace Extractors (10)	1992-2011	LM	1
TCLP 12 position Extractor/Tumbler (2)	1989-2011	LM	1
Gel Permeation Chromatography (GPC) (4)			
J2 Scientific AccuPrep (3)	2005, 2010	LM	2
Gilson (1)	2013	LM	2
Muffle Furnace (2)	2006, 2009	LM	1
Solid Phase Extractors (18) – Horizon SPE-Dex 4790	2003, 2006,2008	LM	4
Microwave Extractor – Mars 6	2014	LM	2
Edmund Buhler 3-Storey top frame VKS 'Shaker table' (1)	2016	LM	1
GC SEMIVOLATILE ORGANICS INSTRUMENT LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Gas Chromatographs (18):			
Agilent 6890 GC with Agilent 7683 Autosampler and Dual ECD Detectors (6)	2001, 2005, 2007,2011	LM	4
Agilent 6890 GC with Agilent 7683 Autosampler and Dual FPD Detectors	2003	LM	3
Agilent 7890A Dual ECD Detectors Agilent 7683B autosampler (4)	2010 - 2014	LM	4
Hewlett-Packard 5890 GC with HP 7673 Autosampler and FID Detector	1995	LM	3
Agilent 6890 with Dual FID Detectors and Agilent 7873 Autosampler (4)	2001, 2005	LM	4
Agilent 7890A Dual NPD Detectors and Agilent 7683B autosampler	2012	LM	2
Varian Ion trap GC/MS:	2003	LM	2
Varian 3800 GC w/CP8400 autosampler	2006	LM	2
Varian Saturn 2100T mass spectrometer	2003	LM	2
Thermo Ion Trap ITQ-90C GC/MS w/TriPlus autosampler	2008	LM	2
GC/MS SEMIVOLATILE ORGANICS INSTRUMENT LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balance - Mettler AB 104-S	2000	MM	6
Gas Chromatograph: Hewlett-Packard 5890 with HP 7673 autosampler and FID Detector	1994	LM	5

Semivolatile GC/MS Systems (11):			
Agilent 6890/5973 with ATAS Optic2 LVI and HP 7673 Autosampler (2)	1997, 2001	LM	5
Agilent 5890/5970 and HP 7673 Autosampler	1990	LM	5
Agilent 5890/5972 with ATAS Optic2 LVI and HP 7673 Autosampler (1)	1993, 1994	LM	5
Agilent 6890/5973 with ATAS Optic3 LVI and HP 7683 Autosampler	2005	LM	5
Agilent 6890/5973 with Agilent PTV Injector and 7683 Autosampler (2)	2007	LM	5
Agilent7890A/5975C with Agilent 7693 Autosampler (4)	2010 - 2011	LM	5
Semivolatile GC/MS/MS – Waters Quattro Micro GC Micromass with Agilent 6890, Agilent PTV Injector, 7683B Autosampler	2008	MM	1
HPLC LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balance - Mettler BB240	1994	MM	8
Drying Oven - Fisher Model 630F	1991	LM	?
Evaporator – Turbo Vap	2009	LM	8
Centrifuge (2)			
Beckman Coulter	2002	LM	8
Eppendorf	2012	LM	8
High-Performance Liquid Chromatographs (3): Agilent 1260 Infinity with Diode Array UV Detector	2011	LM	3
High-Performance LC/MS (5)			
Spectrometer - Thermo Electron TSQ Vantage LC/MS/MS and autosampler	2005	MM	2
API 5000 LC/MS/MS and SIL-20AC autosampler	2008	MM	5
AB Sciex 5500 and Shimadzu DGU 20A5	2011	MM	3
Shimadzu LC/MS 8050 with 2x LC-30AD UHPLC pumps and SIL-30AC MP autosampler	2016	MM	2
Shimadzu LC/MS 8050 with 2x LC-30AD UHPLC pumps and SIL-30AC MP autosampler	2016	MM	2
Agilent 1100 HPLC -UV/Fluorescence detector	2003	LM	1
VOLATILE ORGANICS LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**

Analytical Balance - Mettler PE 160	1989	MM	4
Fisher Vortex Mixer	1989	LM	4
Drying Ovens (1): Boekel 107801	1989	LM	4
Sonic Water Bath - Branson Model 2200	1989	LM	4
Volatile GC/MS Systems (8): Agilent 5890/5970 Tekmar 3000 Purge and Trap Concentrator Dynatech ARCHON 5100 Autosampler Agilent 6890/5973 Tekmar 3100 Purge and Trap Concentrator Encon Centurion Autosampler Agilent 6890/5973 Tekmar Velocity Purge and Trap Concentrator Tekmar Aquatech Autosampler Agilent 7980A/5975C (2) Teledyne Tekmar-Atomx Agilent 6890/5973 Encon Evolution Purge and Trap Concentrator Encon Centurion Autosampler Agilent 7890/5977A Encon Evolution Purge and Trap Concentrator Encon Centurion Autosampler Agilent 7890B/5977B Teledyne Tekmar Atomx	 1989 1995 1996 2001 2001 2001 2005 2005 2005 2010, 2011 2010, 2011 2013 2013 2013 2014 2014 2014 2016 2016	 LM LM LM LM LM LM LM LM LM LM LM LM LM LM LM LM LM LM LM	 4 4 4 4 4 4 4 4 4 3 3 4 4 4 4 4 4 3 3
Agilent 7890 GC with FID Encon Evolution Purge and Trap Concentrator Encon Centurion Autosampler	2013	LM	2
Agilent 7890 GC with FID Encon Evolution Purge and Trap Concentrator Encon Centurion Autosampler	2013 2016	LM	2

AUTOMATED DATA PROCESSING EQUIPMENT			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
1 - WAN: LIMS Sample Manager using Oracle 11gR2 Enterprise RDBMS running on Red Hat Enterprise Linux Advanced Server v.6.6 platform connected via DMVPN circuits (100 Mbps)	2013	LM	NA
1 - Network Server for reporting and data acquisition running Windows Server 2008 R2 with a 1.4 TB capacity, 1 - Application server running Windows Server 2008 R2	2012	LM	NA
Approximately 90+ HP (3015, 4000, 4014, 4050, 4200, 4250, 4300), Dell 1720dn, and Lexmark	2010 - 2015	LM	NA

M5155 printers.			
Approximately 220+ Dell/HP PC workstations running Windows XP/Windows 7 on LAN connected via 100BT/1GigE network	2010 - 2015	LM	NA
Microsoft Office 2013 Professional as the base office application suite for all PC workstations. Some systems using Microsoft Office 2003/2007/2010	1996 - 2014	LM	NA
E-mail via Exchange 2010 with webmail via Outlook Web Access. Microsoft Outlook 2013 is standard email client, with some using Outlook 2010	2011 - 2014	LM	NA
Facsimile Machines - Brother 4750e, Brother 2920, and Brother 1860	2005 - 2008	LM	NA
Copier/Scanners - BizHub 283, BizHub 600, BizHub 601 (2), BizHub 654, BizHUb754e (2), BizHub 951, BizHub 1050.	2005 - 2015	LM	NA
Thru-Put, MARRS, Stealth, Harold, Blackbird, EDDGE, CASLIMS, & LabCoat reporting software systems.	1998 - 2014	LM	NA
Data processing terminals (79) - EnviroQuant, Target, Saturn, MassHunter, Chromeleon, MassLynx, Insight.	1996 - 2016	LM	NA

APPENDIX F - Containers, Preservation and Holding Times

DETERMINATION[a]	MATRIX[b]	CONTAINER[C]	PRESERVATION	HOLDING TIME
Bacterial Tests				
Coliform, Colilert (SM 9223)	W, DW	P, Bottle or Bag	Cool, 4°C, 0.008% $Na_2S_2O_3$[d]	6-24 hours[e]
Coliform, Fecal and Total (SM 9221, 9222D)	W, S, DW	P,G	Cool, 4°C, 0.008% $Na_2S_2O_3$[d]	6-24 hours[e]
Enterococci (Enterolert)	W	P	Cool, 4°C, 0.008% $Na_2S_2O_3$[d]	8 hours
Inorganic Tests				
Acidity (SM 2310B)	W	P,G	Cool, 4°C	14 days[EPA]
Alkalinity (SM 2320B)	W, DW	P,G	Cool, 4°C	14 days[EPA]
Ammonia (SM 4500 NH_3)	W, DW	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Biochemical Oxygen Demand(SM 5210B)	W	P,G	Cool, 4°C	48 hours
Bromate (EPA 300.1)	W, DW	P,G	50mg/L EDA, cool to 4°C	28 days
Bromide (EPA 300.1)	W, DW	P,G	None Required	28 days
Chemical Oxygen Demand (SM 5220C)	W	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Chloride (EPA 300.0)	W, DW	P,G	None Required	28 days
Chloride (EPA 9056)	W, S	P,G	Cool, 4°C	28 days
Chlorine, Total Residual (SM 4500 Cl F)	W, S	P,G	None Required	24 hours
Chlorite (EPA 300.1)	W, DW	P,G	50mg/L EDA, cool to 4°C	14 days
Chlorophyll-A (SM 11200H)	W	G Amber	Cool, 4°C	Analyze immediately
Chromium VI (EPA 7196A)	W	P,G	Cool, 4°C	24 hours
Color (SM 2120B)	W, DW	P,G	Cool, 4°C	48 hours
Cyanide, Total and Amenable to Chlorination (EPA 335.4, 9010, 9012) (SM 4500 CN E,G)	W, S, DW	P,G	Cool, 4°C, NaOH to pH>12, plus 0.6 g Ascorbic Acid	14 days
Cyanide, Weak Acid Dissociable (SM 4500 CN I)	W, S	P,G	Cool, 4°C, NaOH to pH >12	14 days

DETERMINATION[a]	MATRIX[b]	CONTAINER[C]	PRESERVATION	HOLDING TIME
Ferrous Iron (ALS SOP)	W, D	G Amber	Cool, 4°C	24 hours
Fluoride (EPA 300.0, 9056, SM 4500 F-C)	W, S	P,G	Cool, 4°C	28 days
Formaldehyde (ASTM D6303)	W	G Amber	Cool, 4°C	48 hours
Hardness (SM 2340C)	W, DW	P,G	HNO_3 to pH<2	6 months
Hydrogen Ion (pH) (SM 4500H B)	W, DW	P,G	None Required	Analyze immediately
Kjeldahl and Organic Nitrogen (ASTM D3590-89)	W	P,G	Cool, 4°C, H H_2SO_4 to pH<2	28 days
Nitrate (EPA 300.0)	W, DW	P,G	Cool, 4°C	48 hours
Nitrate (EPA 353.2)	W, S	P,G	Cool, 4°C, H_2SO_4 to pH<2	48 hours
Nitrate (EPA 9056)	W, S	P,G	Cool, 4°C	Analyze immediately
Nitrate-Nitrite (EPA 353.2)	W, DW	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Nitrite (EPA 300.0)	W, DW	P,G	Cool, 4°C	48 hours
Nitrite (EPA 353.2)	W, S	P,G	Cool, 4°C, H_2SO_4 to pH<2	48 hours
Nitrite (EPA 9056)	W, S	P,G	Cool, 4°C	Analyze immediately
Nitrocellulose	S	G	Cool, 4°C	28 days
Oil and Grease, Hexane Extractable Material (EPA 1664)	W	G, Teflon Lined Cap	Cool, 4°C, H_2SO_4 or HCL to pH<2	28 days
Organic Carbon, Total (9060 & SM 5310 C)	W	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Organic Carbon, Total (ASTM-D4129)	S	P,G	Cool, 4°C	28 days
Organic Halogens, Adsorbable (EPA 1650B)	W	G, Teflon Lined Cap	Cool, 4°C, HNO_3 to pH<2	6 months
Organic Halogens, Total (EPA 9020)	W	G, Teflon Lined Cap	Cool, 4°C, H_2SO_4 to pH<2, No headspace	28 days
Orthophosphate (SM 4500 P-E)	W, DW	P,G	Cool, 4°C	Analyze immediately
Oxygen, Dissolved (Probe) (SM 4500O G)	W, DW	G, Bottle and Top	None Required	Analyze immediately
Oxygen, Dissolved (Winkler)	W, DW	G, Bottle and Top	Fix on Site and Store in Dark	8 hours
Perchlorate (EPA 314.0)	W, DW ,S	P,G	Protect from temp. extremes	28 days

DETERMINATION[a]	MATRIX[b]	CONTAINER[C]	PRESERVATION	HOLDING TIME
Phenolics, Total (EPA 420.1, 9056)	W, S	G Amber	Cool, 4°C, H_2SO_4 to pH<4	28 days
Phosphorus, Total (EPA 365.3)	W	P,G	Cool, 4°C, H_2SO_4 to pH<2	28 days
Residue, Filterable (TDS) (SM 2540C)	W	P,G	Cool, 4°C	7 days
Residue, Nonfilterable (TSS) (SM 2540D)	W	P,G	Cool, 4°C	7 days
Residue, Settleable (SM 2540F)	W	P,G	Cool, 4°C	48 hours
Residue, Total (SM 2540B)	W	P,G	Cool, 4°C	7 days
Residue, Volatile (EPA 160.4)	W	P,G	Cool, 4°C	7 days
Silica (SM 4500 SiO2 C)	W	P Only	Cool, 4°C	28 days
Specific Conductance (SM 2510 B)	W, DW	P,G	Cool, 4°C	28 days
Sulfate (EPA 300.0)	W, DW	P,G	Cool, 4°C	28 days
Sulfate (EPA 9056)	W, S	P,G	Cool, 4°C	28 days
Sulfide (9030/934)	W, S	P,G	Cool, 4°C, Add Zinc Acetate, plus Sodium Hydroxide to pH>9	7 days
Sulfide (SM 4500 S_2 D)	W	P,G	Cool, 4°C, Add Zinc Acetate, plus Sodium Hydroxide to pH>9	7 days
Sulfide (SM 4500 S_2 F)	W	P,G	Cool, 4°C, Add Zinc Acetate, plus Sodium Hydroxide to pH>9	7 days
Sulfite (SM 4500 SO_3 B)	W	P,G	None Required	24 hours
Sullfides, Acid Voaltile	S	G	Cool, 4°C	14 days
Surfactants (MBAS) (SM 5540 C)	W	P,G	Cool, 4°C	48 hours
Tannin and Lignin (SM 5550B)	W	P,G	Cool, 4°C	28 days
Turbidity (EPA 180.1)	W, DW	P,G	Cool, 4°C	48 hours
Metals				
Arsenic Species 1632	W	G	HCL to pH<2, Cool < 4°C	28 days
Mercury (1631E)	W	F	Cool, 4°C, HCl or H_2SO_4 to pH<2	90 days

DETERMINATION[a]	MATRIX[b]	CONTAINER[C]	PRESERVATION	HOLDING TIME
Mercury (1631E)	S	F	Freeze < -15°C	1 Yr
Mercury (7471)	S	P,G	Cool, 4°C	28 days
Mercury (EPA 245.1, 7470, 7471)	W, DW	P,G	HNO_3 to pH<2	28 days
Metals (200.7, 200.8, 200.9, 6010, 6020)	W, DW	P,G	HNO_3 to pH<2	6 months
Metals (200.7, 200.8, 200.9, 6010, 6020)	S	G, Teflon Lined cap	Cool, 4°C	6 months
Methyl Mercury 1630	W, S, T	F	HCL to pH<2	6 months
Volatile Organics				
Gasoline Range Organics (8015, NWTPH-Gx)	W	G, Teflon-Lined, Septum Cap	Cool, 4°C, HCl to pH<2, No headspace	14 days
Gasoline Range Organics (8015, NWTPH-Gx)	S	G, Teflon-Lined Cap	Cool, 4°C, Minimize Headspace	14 days
Purgeable Halocarbons (624, 8260)	W	G, Teflon-Lined, Septum Cap	No Residual Chlorine Present; HCl to pH<2, Cool, 4°C, No Headspace	14 days
Purgeable Halocarbons (624, 8260)	W	G, Teflon-Lined, Septum Cap	Residual Chlorine Present; 10% $Na_2S_2O_3$, HCl to pH<2, Cool, 4°C	14 days
Purgeable Halocarbons (8260)	S	G, Teflon-Lined Cap	Cool, 4°C, Minimize Headspace	14 days
Purgeable Halocarbons (8260)	S	Method 5035	Terracore/Encore device, Freeze at -20°C Methanol, Cool, 4C	48 hrs to prepare from device, 14 days after preparing.
Purgeable Halocarbons (8260)	S	Method 5035	Sodium Bisulfate Cool, 4°C	48 hrs to prepare, 14 days after preparation
Purgeable Aromatic Hydrocarbons (including BTEX and MTBE 624, 8260)	W	G, Teflon-Lined,Septum Cap, No Headspace	No Residual Chlorine Present: HCl to pH<2, Cool, 4°C, No Headspace	14 days
Purgeable Aromatic Hydrocarbons (including BTEX and MTBE 624, 8260)	W	G, Teflon-Lined,Septum Cap, No Headspace	Residual Chlorine Present: 10% $Na_2S_2O_3$, HCl to pH<2, Cool 4°C	14 days

DETERMINATION[a]	MATRIX[b]	CONTAINER[C]	PRESERVATION	HOLDING TIME
Purgeable Aromatic Hydrocarbons (including BTEX and MTBE 624, 8260)	S	G, Teflon–Lined Cap	Cool, 4°C, Minimize Headspace	14 days
Purgeable Aromatic Hydrocarbons (including BTEX and MTBE 624, 8260)	S	Method 5035	Encore, Freeze at -20°C Methanol, Cool, 4C	48 hr to prepare from Encore, 14 days after preparation.
Purgeable Aromatic Hydrocarbons (including BTEX and MTBE 624, 8260)	S	Method 5035	Sodium Bisulfate, Cool, 4°C	48 hr to prepare from Encore, 14 days after preparation
Acrolein, Acrylonitrile, Acetonitrile (624, 8260)	W	G, Teflon – Lined Septum Cap	Adjust pH to 4–5, Cool, 4°C, No headspace	14 days
2–chloroethyl vinyl ether (8260)	W	G, Teflon – Lined Septum Cap	Cool, 4°C, Minimize Headspace	7 days
Semivolatile Organics				
Nonylphenols	W	G, Teflon–Lined Cap	H_2SO_4 to pH<2, Cool, 4°C	28 days until extraction;40 days after extraction
Organotins (ALS SOP)	W, S	G, Teflon–Lined Cap	Cool, 4°C	7[f] days until extraction;40 days after extraction
Otto Fuel		G, Teflon–Lined Cap	Cool, 4°C	7[f] days until extraction;40 days after extraction
Methanol in Process Liquid NCASI 94.03	L	G, Teflon–Lined Cap	Cool, 4°C	30 days
HAPS – Condensates NCASI 99.01		G, Teflon–Lined Cap	Cool, 4°C	14/30 days
HAPS – Impinger/Canisters NCASI 99.02			Cool, 4°C	21 days
Perfluorinated Compounds HPLC/MS/MS	W	P	Cool, 4°C	14 days until extraction; 40 days after extraction
PBDE/PBB – ROHS GC/MS	W, S, T	G	Cool, 4°C	40 days after extraction

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	HOLDING TIME
Pharma Personal Care Products (EPA 1694)	W, S	Amber G, Teflon-Lined Cap	Cool, < 6°C	7[f] days until extraction; 30 days after extraction
Nitroaromatics and Nitramines (EPA 8330B)	W, S	G, Teflon-Lined Cap	Cool, 4°C	7[f] days until extraction; 40 days after extraction
Nitroaromatics/Nitroamines HPLC/MS/MS	W, S, T	G	Cool, 4°C Tissues < -10 C	7[f] days until extraction; 40 days after extraction
Organic acids HPLC/MS/MS	W	G, Teflon-Lined, Septum Cap	H_2SO_4 to pH<2, Cool, 4[δ]C	14 days
Petroleum Hydrocarbons, Extractable (Diesel-Range Organics) (EPA 8015)	W, S	G, Teflon-Lined Cap	Cool, 4°C	7[f] days until extraction, 40 days after extraction
Alcohols and Glycols (EPA 8015)	W, S	G, Teflon-Lined Cap	Cool, 4°C[g]	7[f] days until extraction; 40 days after extraction
Acid Extractable Semivolatile Organics (EPA 625, 8270)	W	G, Teflon-Lined Cap	Cool, 4°C[g]	7[f] days until extraction; 40 days after extraction
Base/Neutral Extractable Semivolatile Organics (EPA 625, 8270)	W	G, Teflon-Lined Cap	Cool, 4°C[g]	7[f] days until extraction; 40 days after extraction
Acid Extractable Semivolatile Organics (EPA 8270)	S	G, Teflon-Lined Cap	Cool, 4°C[g]	14 days until extraction; 40 days after extraction
Base/Neutral Extractable Semivolatile Organics (EPA 8270)	S	G, Teflon-Lined Cap	Cool, 4°C[g]	14days until extraction; 40 days after extraction
Chlorinated Herbicides (EPA 8151)	W, S	G, Teflon-Lined Cap	Cool, 4°C[g]	7[f] days until extraction; 40 days after extraction
Chlorinated Phenolics (EPA 1653)	W	G, Teflon-Lined Cap	H_2SO_4 to pH<2, Cool, 4[δ]C[g]	30 days until extraction; 30 days after extraction

DETERMINATION[a]	MATRIX[b]	CONTAINER[C]	PRESERVATION	HOLDING TIME
Polynuclear Aromatic Hydrocarbons (EPA 625, 8270)	W, S	G, Teflon-Lined Cap	Cool, 4°C, Store in Dark[g]	7[f] days until extraction; 40 days after extraction
Organochlorine Pesticides and PCBs (EPA 608, 8081, 8082, GC/MS/MS)	W, S	G, Teflon-Lined Cap	Cool, 4°C	7[f] days until extraction; 40 days after extraction
Organophosphorus Pesticides (EPA 8141, GC/MS/MS)	W, S	G, Teflon-Lined Cap	Cool, 4°C, Store in Dark[g]	7[f] days until extraction; 40 days after extraction
Nitrogen- and Phosphorus-Containing Pesticides (EPA 8141)	W,S	G, Teflon-Lined Cap	Cool, 4°C[g]	7[f] days until extraction; 40 days after extraction
Drinking Water Organics				
Purgeable Organics (EPA 524.2)	DW	G, Teflon-Lined, Septum cap	Ascorbic Acid, HCl to pH≤2, Cool, 4°C, No Headspace	14 days
EDB, DBCP, and TCP (EPA 504.1)	W	G, Teflon-Lined Cap	Cool, 4°C, 3 mg $Na_2S_2O_3$, No Headspace	14 days
Chlorinated Herbicides (EPA 515.4)	DW	G, Amber, Teflon-Lined Cap	If Res.Cl, 2mg/40 mL NaS; Cool , <6°C	14 days until extraction; 21 days after extraction
Chlorinated Pesticides (EPA 508.1, 525.2)	DW	G, Amber, Teflon-Lined Cap	50 mg/L NaS, HCl to pH≤ 2;Cool 4°C	14 days until extraction; 30 days after extraction
Diquat and Paraquat (EPA 549.2)	DW	G, Amber, Teflon-Lined Cap	100 mg/L $Na_2S_2O_3$, Res.Cl.Cool 4°C[d]	7 days until extraction; 21 days after extraction
Endothall (EPA 548.1)	DW	G, Amber, Teflon-Lined Cap	Cool, 4°C	7 days until extraction; 14 days after extraction
Haloacetic Acids (EPA 552.2)	DW	G, Amber, Teflon-Lined Cap	100 mg/L NH_4Cl, Cool, 4°C	14 days until extraction; 7 days after extraction
Semivolatile Organics (EPA 525.2)	DW	G, Amber, Teflon-Lined Cap	50 mg/L NaS, HCl to pH≤ 2;Cool, 4°C	14 days until extraction; 30 days after extraction

DETERMINATION[a]	MATRIX[b]	CONTAINER[C]	PRESERVATION	HOLDING TIME
Nitrosoamines (EPA 521)	DW	G, Amber, Teflon-Lined Cap	Dechlorinate at collection[g]	14 days until extraction; 28 days after extraction
Selected Pesticides and Flame Retardants (EPA 527)	DW	G, Amber, Teflon-Lined Cap	See Method, Cool, 4°C	14 days until extraction; 28 days after extraction
Toxicity Characteristic Leaching Procedure (TCLP)				
Semivolatile Organics (EPA 1311/8270)	HW	G, Teflon - Lined Cap	Sample: Cool, 4°C, Store in dark[g]	14 days until TCLP extraction
			TCLP extract: Cool, 4°C, Store in dark[g]	7 days until extraction; 40 days after extraction
Organochlorine Pesticides (EPA 1311/8081)	HW	G, Teflon Lined Cap	Sample: Cool, 4°C	14 days until TCLP extraction
			TCLP extract: Cool, 4°C	7 days until extraction;40 days after extraction
Chlorinated Herbicides (EPA 1311/8151)	HW	G, Teflon Lined Cap	Sample: Cool, 4°C	14 days until TCLP extraction
			TCLP extract: Cool, 4°C	7 days until extraction;40 days after extraction
Mercury(EPA 1311/7470)	HW	P,G	Sample: Cool, 4°C	28 days until extraction
			TCLP extract: HNO_3 to pH<2	28 days after extraction
Metals, except Mercury (EPA 1311/6010)	HW	P,G	Sample: Cool, 4°C	180 days until extraction;
			TCLP extract: HNO_3 to pH<2	14 days until TCLP extraction
Volatile Organics (EPA 1311/8260)	HW	G, Teflon Lined Cap	Sample: Cool, 4°C, Minimize Headspace	14 days until TCLP extraction
			Extract: Cool 4°C, HCL to pH,2, No Headspace	14 days after extraction

a For EPA SW-846 methods the method listed generically, without specific revision suffixes
b DW = Drinking Water, W = Water; S = Soil or Sediment; HW = Hazardous Waste
c P = Polyethylene; G = Glass, F- Fluoropolymer
d For chlorinated water samples
e The maximum holding time dependent upon the geographical proximity of sample source to the lab.
f Fourteen days until extraction for soil, sediment, and sludge samples.
g If the water sample contains residual chlorine, 10% sodium thiosulfate is used to dechlorinate.

APPENDIX G – Standard Operating Procedures

Corporate General and Quality Assurance SOPs

SOP TITLE	SOP ID	Revision
Laboratory Ethics and Data Integrity	CE-GEN001	3.00
(proprietary- client specific)	CE-GEN002	1.00
Records Management Policy	CE-GEN003	2.00
Preventive Action	CE-GEN004	1.00
Document Control	CE-GEN005	2.00
Data Recall	CE-GEN006	2.00
Procurement and Control of Laboratory Services and Supplies	CE-GEN007	2.00
Method Development	CE-GEN008	1.00
Establishing Standard Operating Procedures	CE-GEN009	2.00
Handling Customer Feedback	CE-GEN010	1.00
Assigning and TSR to a Project	CE-GEN011	0.00
Policy for the Use of Accreditation Organization Names, Symbols, and Logos	CE-GEN012	1.00
(proprietary – client specific)	CE-GEN013	0.00
(proprietary- client specific)	CE-GEN014	0.00
Internal Audits	CE-QA001	2.00
Manual Integration Policy	CE-QA002	2.00
Training Policy	CE-QA003	2.00
Qualification of Subcontract Laboratories	CE-QA004	1.00
Laboratory Management Review	CE-QA005	2.00
Proficiency Testing Sample Analysis	CE-QA006	2.00
Making Entries onto Analytical Records	CE-QA007	2.00
Nonconformance and Corrective Action	CE-QA008	2.00
Control Limits	CE-QA009	1.00
Estimation of Uncertainty of Analytical Measurements	CE-QA010	1.00
Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation	CE-QA011	1.00
Quality of Reagents and Standards	CE-QA012	1.00

LABORATORY SOPs

SOP TITLE	SOP ID	Revision
DATA ARCHIVING	ADM-ARCH	6
DOCUMENTING LABORATORY BALANCE AND TEMPERATURE CHECKS	ADM-BAL	7
SAMPLE BATCHES	ADM-BATCH	10
CONTROL CHARTING QUALITY CONTROL DATA	ADM-CHRT	3
DEPARTMENT OF DEFENSE PROJECTS LABORATORY PRACTICES AND PROJECT MANAGEMENT	ADM-DOD	6
DEPARTMENT OF DEFENSE PROJECTS LABORATORY PRACTICES AND PROJECT MANAGEMENT - QSM 5.0	ADM-DOD5	0
LABORATORY DATA REVIEW PROCESS	ADM-DREV	8
CONTINGENCY PLAN FOR LABORATORY EQUIPMENT FAILURE	ADM-ECP	3
METHOD VALIDATION DOCUMENTATION	ADM-MDLC	4
MANUAL INTEGRATION OF CHROMATOGRAPHIC PEAKS	ADM-MI	0
PROJECT MANAGEMENT	ADM-PCM	12
DATA REPORTING AND REPORT GENERATION	ADM-RG	9
REAGENT AND STANDARDS LOGIN AND TRACKING	ADM-RLT	5
SUPPORT EQUIPMENT MONITORING AND CALIBRATION	ADM-SEMC	13
SIGNIFICANT FIGURES	ADM-SIGFIG	8
SOFTWARE QUALITY ASSURANCE AND DATA SECURITY	ADM-SWQADATA	0
ALS KELSO TRAINING PROCEDURE	ADM-TRAIN	2
CHECKING VOLUMETRIC LABWARE	ADM-VOLWARE	4
COLIFORM, FECAL	BIO-9221FC	9
COLIFORM, TOTAL	BIO-9221TC	6
COLIFORM, TOTAL (MEMBRANE FILTER PROCEDURE)	BIO-9222B	0
COLIFORM, FECAL (MEMBRANE FILTER PROCEDURE)	BIO-9222D	4
COLILERT® , COLILERT-18®, & COLISURE®	BIO-9223	9
ENTEROLERT	BIO-ENT	2
HEPTEROTROPHIC PLATE COUNT	BIO-HPC	7
MICROBIOLOGY QUALITY ASSURANCE AND QUALITY CONTROL	BIO-QAQC	16
SHEEN SCREEN/OIL DEGRADING MICROORGANISMS	BIO-SHEEN	3

SOP TITLE	SOP ID	Revision
SEPARATORY FUNNEL LIQUID-LIQUID EXTRACTION	EXT-3510	11
CONTINUOUS LIQUID – LIQUID EXTRACTION	EXT-3520	16
SOLID PHASE EXTRACTION	EXT-3535	7
SOXHLET EXTRACTION	EXT-3540	11
AUTOMATED SOXHLET EXTRACTION	EXT-3541	10
ULTRASONIC EXTRACTION	EXT-3550	11
WASTE DILUTION EXTRACTION	EXT-3580	7
SILICA GEL CLEANUP	EXT-3630	5
GEL PERMEATION CHROMATOGRAPHY	EXT-3640A	9
REMOVAL OF SULFUR USING COPPER	EXT-3660	7
REMOVAL OF SULFUR USING MERCURY	EXT-3660M	4
SULFURIC ACID CLEANUP	EXT-3665	6
CARBON CLEANUP	EXT-CARCU	4
DIAZOMETHANE PREPARATION	EXT-DIAZ	7
FLORISIL CLEANUP	EXT-FLOR	6
ORGANIC EXTRACTIONS GLASSWARE CLEANING	EXT-GC	7
PERCENT LIPIDS IN TISSUE	EXT-LIPID	5
EXTRACTION METHOD FOR ORGANOTINS IN SEDIMENTS, WATER, AND TISSUE	EXT-OSWT	8
PREPARATION OF REAGENTS AND BLANK MATRICES USED IN SEMIVOLATILE ORGANICS ANALYSIS	EXT-REAG	3
ADDITION OF SPIKES AND SURROGATES	EXT-SAS	10
MEASURING SAMPLE WEIGHTS AND VOLUMES FOR ORGANIC ANALYSIS	EXT-WVOL	4
FACILITY AND LABORATORY CLEANING	FAC-CLEAN	2
OPERATION AND MAINTENANCE OF LABORATORY REAGENT WATER SYSTEMS	FAC-WATER	2
FLASHPOINT DETERMINATION – SETAFLASH	GEN-1020	8
COLOR	GEN-110.2	7
TOTAL SOLIDS	GEN-160.3	14
SOLIDS, TOTAL VOLATILE AND PERCENT ASH IN SOIL AND SOLID SAMPLES	GEN-160.4	8
SETTEABLE SOLIDS	GEN-160.5	5

SOP TITLE	SOP ID	Revision
HALIDES, ADSORBABLE ORGANIC (AOX)	GEN-1650	4
GRAVIMETRIC DETERMINATION OF HEXANE EXTRACTABLE MATERIAL (1664)	GEN-1664	10
ALKALINITY TOTAL	GEN-2320	9
HARDNESS, TOTAL	GEN-2340	10
DETERMINATION OF INORGANIC ANIONS IN DRINKING WATER BY ION CHROMATOGRAPHY	GEN-300.1	8
ACIDITY	GEN-305.2	5
PERCHLORATE BY ION CHROMATOGRAPHY	GEN-314.0	14
CHLORIDE (TITRIMETRIC, MERCURIC NITRATE)	GEN-325.3	5
CHLORINE, TOTAL/FREE RESIDUAL	GEN-330.4	3
TOTAL RESIDUAL CHLORINE – METHOD 330.5	GEN-330.5	2
AMMONIA BY FLOW INJECTION ANALYSIS	GEN-350.1	12
NITRATE/NITRITE, NITRITE BY FLOW INJECTION ANALYSIS	GEN-353.2	10
PHOSPHORUS DETERMINATION USING COLORMETRIC PROCEDURE	GEN-365.3	13
PHENOLICS, TOTAL	GEN-420.1	15
AMMONIA AS NITROGEN BY ION SPECIFIC ELECTRODE	GEN-4500 NH3 E	7
DISSOLVED SILICA	GEN-4500 SIO2C	3
SILICA DETERMINATION USING SMARTCHEM METHOD	GEN-4500 SiO2E	2
NITRITE BY COLORIMETRIC PROCEDURE	GEN-4500NO2 B	3
ORTHOPHOSPHATE DETERMINATION USING COLORIMETRIC PROCEDURE	GEN-4500-P-E	2
SULFIDE, METHYLENE BLUE	GEN-4500S2D	3
SULFIDE, TITRIMETRIC (IODINE)	GEN-4500S2F	3
HALOGENS TOTAL AS CHLORIDE BY BOMB COMBUSTION	GEN-5050	3
BIOCHEMICAL OXYGEN DEMAND	GEN-5210B	6
HALIDES, ADSORBABLE ORGANIC (AOX) – SM 5320B	GEN-5320B	3
AQUATIC HUMIC SUBSTANCES	GEN-5510B	1
DETERMINATION OF METHYLENE BLUE ACTIVE SUBSTANCES (MBAS)	GEN-5540C	7
TANNIN AND LIGNIN	GEN-5550	6
HALIDES, TOTAL ORGANIC (TOX)	GEN-9020	9

SOP TITLE	SOP ID	Revision
HALIDES, EXTRACTABLE ORGANIC (EOX)	GEN-9020M	4
TOTAL SULFIDES BY METHYLENE BLUE DETERMINATION	GEN-9030	10
TOTAL CARBON IN SOIL	GEN-ASTM	9
SULFIDES, ACIDS VOLATILE	GEN-AVS	7
HEAT OF COMBUSTION	GEN-BTU	5
CHLOROPHYLL-a BY COLORIMETRY	GEN-CHLOR	3
TOTAL CYANIDES AND CYANIDES AMENABLE TO CHLORINATION	GEN-CN	19
CYANIDE, WEAK ACID DISSOCIABLE	GEN-CNWAD	2
CHEMICAL OXYGEN DEMAND	GEN-COD	9
CONDUCTIVITY IN WATER AND WASTES	GEN-COND	10
CORROSIVITY TOWARDS STEEL	GEN-CORR	2
HEXAVALENT CHROMIUM – COLORIMETRIC	GEN-CR6	13
STANDARD TEST METHODS FOR DETERMINING SEDIMENT CONCENTRATION IN WATER SAMPLES	GEN-D3977	2
CARBONATE (CO3) BY EVOLUTION AND COLUMETRIC TITRATION	GEN-D513-82M	2
SULFIDE, SOLUBLE DETERMINATION OF SOLUBLE SULFIDE IN SEDIMENT	GEN-DIS.S2	3
BULK DENSITY OF SOLID WASTE FRACTIONS	GEN-E1109	1
FREE CYANIDE IN WATER, WASTEWATER, AND SOIL BY MICRODIFFUSION	GEN-FCN	0
FDA EXTRACTABLES	GEN-FDAEX	2
FERROUS IRON IN WATER	GEN-FeII	5
FLUORIDE BY ION SELECTIVE ELECTRODE	GEN-FISE	9
FORMALDEHYDE COLORIMETRIC DETERMINATION	GEN-FORM	3
HYDROGEN PEROXIDE BY PERMANGANATE TITRATION	GEN-H2O2	3
HYDROGEN HALIDES BY ION CHROMATOGTRAPHY (METHOD 26)	GEN-HA26	4
HYDAZINE IN WATER USING COLORIMETRIC PROCEDURE	GEN-HYD	2
TOTAL SULFUR FOR ION CHROMATOGRAPHY	GEN-ICS	3
ION CHROMATOGRAPHY	GEN-IONC	17
COLOR, NCASI	GEN-NCAS	4
NITROCELLULOSE IN SOIL	GEN-NCEL	1

SOP TITLE	SOP ID	Revision
OXYGEN CONSUMPTION RATE	GEN-O2RATE	1
CARBON, TOTAL ORGANIC DETERMINATION (WALKELY BLACK METHOD)	GEN-OSU	3
Ph IN SOIL AND SOLIDS	GEN-pHS	14
Ph IN WATER	GEN-pHW	14
PARTICLE SIZE DETERMINATION - ASTM PROCEDURE	GEN-PSASTM	3
PARTICLE SIZE DETERMINATION	GEN-PSP	8
SULFIDES, REACTIVE	GEN-RS	5
TOTAL SULFIDE BY PSEP	GEN-S2PS	2
SULFITE	GEN-SO3	3
SPECIFIC GRAVITY	GEN-SPGRAV	2
SUBSAMPLING AND COMPOSITING OF SAMPLES	GEN-SUBS	6
SOLIDS, TOTAL DISSOLVED (TDS)	GEN-TDS	12
THIOCYANATE	GEN-THIOCN	2
NITROGEN, TOTAL AND SOLUBLE KJELDAHL	GEN-TKN	15
TOTAL NITROGEN AND TOTAL PHOSPHORUS BY ALKALINE PERSULFATE DIGESTION NCASI METHOD TNTP-W10900	GEN-TNTP	1
TOTAL ORGANIC CARBON IN WATER	GEN-TOC	14
SOLIDS, TOTAL SUSPENDED (TSS)	GEN-TSS	12
TURBIDITY MEASUREMENT	GEN-TURB	7
GLASSWASHING FOR INORGANIC ANALYSES	GEN-WASH	5
PHARMACEUTICALS, PERSONAL CARE PRODUCTS AND ENDOCRINE DISRUPTING COMPOUNDS BY HPLC/TANDEM MASS SPECTROMETRY (HPLC/MS/MS)	LCP-1694	5
DETERMINATION OF SELECTED PERFLUORINATED ALKYL ACIDS IN DRINKING WATER BY SOLID PHASE EXTRACTION AND TANDEM (LC/MS/MS)	LCP-537	3
DETERMINATION OF HORMONES IN DRINKING WATER BY SOLID PHASE EXTRACTION AND LIQUID CHROMATOGRAPHY ELECTROSPRAY IONIZATION	LCP-539	3
PERCHLORATE IN WATER, SOILS, AND SOLID WASTE USING LIQUID CHROMATOGRAPHY TANDEM MASS SPECTROMETRY (LC/MS/MS)	LCP-6850	0
ALDEHYDES BY HPLC	LCP-8315	7
Quantitative Determination of Carbamate Pesticides in Solid Matrices by High Performance Liquid Chromatography/Tandem Mass Spectrometry (HPLC/MS/MS)	LCP-8321(S)	1
Determination of Carbamates in Water by EPA 8321 Using LC Tandem Mass Spectrometry	LCP-8321W	2
NITROAROMATICS AND NITRAMINES BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY(HPLC)	LCP-8330B	5
Acrylamide by High Performance Liquid Chromatography/tandem mass spectrometry (HPLC/MS/MS)	LCP-ACRYL	2

SOP TITLE	SOP ID	Revision
Dioctyl sulfosuccinate by High Performance Liquid Chromatography/tandem mass spectrometry (HPLC/MS/MS)	LCP-DOS	5
QUANTITATION OF NITROAROMATICS AND NITRAMINES IN WATER, SOIL, AND TISSUE BY LIQUID CHROMATOGRAPHY AND TANDEM MASS SPECTROMETRY (LC-MS/MS)	LCP-LCMS4	3
NITROGUANIDINE BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY	LCP-NITG	7
QUANTITATION OF NITROPHENOLS IN SOILS BY LIQUID CHROMATOGRAPHYAND TANDEM MASS SPECTROMETRY (LC/MS/MS)	LCP-NITRO	3
ORGANIC ACIDS IN AQUEOUS MATRICES BY HPLC	LCP-OALC	5
QUANTITATIVE DETERMINATION OF OPTICAL BRIGHTENER 220 By High Performance Liquid Chromatography (HPLC)	LCP-OPBr	1
OXYANIONS IN WATER USING LIQUID CHROMATOGRAPHY TANDEM MASS SPECTROMETRY (LC/MS/MS)	LCP-OXY	1
PERFLUORINATED COMPOUNDS BY HPLC/MS/MS	LCP-PFC	6
DETERMINATION OF PHTHALATES IN FOOD BY LIQUID CHROMATOGRAPHY TANDEM MASS SPECTROMETRY (LC/MS/MS)	LCP-PHT	1
PICRIC ACID AND PICRAMIC ACID BY HPLC	LCP-PICRIC	3
METHYL MERCURY IN SOIL AND SEDIMENT BY ATOMIC FLUORESCENCE SPECTROMETRY	MET-1630S	3
METHYL MERCURY IN TISSUE BY ALCOHOLIC POTASSIUM HYDROXIDE DIGESTION, ETHYLATION, PURGE AND TRAP, AND COLD VAPOR ATOMIC FLUORESCENCE	MET-1630T	2
METHYL MERCURY IN WATER BY ATOMIC FLUORESCENCE SPECTROMETRY	MET-1630W	3
MERCURY IN WATER BY OXIDATION, PURGE&TRAP, AND COLD VAPOR ATOMIC FLUORES. SPECTROMETRY	MET-1631	14
DETERMINATION OF ARSENIC SPECIES BY HYDRIDE GENERATION CRYOGENIC TRAPPING GAS CHROMATOGRAPHY ATOMIC ABSORPTION SPECTROPHOTOMETRY	MET-1632	3
MERCURY IN WATER	MET-245.1	14
METALS DIGESTION	MET-3010A	13
METALS DIGESTION	MET-3020A	16
METALS DIGESTION	MET-3050B	15
CLOSED VESSEL OIL DIGESTION	MET-3051M	4
CLOSED VESSEL DIGESTION OF SILICEOUS AND ORGANICALLY BASED MATRICIES	MET-3052M	2
DETERMINATION OF METALS & TRACE ELEMENTS BY INDUCTIVELY COUPLED PLASMA-MS (METHOD 6020)	MET-6020	17
ARSENIC BY BOROHYDRIDE REDUCTION ATOMIC ABSORPTION	MET-7062	4
METALS DIGESTION FOR HEXAVALENT CHROMIUM	MET-7195	10
MERCURY IN LIQUID WASTE	MET-7470A	16
MERCURY IN SOLID OR SEMISOLID WASTE	MET-7471	17
SELENIUM BY BOROHYDRIDE REDUCTION ATOMIC ABSORPTION	MET-7742	4
AUTOFLUFF	MET-AUTOFLU	3

SOP TITLE	SOP ID	Revision
BIOACCESSIBILITY OF METALS IN SOIL AND SOLID WASTE	MET-BIOACC	2
METALS DIGESTION OF AQUEOUS SAMPLES	MET-DIG	16
SAMPLE FILTRATION FOR METALS ANALYSIS	MET-FILT	4
METALS LABORATORY GLASSWARE CLEANING	MET-GC	6
DETERMINATION OF TRACE METALS BY GRAPHITE FURNACE ATOMIC ABSORPTION SPECTROMETRY (GFAA)	MET-GFAA	21
DETERMINATION OF METALS AND TRACE ELEMENTS BY ICP/AES	MET-ICP	26
DETERMINATION OF METALS & TRACE ELEMENTS BY INDUCTIVELY COUPLED PLASMA-MS (METHOD 200.8)	MET-ICPMS	17
TRACE METALS IN WATER BY PRECONCENTRATION USING REDUCTIVE PRECIPITATION FOLLOWED BY ICP-MS	MET-RPMS	8.1
METALS AND SEMIVOLATILES SPLP EXTRACTION (EPA METHOD 1312)	MET-SPLP	2
WASTE EXTRACTION TEST (WET) PROCEDURE (STLC) for NONVOLATILE and SEMIVOLATILE PARAMETERS	MET-STLC	3
METALS AND SEMIVOLATILES TCLP EXTRACTION (EPA METHOD 1311)	MET-TCLP	9
SAMPLE PREPARATION OF BIOLOGICAL TISSUES FOR METALS ANALYSIS BY GFAA, ICP-OES, AND ICP-MS	MET-TDIG	5
TISSUE SAMPLE PREPARATION	MET-TISP	10
ANALYSIS OF WATER AND SOLID SAMPLES FOR ALIPHATIC HYDROCARBONS	PET-ALIPHAT	2
ANALYSIS OF WATER, SOLIDS AND SOLUBLE WASTE SAMPLES FOR SEMI-VOLATILE FUEL HYDROCARBONS	PET-SVF	14
ANALYSIS OF WATER AND SOLIDS SAMPLES FOR TOTAL PETROLEUM HYDROCARBONS	PET-TPH	2
ANALYSIS OF SOLID AND AQUEOUS SAMPLES FOR STATE OF WISCONSIN DIESEL RANGE ORGANICS	PHC-WIDRO	5
GC-FID IMPURITIES IN METHYLSULFONYLMETHANE (MSM)	PHM-MSM	5
ASSAY FOR METHYLSULFONYLMETHANE (MSM)	PHM-MSMAssay	0
OPERATION, CALIBRATION, AND MAINTENANCE OF THE METTLER TOLEDO DL38 TITRATOR	PHM-KF	5
OPERATION, CALIBRATION, AND MAINTENANCE OF THE STANFORD RESEARCH SYSTEMS MPA100 OPTIMELT AUTOMATED MELTING POINT SYSTEM	PHM-MP	6
BOTTLE ORDER PREPARATION AND SHIPPING	SMO-BORD	17
SAMPLE DISPOSAL	SMO-DISP	12
FOREIGN SOILS HANDLING TREATMENT	SMO-FSHT	11
SAMPLE RECEIVING	SMO-GEN	33
SAMPLE TRACKING AND INTERNAL CHAIN OF CUSTODY	SMO-SCOC	15
ORGANOCHLORINE PESTICIDES AND PCBs (METHOD 608)	SOC-608	8
1,2-DIBROMOETHANE (EDB) AND 1,2-DIBROMO-3-CHLORO-PROPANE (DBCP) IN AQUEOUS SAMPLES BY MICROEXTRACTION AND GAS CHROMATOGRAPHY	SOC-8011	1

SOP TITLE	SOP ID	Revision
1,2-DIBROMOETHANE (EDB) AND 1,2-DIBROMO-3-CHLORO-PROPANE (DBCP) IN SOLIDS BY MICROEXTRACTION AND GAS CHROMATOGRAPHY	SOC-8011S	1
GLYCOLS	SOC-8015	12
ORGANOCHLORINE PESTICIDES BY GAS CHROMATOGRAPHY: CAPILLARY COLUMN TECHNIQUE	SOC-8081	19
PCBS AS AROCLORS	SOC-8082Ar	17
CONGENER-SPECIFIC DETERMINATION OF PCBS BY GC/ECD	SOC-8082Co	14
DETERMINATION OF NITROGEN OR PHOSPHORUS CONTAINING PESTICIDES	SOC-8141	14
CHLORINATED HERBICIDES	SOC-8151	16
CHLORINATED PHENOLS METHOD 8151 MODIFIED	SOC-8151M	11
METHANOL IN PROCESS LIQUIDS AND STATIONARY SOURCE EMISSIONS	SOC-9403	9
HAZARDOUS AIR POLLUTANTS (HAPS) IN PULP AND PAPER INDUSTRY CONDENSATES	SOC-9901	6
HAPS AND OTHER COMPOUNDS IN IMPINGER/CANISTER SAMPLES FROM WOOD PRODUCTS FACILITIES	SOC-9902	5
ALCOHOLS	SOC-ALC	3
BUTYLTINS	SOC-BUTYL	13
CALIBRATION OF INSTRUMENTS FOR ORGANICS CHROMATOGRAPHIC ANALYSES	SOC-CAL	9
CONFIRMATION PROCEDURE FOR GC AND HPLC ANALYSES	SOC-CONF	7
DETERMINATION OF OTTO FUEL II IN WATER	SOC-OTTO	2
PREPARATION OF POLYETHYLENE (PE) PASSIVE SAMPLERS WITH PERFORMANCE REFERENCE COMPOUNDS (PRC) LOADING	SOC-PE/PRC	0
SEMI-VOLATILE ORGANICS SCREENING	SOC-SCR	5
1,2-DIBROMOETHANE, 1,2-DIBROMO-3-CHLOROPROPANE, AND 1,2,3-TCP BY GC	SVD-504	11
ORGANOCHLORINE PESTICIDES AND PCBS IN DRINKING WATER	SVD-508_1	9
CHLORINATED HEBICIDES IN DRINKING WATER	SVD-515.4	10
N-NITROSAMINES BY GC/MS/MS	SVD-521	7
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS (METHOD 525.2)	SVD-525	10
ENDOTHALL IN DRINKING WATER BY GC/MS	SVD-548	11
DIQUAT AND PARAQUAT BY HPLC	SVD-549	8
HALOACETIC ACIDS IN DRINKING WATER	SVD-552	8
CHLORINATED PHENOLICS BY IN-SITU ACETYLATION AND GC/MS	SVM-1653A	10
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS	SVM-625	8

SOP TITLE	SOP ID	Revision
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS – METHOD 8270D	SVM-8270D	5
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS – LOW LEVEL PROCEDURE	SVM-8270L	10
POLYNUCLEAR AROMATIC HYDROCARBONS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY SIM	SVM-8270P	10
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS SELECTED ION MONITORING	SVM-8270S	8
ANTRHAQUINONE IN PAPERBOARD BY GC/MS SELECTED ION MONITORING	SVM-AQ	0
QUANTITATIVE GEOCHEMICAL BIOMARKERS BY GC/MS SELECTIVE ION MONITORING	SVM-BIO	1
OCTAMETHYLCYCLOTETRASILOXANE (D4) IN AQUEOUS SAMPLES BY GC/MS	SVM-D4AQ	1
OCTAMETHYLCYCLOTETRASILOXANE (D4) IN SEDIMENTS AND BIOSOLIDS BY GC/MS	SVM-D4SO	1
OCTAMETHYLCYCLOTETRASILOXANE (D4) IN BIOLOGICAL MATRICES BY GC/MS	SVM-D4TI	1
DIISOPROPYL METHYLPHOSPHONATE BY GC/MS SELECTIVE ION MONITORING	SVM-DIMP	0
NONYLPHENOLS ISOMERS AND NONYLPHENOL ETHOXYLATES	SVM-NONYL	6
ORGANOPHOSPHOROUS PESTICIDES BY GC/MS/MS	SVM-OPPMS2	2
CHLORINATED PESTICIDES BY GC/MS/MS	SVM-PESTMS2	5
POLYBROMINATED DIPHENYL ETHERS (PBDEs) AND POLYBROMINATED BIPHENYLS (PBBs) BY GC/MS	SVM-ROHS	2
1,2,3-TRICHLOROPROPANE BY ISOTOPE DILUTION-GC/MS SIM	SVM-TCP	0
PURGE AND TRAP FOR AQUEOUS SAMPLES	VOC-5030	10
PURGE AND TRAP/EXTRACTION FOR VOC IN SOIL AND WASTE SAMPLES , CLOSED SYSTEM	VOC-5035	11
VOLATILE ORGANIC COMPOUNDS BY GC/MS	VOC-524.2	17
VOLATILE ORGANIC COMPOUNDS IN WATER BY GC/MS SIM	VOC-524.2SIM	0
VOLATILE ORGANIC COMPOUNDS BY GC/MS	VOC-624	13
VOLATILE ORGANIC COMPOUNDS BY GC/MS	VOC-8260	19
VOLATILE ORGANIC COMPOUNDS BY GC/MS SELECTIVE ION MONITORING	VOC-8260S	3
VOA STORAGE BLANKS	VOC-BLAN	10
SAMPLE SCREENING FOR VOLATILE ORGANIC COMPOUNDS IN SOIL, WATER AND MISC. MATRICES	VOC-BVOC	8
GASOLINE RANGE ORGANICS BY GAS CHROMATOGRAPHY	VOC-GRO	11
ZERO HEADSPACE EXTRACTION (EPA METHOD 1311)	VOC-ZHE	8

APPENDIX H – Data Qualifiers

Inorganic Data Qualifiers

*	The result is an outlier. See case narrative.
#	The control limit criteria is not applicable. See case narrative.
B	The analyte was found in the associated method blank at a level that is significant relative to the sample result as defined by the DOD or NELAC standards.
E	The result is an estimate amount because the value exceeded the instrument calibration range.
J	The result is an estimated value.
U	The analyte was analyzed for, but was not detected ("Non-detect") at or above the MRL/MDL. *DOD-QSM 4.2 definition* : Analyte was not detected and is reported as less than the LOD or as defined by the project. The detection limit is adjusted for dilution.
i	The MRL/MDL or LOQ/LOD is elevated due to a matrix interference.
X	See case narrative.
Q	See case narrative. One or more quality control criteria was outside the limits.
H	The holding time for this test is immediately following sample collection. The samples were analyzed as soon as possible after receipt by the laboratory.

Metals Data Qualifiers

#	The control limit criteria is not applicable. See case narrative.
J	The result is an estimated value.
E	The percent difference for the serial dilution was greater than 10%, indicating a possible matrix interference in the sample.
M	The duplicate injection precision was not met.
N	The Matrix Spike sample recovery is not within control limits. See case narrative.
S	The reported value was determined by the Method of Standard Additions (MSA).
U	The analyte was analyzed for, but was not detected ("Non-detect") at or above the MRL/MDL. *DOD-QSM 4.2 definition* : Analyte was not detected and is reported as less than the LOD or as defined by the project. The detection limit is adjusted for dilution.
W	The post-digestion spike for furnace AA analysis is out of control limits, while sample absorbance is less than 50% of spike absorbance.
i	The MRL/MDL or LOQ/LOD is elevated due to a matrix interference.
X	See case narrative.
+	The correlation coefficient for the MSA is less than 0.995.
Q	See case narrative. One or more quality control criteria was outside the limits.

Organic Data Qualifiers

*	The result is an outlier. See case narrative.
#	The control limit criteria is not applicable. See case narrative.
A	A tentatively identified compound, a suspected aldol-condensation product.
B	The analyte was found in the associated method blank at a level that is significant relative to the sample result as defined by the DOD or NELAC standards.
C	The analyte was qualitatively confirmed using GC/MS techniques, pattern recognition, or by comparing to historical data.
D	The reported result is from a dilution.
E	The result is an estimated value.
J	The result is an estimated value.
N	The result is presumptive. The analyte was tentatively identified, but a confirmation analysis was not performed.
P	The GC or HPLC confirmation criteria was exceeded. The relative percent difference is greater than 40% between the two analytical results.
U	The analyte was analyzed for, but was not detected ("Non-detect") at or above the MRL/MDL. *DOD-QSM 4.2 definition* : Analyte was not detected and is reported as less than the LOD or as defined by the project. The detection limit is adjusted for dilution.
i	The MRL/MDL or LOQ/LOD is elevated due to a chromatographic interference.
X	See case narrative.
Q	See case narrative. One or more quality control criteria was outside the limits.

Additional Petroleum Hydrocarbon Specific Qualifiers

F	The chromatographic fingerprint of the sample matches the elution pattern of the calibration standard.
L	The chromatographic fingerprint of the sample resembles a petroleum product, but the elution pattern indicates the presence of a greater amount of lighter molecular weight constituents than the calibration standard.
H	The chromatographic fingerprint of the sample resembles a petroleum product, but the elution pattern indicates the presence of a greater amount of heavier molecular weight constituents than the calibration standard.
O	The chromatographic fingerprint of the sample resembles an oil, but does not match the calibration standard.
Y	The chromatographic fingerprint of the sample resembles a petroleum product eluting in approximately the correct carbon range, but the elution pattern does not match the calibration standard.
Z	The chromatographic fingerprint does not resemble a petroleum product.

APPENDIX I – Controlled and Normative Documents

Internal QA Documents	Location
Quality Assurance Manual	Q:\QA Manual\QAM.rXX.DOC
ALS-Kelso Certifications/Accreditations	Cert_kel.xls (QA Dept.)
MDL/LOD/LOQ Tracking Spreadsheet	MDL_LIST.(*date*).xls
Technical Training Summary Database	TrainDat.mdb
Approved Signatories List	QAM App A
Personnel resumes/qualifications	HR Department
Personnel Job Descriptions	HR Department
ALS – Kelso Data Quality Objectives	Kelso DQO table – mmddyy.xls
Master Logbook of Laboratory Logbooks	QA Masterlog-001
Standard Operating Procedures and Spreadsheet	1_ Kelso SOP.xls
Proficiency Testing Schedule and Tracking Spreadsheet	PT_Schedule.xls
External Normative Documents	**Location**
USEPA Manual for the Certification of Laboratories Analyzing Drinking Water, 5th Edition, EPA 815-B-97-001 (January 2005	QA Department
USEPA 40 CFR Part 136, Guidelines for Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, and EPA Method Update Rule 2007 and 2012.	QA Department and online access
USEPA 40 CFR Part 141, National Primary Drinking Water Regulations and EPA Method Update Rule 2007.	QA Department and online access
National Environmental Laboratory Accreditation Program (NELAP), 2003 Quality Standards.	QA Department
TNI: TNI Standard – Environmental Laboratory Sector, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, EL-V1-2009.	QA Department
Quality Standards. American National Standard General requirements for the competence of testing and calibration laboratories, ANSI/ISO/IEC 17025:2005(E)	QA Department
DoD Quality Systems Manual for Environmental Laboratories, Versions 4.2 and 5.0	QA Department and online access
Analytical Methods (see References section)	Laboratory Departments and Online access

APPENDIX J - Laboratory Accreditations

The list of accreditations, certifications, licenses, and permits existing at the time of this QA Manual revision is given below, followed by the entire primary NELAP and DOD ELAP accreditations (un-numbered attachments). Current accreditation information is available at any time by contacting the laboratory or viewing the ALS Global website www.alsglobal.com.

Program	Number
National Programs	
DoD ELAP	L14-51-R2
ISO 17025	L14-50
State Programs	
Alaska DEC UST	UST-040
Arizona DHS	AZ0339
Arkansas - DEQ	88-0637
California DHS	2795
Florida DOH	E87412
Hawaii DOH	-
Louisiana DEQ	3016
Maine DHS	WA01276
Minnesota DOH	053-999-457
Montana DPHHS	CERT0047
Nevada DEP	WA012762015-3
New Jersey DEP	WA005
New York DoH	12060
North Carolina DWQ	605
Oklahoma DEQ	9801
Oregon - DOH (primary NELAP)	WA100010
South Carolina DHEC	61002
Texas CEQ	T104704427-14-7
Utah	WA012762015-4
Washington DOE	C544
Wyoming (EPA Region8)	-
Miscellaneous	
Foreign Soil Permit	USDA
Plant Import Permit	USDA
Controlled Substances Permit	US DEA
Controlled Substances Permit	WA DOH

END

OF

DOCUMENT

ERA
A Waters Company

Reference Materials

▪ Certificate of Analysis ▪

Product: Metals in Soil
Catalog Number: 540
Lot No. D087-540
Certificate Issue Date: September 30, 2014
Expiration Date: April 30, 2018
Revision Number: Original

CERTIFICATION

Parameter	Total Concentration	Certified Value[1]	Uncertainty[2]	QC Performance Acceptance Limits[3]	PT Performance Acceptance Limits[4]
	mg/kg	mg/kg	%	mg/kg	mg/kg
Aluminum	68100	7930	4.25	3980 - 11900	3090 - 12800
Antimony	215	105	18.4	D.L. - 211	21.4 - 267
Arsenic	111	98.5	9.27	76.6 - 121	68.3 - 143
Barium	739	308	5.62	254 - 362	228 - 388
Beryllium	71.3	66.0	5.50	54.8 - 77.1	48.6 - 83.4
Boron	171	137	5.13	99.3 - 174	82.1 - 194
Cadmium	162	146	5.79	121 - 172	107 - 185
Calcium	24800	6610	5.32	5530 - 7680	4900 - 8320
Chromium	421	182	6.02	145 - 219	129 - 236
Cobalt	183	162	5.38	135 - 188	120 - 203
Copper	122	106	9.95	86.4 - 126	79.0 - 133
Iron	42900	14400	18.7	6350 - 22400	5120 - 23600
Lead	125	130	10.2	107 - 153	94.2 - 165
Magnesium	8840	2640	4.21	2000 - 3290	1700 - 3590
Manganese	981	410	7.29	333 - 488	313 - 508
Mercury	6.66	7.10	14.8	5.23 - 8.97	3.64 - 10.6
Molybdenum	192	164	7.33	129 - 198	116 - 211
Nickel	190	149	4.65	123 - 175	109 - 189
Potassium	19800	2550	6.34	1760 - 3330	1550 - 3540
Selenium	169	154	5.78	120 - 188	104 - 204
Silver	44.1	40.9	19.4	30.7 - 51.0	27.0 - 54.7
Sodium	19500	2480	13.6	1750 - 3200	1620 - 3330
Strontium	221	84.8	12.5	68.2 - 101	59.5 - 110
Thallium	198	175	8.42	137 - 212	120 - 229

ACCREDITED ACCREDITED

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Parameter	Total Concentration	Certified Value[1]	Uncertainty[2]	QC Performance Acceptance Limits[3]	PT Performance Acceptance Limits[4]
	mg/kg	mg/kg	%	mg/kg	mg/kg
Tin	115	102	4.02	79.0 - 126	58.4 - 146
Titanium	3300	398	13.7	115 - 681	110 - 687
Vanadium	182	96.7	12.5	74.7 - 119	62.9 - 131
Zinc	173	191	10.1	159 - 223	133 - 249

ANALYTICAL VERIFICATION

Parameter	Certified Value[1]	Proficiency Testing Study			NIST Traceability	
		Mean	Recovery[5]	n	SRM Number	Recovery
	mg/kg	mg/kg	%			%
Aluminum	7930	7930	100	145	-	-
Antimony	105	105	100	156	-	-
Arsenic	98.5	98.5	100	186	-	-
Barium	308	308	100	171	-	-
Beryllium	66.0	66.0	100	163	-	-
Boron	137	137	100	112	-	-
Cadmium	146	146	100	187	-	-
Calcium	6610	6610	100	129	-	-
Chromium	182	182	100	181	-	-
Cobalt	162	162	100	160	-	-
Copper	106	106	100	183	-	-
Iron	14400	14400	100	146	-	-
Lead	130	130	100	191	-	-
Magnesium	2640	2640	100	132	-	-
Manganese	410	410	100	166	-	-
Mercury	7.10	7.10	100	123	-	-
Molybdenum	164	164	100	165	-	-
Nickel	149	149	100	183	-	-
Potassium	2550	2550	100	136	-	-
Selenium	154	154	100	181	-	-
Silver	40.9	40.9	100	171	-	-

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Parameter	Certified Value[1]	Proficiency Testing Study			NIST Traceability	
		Mean	Recovery[5]	n	SRM Number	Recovery
	mg/kg	mg/kg	%			%
Sodium	2480	2480	100	129	-	-
Strontium	84.8	84.8	100	111	-	-
Thallium	175	175	100	159	-	-
Tin	102	102	100	120	-	-
Titanium	398	398	100	117	-	-
Vanadium	96.7	96.7	100	158	-	-
Zinc	191	191	100	181	-	-

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1. The **Certified Values** are equal to the mean recoveries for the parameters as determined in an interlaboratory round robin study based on all applicable digestion techniques reported in the study. The Certified Values are based on an "as received" basis, assuming 100% solids content. The certified values are monitored and purchasers will be notified of any significant changes resulting in recertification or withdrawal of this certified reference material during the period of validity of this certificate.

2. The **Uncertainty** is the total propagated uncertainty at the 95% confidence interval. The uncertainty is based on the preparation and internal analytical verification of the product by ERA, multiplied by a coverage factor. The uncertainty applies to the product as supplied and does not take into account any required or optional dilution and/or preparations the laboratory may perform while using this product.

3. The **QC Performance Acceptance Limits (QC PALs™)**are based on actual historical data collected in ERA's Proficiency Testing program. The QC PALs™ reflect any inherent biases in the methods used to establish the limits and closely approximate a 95% confidence interval of the performance that experienced laboratories should achieve using accepted environmental methods. Use the QC PALs™ to realistically evaluate your performance against your peers.

4. The **PT Performance Acceptance Limits (PT PALs™)** are calculated using the regression equations and fixed acceptance criteria specified in the NELAC proficiency testing requirements. Use the PT PALs™ when analyzing this QC standard alongside USEPA and NELAC compliant PT standards. Please note that many PT study acceptance limits are concentration dependent (some non-linearly) and, therefore, the acceptance limits of this QC standard and any PT standard may differ relative to their difference in concentrations.

5. The **PT Data/Traceability** data include the mean value, percent recovery and number of data points reported by the laboratories in our Proficiency Testing study compared to the Certified Values. In addition, where NIST Standard Reference Materials (SRMs) are available, each analyte has been analytically traced to the NIST SRM listed. This product is traceable to the lot numbers of its starting materials. All gravimetric and volumetric measurements related to its manufacture are traceable to NIST through an unbroken chain of comparisons.
Traceability Recovery (%) = [(% recovery certified standard)/(% recovery NIST SRM)]*100
The traceability data shown were compiled by analyzing the ERA standards or their associated stock solutions against the applicable NIST SRMs.

6. The **Total Concentrations** are equal to the background concentrations in the blank soil matrix (measured using neutron activation, XRF, and total acid digestion techniques), plus the amount of each analyte spiked onto the soil. For Trace Metals, the values listed are only ""Theoretical Values"" based upon the methodologies listed.

7. For additional information on this product such as intended use, instructions for use, level of homogeneity, and safety information, please refer to the provided Instruction Sheet

If you have any questions or need technical assistance, please call ERA technical assistance at 1-800-372-0122 or send an email to info@eraqc.com.

Certifying Officer

Tom Widera

Thomas Widera

Quality Officer

David Kilhefner

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National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 1547

Peach Leaves

This Standard Reference Material (SRM) is intended primarily for use in evaluating the reliability of analytical methods for the determination of major, minor, and trace elements in botanical materials, agricultural food products, and materials of similar matrix. A unit of SRM 1547 consists of 50 grams of dried peach leaves of the Coronet variety.

Certified and Noncertified Concentrations of Constituent Elements: The certified concentrations of the constituent elements are given in Table 1. These concentrations are based on the agreement of results from at least two independent analytical methods or the mean of results from a method of known accuracy. Noncertified concentrations of constituent elements are provided for information only in Table 2.

Notice and Warnings to Users

Expiration of Certification: This certification is valid for five years from the date of shipment. Should any of the certified values change before the expiration of the certification, purchasers will be notified by NIST. Please return the attached registration card to facilitate notification.

Stability: This material was radiation sterilized (^{60}Co) at an estimated minimum dose of 27.8 kGy for microbiological control. However, its stability has not be rigorously assessed. NIST will monitor this material and will report any substantive changes in certification to the purchasers.

Storage: The material should be kept tightly closed in its original bottle and stored in the dark at a temperature between 10 and 30 °C. It should not be exposed to intense sources of radiation. Ideally, the bottle should be kept in a desiccator under the conditions indicated above.

Use: The bottle should be thoroughly mixed by rotating and/or rolling the bottle before each use. Allow the contents to settle for one minute prior to opening. A minimum sample of 150 mg of the dried material, dried as described in the section on "Instructions for Drying", should be used to relate analytical determinations to the certified values in this certificate. In some cases, especially for volatile elements such as mercury, it is preferable to analyze samples from the bottle without drying, determine the moisture content on a separate sample from the same bottle, and correct the analytical results to a dry weight basis.

Dissolution of SRM 1547: Digestion procedures should be designed to avoid loss of volatile elements, such as arsenic, mercury, etc. Digestion of the SRM in nitric and perchloric acids was found to be incomplete with a small residue of siliceous material remaining. This residue must be considered an integral part of the SRM and should be dissolved with a small amount of hydrofluoric acid to obtain total dissolution.

Coordination of all analytical measurements used in the characterization of this SRM was performed by D.A. Becker of the NIST Inorganic Analytical Research Division.

Statistical analysis of the experimental data was performed by W. Guthrie and S.B. Schiller of the NIST Statistical Engineering Division.

The technical and support aspects involved in the certification and issuance of this SRM were coordinated through the Standard Reference Materials Program by R. Alvarez and T.E. Gills.

Gaithersburg, MD 20899
January 22, 1993
(Revision of certificate dated 7-2-91)

William P. Reed, Chief
Standard Reference Materials Program

Instructions for Drying: Samples of this SRM must be dried only by one of the following two procedures.

1. Drying in a desiccator at room temperature (approximately 22 °C) for 120 hours over fresh anhydrous magnesium perchlorate. The sample depth should not exceed one cm.

2. Freeze drying for 24 hours at a pressure of 13.3 Pa or lower and a shelf temperature of -5 °C or lower after having frozen the sample (not to exceed one cm in depth) at -40 °C or lower for at least one hour. At the end of the 24-hour period, samples are placed immediately in a desiccator with fresh anhydrous magnesium perchlorate. Samples are weighed after allowing a minimum of four hours to establish temperature equilibrium.

NOTE: Vacuum drying at room temperature and oven drying at elevated temperatures have resulted in excessive weight losses and therefore are not recommended.

Homogeneity Assessment: Samples from randomly selected bottles of SRM 1547 were tested for homogeneity by instrumental neutron activation analysis. No evidence of chemically significant inhomogeneity was observed (Ref. 1).

Table 1 Certified Concentrations of Constituent Elements

Element	Concentration, wt. percent		
Calcium	1.56	±	0.02
Magnesium	0.432	±	0.008
Nitrogen (Total)	2.94	±	0.12
Phosphorus	0.137	±	0.007
Potassium	2.43	±	0.03

Element	Concentration, µg/g			Element	Concentration, µg/g		
Aluminum	249	±	8	Mercury	0.031	±	0.007
Arsenic	0.060	±	0.018	Molybdenum	0.060	±	0.008
Barium	124	±	4	Nickel	0.69	±	0.09
Boron	29	±	2	Rubidium	19.7	±	1.2
Cadmium	0.026	±	0.003	Selenium	0.120	±	0.009
Chlorine	360	±	19	Sodium	24	±	2
Copper	3.7	±	0.4	Strontium	53	±	4
Iron	218	±	14	Vanadium	0.37	±	0.03
Lead	0.87	±	0.03	Zinc	17.9	±	0.4
Manganese	98	±	3				

Certified Concentrations and Uncertainties: The certified concentrations are equally weighted means of results from two or more different analytical methods or the mean of results from a method of known accuracy. In the case of two or more methods, each uncertainty is the sum of a 95% confidence limit and an allowance for systematic error between the methods used. In the case of a method of known accuracy, each uncertainty is the sum of a 95% confidence limit and the known systematic error of the method.

Table 2. Noncertified Concentrations of Constituent Elements

Elements other than those certified are present in this material. Those that were determined but not certified are provided as additional information on the composition. Although total nitrogen is certified, nitrogen determined by the Kjeldahl procedure is not.

Element	Concentration wt. percent
*Nitrogen (Kjeldahl)	(2.96)
Sulfur	(0.2)

Element	Concentration, µg/g	Element	Concentration, µg/g
Antimony	(0.02)	Lanthanum	(9)
Bromine	(11)	Neodymium	(7)
Cerium	(10)	Samarium	(1)
Chromium	(1)	Scandium	(0.04)
Cobalt	(0.07)	Terbium	(0.1)
Europium	(0.17)	Thorium	(0.05)
Gadolinium	(1)	Tin	(<0.2)
Iodine	(0.3)	Uranium	(0.015)
		Ytterbium	(0.2)

*Method Reference. Official Methods of Analysis of the Association of Official Analytical Chemists, Arlington, VA, 14th Ed., 1984, p.16, Nitrogen (Total) in Fertilizers, Kjedlahl Method (Final Action): Method 2.057, Improved Method for Nitrate Free Samples. Samples were dried as described in procedure 1 under "Instructions for Drying".

Source and Preparation of Material: The plant material for this SRM was collected and prepared under the direction of R.A. Isaac, Soil Testing & Plant Analysis Laboratory, The University of Georgia College of Agriculture. Leaves, representative of healthy Georgia peach trees, variety "Coronet" were picked from a field in Peach County, Georgia approximately 150 miles south of Athens, Georgia. Fungicide and insecticide sprays were controlled to minimize heavy metal contamination. The leaves were dried and ground in a stainless steel mill to pass a 1 mm screen. At NIST, the ground leaves were jet milled and air classified to a particle size of approximately 75 µm (200 mesh). After mixing in a large blender, the leaves were irradiated with cobalt-60 radiation to a minimize absorbed dose of 27.8 kGy for microbiological control and bottled.

Table 3. Methods and Analysts for Certified Elemental Determinations

Element	Method Code	Element	Method Code
Aluminum	ICP INAA	Mercury	CVAAS RNAA
Arsenic	HGAAS RNAA	Molybdenum	IDICPMS RNAA
Barium	IDICPMS INAA	Nickel	LEIS IDICPMS RNAA
Boron	IDICPMS PGAA	Nitrogen	KJEL PGAA
Cadmium	RNAA IDICPMS	Phosphorus	DCPES ICP
Calcium	IDTIMS INAA	Potassium	IDTIMS INAA
Chlorine	INAA PGAA	Rubidium	DCPES INAA
Copper	POL RNAA	Selenium	HGAAS INAA RNAA
Iron	INAA DCPES	Sodium	FAES INAA
Lead	IDTIMS	Strontium	IDICPMS INAA
Magnesium	IDTIMS INAA FAAS	Vanadium	INAA RNAA
Manganese	LEIS INAA	Zinc	POL INAA

Methods Used for Analysis of SRM 1547:

CVAAS = Cold-Vapor Atomic Absorption Spectrometry
DCPES = Direct Current Plasma Emission Spectrometry
FAAS = Flame Atomic Absorption Spectrometry
FAES = Flame Atomic Emission Spectrometry
GFAAS = Graphite Furnace Atomic Absorption Spectrometry
HGAAS = Hydride Generation Atomic Absorption Spectrometry
ICP = Inductively-Coupled Plasma Emission Spectrometry
IDICPMS = Isotope Dilution, Inductively Coupled Plasma Mass Spectrometry
IDTIMS = Isotope Dilution, Thermal Ionization Mass Spectrometry

INAA = Instrumental Neutron Activation Analysis
KJEL = Kjeldahl Nitrogen Determination

LEIS= Laser-Enhanced Ionization Spectrometry
PGAA = Prompt Gamma Activation Analysis
POL = Polarography
RNAA = Radiochemical Neutron Activation Analysis

Analysts, National Institute of Standards and Technology

I.L. Barnes	P.J. Paulsen
E.S. Beary	K.W. Pratt
D.A. Becker	T.A. Rush
D.S. Braverman	G. Sleater
C.A. Clements	S.F. Stone
R.R. Greenberg	G.C. Turk
L.B. Jassie	T.W. Vetter
H.M. Kingston	R.D. Vocke
J.R. Moody	L.J. Wood
T.J. Murphy	Xu Zhen

Cooperating Analysts

D.L. Anderson, Center for Food Safety and Applied Nutrition, U.S. Food and Drug Administration, Washington, DC

A.R. Byrne, Jozef Stefan Institute, Ljubljana, Yugoslavia

J. Kucera, Nuclear Research Institute, Rez, Czechoslavia

N. Miller-Ihli, Nutrient Composition Laboratory, U.S. Department of Agriculture, Beltsville, MD

B. Smodis, Jozef Stefan Institute, Ljubljana, Yugoslavia

REFERENCE

1. Becker, D.A., Homogeneity and Evaluation of the New NIST Leaf Certified Reference Materials, in Nuclear Analytical Methods in the Life Sciences, R. Zeisler and V.P. Guinn, eds. Clifton, NJ; Humana Press, 1990, 571-577. [Proceedings of the International Conference, "Nuclear Analytical Methods in the Life Sciences", held at NIST, Gaithersburg, MD, April 1989.]

National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 1573a

Tomato Leaves

This Standard Reference Material (SRM) is intended primarily for use in evaluating the reliability of analytical methods for the determination of major, minor, and trace elements in botanical materials, agricultural food products, and materials of similar matrix. A unit of SRM 1573a consists of 50 g of dried tomato leaves.

Certified and Noncertified Concentrations of Constituent Elements: The certified concentrations of the constituent elements are given in Table 1. These concentrations are based on the agreement of results from at least two independent analytical methods or the mean of results from a method of known accuracy. Noncertified concentrations of constituent elements are provided for information only in Table 2.

NOTICE AND WARNINGS TO USERS

Expiration of Certification: This certification is valid for five years from the date of shipment. Should any of the certified values change before the expiration of the certification, purchasers will be notified by NIST. Please return the attached registration card to facilitate notification.

Stability: This material was radiation sterilized (^{60}Co) at an estimated minimum dose of 25 kGy (2.5 Mrads) for microbiological control. However, its stability has not been rigorously assessed. NIST will monitor this material and will report any substantive changes in certification to the purchaser.

Storage: The material should be kept tightly closed in its original bottle and stored in the dark at a temperature between 10 and 30 °C. It should not be exposed to intense sources of radiation. Ideally, the bottle should be kept in a desiccator under the conditions indicated above.

Use: The bottle should be thoroughly mixed by rotating and/or rolling the bottle before each use. Allow the contents to settle for one minute prior to opening. A minimum sample of 150 mg of the dried material, dried as described in the section on "Instructions for Drying", should be used to relate analytical determinations to the certified values in this certificate. In some cases, especially for volatile elements such as mercury, it is preferable to analyze samples from the bottle without drying, determine the moisture content on a separate sample from the same bottle, and correct the analytical results to a dry weight basis.

Dissolution: Digestion procedures should be designed to avoid loss of volatile elements, such as arsenic, mercury, etc. Digestion of the SRM in nitric and perchloric acids was found to be incomplete with a small residue of siliceous material remaining. This residue must be considered an integral part of the SRM and should be dissolved with a small amount of hydrofluoric acid to obtain total dissolution.

Coordination of all analytical measurements used in the characterization of this SRM was performed by D.A. Becker of the NIST Inorganic Analytical Research Division.

Statistical analysis of the experimental data was performed by W. Guthrie of the NIST Statistical Engineering Division.

The technical and support aspects involved in the certification and issuance of this SRM were coordinated through the Standard Reference Materials Program by R.A. Alvarez and T.E. Gills.

Gaithersburg, MD 20899
October 19, 1993

Thomas E. Gills, Acting Chief
Standard Reference Materials Program

(over)

Instructions for Drying: Samples of this SRM must be dried only by one of the following two procedures.

1. Drying in a desiccator at room temperature (approximately 22 °C) for 120 h over fresh anhydrous magnesium perchlorate. The sample depth should not exceed 1 cm.

2. Freeze drying for 24 h at a pressure of 13.3 Pa or lower and a shelf temperature of -5 °C or lower after having frozen the sample (not to exceed 1 cm in depth) at -40 °C or lower for at least 1 h. At the end of the 24 h period, samples are placed immediately in a desiccator with fresh anhydrous magnesium perchlorate. Samples are weighed after allowing a minimum of 4 h to establish temperature equilibrium.

NOTE: Vacuum drying at room temperature and oven drying at elevated temperatures have resulted in excessive weight losses and therefore are not recommended.

Homogeneity Assessment: Homogeneity was assessed by careful evaluation of analytical data used for certification. No evidence of chemically or statistically significant inhomogeneity was observed.

Table 1. Certified Concentrations of Constituent Elements

Element	Concentration, Wt %*
Calcium	5.05 ± 0.09
Nitrogen (Total)	3.03 ± 0.15
Phosphorus	0.216 ± 0.004
Potassium	2.70 ± 0.05

Element	Concentration, mg/kg	Element	Concentration, mg/kg
Aluminum	598 ± 12	Mercury	0.034 ± 0.004
Antimony	0.063 ± 0.006	Nickel	1.59 ± 0.07
Arsenic	0.112 ± 0.004	Rubidium	14.89 ± 0.27
Boron	33.3 ± 0.7	Selenium	0.054 ± 0.003
Cadmium	1.52 ± 0.04	Sodium	136 ± 4
Chromium	1.99 ± 0.06	Vanadium	0.835 ± 0.010
Cobalt	0.57 ± 0.02	Zinc	30.9 ± 0.7
Copper	4.70 ± 0.14		
Iron	368 ± 7		
Manganese	246 ± 8		

*Wt % = mg/kg x 10^{-4}

Certified Concentrations and Uncertainties: The certified concentrations are equally weighted means of results from two or more different analytical methods or the mean of results from a method of known accuracy. In the case of two or more methods, each uncertainty is the sum of a 95% confidence limit and an allowance for systematic error between the methods used. In the case of a method of known accuracy, each uncertainty is the sum of a 95% confidence limit and the known systematic error of the method.

Table 2. Noncertified Concentrations of Constituent Elements

Elements other than those certified are present in this material. Those that were determined but not certified are provided as additional information on the composition. Although total nitrogen is certified, nitrogen determined by the Kjeldahl procedure is not.

Element	Concentration Wt %
Hydrogen	(5.2)
Magnesium	(1.2)
*Nitrogen (Kjeldahl)	(2.92)
Sulfur	(0.96)

Element	Concentration, mg/kg	Element	Concentration, mg/kg
Barium	(63)	Lanthanum	(2.3)
Bromine	(1300)	Molybdenum	(0.46)
Cerium	(2)	Samarium	(0.19)
Cesium	(53)	Scandium	(0.1)
Chlorine	(6600)	Silver	(0.017)
Gadolinium	(0.17)	Strontium	(85)
Hafnium	(0.14)	Thorium	(0.12)
Iodine	(0.85)	Uranium	(35)

*Method Reference. Official Methods of Analysis of the Association of Official Analytical Chemists, Arlington, VA, 14th Ed., 1984, p.16, Nitrogen (Total) in Fertilizers, Kjeldahl Method (Final Action): Method 2.057, Improved Method for Nitrate Free Samples. Samples were dried as described in procedure 1 under "Instructions for Drying".

Source and Preparation of Material: The plant material for this SRM was collected and prepared under the direction of C.B. Smith, Plant Analysis Laboratory, The Pennsylvania State University, University Park, PA. The tomato leaves were selected from "Count II" tomato plants grown in three (3) lime and fertilizer experiments covering about three (3) acres at the Horticultural Research Farm at Rock Springs, PA. Mature leaves were selected primarily from guard plants which had not received any treatment in order to obtain as uniform material as possible. Twenty four (24) batches of leaves were collected in paper or plastic containers. Since the leaves averaged only about 11% dry weight, about three (3) tons of leaves had to be collected. Fungicide sprays containing manganese, zinc, and copper were avoided in order to prevent trace element contamination of the sample.

After each collection, the sample was transported to the Plant Analysis Laboratory and washed as soon as possible (usually the same day). Most of the soil contamination was removed in a water spray and then the sample was dipped in a detergent solution, and rinsed in tap water and 3 successive rinses of distilled water.

The washed sample was drained and then placed in large pasteboard trays for drying in ovens at 60-70 °C. Drying had to be done quickly to avoid decomposition. The sample was then ground to pass a 40-mesh screen in a Wiley mill. A representative sample was taken from each batch for analysis using the Technicon Autoanalyzer with manual digestion for nitrogen and an ICP emission spectrometer for 12 other elements. These analyses allowed for a check on each batch before it was mixed with others.

The sample was placed in six 55-gallon drums with plastic liners for shipment to NIST. Each drum contained an equal portion from each of the 24 batches.

At NIST, the ground leaves were jet milled and air classified to a particle size of approximately 75 μm (200 mesh). After mixing in a large blender, the leaves were irradiated with cobalt-60 radiation to a minimum absorbed dose of 25 kGy for microbiological control and bottled.

Table 3. Methods and Analysts for Certified Elemental Determinations

Element	Method Code	Element	Method Code
Aluminum	ICP-AES INAA	Mercury	CVAAS RNAA
Antimony	INAA RNAA	Nickel	ID-ICPMS RNAA
Arsenic	FIA-HAAS RNAA	Nitrogen	KJEL PGAA
Boron	ID-ICPMS PGAA	Phosphorus	COL ICP-AES
Cadmium	ID-ICPMS PGAA RNAA	Potassium	INAA PGAA
Calcium	ID-TIMS INAA	Rubidium	ID-TIMS INAA
Chromium	INAA RNAA	Selenium	FIA-HAAS INAA RNAA
Cobalt	INAA RNAA	Sodium	FAES INAA
Copper	ICP-AES RNAA	Vanadium	ID-TIMS INAA
Iron	ICP-AES INAA	Zinc	ICP-AES INAA
Manganese	LEAFS INAA		

Methods Used for Analysis of SRM 1573a:

COL = Colorimetry
CVAAS = Cold-Vapor Atomic Absorption Spectrometry
FAES = Flame Atomic Emission Spectrometry
FIA-HAAS = Flow Injection-Hydride Generation Atomic Absorption Spectrometry
ICP-AES = Inductively-Coupled Plasma Atomic Emission Spectrometry
ID-ICPMS = Isotope Dilution, Inductively Coupled Plasma Mass Spectrometry
ID-TIMS = Isotope Dilution, Thermal Ionization Mass Spectrometry
INAA = Instrumental Neutron Activation Analysis
KJEL = Kjeldahl Nitrogen Determination
LEAFS = Laser-Excited Atomic Fluorescence Spectrometry
PGAA = Prompt Gamma Activation Analysis
RNAA = Radiochemical Neutron Activation Analysis

Analysts, National Institute of Standards and Technology

E.S. Beary
C.M. Beck II
D.A. Becker
D.S. Braverman
M.S. Epstein
J.D. Fassett
K.M. Garrity
R.R. Greenberg
R.M. Lindstrom
E. Mackey
J.R. Moody
K.E. Murphy
P.J. Paulsen
T.A. Rush
R. Saraswati
J.M. Smeller
G.C. Turk
T.W. Vetter
R.D. Vocke
R.L. Watter, Jr.
L.J. Wood

Cooperating Analysts

D.L. Anderson, Center for Food Safety and Applied Nutrition, U.S. Food & Drug Administration, Washington, DC

A.R. Byrne, Jozef Stefan Institute, Ljubljana, Slovenia

J. Kucera, Nuclear Research Institute, Rez, Czechoslovakia

B. Smodis, Jozef Stefan Institute, Ljubljana, Slovenia

ALS Standard Operating Procedure

DOCUMENT TITLE:	MERCURY BY OXIDATION, PURGE AND TRAP, AND COLD VAPOR ATOMIC FLUORESCENCE SPECTROMETRY
REFERENCED METHOD:	EPA 1631E
SOP ID:	MET-1631
REVISION NUMBER:	14
EFFECTIVE DATE:	12/29/2015

ALS-Kelso SOP Annual Review Statement

SOP Code: MET-1631
Revision: Rev.14

An annual review of the SOP listed was completed on (date): **2/24/17**

☒ The SOP reflects current practices and requires no procedural changes.

Supervisor: JDB Date: 2-24-17

☐ Revision of the SOP is needed to reflect current practices. Draft revisions are listed below.

SOP Section Number	**Description of Revision Needed**	**Date Procedure Change Implemented**	**Supervisor Initials** Indicating Approval of Revision

MERCURY BY OXIDATION, PURGE AND TRAP, AND COLD VAPOR ATOMIC FLUORESCENCE SPECTROMETRY

ALS-KELSO

SOP ID:	MET-1631	Rev. Number:	14	Effective Date:	12/29/2015

Approved By: _____ Date: 12/11/15
Department Manager/Technical Director – Jeff Coronado

Approved By: _____ Date: 12/11/15
QA Manager – Carl Degner

Approved By: _____ Date: 12/13/15
Laboratory Director – Jeff Grindstaff

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ANNUAL REVIEW

SIGNATURES BELOW INDICATE NO PROCEDURAL CHANGES HAVE BEEN MADE TO THE SOP SINCE THE APPROVAL DATE ABOVE. THIS SOP IS VALID FOR TWELVE ADDITIONAL MONTHS FROM DATE OF THE LAST SIGNATURE UNLESS INACTIVATED OR REPLACED BY SUBSEQUENT REVISIONS.

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MERCURY BY OXIDATION, PURGE AND TRAP, AND COLD VAPOR ATOMIC FLUORESCENCE SPECTROMETRY

1. SCOPE AND APPLICATION

1.1. This procedure is used to determine the concentration of Mercury in water, soil, tissues, aqueous and non-aqueous wastes, and sediment samples using EPA Method 1631E and its appendix.

1.2. Method 1631 is a cold vapor atomic fluorescence procedure used in determining the total mercury (Hg) in filtered and unfiltered water samples. This method is designed for the measurement of total Hg in the range of 0.2 - 100 ng/L. With use of an additional calibration standard the range may be extended to 400 ng/L.

1.3. Total mercury as defined by this method means all BrCl-oxidizable mercury forms and species found in aqueous solution. This includes but is not limited to Hg(II), Hg(0), strongly organocomplexed Hg(II) compounds, adsorbed particulate Hg and several covalently bound organomercurials (i.e., CH_3HgCl, $(CH_3)_2Hg$, and $C_6H_5HgOOCCH_3$). The recovery of Hg bound within microbial cells may require the addition step of UV photo-oxidation.

1.4. In cases where there is a project-specific quality assurance plan (QAPP), the project manager identifies and communicates the QAPP-specific requirements to the laboratory. In general, project specific QAPP's supersede method specified requirements. An example of this are projects falling under DoD ELAP. QC requirements defined in the SOP *Department of Defense Projects – Laboratory Practices and Project Management (ADM-DOD)* may supersede the requirements defined in this SOP.

2. METHOD SUMMARY

2.1. Samples are collected in new lab cleaned 500 mL Fluorinated LPE bottles. The samples are preserved with 1:1 HCl in the field, or upon receipt at the lab. Samples are prepared for analysis by the addition of 2.5 ml of BrCl solution to the 500 mL sample bottle. After oxidation the samples are pre-reduced with NH_2OH□HCl to destroy free halogens, and then reduced with $SnCl_2$ to convert Hg(II) to volatile Hg(0). Solid samples are prepared by HNO_3/ H_2SO_4 digestion then diluted with dilute BrCl solution to 40 mL, and a aliquot of 5ml is taken. The Hg(0) is separated from solution by purging with argon onto a gold trap. The trapped Hg is then thermally desorbed from the gold trap into an Ar gas stream that carries the Hg into the cell of the Brook Rand Model III cold-vapor atomic fluorescence spectrometer (CVAFS) for detection.

3. DEFINITIONS

3.1. Analysis Sequence - Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration or calibration verification followed by sample analyses interspersed with calibration standards.

3.2. Batch - A batch of samples is a group of environmental samples that are prepared and/or analyzed together as a unit with the same process and personnel using the same lot(s) of reagents. It is the basic unit for analytical quality control.

3.2.1. Preparation Batch - A preparation batch is composed of one to twenty field samples, all of the same matrix, and with a maximum time between the start of processing of the first and last samples in the batch to be 24 hours.

3.2.2. Analysis Batch - Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration (initial or continuing verification) followed by sample extracts interspersed with calibration standards (CCBs, CCVs, etc.) The sequence ends when the set of samples has been injected or when qualitative and/or quantitative QC criteria indicate an out-of-control situation.

3.3. Sample

3.3.1. Field Sample - An environmental sample collected and delivered to the laboratory for analysis; a.k.a., client's sample.

3.3.2. Laboratory Sample - A representative portion, aliquot, or subsample of a field sample upon which laboratory analyses are made and results generated.

3.4. Quality System Matrix - The *matrix* of an environmental sample is distinguished by its physical and/or chemical state and by the program for which the results are intended. The following sections describe the matrix distinctions. These matrices shall be used for purpose of batch and quality control requirements.

3.4.1. Aqueous - Any groundwater sample, surface water sample, effluent sample, and TCLP or other extract. Specifically excluded are samples of the drinking water matrix and the saline/estuarine water matrix.

3.4.2. Drinking water - Any aqueous sample that has been designated a potable or potential potable water source.

3.4.3. Saline/Estuarine water - Any aqueous sample from an ocean or estuary or other salt-water source.

3.4.4. Nonaqueous Liquid - Any organic liquid with <15% settleable solids.

3.4.5. Animal tissue - Any tissue sample of an animal, invertebrate, marine organism, or other origin; such as fish tissue/organs, shellfish, worms, or animal material.

3.4.6. Solids - Any solid sample such as soil, sediment, sludge, and other materials with >15% settleable solids.

3.4.7. Chemical waste - Any sample of a product or by-product of an industrial process that results in a matrix not described in one of the matrices in Sections 3.3.1 through 3.3.6. These can be such matrices as non-aqueous liquids, solvents, oil, etc.

3.4.8. Miscellaneous matrices – Samples of any composition not listed in 3.3.1 – 3.3.7. These can be such matrices as plant material, paper/paperboard, wood, auto fluff, mechanical parts, filters, wipes, etc. Such samples shall be batched/grouped according to their specific matrix.

3.5. Matrix Spike/Duplicate Matrix Spike (MS/DMS) Analysis - In the matrix spike analysis, predetermined quantities of target analytes are added to a sample matrix prior to sample preparation and analysis. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the method used for the analysis. Duplicate samples are spiked, and analyzed as a MS/DMS pair. Percent recoveries are calculated for each of the analytes detected. The relative percent difference (RPD) between the duplicate spikes (or samples) is calculated and used to assess analytical precision. The concentration of the spike should be at the mid-point of the calibration range or at levels specified by a project analysis plan.

3.6. Laboratory Duplicates (DUP) – Duplicates are additional replicates of samples that are subjected to the same preparation and analytical scheme as the original sample. The relative percent difference (RPD) between the sample and its duplicate is calculated and used to assess analytical precision.

3.7. Method Blank (MB) - The method blank is an artificial sample composed of analyte-free water or solid matrix and is designed to monitor the introduction of artifacts into the analytical process. The method blank is carried through the entire analytical procedure.

3.8. Laboratory Control Samples (LCS) – The LCS is an aliquot of analyte free water or analyte free solid to which known amounts target analytes are added. The LCS is prepared and analyzed in exactly the same manner as the samples. The percent recovery is compared to established limits and assists in determining whether the batch is in control.

3.9. Independent Verification Standard (ICV) - A mid-level standard injected into the instrument after the calibration curve and prepared from a different source than the initial calibration standards. This is used to verify the validity of the initial calibration standards

3.10. Continuing Calibration Verification Standard (CCV) - A mid-level standard analyzed at specified intervals. Used to verify that the initial calibration curve is still valid for quantitative purposes.

3.11. Instrument Blank (CCB) - The instrument blank (also called continuing calibration blank) is a volume of clean solvent analyzed on each column and instrument used for sample analysis. The purpose of the instrument blank is to determine the levels of contamination associated with the instrumental analysis itself, particularly with regard to the carry-over of analytes from standards or highly contaminated samples into subsequent sample analyses.

3.12. Duplicates and Duplicate Matrix Spikes are additional replicates of samples that are subjected to the same preparation and analytical scheme as the original sample. Depending on the method of analysis, either a duplicate analysis (and/or a matrix spiked sample) or a matrix spiked sample and duplicate matrix spiked sample (MS/DMS) are analyzed.

3.13. Standard Reference Material (SRM) – A material with specific certification criteria and is issued with a certificate or certificate of analysis that reports the results of its characterizations and provides information regarding the appropriate use(s) of the material. An SRM is prepared and used for three main purposes: (1) to help develop accurate methods of analysis; (2) to calibrate measurement systems used to facilitate exchange of goods, institute quality control, determine performance characteristics, or measure a property at the state-of-the-art limit; and (3) to ensure the long-term adequacy and integrity of measurement quality assurance programs.

3.14. Ongoing Precision Recovery (OPR) - A standard analyzed at specified intervals and used to verify the ongoing validity of the instrument calibration.

3.15. Calibration Verification (VER) – is spiked reagent water sample (aqueous blank spike) and is used to determine that the CVAFS remains in control.

4. INTERFERENCES

4.1. It is imperative that extreme care be taken to avoid contamination when collecting and analyzing ambient water samples for trace mercury. Potential sources of contamination during sampling include: metallic or metal-containing labware, containers, sampling equipment, reagents, and reagent water; improperly cleaned and stored equipment, labware, and reagents; and atmospheric inputs such as dirt and dust. Even human contact can be a source of trace mercury contamination (e.g., mercury fillings).

4.2. Within the laboratory, interferences from contaminated reagents must be minimized. Pre-screened acids and reagent grade chemicals are used to prepare the reagents. To minimize contamination all sample preparation should be performed in the Class 100 clean hood. Before a given batch of samples is processed, all work surfaces in the hood or clean bench where the samples will be processed should be cleaned by wiping with a lint-free cloth or wipe soaked with reagent water.

4.3. All apparatus used for determination of mercury at ambient water quality criteria levels must be nonmetallic, free of material that may contain metals, or both.

4.4. Samples are taken in fluorinated LPE containers. These containers have a thin coating of a fluoropolymer.

4.5. Water vapor may collect in the gold trap and subsequently condense in the fluorescence cell upon desorption, giving a false peak due to scattering of excitation radiation. Condensation can be avoided by pre-drying the gold trap, and by discarding those traps that tend to absorb large quantities of water vapor.

4.6. Bottle blanks-- Laboratory cleaned bottles are used for method blanks associated with each batch of samples confirming the absence of contamination on an ongoing basis.

5. SAFETY

5.1. All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this procedure. This includes the use of personnel protective equipment, such as, safety glasses, lab coat and the correct gloves.

5.2. Chemicals, reagents and standards must be handled as described in the ALS safety policies, approved methods and in MSDSs where available. Refer to the ALS Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

5.3. Hydrochloric and/or Nitric Acid are used in this method. These acids are extremely corrosive and care must be taken while handling them. A face shield should be used while pouring acids. And safety glasses should be worn while working with the solutions. Lab coat and gloves should always be worn while working with these solutions.

5.4. Chronic mercury exposure may cause kidney damage, muscle tremors, spasms, personality changes, depression, irritability and nervousness. Organomercurials may cause permanent brain damage. Because of the toxicological and physical properties of the Hg, pure standards should be handled only by highly trained personnel thoroughly familiar with handling and cautionary procedures and the associated risks.

5.5. ALS purchases a dilute standard solution of Hg for this method. If primary solutions are prepared, they must be prepared in a hood.

5.6. Hands should be washed thoroughly after each manipulation and before breaks.

5.7. If background contamination is encountered, then the cleanliness of work surfaces and tools may be assessed by wiping the surface with a piece of filter paper. Extraction and analysis by this method can achieve a limit of detection of less than 1 ng per wipe. Less than 0.1 mg per wipe indicates acceptable cleanliness; anything higher warrants further cleaning. More than 10 mg on a wipe constitutes an acute hazard and requires prompt cleaning before further use of the equipment or work space, and indicates that unacceptable work practices have been employed.

6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION AND STORAGE

6.1. ALS laboratory staff does not collect samples. Samples are collected by field sampling staff of ALS customers using their sampling plans and procedures. In some cases, persons collecting samples may be required to be certified by regulatory bodies. Samples are either field- or laboratory-preserved by adding 5 mL of 6N (1:1) HCl to a 500 mL Fluorinated LPE bottle. Upon receipt at the laboratory, samples are taken to the clean room and BrCl (2.5ml/500ml) is added.

6.2. Samples that are acid-preserved only may lose Hg to coagulated organic materials in the water or the Hg may be condensed on the walls (Reference 19.11). The bottle should be vigorously shaken before sub-sampling to re-suspend the organic matter.

6.3. All handling of the samples in the laboratory should be undertaken in a mercury-free clean bench.

6.4. Water samples are typically stored at 4 ± 2ºC until analysis. However, this is not a method requirement and samples may be stored at room temperature if necessary. The maximum holding time for mercury in unpreserved aqueous samples is 48 hrs. If a sample is oxidized in the sample bottle, the time to preservation can be extended to 28 days. Once preserved, aqueous samples have maximum holding time of 90 days. A sample collected for dissolved trace level mercury should be filtered in the laboratory within 24 hours of the time of collection. Solid samples have a holding time of 1 year if stored in a freezer at < -15ºC. Samples that are freeze dried have a holding time of 1 year, also.

6.5. Soil samples are aliquoted and weighed for analysis upon receipt. Prepared samples are stored at < -15º C for up to 1 year before digestion and analysis.

6.6. Tissue samples are homogenized then lyophilized. The lyophilized sample may be stored at room temperature in a low level mercury atmosphere for up to 1 year.

6.7. Field blanks – Field blanks are used to demonstrate that samples have not been contaminated by the sample collection and transport activities.

6.7.1. Analyze the field blank(s) shipped with each set of samples (samples collected from the same site at the same time, to a maximum of 10 samples). Analyze the blank immediately before analyzing the samples in the batch.

6.7.2. If Hg or any potentially interfering substance is found in the field blank at a concentration equal to or greater than the MRL (Table 1) or greater than one-fifth the level in the associated sample, whichever is greater, results for associated samples may be the result of contamination and may not be reported or otherwise used for regulatory compliance purposes.

6.7.3. If contamination of the field blanks and associated samples is known or suspected, the laboratory should communicate this to the sampling team so that the source of contamination can be identified and corrective measures taken before the next sampling event.

6.8. Equipment blanks—before any sampling equipment is used at a given site, equipment blanks (bottle blanks and sampler check blanks) must be submitted to the laboratory to demonstrate that the sampling equipment is free from contamination. Sampler check blanks are generated by processing reagent water through the sampling devices using the same procedures that are used in the field. Refer to Method 1631E for details.

7. STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

7.1. Reagent grade chemicals shall be used in all tests. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lowering the accuracy of the determination. The preparation for all laboratory prepared reagents and solutions must be documented in a laboratory logbook. Refer to the SOP *Reagent/Standards Login and Tracking (ADM-RTL)* for the complete procedure and documentation requirements.

7.1.1. Reagent water: Water in which mercury is not detected by this method; 18-megaohm ultra-pure deionized water starting from a pre-purified (laboratory DI water) source.

7.1.2. Argon: Grade 5.0 (ultra high-purity, GC grade) inert gas that has been further purified by the removal of Hg using a gold-coated sand trap.

7.1.3. Hydrochloric acid: trace-metal purified reagent HCl containing less than 5 pg/mL Hg.

7.1.4. Hydroxylamine hydrochloride: Dissolve 150 g of $NH_2OH\cdot HCl$ in reagent water and bring to 500 mL. This solution may be purified by the addition of 0.5 mL of $SnC1_2$ solution and purging overnight at 350 mL/min. with Hg-free Ar. Store tightly capped.

7.1.5. Stannous chloride: Bring 100 g of $SnCl_2\cdot 2H_2O$ and 75 mL concentrated HCl to 500 mL with reagent water. Purge overnight with mercury-free Ar at 350 mL/min. to remove all traces of Hg. Store tightly capped.

7.1.6. Bromine monochloride (BrCl): Dissolve 5.4 g of reagent grade KBr in 500 mL of low-Hg HCl. Place a clean magnetic stir bar in the bottle and stir for approximately 1 hr in a fume hood. Slowly add 7.6 g reagent grade $KBrO_3$ to the acid with stirring. When all of the $KBrO_3$ has been added, the solution color should change from yellow to red to orange. Loosely cap the bottle, and allow to stir another hour before tightening the lid. KBr and $KBrO_3$ are purified by heating to 250°C overnight in an oven.

CAUTION: This process generates copious quantities of free halogens ($C1_2$, Br_2, BrCl), which are released from the bottle. Add the $KBrO_3$ SLOWLY in a fume hood!

7.1.7. HNO_3/H_2SO_4 solution: In a fume hood, slowly add 300ml of concentrate H_2SO_4 to 700ml of concentrate HNO_3 in a Teflon bottle.

Warning: This mixture gets hot and emits fumes.

7.1.8. 0.02 N Bromine monochloride solution: Dilute 100mls of concentrated BrCl solution to 1000ml with reagent water in a Teflon bottle.

7.2. Stock standard solutions may be purchased from a number of vendors. All reference standards, where possible, must be traceable to SI units or NIST certified reference materials. The vendor-assigned expiration date is used.

7.2.1. Stock mercury standard: Stock mercury standards (1,000 ppm) are obtained from CPI International, Santa Rosa, CA, P/N 4400-1000331 and Inorganics Ventures, Inc., Lakewood, NJ, P/N CGHG1-5. A NIST-certified 10,000 ppm aqueous Hg solution (NBS-3133) is also available. These solutions are stable until the expiration date.

7.2.2. Secondary Hg standard: Dilute 0.100 mL of the stock solution (1,000 ppm) to 100 mL in water containing 2.5 mL of BrCl. This solution contains 1.00 ug/mL (1.00 ppm) Hg. Keep in a tightly closed fluoropolymer bottle. This expiration date is the same as the stock standard.

7.2.3. Working Hg standard A: Dilute 1.00 mL of the secondary Hg standard to 100 mL in a class A volumetric flask with reagent water containing 2.5% by volume BrCl solution. This solution contains 10.0 ng/mL and should be replaced monthly.

7.2.4. Working Hg standard B: Dilute 1.00 mL of the secondary Hg standard to 1000 mL with reagent water containing 2.5% by volume BrCl solution (10 mL BrCl/1000mL). This solution contains 0.1 ng/mL and should be replaced monthly.

8. APPARATUS AND EQUIPMENT

8.1. Cold vapor atomic fluorescence spectrometer (CVAFS): Brooks-Rand (Seattle, WA) Model III CVAFS, or equivalent.

8.2. Autosampler: Brooks Rand (Seattle, WA) Model 17400.

8.3. Purge and Trap Module: Brooks Rand (Seattle, WA) MERX Total-Hg Purge and Trap Module.

8.4. Flowmeter, with needle valve capable of reproducibly keeping the carrier gas flow rate at 60 mL/min.

8.5. Pyrex bubbler with 4 way Teflon stopcock, 220 mL (Brooks-Rand, Seattle, WA, part no. AF-32 or equivalent)

8.6. Flow meter/needle valve capable of controlling and measuring gas flow rate to the purge vessel at 350 ± 50 mL/min.

8.7. Fluoropolymer fittings: Connections between components and columns are made using 6.4-mm o.d. fluoropolymer tubing and fluoropolymer friction-fit or threaded tubing connectors. Connections between components requiring mobility are made with 3.2-mm o.d. fluoropolymer tubing because of its greater flexibility.

8.8. Acid fume pre-trap: 10-cm long x 0.9-cm i.d. fluoropolymer tube containing 2-3 g of reagent grade, non-indicating, ~14 mesh soda lime chunks, packed between wads of silanized glass wool. This trap must be cleaned of Hg by placing on the output of a bubbler containing reagent water and $SnCl_2$ and purging for 45 minutes with Ar.

8.9. Gold-coated sand trap or gold wire trap: 10-cm x 6.5-mm o.d. x 4-mm i.d. quartz tubing. The tube is filled with 3.4 cm of gold-coated 45/60 mesh quartz sand (Brooks Rand, Ltd., Seattle, WA, Part No. AF-20 or equivalent). The ends are plugged with quartz wool. A gold wire trap is also available from Brooks Rand (Part No. AF-19).

8.10. Traps are fitted with 6.5-mm i.d. fluoropolymer friction-fit sleeves for making connection to the system. When traps are not in use, fluoropolymer end plugs are inserted in trap ends to eliminate contamination. At least 16 traps are needed for efficient operation.

8.11. Heating of gold-coated sand traps: To desorb Hg collected on the traps, heat for 2-3 minutes to 450-500°C (a barely visible red glow when the room is darkened) with a coil consisting of 75 cm of 24-gauge Nichrome wire at a potential of 10 Vac. Potential is applied and finely adjusted with a variable transformer.

8.12. Air blower: After heating, the trap is cooled by blowing air from a small blower positioned immediately below the trap.

8.13. Computer (386 or better) and Windows Mercury Guru software to record and integrate the signal from the spectrometer.

8.14. Pipettors: All-plastic pneumatic fixed-volume and variable pipettors in the range of 10 uL to 5.0 mL.

8.15. Analytical balance capable of weighing to the nearest 0.001 g

8.16. Hot Block that is able to maintain temperature of 100 Celsius.

8.17. 40mL Precleaned Clear VOA Vials.

9. PREVENTIVE MAINTENANCE

9.1. All maintenance activities are recorded in a maintenance logbook kept for each instrument. Pertinent information (serial numbers, instrument I.D., etc.) must be in the logbook. This includes the routine maintenance described in section 9. The entry in the log must include: date of event, the initials of who performed the work, and a reference to analytical control.

9.2. Broad or asymmetrical peaks indicate a problem with the desorption train, such as low gas flow rate, water vapor on the trap(s), or an analytical column damaged by chemical fumes or overheating.

9.3. Damage to a trap is also indicated by a sharp peak, followed by a small, broad peak.

9.4. If the trap has been damaged, it and the fluoropolymer tubing downstream from it should be discarded because of the possibility of gold migration on downstream surfaces.

9.5. Gold traps should be tracked by unique identifiers so that any trap producing poor results can be quickly recognized and discarded. Follow the procedure in the Brooks Rand Manual for replacing gold traps for the Total Hg Purge and Trap Module.

9.6. The Teflon Liquid collection trap should be emptied prior to use each day to prevent liquid from damaging the analytical gold sand traps.

9.7. Every day prior to use replace the Soda Lime in the Soda Lime trap to prevent moisture in argon carrier to enter the analytical system.

9.8. Depending on usage the Mercury Source Lamp in the CVAFS unit will need to be changed. This procedure is found in the Brooks Rand Manual.

10. RESPONSIBILITIES

10.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. The acceptance criteria for test performance are listed in Table 1. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10.2. It is the responsibility of the department supervisor/manager to document analyst training and method proficiency.

11. PROCEDURE

11.1. An analytical batch is up to 20 field samples that are oxidized with the same reagents and analyzed within the same 12-hour shift. The analytical batch contains the following standards, samples, and blanks, in order:

Water Run Manual System:

5 ng/L OPR and bubbler blank
Water blank 1
Method blank 1
5 ng/L QCS and bubbler blank
Up to 10 samples

Matrix Spike
Duplicate Matrix Spike
Method blank 2
OPR (optional)
Up to 10 additional samples
Matrix Spike
Duplicate Matrix Spike
Method blank 3
5 ng/L OPR and bubbler blank

Soil Run Manual System:

Calibration Verification (VER) and bubbler blank
Method Blank 1
Method Blank 2
OPR (use to demonstrate end to end analytical system)
QCS or SRM
Up to 10 samples
Matrix Spike
Duplicate Matrix Spike
Calibration Verification (VER) and bubbler blank
Up to 10 additional samples
Matrix Spike
Duplicate Matrix Spike
Method blank 3
OPR (use to demonstrate end to end analytical system)
Calibration Verification (VER) and bubbler blank

For more than 20 samples in a 12 hour shift, the sequence is repeated.

Water Run MERX System:

Calibration/Bubbler Blank
Calibration/Bubbler Blank
Calibration/Bubbler Blank
Calibration/Bubbler Blank
5pg STD
10pg STD
25pg STD
100pg STD
500pg STD
2500pg STD
10000pg STD
OPR 5ng/L
QCS 5ng/L
Method Blank 1
Up to 10 samples
Matrix Spike
Matrix Spike Dup
Method Blank 2
Up to 10 samples
Matrix Spike
Matrix Spike Dup

Method Blank 3
OPR 5ng/L

For more than 20 samples, the sequence is repeated from the first OPR.

Soil Run MERX System:

Calibration/Bubbler Blank
Calibration/Bubbler Blank
Calibration/Bubbler Blank
Calibration/Bubbler Blank
5pg STD
10pg STD
25pg STD
100pg STD
500pg STD
2500pg STD
10000pg STD
VER 5ng/L
Method Blank 1
Method Blank 2
OPR 5ng/L (use to demonstrate end to end analytical system)
QCS 5ng/L/MESS/TORT
Up to 10 samples
Matrix Spike
Matrix Spike Dup
VER 5ng/L
Up to 10 samples
Matrix Spike
Matrix Spike Dup
Method Blank 3
OPR 5ng/L (use to demonstrate end to end analytical system)
VER 5ng/L

For more than 20 samples, the sequence is repeated from the first VER.

11.2. Reagent blanks are analyzed in triplicate when there is a change in reagent(s) to verify its purity.

11.3. Sample Preparation

11.3.1. Record all sample preparation and sample information on the applicable benchsheet. This includes acid mixture tracking documentation.

11.3.2. Water samples are oxidized by adding 2.5 mL of BrCl per 500 mL bottle, however more may be required for complete oxidation. If more than 5.0 mL of BrCl is used then a method blank is prepared with the equivalent amount of BrCl. Digest at room temperature for 12 hours. Shorter digestion times can be achieved by using elevated temperatures. For example, 70°C for 2 hours is adequate.

11.3.3. Some highly organic matrices, such as sewage effluent, will require dilution as much as 1:10, additional BrCl, and longer oxidation times. The oxidation must be continued until excess BrCl remains. This can be checked using starch-iodide paper. Again a method blank is created using the equivalent amount of BrCl.

11.3.4. Solid samples are prepared by weighing 400mg of sample into a 40ml vial. To each sample, add 10.0 ml of HNO_3/H_2SO_4 solution. A tapered glass marble is placed over the digestion vessel. The sample vials are placed in a cold digestion Hot Block which is then switched on and set to 100˚C. After the Hot Block and sample vials reach 100˚ C they are refluxed for 2-3 hours. Once the samples have cooled they are diluted to 40 mL with 0.02 N BrCl solution. The samples then need to sit for At least 4 hours prior to analysis. This is to allow for complete oxidation of the methyl Hg.

11.3.5. Matrix spikes and matrix spike duplicates: For each batch of 10 or fewer samples, aliquot two additional 100-mL portions for the manual system, 25mL for the MERX system from a randomly selected sample, spike at the level specified in Section 12.3.3, and process in the same manner as the samples.

11.3.6. Method Blank: Three method blanks are prepared with each batch of samples. The method blanks are prepared in lab cleaned 500 mL sample bottles chosen at random, thus serving as a check on the bottle washing procedure.

11.3.7. Water blank: For the manual system at the same time as the samples are analyzed, prepare a water blank by adding 0.5 mL of BrCl to 100 mL in one of the sample bubblers. The mercury content of the water blank is used to correct the OPR, QCS, method blank values, and dilutions. Water blanks may not be used for Wisconsin samples; instead prepurged DI water will be used for OPR, QCS, MBs and dilution water.

11.4. At the beginning of each 12 hour shift, attach lime traps to the bubbler and purge with Ar for 20 minutes to clean for the manual system. For the MERX system replace soda lime in the soda lime trap.

11.5. Instrument Initialization--Allow the instrument to warm-up for 30 minutes before attempting to analyze samples. Instrument may be left on overnight and therefore will not need 30 minutes to warm.

NOTE: Purging of halogens onto the gold trap will result in damage and low or irreproducible results.

11.6. Sample Analysis

11.6.1. Water Samples Manual System

11.6.1.1. Aliquot 100 mL of BrCl-oxidized sample to a bubbler and, add 0.2 mL of NH_2OH. Cap swirl the sample. The yellow color will disappear, indicating the destruction of the BrCl.

11.6.1.2. Connect a soda lime trap and a gold trap to the bubbler, add 1.0 mL of $SnCl_2$ solution, and purge the sample with Ar for 20 min. at 300-400 mL/min. At the end of the sparging time, remove the gold trap, plug the ends and save for analysis (See Section 11.7). Use the Bubbler blanks to correct the sample

measurements and the water blank to correct the QCS, OPR, and Method Blank measurements. Turn of the argon valve and disconnect the argon lines from each bubbler. Each bubbler's base and stem is then rinsed three times with DI water. After reconnecting the argon lines the bubblers are ready for the next analysis.

11.6.1.3. Repeat 11.6.1.1 and 11.6.1.2 for each standard, blank, QC sample, and field sample.

11.6.2. Water Samples MERX System

11.6.2.1. Aliquot 25mL of BrCl-oxidized sample to a 40mL clear vial and add 0.1mL NH2OH, and 0.1mL SnCl2 solution. Cap the vial and place into autosampler rack and then place the autosampler rack onto the autosampler.

11.6.2.2. Repeat 11.6.2.1 for each standard, blank. QC sample and field sample.

11.6.2.3. Open Hg Guru Software and fill out batch information tab and run information tab. Then connect to the instrument, turn on instrument gases, and measure noise.

11.6.2.4. Select number of samples to analyze on the automation tab and then press the start batch tab on the top left corner of the Hg Guru software.

11.6.2.5. Use the Calibration/Bubbler Blanks to correct all measurements.

11.6.3. Solid samples Manual System - A 100ml portion of water is pre-purged with 1.0 ml $SnCl_2$ solution for 5 minutes. This is required for all samples and standards that are purged. Connect a soda lime trap and a gold trap to the bubbler. A VER is ran every ten samples and at the beginning and end of the run. A 5ml aliquot of the samples and standards are purged for 20 min. at 300-400 ml/min. They are sparged according to the run order (See Section 11.1). At the end of the sparging time, remove the gold trap, plug the ends and save for analysis (See Section 11.7). Use the Bubbler blanks to correct the sample measurements.

11.6.4. Solid Samples MERX System

11.6.4.1. Add 5.0mL of sample to 20mL of DI water in a 40mL clear vial. Then add 0.2mL NH2OH, and 0.1mL SnCl2 solution. Cap the vial and place into autosampler rack and then place the autosampler rack onto the autosampler.

11.6.4.2. Repeat 11.6.2.1 for each standard, blank. QC sample and field sample.

11.6.4.3. Open Hg Guru Software and fill out batch information tab and run information tab. Then connect to the instrument, turn on instrument gases, and measure noise.

11.6.4.4. Select number of samples to analyze on the automation tab and then press the start batch tab on the top left corner of the Hg Guru software.

11.6.4.5. Use the Calibration/Bubbler Blanks to correct all measurements.

11.7. Desorption of Hg from the gold trap for the Manual System

11.7.1. Remove the plugs from the gold trap, place the Nichrome wire coil around the trap and connect it into the analyzer train between the incoming Hg-free argon and the detector.

11.7.2. Pass argon through the trap at a flow rate of approximately 30 mL/min. for 45-60 seconds to drive off condensed water vapor.

11.7.3. Apply electrical current (9.5-10 V DC) to the Nichrome coil around the gold trap and begin data collection. The applied electrical current will thermally desorb the Hg (as Hg(0)) from the trap into the detector.

11.7.4. After the 3-min. desorption time, stop data collection, turn off the current to the coil, and cool the trap (about one minute) using the cooling fan.

11.8. Place the next gold trap in line and proceed with analysis of the next sample.

Peaks generated using this technique should be very sharp and almost symmetrical. Mercury elutes at approximately 2 min. and has a width at half-height of about 11 seconds.

11.9. Calibration and standardization

11.9.1. The calibration must contain five or more non-zero points and the results of the analysis of 4 bubbler blanks. The lowest calibration point must be equivalent to the MRL, or lower. For the manual system a maximum of 4 bubblers may be used for calibration.

11.9.2. Using the procedure in Section 11.6, standards are analyzed by the addition of aliquots of Hg working standard A (8.10) and Hg working standard B (8.11) directly into 100 mL of previously purged water in the bubbler.

11.9.2.1. For the manual system add 0.2 mL, 0.5 mL and 2.0 mL of working standard B and 1.0 mL $SnCl_2$ to three separate bubblers. Swirl to produce a standard of 0.2, 0.5 and 2.0 ng/L. Purge under the normal operating conditions described above. Sequentially follow with the addition of aliquots of 0.05, 0.20, 0.50, and 1.5 mL of working standard A plus 1.0 mL of $SnCl_2$ to produce standards of 5.0, 20.0, 50.0, and 150 ng/L.

11.9.2.2. For the MERX system add 0.05mL, 0.1mL, 0.25mL, 1.0mL of working standard B and 0.1mL NH_2OH, and 0.1mL $SnCl_2$ to four separate 40mL clear vials. Sequentially follow with the addition of aliquots of 0.05mL, 0.25mL and 1.0mL of working standard A plus 0.1mL NH_2OH, and 0.1mL $SnCl_2$.

11.9.3. For each point, subtract the mean peak area of the Calibration/Bubbler Blanks for the batch from the area of each standard. Calculate the calibration factors (CF) for Hg in each of the standards as follows:

$$CF = \frac{(C_s)}{(A_{Corr})}$$

where C_s = Concentration of the standard
A_{corr} = Bubbler blank corrected peak area

Calculate the relative standard deviation (RSD) of the calibration factor over the six-point range.

11.9.4. Calibration criteria are as follows:

(a) There must be a minimum of five non-zero calibration points.
(b) The difference between successive calibration points must be no greater than a factor of 10 and no less than a factor of 2 and should be approximately evenly spaced on a logarithmic scale over the calibration range.
(c) The relative standard deviation (RSD) of the calibration factors for all calibration points must be less than 15%.
(d) The calibration factor for any calibration point at a concentration greater than 100 ng/L must be within ±15% of the average calibration factor for the points at or below 100
(e) The calibration factor for any point <0.5 ng/L must be within 25% of the average calibration factor for all points.
(f) If calibration is to a higher range and this procedure is used for regulatory compliance, the MRL must be less than one-third the regulatory compliance limit.

11.9.5. Ongoing precision and recovery (OPR)—Perform the ongoing precision and recovery test (12.3.1) to verify calibration prior to, and after in each analytical batch.

12. QA/QC REQUIREMENTS

12.1. Initial demonstration of capability

12.1.1. Laboratory performance is compared to the established performance criteria listed in Table 1. The analyst shall make an initial demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as follows:

12.1.2. Initial precision and recovery (IPR). To establish the ability to generate acceptable precision and accuracy, the analyst shall perform the following operations:

12.1.2.1. Analyze four replicates of the working Hg standard (Section 8.10) according to the procedure beginning in Section 11.3 – 11.7. These four replicates are prepared the same as samples are prepared.

12.1.2.2. Using the results of the set of four analyses, compute the average percent recovery (X), and the standard deviation of the percent recovery (s) for total Hg.

12.1.2.3. Compare s and X with the corresponding limits for initial precision and recovery in Table 1. If s and X meet the acceptance criteria, system

performance is acceptable and sample analysis may begin. If, however, s exceeds the precision limit or X falls outside the acceptance range, system performance is unacceptable. Correct the problem and repeat the test.

12.2. Method Detection Limits

12.2.1. A method detection limit (MDL) study must be undertaken before analysis of samples begins. To establish detection limits that are precise and accurate, the analyst must perform the following procedure. Spike a minimum of seven blank replicates with a MDL spiking solution (at or below the MRL) and analyze. Refer to the SOP CE-QA011, *Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantification*.

12.2.2. Calculate the average concentration found (x) in the sample concentration, and the standard deviation of the concentrations for each analyte. Calculate the MDL for each analyte using the correct T value for the number of replicates. The MDL study must be verified annually.

12.3. Ongoing QC Samples required are described in the ALS-Kelso *Quality Assurance Manual,* in the *SOP for Sample Batches*, and in method 1631E. An analytical batch is a set of samples oxidized with the same batch of reagents, and analyzed during the same 12-hour shift. A batch may be from 1 to 20 samples. Each batch must be accompanied by three method blanks, two OPR samples, and a QCS. In addition, there must be one MS/MSD pair analyzed for every 10 samples.

12.3.1. Ongoing precision and recovery (OPR): To demonstrate that the analysis system is in control and that acceptable precision and accuracy is being maintained within each analytical batch, the analyst shall perform the following operations:

12.3.1.1. Analyze the low-level Hg (5 ppt) working standard (Section 8.10) before and after analysis of each analytical batch according to the procedure beginning in Section 11. Subtract the mean peak area of the bubbler blank and water blanks (water blanks are subtracted only for the manual system) from the area for the standard and compute the concentration for the blank-subtracted standard. (Note: bubbler and water blank corrections are done automatically by the instrument software.)

12.3.1.2. Run a bubbler blank after each OPR and QCS sample for the manual system. There must be at least 3 per analytical batch of 20 samples. See section 12.4.4.1. For the MERX system bubbler blanks are run at the beginning of the analytical run.

12.3.1.3. Compare the concentration with the limits for ongoing precision and recovery in Table 1. If the concentration is in the range specified, the analysis system is in control and analysis of samples and blanks may proceed. If, however, the concentration is not in the specified range, the analytical process is not in control. Correct the problem and repeat the ongoing precision and recovery test.

12.3.2. Quality control sample (QCS): A QCS from a source different than the Hg used to produce the standards (OPR and working standards) must be analyzed at the beginning of each analytical batch. The acceptance criteria for the water QCS is 77-

123%. For soils and tissues, a reference material is routinely used and the acceptance criterion is 70-130%.

12.3.3. Matrix spike (MS) and matrix spike duplicate (MSD):

12.3.3.1. To assess the performance of the method on a given sample matrix, spike, in duplicate, a minimum of 10% (1 sample in 10) from a given sampling site or, if for compliance monitoring, from a given discharge. Blanks (e.g., field blanks) may not be used for MS/MSD analysis. A spike level of 5 ng/L has been found to acceptable for most samples.

Note: If, as in compliance monitoring, the concentration of Hg in the sample is being checked against a regulatory compliance limit, the spiking level shall be at that limit or at 1-5 times the background concentration in the sample, whichever is greater. If the sample concentration is not being checked against a regulatory limit, the spike shall be at 1-5 times the background concentration in the sample.

12.3.3.2. Spike two sample aliquots (MS and MSD) with the spiking solution and analyze these aliquots to determine the concentration after spiking (A).

12.3.3.3. Calculate the percent recovery (P) in each aliquot using the following equation:

$$100\ (A-B)/T$$

where:
A = Measured concentration of analyte after spiking
B = Measured concentration of analyte before spiking
T = True concentration of the spike

12.3.3.4. Compare the percent recovery with the QC acceptance criteria in Table 1. If the results of spike fail the acceptance criteria, and recovery for the OPR standard for the analytical batch is within the acceptance criteria in Table 1, interference may be present. The result may not be reported for regulatory compliance purposes. If the interference can be attributed to sampling, the site or discharge should be re-sampled. If the interference can be attributed to a laboratory error or deficiency, the analyst must take corrective action and repeat analysis of the associated samples (10 per MS/MSD pair) and MS/MSD.

12.3.3.5. If the results of both the spike and the OPR test fail the acceptance criteria, the analytical system is judged to be out of control. The analyst must identify and correct the problem and reanalyze the sample batch.

12.3.3.6. Relative percent difference between duplicates: Compute the relative percent difference (RPD) between the MS and MSD according to the following equation using the concentrations found in the MS and MSD. Do not use the recoveries calculated in Section 12.4.4.3 for this calculation because the RPD is inflated when the background concentration is near the spike concentration.

RPD = 200 x (D1-D2)/(D1+ D2)

Where:

Dl = concentration of Hg in the MS sample
D2 = concentration of Hg in the MSD sample

The RPD for the MS/MSD pair shall meet the acceptance criterion in Table 1. If the criterion is not met, the system is judged to be out of control. The problem must immediately be identified and corrected, and the analytical batch reanalyzed.

12.3.4. Blanks—Blanks are critical to the reliable determination of Hg at low levels. The sections below give the minimum requirements for analysis of blanks.

12.3.4.1. Bubbler blanks—Bubbler blanks are analyzed to demonstrate freedom from system contamination. At least three bubbler blanks must be run per analytical batch. One bubbler blank must be analyzed following each OPR.

12.3.4.1.1. Immediately after analyzing a sample for Hg, place a clean gold trap on the bubbler, purge and analyze the sample a second time using the procedure in Section 11, and determine the amount of Hg remaining in the system.

12.3.4.1.2. If the bubbler blank is found to contain more than 50 pg Hg, the system is out of control. The problem must be investigated and remedied, and the samples run on that bubbler must be reanalyzed. If the blanks from other bubblers contain less than 50 pg Hg, the data associated with those bubblers remain valid.

12.3.4.1.3. The mean result for all bubbler blanks (from bubblers passing the specification above) in an analytical batch (at least three bubbler blanks) is calculated at the end of the batch. The mean result must be < 25 pg with a standard deviation of < 10 pg for the batch to be considered valid.

12.3.4.1.4. If Hg in the bubbler blank exceeds the acceptance criteria, the system is out of control, and the problem must be resolved and the samples reanalyzed. Usually, the bubbler blank is too high for one of the following reasons:

- Bubblers need rigorous cleaning;
- Soda-lime is contaminated; or
- Carrier gas is contaminated.

12.3.4.2. Method/Bottle Blank: Three method blanks are prepared with each batch of sample by adding 2.5 mL BrCl to 500 mL of deionized water in a laboratory cleaned Fluorinated LPE bottle. When samples require more BrCl for preservation than a method blank at the same level is prepared to accompany those samples for analysis. The method blank uses bottles chosen at

random from all sample bottles and serves as a check on the bottle washing procedure.

12.3.4.3. Water blank: At the same time as the samples are analyzed, prepare a water blank by adding 0.5 mL of BrCl to 100 mL of DI water in one of the bubblers. The mercury content of the water blank is used to correct the OPR, QCS, and Method Blank values. One water blank is prepared at the beginning of purging samples.

12.4. Reagent blanks—The Hg concentration in reagent blanks must be determined on solutions of reagents by adding these reagents to reagent water in the bubbler.

12.4.1. Reagent blanks are required when the batch of reagents (bromine monochloride plus hydroxylamine hydrochloride) are prepared, with verification in triplicate for each new batch of reagents is needed.

12.4.2. Add aliquots of BrCl (0.5 mL), NH_2 OH (0.2 mL) and $SnCl_2$ (1.0 mL) to previously purged water in the bubbler for the manual system, for the MERX system add to 25mL of DI water in 40mL Clear VOA Vial. In order to evaluate the reagents as a potential source of contamination, the amount of reagent added to the reagent blank(s) must be the same as the amount of reagent added to the sample(s).

12.4.3. The presence of more than 20 pg of Hg indicates a problem with the reagent solution. The purging of certain reagent solutions, such as $SnCl_2$ or NH_2 OH with mercury-free argon can reduce Hg to acceptable levels. Because BrCl cannot be purified, a new batch should be made from different reagents and should be tested for Hg levels if the level of Hg in the BrCl solution is too high.

12.5. As part of the QC program for the laboratory, method precision and accuracy for samples should be assessed and records maintained. Update the accuracy assessment regularly.

13. DATA REDUCTION AND REPORTING

13.1. Quantitation

13.1.1. Calculate the concentration of Hg in each sample directly from the mean calibration factor:

$$Hg(ng/L) = \frac{(A_s - \overline{A}_{BB}) \, x \, CF_m}{V}$$

where: A_s = peak area (or height) for Hg in sample
A_{BB} = peak area (or height) for Hg in bubbler blank (mean)
CF_m = Mean calibration factor
V = Volume of sample

13.1.2. Report results for Hg in reagent blanks separately.

13.2. Report results for samples in ng/L to two significant figures (three if >10 ng/L) for total Hg found above the MRL (See Table 1). Report results below the MRL but above the MDL as

estimated values (J flagged). Report results below the MRL as ND, unless the project specifies reporting to the MDL.

13.3. If the result over the calibration range, prepare and analyze a diluted sample using the appropriate dilution factor to bring within range. For water samples, dilute the selected sample aliquot in reagent water and analyze. For soils, dilute an appropriate smaller aliquot of the soil digestate into reagent water and analyze. For samples far over range, an alternate procedure may be more appropriate. Consult with the Project Chemist before using another method.

13.4. Data Review and Assessment

13.4.1. Refer to the *SOP for Laboratory Data Review Process* for general instructions for data review.

13.4.2. It is the analyst's responsibility to review analytical data to ensure that all quality control requirements have been met for each analytical run. Results for QC analyses are calculated and recorded as specified in section 12.

13.4.3. Following primary data interpretation and calculations, all data is reviewed by a secondary analyst. Following generation of the report, the report is also reviewed. Refer to the SOP for *Laboratory Data Review Process* for details. The person responsible for final review of the data report and/or data package should assess the overall validity and quality of the results and provide any appropriate comments and information to the Project Chemist to inclusion in the report narrative.

13.5. Reporting

13.5.1. Refer to the SOP for *Data Reporting and Report Generation* for reporting guidelines.

13.5.2. Reports are generated in Excel© by compiling the SMO login from CASLIMS and then entering sample information. The forms generated may be ALS standard reports, DOD, or client-specific reports. The compiled data from Excel© file are also used to create EDDs.

14. CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

14.1. Refer to the SOP for *Nonconformity and Corrective Action* (CE-QA008) for procedures for corrective action. Personnel at all levels and positions in the laboratory are to be alert to identifying problems and nonconformities when errors, deficiencies, or out-of-control situations are detected.

14.2. Handling out-of-control or unacceptable data-See Table 2

14.2.1. On-the-spot corrective actions that are routinely made by analysts and result in acceptable analyses should be documented as normal operating procedures, and no specific documentation need be made other than notations in laboratory maintenance logbooks, runlogs, for example.

14.2.2. Some examples when documentation of a nonconformity is required using a Nonconformity and Corrective Action Report (NCAR):

- Quality control results outside acceptance limits for accuracy and precision
- Method blanks or continuing calibration blanks (CCBs) with target analytes above acceptable levels
- Sample holding time missed due to laboratory error or operations
- Deviations from SOPs or project requirements
- Laboratory analysis errors impacting sample or QC results
- Miscellaneous laboratory errors (spilled sample, incorrect spiking, etc.)
- Sample preservation or handling discrepancies due to laboratory or operations error

15. METHOD PERFORMANCE

15.1. This method was validated through single laboratory studies of accuracy and precision. Refer to the reference method for additional method performance data available.

15.2. The method detection limit (MDL) is established using the procedure described in the SOP CE-QA011, *Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantification*. Method Reporting Limits are established for this method based on MDL studies and as specified in the ALS Quality Assurance Manual.

16. POLLUTION PREVENTION AND WASTE MANAGEMENT

16.1. It is the laboratory's practice to minimize the amount of solvents, acids, and reagents used to perform this method wherever feasibly possible. Standards are prepared in volumes consistent with methodology and only the amount needed for routine laboratory use is kept on site. The threat to the environment from solvents and/or reagents used in this method can be minimized when recycled or disposed of properly.

16.2. The laboratory will comply with all Federal, State, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the ALS Environmental Health and Safety Manual.

16.3. This method uses acid. Waste acid is hazardous to the sewer system and to the environment. All acid waste must be neutralized to a pH of 2.5-12 prior to disposal down the drain. The neutralization step is considered hazardous waste treatment and must be documented on the treatment by generator record. See the ALS EH&S Manual for details.

17. TRAINING

17.1. Refer to the *SOP for ALS KELSO TRAINING PROCEDURE* for documentation of training.

17.2. Training outline

17.2.1. Review literature (see references section). Read and understand the SOP. Also review the applicable MSDS for all reagents and standards used. Following these reviews, observe the procedure as performed by an experienced analyst at least three times.

17.2.2. The next training step is to assist in the procedure under the guidance of an experienced analyst. During this period, the analyst is expected to transition from a

role of assisting, to performing the procedure with minimal oversight from an experienced analyst.

17.2.3. Perform initial precision and recovery (IPR) study as described above for water samples. Summaries of the IPR are reviewed and signed by the supervisor. Copies may be forwarded to the employee's training file. For applicable tests, IPR studies should be performed in order to be equivalent to NELAC's Initial Demonstration of Capability.

17.3. Training is documented following the *SOP for Documentation of Training*.

NOTE: When the analyst training is documented by the supervisor on internal training documentation forms, the supervisor is acknowledging that the analyst has read and understands this SOP and that adequate training has been given to the analyst to competently perform the analysis independently.

18. METHOD MODIFICATIONS

18.1. For the MERX system the SnCl volume and NH_2OH are reduced due to a smaller sample aliquot.

19. REFERENCES

19.1. *Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry,* Method 1631, Revision E; USEPA, August 2002.

20. CHANGES SINCE THE LAST REVISION

20.1. *Change Request dated 12/11/15: Section 11.1, changed calibration standard units from ng to pg (Water Run Merx System and Soil Run System).*

TABLE 1
DQOs and Acceptance Criteria

	Waters	Soils/Tissue
Method Detection Limit	See DQO Table[a]	See DQO Table[a]
Method Reporting Limit	0.5 ng/L[b]	1.0 ug/Kg
Initial Precision and Recovery		
Precision (s)	<21%	< 30%
Recovery	79-121%	70 – 130%
Matrix Spike/Matrix Spike Duplicate		
Recovery	71-125%	70-130%
Relative Percent Difference	< 24%	< 30%
Ongoing Precision and Recovery	77-123%	70 -130 %

a. Method 1631E states that the method detection limit has been determined to be 0.2 ng/L when no interferences are present.
b. Method 1631E states "the minimum level of quantitation (ML) has been established as 0.5 ng/L." The MDL and calibration can support the use of a 0.5 ng/L MRL if required by regulation or project protocols. With use of an additional calibration standard the range may be extended to 200 ng/L if necessary.

TABLE 2

Summary of Corrective Actions				
Method Reference	Control	Specification and Frequency	Acceptance Criteria	Corrective Action
1631E	ICAL	Prior to sample analysis: 5 pts + 4 bubbler blanks	% RSD ≤ 15% > 100ng/L ± 15%, < 5 ng/L ± 25%	Correct problem then repeat ICAL
1631E	OPR	Prior to and at end of run	See Table 1	If fails, correct problem and reanalyze or repeat initial calibration.
1631E	Bubbler blank	After each OPR and QCS for manual system	< 50 pg	If > 50 pg, correct and reanalyze
1631E	QCS	Analyze at start of run	See Table 1	If fails, correct problem and reanalyze.
1631E	Method/Bottle Blank	3/batch	< MRL	If target exceeds MRL, re-analyze.
1631E	Water Blank	1/Batch for manual system	< MRL	If target exceeds MRL, re-analyze.
1631E	Matrix Spike/MS Dup	Include with each analysis batch (up to 10 samples)	See Table 1	Evaluate data to determine if the there is a matrix effect or analytical error. If matrix caused, dilute & rerun. If lab error, reanalyze

ALS Standard Operating Procedure

DOCUMENT TITLE:	METALS DIGESTION
REFERENCED METHOD:	EPA 3020A
SOP ID:	MET-3020A
REVISION NUMBER:	17
EFFECTIVE DATE:	2/5/17

METALS DIGESTION EPA 3020A

ALS-KELSO

SOP ID:	MET-3020A	Rev. Number:	17	Effective Date:	2/5/17

Approved By: _____ Date: 1/31/17
Department Supervisor/Technical Director - Jeff Coronado

Approved By: _____ Date: 2/1/17
QA Manager - Carl Degner

Approved By: _____ Date: 2/1/17
Laboratory Director - Jeff Grindstaff

Issue Date: _____	Doc Control ID#: _____	Issued To: _____

ANNUAL REVIEW

SIGNATURES BELOW INDICATE NO PROCEDURAL CHANGES HAVE BEEN MADE TO THE SOP SINCE THE APPROVAL DATE ABOVE. THIS SOP IS VALID FOR TWELVE ADDITIONAL MONTHS FROM DATE OF THE LAST SIGNATURE UNLESS INACTIVATED OR REPLACED BY SUBSEQUENT REVISIONS.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

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METALS DIGESTION EPA 3020A

1. SCOPE AND APPLICATION

1.1. This procedure uses techniques described in Method 3020A for acid digestion used to prepare aqueous samples, and wastes that contain suspended solids for analysis by furnace atomic absorption spectroscopy (see SOP MET-GFAA for methods and target elements) or ICP-MS (see SOPs MET-ICPMS and MET-6020 for methods and target elements). This procedure is used to determine total metals.

1.2. In cases where there is a project-specific quality assurance plan (QAPP), the project manager identifies and communicates the QAPP-specific requirements to the laboratory. In general, project specific QAPP's supersede method specified requirements. An example of this are projects falling under DOD ELAP. QC requirements defined in the SOP *Department of Defense Projects – Laboratory Practices and Project Management* (ADM-DOD) may supersede the requirements defined in this SOP.

2. METHOD SUMMARY

2.1. Nitric acid is added to a representative aliquot of sample and refluxed in a beaker. This step is repeated until the digestate is light in color or until the color has stabilized.

2.2. After the digestate has been brought to a low volume, it is cooled and diluted to final volume containing approximately 6% (v/v) nitric acid.

3. DEFINITIONS

3.1. Batch - A batch of samples is a group of environmental samples that are prepared and/or analyzed together as a unit with the same process and personnel using the same lot(s) of reagents. It is the basic unit for analytical quality control.

3.2. Preparation Batch - A preparation batch is composed of one to twenty field samples, all of the same matrix, meeting the criteria in Section 3.3 and with a maximum time between the start of processing of the first and last samples in the batch to be 24 hours.

3.3. Sample

3.3.1. Field Sample - An environmental sample collected and delivered to the laboratory for analysis; a.k.a., client's sample.

3.3.2. Laboratory Sample - A representative portion, aliquot, or subsample of a field sample upon which laboratory analyses are made and results generated.

3.4. Quality System Matrix - The matrix of an environmental sample is distinguished by its physical and/or chemical state and by the program for which the results are

intended. The following sections describe the matrix distinctions. These matrices shall be used for purpose of batch and quality control requirements.

3.4.1. Aqueous - Any groundwater sample, surface water sample, effluent sample, and TCLP or other extract. Specifically excluded are samples of the drinking water matrix and the saline/estuarine water matrix.

3.4.2. Drinking water - Any aqueous sample that has been designated a potable or potential potable water source.

3.4.3. Saline/Estuarine water - Any aqueous sample from an ocean or estuary or other salt-water source.

3.5. Method Blank (MB) - The method blank is an artificial sample composed of analyte-free water or solid matrix and is designed to monitor the introduction of artifacts into the analytical process. The method blank is carried through the entire analytical procedure.

3.6. Laboratory Control Samples (LCS) – The LCS is an aliquot of analyte free water or analyte free solid to which known amounts target analytes are added. The LCS is prepared and analyzed in exactly the same manner as the samples. The percent recovery is compared to established limits and assists in determining whether the batch is in control.

3.7. Matrix Spike/Duplicate Matrix Spike (MS/DMS) Analysis - In the matrix spike analysis, predetermined quantities of target analytes are added to a sample matrix prior to sample preparation and analysis. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the method used for the analysis. Samples are split into duplicates, spiked, and analyzed as a MS/DMS pair. Percent recoveries are calculated for each of the analytes detected. The relative percent difference (RPD) between the duplicate spikes (or samples) is calculated and used to assess analytical precision. The concentration of the spike should be at 3- 5 times the method reporting limit or at levels specified by a project analysis plan.

3.8. Laboratory Duplicates (DUP) – Duplicates are additional replicates of samples that are subjected to the same preparation and analytical scheme as the original sample. The relative percent difference (RPD) between the sample and its duplicate is calculated and used to assess analytical precision.

3.9. Standard Reference Material (SRM) – A material with specific certification criteria and is issued with a certificate or certificate of analysis that reports the results of its characterizations and provides information regarding the appropriate use(s) of the material. An SRM is prepared and used for three main purposes: (1) to help develop accurate methods of analysis; (2) to calibrate measurement systems used to facilitate exchange of goods, institute quality control, determine performance characteristics, or measure a property at the state-of-the-art limit; and (3) to ensure the long-term adequacy and integrity of measurement quality assurance program.

3.10. Critical-volume measurement: Any measurement of volume which has a direct impact on the quantification of target parameters. Examples are: Measurement of

standards, sample aliquots, QC standards and spiking solutions, measurement of sample volume, final extract or digestate volume.

3.11. Non-Volumetric Labware: Any container used for measuring initial sample volume or final extract or digestate volume. Examples of this are the centrifuge tubes used to bring metals digestions to final volume and disposable serological pipets.

3.12. Manufacturer Lot: Labware with unique manufacturer identification referred to as a lot number.

4. INTERFERENCES

4.1. Refer to the determinative method for a discussion of interferences.

5. SAFETY

5.1. All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this procedure. This includes the use of personnel protective equipment, such as, safety glasses, lab coat and the correct gloves.

5.2. Chemicals, reagents and standards must be handled as described in the ALS safety policies, approved methods and in SDSs where available. Refer to the ALS Chemical Hygiene Plan and the appropriate SDS prior to beginning this method.

5.3. Hydrochloric and/or Nitric Acid are used in this method. These acids are extremely corrosive and care must be taken while handling them. A face shield should be used while pouring acids. And safety glasses should be worn while working with the solutions. Lab coat and gloves should always be worn while working with these solutions.

6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION AND STORAGE

6.1. Samples are typically collected in plastic containers. Aqueous samples are preserved with nitric acid (pH<2), then refrigerated at 4 ± 2°C from receipt until analysis. Samples acidified in the laboratory must be held for 24 hours, and then verified to be at pH < 2 prior to sample for analysis. The pH of samples preserved by the laboratory must be checked and recorded in the sample specific comment field of the digestion bench sheet. If the pH still remains ≥2 check with the digestion lab supervisor for corrective action.

6.2. Metals holding time is six months from sample collection until analysis.

7. STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

7.1. Reagent grade chemicals shall be used in all tests. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lowering the accuracy of the determination. The preparation for all laboratory prepared reagents and solutions must be documented in a

laboratory logbook. Refer to the SOP *Reagent/Standards Login and Tracking* (ADM-RTL) for the complete procedure and documentation requirements.

7.2. Reagent water: ASTM Type I water (resistivity ≥18 MΩ-cm, conductivity ≤0.056 uS/cm).

7.3. All stocks, working solutions and sample dilutions should be prepared using deionized water (DI) conforming to ASTM Type I reagent water. For more information on reagent water generation, refer to the related SOP, Operation and Maintenance of Laboratory Reagent Water Systems.

7.4. Concentrated Nitric Acid: J.T. Baker "Instra-analyzed", Trace Metals Grade.

7.5. Standards

7.5.1. Stock standard solutions may be purchased from a number of vendors. All reference standards, where possible, must be traceable to SI units or NIST certified reference materials. The vendor-assigned expiration date is used.

7.5.2. Metals spiking solutions: Four spiking solutions are needed to prepare the matrix spike standards: k-met 1/100 QCP-CICV-1; k-met 5 µg/mL Sb; k-met 1/100 QCP-CIV3; k-met Mo/U 10 ppm.

7.5.3. Follow the formulations laid out on the ICP-MS LCSW and Spiking Solutions Form (see Table A). These solutions are prepared in acid rinsed Class A volumetric flasks using purchased custom mixed standards or 1000 ppm single analyte standards. Aliquots are made using acid rinsed Class A volumetric pipettes of the appropriate size.

7.5.4. k-met 1/100 QCP - CICV-1.: Fill a 500 mL volumetric flask approximately half full with reagent water. Next add 5.0 mL of QCP-CICV-1, dilute with reagent water mix thoroughly and transfer to a 500 mL Teflon bottle for storage. The solution expiration date is determined by the earliest expiration date of any single component in the solution.

7.5.5. k-met 1/100 QCP - CICV-3: Fill a 500 mL volumetric flask approximately half full with reagent water. Next add 5.0 mL of QCP-CICV-3 dilute with reagent water mix thoroughly and transfer to a 500 mL Teflon bottle for storage. The solution expiration date is determined by the earliest expiration date of any single component in the solution.

7.5.6. k-met 5 µg/mL Sb, fill a 500 mL volumetric flask approximately half full with reagent water. Next add 2.5 mL of 1000 ppm Sb, dilute with reagent water mix thoroughly and transfer to a 500 mL Teflon bottle for storage. The solution expiration date is determined by the earliest expiration date of any single component in the solution.

7.5.7. k-met Mo/U 10 ppm. Fill a 200 mL volumetric flask approximately half full with reagent water, add 2.0 mL of nitric acid and mix. Next add 2.0 mL of 1000 ppm Uranium, Molybdenum dilute with reagent water mix thoroughly and transfer to a 200 mL Teflon bottle for storage. The solution expiration

date is determined by the earliest expiration date of any single component in the solution.

7.5.8. Alternate 200.8 Solution: Fill a 100 mL volumetric flask approximately half full with reagent water, add 2.0 mL of Ultrex nitric acid and a trace amount of HF and mix. Add 10 mL of alternate 200.8 solution and dilute with reagent water and mix thoroughly and transfer to a 125 mL Teflon bottle for storage. The solution expiration date is determined by the earliest expiration date of any single component in the solution.

8. APPARATUS AND EQUIPMENT

8.1. Borosilicate glass beakers, 150 mL.

8.2. Borosilicate watch glasses.

8.3. Hot Plates: "Thermolyne Cimerac 3", calibrated to maintain 95°C ± 2°C.

8.4. 50 mL Evergreen® disposable (or equivalent) Centrifuge Tubes.

8.4.1. Evergreen® Centrifuge tubes are used for "critical volume measurement" (initial and/or final digestate volumes), therefore, an Accuracy and Precision verification check must be performed with each new vendor and with each new Manufacturer's Lot prior to use. For pre-testing, water is filled to the 50 mL mark and the water's mass is gravimetrically determined. Pre-testing is also performed for the 25 mL volume by filling water to the 25 mL mark and the water's mass is also gravimetrically determined. Refer to the SOP for *Checking Volumetric Labware* (ADM-VOLWARE), for further detailed instructions. Performance data must meet the Accuracy and Precision requirements specified for non-Volumetric labware in the referenced SOP.

8.4.2. A logbook to document the raw measurements for accuracy and precision must include the labware I.D. or lot number of the centrifuge tube, the initials of the person performing the calibration, the date of calibration, and the accuracy and precision data. The logbook must be reviewed and initialed by the Department Supervisor on a quarterly basis.

8.4.3. An Excel spreadsheet located at: G/QA/QA_Forms/Volumetric labware, can be used to record and calculate the results. Once verification is complete, a copy of the spreadsheet is printed and inserted into the laboratory logbook. Data will be maintained following ALS data entry and document control procedures. See SOP *Making Entries onto Analytical Records* (CE-QA007).

9. PREVENTIVE MAINTENANCE

9.1. All maintenance activities are recorded in a maintenance logbook. Pertinent information must be in the logbook. Maintenance entries should include date, symptom of problem, corrective actions, and description of maintenance, date, and name. The log should contain a reference to return to analytical control.

9.2. Maintenance for this procedure is generally limited to glassware cleaning, pipette monitoring, and hot plate calibration. Procedures for glassware washing are described in the SOP for *Metals Laboratory Glassware Cleaning* (MET-GC). Procedures for pipette monitoring are given in the SOP *Checking Volumetric Labware* (ADM-VOLWARE).

9.3. Each hotplate is uniquely identified and temperature monitored with each digestion batch. To perform the calibration, a certified thermometer is placed in a container half filled with mineral oil, which is then placed in the center of the hotplate or block digestor. A clamp is used to ensure the thermometer does not touch the bottom of the beaker. The temperature is turned to the 95˚C setting and the mineral oil is allowed to come to temperature. The analyst will verify that the hotplate gives a temperature of 95˚C ± 2˚C. If not, the thermostat is adjusted until the thermometer reads and maintains 95˚C ± 2˚C. The thermostat is then marked to clearly indicate the correct setting to be used during sample digestion. The thermostat position and thermometer reading are recorded for each unit in a logbook.

10. RESPONSIBILITIES

10.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10.2. It is the responsibility of the department supervisor/manager to document analyst training. Documenting method proficiency is also the responsibility of the department supervisor/manager.

11. PROCEDURE

11.1. Record all digestion and sample information on the applicable bench sheet.

11.2. Shake the sample to mix. Measure a 25 mL aliquot, using a 50mL centrifuge tube, into a 150mL beaker.

11.3. Add the appropriate spiking solutions directly into the designated MS and LCS samples prior to addition of water or other reagents. The amount and mix of spiking solutions are determined during the initial batch set up in LIMs. Typically this is 0.25 ml - 0.05mL appropriate spiking solution. Fill out a spiking data sheet and keep it with the digestion data sheets.

11.3.1. Pipette tips require pre-cleaning. This is done by rinsing 3 times with Ultrex HNO_3, and then 3 times with DI water. Dispose of the rinse into an acid waste container.

11.4. Add 0.750 mL of concentrated HNO_3, place beaker on hot plate at 95°C and evaporate to a low volume (5-10mL), making certain the sample does not boil and that no portion of the bottom of the beaker is allowed to go dry.

11.5. Cool the beaker, add another 0.750 mL portion of HNO, cover with a watch glass and heat so that a gentle reflux action occurs. (CAUTION: Do not allow sample to go dry. Should this happen, discard and re-prepare sample.)

11.6. Continue refluxing until digestion is complete. (Additional acid may be required). Reduce the sample volume to 5 ml. Remove the beaker and add approximately 10 ml of DI water. Continue warming on the hot plate for 10 to 15 minutes to solubilize any residue.

11.7. Remove the beaker from the hot plate and allow to cool. Quantitatively transfer the sample from the beaker to a 50mL centrifuge tube by rinsing down the sides of the beaker and the watch glass 3 times with DI water. Dilute to the 25 mL mark on the centrifuge tube with reagent water. (Note: The 50mL graduation on the Evergreen disposable centrifuge tubes are checked for accuracy on a per batch basis). If any insoluble material is present, let the material settle or centrifuge before analysis. If immediate analysis is necessary the digestates may be centrifuged to remove insoluble material.

11.8. SPLP Extraction digestion for ICP-MS.

11.8.1. Use 25% HCL Acid -rinsed 50 mL centrifuge tubes in Hot Block (Clean Room).

11.8.2. Initial Volume: 5.00 mL. Final Volume: 25 mL.

11.8.3. Method Blank (DI Water) LCS (DI Water).

11.8.4. Method Blank Extraction Blank.

11.8.5. LCS and Spikes

0.1mL ALT 200.8 Solution.
0.1mL Mo/U 10 ppm.
0.5mL 1/100 QCP-CICV-3.
0.5mL 1/100 QCP-CICV-1.
0.5mL Sb 5 ug/mL Sb.

11.8.6. After spiking the Spike and LCS samples add 1.50 mL of concentrated acid and digest for 1 hour.

11.8.7. Remove from Hot Block and let cool, add another 1.50 mL of Concentrated HNO3 acid and digest for 45 minutes.

11.8.8. Remove from the Hot Block and let cool, add 0.05mL of Concentrated HCL and digest for 15 minutes.

11.8.9. Dilute to Final Volume of 25mL with DI H20.

11.8.10. Matrix:
6% HNO3;
0.05mL HCL *

Note: *= Add 0.05mL HCL only if digesting samples from specific clients which require this addition..

12. QA/QC REQUIREMENTS

12.1. Initial Precision and Recovery Validation

12.1.1. The accuracy and precision of the procedure must be validated before analyses of samples begin, or whenever significant changes to the procedures have been made. To do this, four water samples are spiked with the LCS spike solution, then prepared and analyzed. Performance is acceptable if the % recovery and % RSD meet LCS (or method) acceptance limits.

12.2. Ongoing QC Samples required are described in the ALS-Kelso Quality Assurance Manual and in the SOP for *Sample Batches* (ADM-BATCH). Additional QC Samples may be required in project specific quality assurance plans (QAPP). General QA requirements for DoD QSM are defined in the laboratory SOP, Department of Defense Projects – Laboratory Practices and Project Management (ADM-DOD). General QC Samples are:

12.2.1. Method Blank

12.2.1.1. A method blank is extracted and analyzed with every batch of 20 (or fewer) samples to demonstrate that there are no method interferences. If the method blank shows any hits above the reporting limit, corrective action must be taken. Corrective action includes recalculation, reanalysis, system cleaning, or re-extraction and reanalysis. For some project specific needs, exceptions may be noted and method blank results above the MRL may be reported for common lab contaminants.

12.2.2. Lab Control Sample (LCS)

12.2.2.1. The laboratory control sample is composed of analyte-free water into which is spiked a number of appropriate target analytes. The LCS is designed to monitor the accuracy of the procedure. The concentration of the spike in the LCS matrix should be at 5 to 10 times the MRL or at levels specified by a project analysis plan.

12.2.2.2. Digest one laboratory control sample with each sample batch. Use the appropriate dilution of Inorganic Ventures ICV solutions for the liquid laboratory control sample (LCSW.)

12.2.3. Duplicate/Matrix Spike

12.2.3.1. Digest one Duplicate and one Matrix Spike sample with each sample matrix. Prepare one Duplicate and Spike sample per digestion batch, or per twenty samples, whichever is more frequent.

12.2.3.2. Depending on project specific requirements a matrix spike (MS) and duplicate matrix spike (DMS) may be prepared and analyzed with every batch of 20 (or fewer) samples. The MS/DMS is prepared by adding a known volume of the matrix spike solution to the sample and determining the spiked sample concentration.

12.3. The 95°C hotplate temperature must be monitored and documented on a per-batch basis. The actual measured temperature, thermometer correction factor, and corrected temperature must all be recorded.

13. DATA REDUCTION AND REPORTING

13.1. Digestion data sheets including volumes used are completed and a batch lot number is assigned and attached to the data sheet. The Manufacturer's lot numbers for the reagents used are added to the digestion data sheet.

13.1. Spiking sheets are included with the digestion bench sheet for reference.

13.2. Following primary data interpretation and calculations, all data is reviewed by a secondary analyst. Following generation of the report, the report is also reviewed. Refer to the *SOP for Laboratory Data Review Process* (ADM-DREV) for details. The person responsible for final review of the data report and/or data package should assess the overall validity and quality of the results and provide any appropriate comments and information to the Project Manager to inclusion in the report narrative.

13.3. It is the analyst's responsibility to review analytical data to ensure that all quality control requirements have been met for each analytical run. Results for QC analyses are calculated and recorded as specified in this SOP. Average, RPD, spike level and spike recovery are entered on the analytical spreadsheet for corresponding samples. All data will be initialed, dated and attached to required data quality worksheet.

14. CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

14.1. Refer to the SOP for *Non Conformance and Corrective Action* (CE-QA008) for procedures for corrective action. Personnel at all levels and positions in the laboratory are to be alert to identifying problems and nonconformities when errors, deficiencies, or out-of-control situations are detected.

14.2. Handling out-of-control or unacceptable data

14.2.1. On-the-spot corrective actions that are routinely made by analysts and result in acceptable analyses should be documented as normal operating procedures, and no specific documentation need be made other than notations in laboratory maintenance logbooks, run-logs, for example.

14.2.2. Some examples when documentation of a nonconformity is required using a Nonconformity and Corrective Action Report (NCAR):

- Quality control results outside acceptance limits for accuracy and precision.
- Method blanks or continuing calibration blanks (CCBs) with target analytes above acceptable levels.
- Sample holding time missed due to laboratory error or operations.
- Deviations from SOPs or project requirements.
- Laboratory analysis errors impacting sample or QC results.
- Miscellaneous laboratory errors (spilled sample, incorrect spiking, etc.).
- Sample preservation or handling discrepancies due to laboratory or operations error.

15. METHOD PERFORMANCE

15.1. Available method performance data is given in the reference method. In addition, this procedure was validated through single laboratory studies of accuracy and precision as in the determinative procedure.

16. POLLUTION PREVENTION AND WASTE MANAGEMENT

16.1. It is the laboratory's practice to minimize the amount of solvents, acids, and reagents used to perform this method wherever feasibly possible. Standards are prepared in volumes consistent with methodology and only the amount needed for routine laboratory use is kept on site. The threat to the environment from solvents and/or reagents used in this method can be minimized when recycled or disposed of properly.

16.2. The laboratory will comply with all Federal, State, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the ALS Chemical Hygiene Plan and ALS-Kelso Lab Waste management Plan.

16.3. Hydrochloric and/or Nitric Acid are used in this method. These acids are extremely corrosive and care must be taken while handling them. A face shield must be used while using concentrated acids. And safety glasses should be worn while working with the solutions. Lab coat and gloves should always be worn while working with these solution.

17. TRAINING

17.1. Training outline

17.1.1. Review literature (see references section). Read and understand the SOP. Also review the applicable MSDS for all reagents and standards used. Following these reviews, observe the procedure performed by an experienced analyst at least three times.

17.1.2. The next training step is to assist in the procedure under the guidance of an experienced analyst. During this period, the analyst is expected to transition from a role of assisting, to performing the procedure with minimal oversight from an experienced analyst.

17.1.3. Perform initial precision and recovery (IPR) study as described above for water samples. Summaries of the IPR are reviewed and signed by the supervisor. Copies may be forwarded to the employee's training file. For applicable tests, IPR studies should be performed in order to be equivalent to NELAC's Initial Demonstration of Capability.

17.2. Training is documented following the SOP *ALS-Kelso Training Procedure* (ADM-TRAIN).

17.2.1. When the analyst training is documented by the supervisor on internal training documentation forms, the supervisor is acknowledging that the analyst has read and understands this SOP and that adequate training has been given to the analyst to competently perform the analysis independently.

18. METHOD MODIFICATIONS

18.1. Section 11: The lab uses 25 mL of sample. All acids added to the samples are adjusted accordingly to the 25 mL final volume.

18.2. Digests are not covered with a watch glass during the evaporation process.

19. REFERENCES

19.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods. EPA SW-846, 3rd Edition, Update 1, Method 3020A, July 1992.

20. CHANGES SINCE THE LAST REVISION

20.1. Various typographical, grammatical, and format revisions.

20.2. Revised to reflect name changes to SDS and Chemical Hygiene Plan.

20.3. Section 7.5.8 - Added use of an alternate 200.8 spiking solution per procedural change request submitted 12/22/2016.

20.4. Section 11.8 - Added SPLP Extraction digestion into the SOP per procedural change request submitted 12/15/2016.

Table A

	ICP-MS LCSW AND SPIKING SOLUTIONS	
5.00mL to 500mL Dilution of Inorganics Ventures QCP-CICV-1		
	k-met 1/100 QCP-CICV-1	
Analyte	Concentration in solution (ppb)	Concentration in digest (ppb)
Al	10000	100
Ba	10000	100
Co	2500	25
Mn	2500	25
Ni	2500	25
V	2500	25
Zn	2500	25
Cu	1250	12.5
Ag	1250	12.5
Cr	1000	10
Be	250	2.5
2.50mL to 500mL Dilution of 1000ppm Sb		
	k-met 5ug/mL Sb	
Analyte	Concentration in solution (ppb)	Concentration in digest (ppb)
Sb	5000	50
5.00mL to 500mL Dilution of Inorganics Ventures QCP-CICV-3		
	k-met 1/100 QCP-CICV-3	
Analyte	Concentration in solution (ppb)	Concentration in digest (ppb)
As	5000	50
Pb	5000	50
Se	5000	50
Tl	5000	50
Cd	2500	25
2.00mL to 200mL Dilution of 1,000 ppm Mo and 1,000 ppm U		
	k-met Mo/U 10ppm	
Analyte	Concentration in solution (ppb)	Concentration in digest (ppb)
Mo	10000	20
U	10000	20

ALS Standard Operating Procedure

DOCUMENT TITLE:	METALS DIGESTION
REFERENCED METHOD:	EPA 3050B
SOP ID:	MET-3050B
REVISION NUMBER:	15
EFFECTIVE DATE:	5/29/16

ALS-Kelso SOP Annual Review Statement

SOP Code: MET-3050B
Revision: 15

An annual review of the SOP listed was completed on (date): **5-8-17**

☒ The SOP reflects current practices and requires no procedural changes.

Supervisor: Date:

☐ Revision of the SOP is needed to reflect current practices. Draft revisions are listed below.

SOP Section Number	**Description of Revision Needed**	**Date Procedure Change Implemented**	**Supervisor Initials** Indicating Approval of Revision
			.

METALS DIGESTION

ALS-KELSO

SOP ID:	MET-3050B	Rev. Number:	15	Effective Date:	5/29/16

Approved By: _____ Date: 5/11/16
Department Manager/Technical Director - Jeff Coronado

Approved By: _____ Date: 5/11/16
QA Manager - Carl Degner

Approved By: _____ Date: 5/12/16
Laboratory Director - Jeff Grindstaff

Issue Date: _____ Doc Control ID#: _____ Issued To: _____

ANNUAL REVIEW

SIGNATURES BELOW INDICATE NO PROCEDURAL CHANGES HAVE BEEN MADE TO THE SOP SINCE THE APPROVAL DATE ABOVE. THIS SOP IS VALID FOR TWELVE ADDITIONAL MONTHS FROM DATE OF THE LAST SIGNATURE UNLESS INACTIVATED OR REPLACED BY SUBSEQUENT REVISIONS.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

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METALS DIGESTION

1. SCOPE AND APPLICATION

1.1. This procedure describes Method 3050B for acid digestion of sediments, sludges, and soil samples for analysis by Flame AA (Methods 7470-Pb, 7742-Se, 7062-As), ICP-OES (Methods 6010 and 200.7), and ICP-MS (Methods 200.8 and 6020), a procedure designated for "Total Metals" analysis. This procedure is not a *total digestion* technique, but extracts "environmentally available" elements from the sample of interest.

1.2. Preparation of samples for analysis by GFAA, refer to the SOP, MET-GFAA.

1.3. In cases where there is a project-specific quality assurance plan (QAPP), the project manager identifies and communicates the QAPP-specific requirements to the laboratory. In general, project specific QAPP's supersede method specified requirements. An example of this are projects falling under DOD ELAP or project which require older versions of EPA methods (i.e. 6010B). QC requirements defined in the SOP *Department of Defense Projects – Laboratory Practices and Project Management (ADM-DOD)* may supersede the requirements defined in this SOP.

2. METHOD SUMMARY

2.1. One-gram equivalent dry weight sediment, sludge, or soil samples are digested with repeated additions of nitric acid (HNO3) and hydrogen peroxide (H2O2). For ICP-OES, flame AA, and ICP-MS analysis, hydrochloric acid (HCl) is added to the initial digestate and the sample is refluxed prior to dilution to a final volume of 100 mL.

2.2. For GFAA analysis the resultant digestate is reduced in volume while heating and then diluted to a final volume of 100 mL.

3. DEFINITIONS

3.1. **Batch** - A batch of samples is a group of environmental samples that are prepared and/or analyzed together as a unit with the same process and personnel using the same lot(s) of reagents. It is the basic unit for analytical quality control.

3.2. **Preparation Batch** - A preparation batch is composed of one to twenty field samples, all of the same matrix, and with a maximum time between the start of processing of the first and last samples in the batch to be 24 hours.

3.3. **Sample**

3.3.1. Field Sample - An environmental sample collected and delivered to the laboratory for analysis; a.k.a., client's sample.

3.3.2. Laboratory Sample - A representative portion, aliquot, or subsample of a field sample upon which laboratory analyses are made and results generated.

3.4. **Quality System Matrix** - The *matrix* of an environmental sample is distinguished by its physical and/or chemical state and by the program for which the results are intended. The following sections describe the matrix distinctions. These matrices shall be used for purpose of batch and quality control requirements.

3.4.1. Solids - Any solid sample such as soil, sediment, sludge, and other materials with >15% settleable solids.

3.5. **Laboratory Control Sample (LCS)** - A laboratory blank that has been fortified with target analyte and used to determine that the analysis is in control.

3.6. **Matrix Spike (MS)** - In the matrix spike analysis, predetermined quantities of target analytes are added to a sample matrix prior to sample preparation and analysis. The percent recovery is calculated. The MS is used to evaluate the effects of the sample matrix on the method used for the analysis. The concentration of the spike should be at three to five times the sample result or at levels specified by a project analysis plan.

3.7. **Duplicate Sample (DUP)** - A laboratory duplicate. The duplicate sample is a separate field sample aliquot that is processed in an identical manner as the sample proper. The relative percent difference between the samples is calculated and used to assess analytical precision.

3.8. **Method Blank (MB)** - The method blank is an artificial sample composed of analyte-free water or solid matrix and is designed to monitor the introduction of artifacts into the analytical process. The method blank is carried through the entire analytical procedure.

4. INTERFERENCES

4.1. Refer to the determinative method for a discussion of interferences.

5. SAFETY

5.1. All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this procedure. This includes the use of personnel protective equipment, such as, safety glasses, lab coat and the correct gloves.

5.2. Chemicals, reagents and standards must be handled as described in the ALS safety policies, approved methods and in MSDSs where available. Refer to the ALS Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

5.3. Hydrochloric and/or Nitric Acid are used in this method. These acids are extremely corrosive and care must be taken while handling them. A face shield must be used while pouring concentrated acids. And safety glasses should be worn while working with the solutions. Lab coat and gloves should always be worn while working with these solutions.

6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION AND STORAGE

6.1. Samples may be collected in plastic or glass jars. Non-aqueous samples are refrigerated at 4 ± 2°C from receipt until analysis.

6.2. The recommended holding time is 6 months from the day of sampling.

7. STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

7.1. Reagent grade chemicals shall be used in all tests. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lowering the accuracy of the determination. The preparation for all laboratory prepared reagents and solutions must be documented in a laboratory logbook. Refer to the SOP *Reagent/Standards Login and Tracking (ADM-RTL)* for the complete procedure and documentation requirements.

7.2. All stocks, working solutions and sample dilutions should be prepared using deionized water (DI) conforming to ASTM Type I reagent water. For more information on reagent water generation, refer to the related SOP, Operation and Maintenance of Laboratory Reagent Water Systems.

7.3. Reagent water: ASTM Type I water (resistivity $\geq$18 M Ωcm, conductivity $\leq$0.056 uS/cm).

7.4. Concentrated Nitric Acid: J.T. Baker "Instra-analyzed", Trace Metals Grade

7.5. Concentrated Hydrochloric Acid: EMD GR ACS

7.6. Hydrogen Peroxide (30%): EMD GR ACS

7.7. Standards

7.7.1. Stock standards may be purchased from a number of vendors. All reference standards, where possible, must be traceable to SI units or NIST certified reference materials. The vendor assigned expiration date is used.

7.7.2. Metals spiking solutions: Five spiking solutions are needed to prepare the matrix spike sample; SS1, SS2, SS3, SS4, and SS5.

7.7.3. Follow the formulations laid out on the "Metals Spike Form" (see attached Table A). These solutions are prepared in acid rinsed Class A volumetric flasks using purchased custom mixed standards or 1000 ppm single analyte standards. Aliquots are made using acid rinsed Class A volumetric pipettes of the appropriate size.

7.7.4. SS1 (Al, Ag, Ba, Be, Cd, Co, Cr, Cu, Fe, Pb, Mn, Ni, Sb, V, and Zn): Fill a 1000 mL volumetric flask approximately half full with reagent water, add 50 mL of nitric acid and mix. Next add 100 mL of the custom mixed standard (CAS-CAL-14) purchased from "Inorganic Ventures". In addition add 50 mL of 1000 ppm Antimony(use the Antimony standard that does not contain HCL.) Dilute to volume with reagent water, mix thoroughly and transfer to a 1000 mL Teflon bottle for storage. The solution expiration date is determined by the earliest expiration date of any single component in the solution.

7.7.5. SS2 (GFAA As, Cd, Cu, Pb, Se, Tl): Fill a 500 mL volumetric flask approximately half full with reagent water, add 25 mL of nitric acid and mix. Next add 2.0 mL each of 1000 ppm Arsenic, Cadmium, Copper, Lead, Selenium, and Thallium. Dilute to volume with reagent water, mix thoroughly and transfer to a 500 mL Teflon bottle for storage. The solution expiration date is determined by the earliest expiration date of any single component in the solution.

7.7.6. SS3 (As, Se, Tl, and Hg): Fill a 500 mL volumetric flask approximately half full with reagent water, add 25 mL of nitric acid and mix. Next add 50 mL each of 1000 ppm Arsenic, Selenium, and Thallium. Add 6.0mL of 1000ppm Hg. Dilute to volume with reagent water, mix thoroughly and transfer to a 500 mL Teflon bottle for storage. The solution expiration date is determined by the earliest expiration date of any single component in the solution.

7.7.7. SS4 (B, Mo): Fill a 500 mL volumetric flask approximately half full with reagent water, add 25 mL of nitric acid and mix. Next add 50 mL each of 1000 ppm Boron and Molybdenum. Dilute to volume with reagent water, mix thoroughly and transfer to a 500 mL Teflon bottle for storage. The solution's expiration date is determined by the earliest expiration date of any single component in the solution.

7.7.8. SS5 (K,Na,Mg,Ca): Fill a 200 mL volumetric flask approximately half full with reagent water add 10.0 mL of nitric acid and mix. Next add 20 mL each of 10,000 ppm Potassium, Sodium, Magnesium and Calcium. Dilute to volume with reagent water, mix thoroughly and transfer to a 250 mL Teflon bottle for storage. The solution's expiration date is determined by the earliest expiration date of any single component in the solution.

7.8. Metals reference material (ERA Priority PollutnT/CLP Inorganic Soil) for use as the laboratory control sample. The expiration date is assigned by the manufacturer.

7.9. Teflon beads, Teflon boiling chips, or other suitable blank material.

8. APPARATUS AND EQUIPMENT

8.1. 125 mL plastic cup beaker cup, calibrated at 50mL and 100mL

8.2. Borosilicate watch glasses

8.3. Block Digester, calibrated to maintain 95˚C ± 2˚C

8.4. Hot Plates: "Thermolyne Cimerac 3", calibrated to maintain 95˚C ± 2˚C

8.5. Laboratory balance, top-loader capable of reading 0.01g

8.6. Digestion tubes, 125 mL - Environmental Express. An accuracy and precision verification check must be made with each new vendor lot prior to use. Refer to the SOP for *Checking Volumetric Labware ADM-VOLWARE*, for further detailed instructions. Performance data must meet the accuracy and precision requirements specified in Table 1 (*ADM-VOLWARE*) for non-volumetric labware used for measuring initial and/or final digestate volumes.

8.7. USS # 10 sieve.

9. PREVENTIVE MAINTENANCE

9.1. All maintenance activities are recorded in a maintenance logbook. Pertinent information must be in the logbook. Maintenance entries should include date, symptom of problem,

corrective actions, and description of maintenance, date, and name. The log should contain a reference to return to analytical control.

9.2. Maintenance for this procedure is generally limited to glassware cleaning, pipet monitoring, and hot plate calibration. Procedures for glassware washing are described in the SOP for Metals Laboratory Glassware Cleaning (MET-GC). Procedures for pipet monitoring are given in the SOP for Checking Volumetric Labware, (ADM-VOLWARE).

9.3. Each hotplate or block digester is uniquely identified and the temperature is verified with each batch of samples. To perform the verification, a certified thermometer is placed in a container half filled with mineral oil, which is then placed in the center of the hotplate or block digester. The thermometer does not touch the bottom of the container. The temperature is turned to the 95˚C setting and the mineral oil is allowed to come to temperature. The analyst will verify that the hotplate gives a temperature of 95˚C ± 2˚C. If not, the thermostat is adjusted until the thermometer reads and maintains 95˚C ± 2˚C. The thermostat is then marked to clearly indicate the correct setting to be used during sample digestion (when using Hot Plates.). Each hot Block has an assigned calibrated thermometer. The Temperature and the correction factor of the assigned thermometer is recorded on the digestion bench sheet.

10. RESPONSIBILITIES

10.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10.2. It is the responsibility of the department supervisor/manager to document analyst training.

11. PROCEDURE

11.1. Record all digestion and sample information on the applicable benchsheet.

11.2. Mix the sample thoroughly to achieve homogeneity. Sieve if necessary using a USS #10 sieve.

11.3. It can be difficult to obtain a representative sample with wet or damp materials. As per Method 3050B, wet samples may be dried, crushed, and ground to reduce subsample variability, however, drying is not recommended since drying may affect the extraction of the analytes of interest in the sample.

11.4. Weigh approximately 1g of sample into a 125ml plastic beaker cup and record the weight to the nearest 0.01g. For sludge's and sediments that have high moisture content, use more sample. A plastic 10.0 mL disposable pipette is used to measure 10.0 mL of sample. The volume and weight of the pipetted sample is recorded. In cases where the sludge is very thick a 10.0 mL graduated cylinder may be used. The objective is to use about 1g of dry weight sample. For analysis of Lead by Flame AA, use about 2.5g of dry wt. sample and change the final dilution volume to 50ml. This will achieve a lower detection limit needed for

most projects. At this point add the appropriate spiking solutions directly onto the designated spike sample prior to addition of reagents.

11.5. Add 5ml reagent water and 5ml concentrated HNO_3. Place in a hot block, cover and reflux (without boiling) at 95ºC for 10 to 15 minutes. Allow the sample to cool. Add 5ml of concentrated HNO_3, cover and reflux for 30 minutes. If brown fumes are generated, indicating oxidation of the sample by HNO_3, repeat the addition of 5ml of HNO_3 and reflux over and over until no brown fumes are given off. Reduce the digestate volume to approximately 5 mL without boiling or digest for two hours maintaining a covering of solution over the bottom of the beaker at all times. If this occurs discard the digestate and begin with a new sample aliquot.

Note: The 95˚C hot block temperature must be monitored and documented on a per-batch basis. The actual measured temperature, thermometer correction factor, and corrected temperature must all be recorded.

Note: All Wisconsin samples must digest for 2 hours after generation of brown fumes has ceased.

Note: Wipe samples do not require the final two hour digestion.

11.6. Cool the sample and add 3 mL of 30% H_2O_2. Cover and heat to start the peroxide reaction. Care must be taken to ensure that losses do not occur due to excessive effervescence. Heat in the hot block until effervescence subsides. Remove from hot block and cool the beaker.

11.7. Continue to add 30% H202 in 3mL aliquots with warming until the effervescence is minimal, or until the general sample appearance is unchanged. Do not add more than 10ml of 30% H202. When the peroxide additions are complete cover the sample with a watch glass and continue heating the acid-peroxide digestate until the volume has been reduced to approximately 5 mL or heat at 95˚C plus or minus 5 degrees without boiling for 2 hours. Do not let the samples go to dryness, by ensuring the solution covers the bottom of the vessel at all times.

Note: All Wisconsin samples must digest for 2 hours after the final peroxide addition.

Note: Wipe samples do not require the final two hour digestion.

If the sample is being prepared for analysis by ICP-OES, Flame AA or ICP-MS, add 10 mL of concentrated HCl. Cover and reflux for 15 minutes at 95 ˚C. After cooling, the samples may be diluted to 100 mL with ASTM Type I reagent water (resistivity $\geq$18 MΩ-cm, conductivity $\leq$0.056 uS/cm) in a 125 mL plastic beaker cup.

Note: For method 7062 and 7742 samples, the 3050B soil digestion is modified as follows: After the final peroxide addition (i.e. before the final reduction stage) add 5.0mL of concentrated hydrochloric acid and reduce the digestate volume to less than 5.0mL, but not to dryness. After cooling, dilute the digestate to 100mL with reagent water.

11.8. Cover and reflux the Flame AA and ICP samples for 15 minutes at 95ºC. After cooling, the samples may be diluted to 100ml for ICP analysis, or 50ml for Flame AA analysis.

11.9. Particulates in the digestates that may clog the nebulizer are allowed to settle overnight, or the digestates may be centrifuged.

11.10. To improve the solubility for Antimony, Barium, Lead and Silver, the following modification of the digestion procedure may be used as directed by the client or project chemist.

11.10.1. Weigh (to the nearest 0.01g) 1.00 g of sample into a 125ml plastic cup. For sludges and sediments that have high moisture content, use more sample. The objective is to use about 1g of dry weight sample.

11.10.2. Add 2.5mL HNO_3 and 10mL HCl and cover with a watch glass. Reflux for 15 minutes.

11.10.3. Filter the digestate through Whatman No. 41or equivalent filter paper and collect in a 100mL volumetric flask. Wash the filter paper, while still in the funnel, with no more than 5mL of hot (95°) HCl, and then with 20mL of hot (95°) reagent water. Collect washing in the same volumetric flask.

11.10.4. Remove the filter and residue from the funnel, and place them back in the beaker. Add 5mL HCl, cover and heat at 95° ± 5° until the filter paper dissolves. Remove from the heat and wash the cover and sides with reagent water.

11.10.5. Filter the residue and collect the filtrate in the same 100mL flask. Allow to cool, then dilute to volume.

11.10.6. If precipitation occurs in the flask upon cooling, do not dilute to volume. Instead, add up to 10mL of HCl to dissolve the precipitate. After precipitate is dissolved, dilute to volume with water.

12. QA/QC REQUIREMENTS

12.1. Initial Precision and Recovery Validation

12.1.1. The accuracy and precision of the procedure must be validated before analyses of samples begin, or whenever significant changes to the procedures have been made. To do this, four blank matrix samples are spiked with the LCS spike solution, then prepared and analyzed.

12.2. Monitor Hot Blocks and Hotplates on a per batch basis. Report all deficiencies to the Lab Manager. Corrective action must be taken.

12.3. Digest one laboratory control sample with each batch. Weigh 1.00 g of the current lot of Environmental Resource Associates PriorityPollutant/CLP Inorganic Soil prepared reference material into a 150 mL beaker and digest as per the procedure.

12.4. Digest one preparation blank (method blank) per digestion batch, or per 20 samples whichever is more frequent. For the method blank, use Teflon beads, Teflon boiling chips, or other suitable solid blank material and follow the digestion procedures.

12.5. Digest one duplicate and one spiked sample with each sample matrix. Prepare one duplicate and spike sample per each digestion batch, or per twenty samples whichever is more

frequent. At times, specific samples will be assigned as duplicates of spikes depending on client requirements.

12.6. Soil spikes for ICP and ICP-MS are prepared by adding 2.0 mL of SS1, and 1.0 mL of SS3, SS4 and SS5 directly to the sample aliquot, prior to the addition of any water or acid. Fill out a spiking data sheet and keep it with the digestion data sheets.

12.6.1. For GFAA digestions 2.0 mL of SS2 is added to the sample aliquot designated as the matrix spike sample. The matrix spike sample is then digested as per the procedure.

13. DATA REDUCTION AND REPORTING

13.1. Digestion data sheets including weights and volumes used and reagents/acids are completed and a prep run number or batch lot number is assigned and attached to the data sheet. The lot numbers for the reagents used are added to the digestion data sheet (see Attachments).

13.2. Spiking sheets are included (See Attachments).

13.3. Data Review and Assessment

13.3.1. Refer to the *SOP for Laboratory Data Review Process* for general instructions for data review.

13.3.2. It is the supervisor's responsibility to ensure that digestions data is reviewed to ensure that all quality control requirements have been met and documentation is complete.

14. CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

14.1. Refer to the SOP for *Nonconformity and Corrective Action* (CE-QA008) for procedures for corrective action. Personnel at all levels and positions in the laboratory are to be alert to identifying problems and nonconformities when errors, deficiencies, or out-of-control situations are detected.

14.2. Handling out-of-control or unacceptable data

14.2.1. On-the-spot corrective actions that are routinely made by analysts and result in acceptable analyses should be documented as normal operating procedures, and no specific documentation need be made other than notations in laboratory maintenance logbooks, runlogs, for example.

14.2.2. Some examples when documentation of a nonconformity is required using a Nonconformity and Corrective Action Report (NCAR):

- Quality control results outside acceptance limits for accuracy and precision
- Method blanks or continuing calibration blanks (CCBs) with target analytes above acceptable levels
- Sample holding time missed due to laboratory error or operations
- Deviations from SOPs or project requirements

- Laboratory analysis errors impacting sample or QC results
- Miscellaneous laboratory errors (spilled sample, incorrect spiking, etc.)
- Sample preservation or handling discrepancies due to laboratory or operations error

15. METHOD PERFORMANCE

15.1. This method was validated through single laboratory studies of accuracy and precision. Refer to the reference method for additional method performance data available.

15.2. The method detection limit (MDL) is established using the procedure described in the SOP CE-QA011, *Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantification*. Method Reporting Limits are established for this method based on MDL studies and as specified in the ALS Quality Assurance Manual.

16. POLLUTION PREVENTION AND WASTE MANAGEMENT

16.1. It is the laboratory's practice to minimize the amount of solvents, acids, and reagents used to perform this method wherever feasibly possible. Standards are prepared in volumes consistent with methodology and only the amount needed for routine laboratory use is kept on site. The threat to the environment from solvents and/or reagents used in this method can be minimized when recycled or disposed of properly.

16.2. The laboratory will comply with all Federal, State, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the ALS Environmental Health and Safety Manual.

16.3. This method uses acid. Waste acid is hazardous to the sewer system and to the environment. All acid waste must be neutralized to a pH of 2.5-12 prior to disposal down the drain. The neutralization step is considered hazardous waste treatment and must be documented on the treatment by generator record. See the ALS EH&S Manual for details.

17. TRAINING

17.1. Training outline

17.1.1. Review literature (see references section). Read and understand the SOP. Also review the applicable MSDS for all reagents and standards used. Following these reviews, observe the procedure as performed by an experienced analyst at least three times.

17.1.2. The next training step is to assist in the procedure under the guidance of an experienced analyst. During this period, the analyst is expected to transition from a role of assisting, to performing the procedure with minimal oversight from an experienced analyst.

17.1.3. Perform initial precision and recovery (IPR) study as described above for water samples. Summaries of the IPR are reviewed and signed by the supervisor. Copies may be forwarded to the employee's training file. For applicable tests, IPR studies should be performed in order to be equivalent to NELAC's Initial Demonstration of Capability.

17.2. Training is documented following the *SOP ADM-TRAIN*.

NOTE: When the analyst training is documented by the supervisor on internal training documentation forms, the supervisor is acknowledging that the analyst has read and understands this SOP and that adequate training has been given to the analyst to competently perform the analysis independently.

18. METHOD MODIFICATIONS

18.1. The method uses 2 mL of water and 3 mL of H202. The lab does not add the 2 mL of water. 3.0 mL aliquots of 30% H202 in lieu of 1.0 mL aliquots are added subsequently.

19. REFERENCES

19.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods. EPA SW-846, 3rd Edition, Final Update III, Method 3050B, December 1996.

19.2. Table A - METALS SPIKING SOLUTIONS CONCENTRATIONS FORM

20. CHANGES SINCE THE LAST REVISION

20.1. Signature page: Updated Quality Assurance Manager.
20.2. Section 11.5: Added note regarding wipe sample digestion.
20.3. Section 11.7: Added note regarding wipe sample digestion.
20.4. Added DOD/project specific paragraph in Scope and Application.
20.5. Section 1.1: Removed reference to second sample preparation step.
20.6. Section 2.1: Included ICP-MS with ICP-OES preparation step (HCl).
20.7. Section 11: Deleted former sections 11.8 and 11.9.

TABLE A
METALS SPIKING SOLUTIONS CONCENTRATIONS FORM

Solution Name	Element	mL of 1000ppm Solution	Final Volume	Solution Conc. mg/L	Enter ml Added
K-MET SS1 *** Add after HNO3 and before cas cal -14 when making the solution	HNO3	50.0	1000ml	-	
	Al	100*	1000ml	200	
	Ag	100*	1000ml	5	
	Ba	100*	1000ml	100	
	Be	100*	1000ml	5	
	Cd	100*	1000ml	5	
	Co	100*	1000ml	50	
	Cr	100*	1000ml	20	
	Cu	100*	1000ml	25	
	Fe	100*	1000ml	100	
	Pb	100*	1000ml	50	
	Mn	100*	1000ml	50	
	Ni	100*	1000ml	50	
	Sb***	50	1000ml	50	
	V	100*	1000ml	50	
	Zn	100*	1000ml	50	
K-MET SS2	HNO3	25.0	500ml	-	
	As	2.0	500ml	4	
	Cd	2.0	500ml	4	
	Pb	2.0	500ml	4	
	Se	2.0	500ml	4	
	Tl	2.0	500ml	4	
	Cu	2.0	500ml	4	
K-MET SS3	HNO3	25.0	500ml	-	
	As	50.0	500ml	100	
	Se	50.0	500ml	100	
	Tl	50.0	500ml	100	
	Hg	6	500ml	12	
K-MET SS4	HNO3	25	500ml	-	
	B	50	500ml	100	
	Mo	50	500ml	100	
K-MET SS5	HNO3	10.0	200ml	-	
	K**	20	200ml	1000	
	Na**	20	200ml	1000	
	Mg**	20	200ml	1000	
	Ca**	20	200ml	1000	

K-MET GFLCSW	HNO3	10.0	1000ml	-	
	As, Pb, Se, Tl	5.0	1000ml	2.5	
	Cd	-	-	1.25	
	Cu	2.5	1000ml	2.5	
K-MET QCP-CICV-1	Ca, Mg, Na, K	no dilution	-	2500	
	Al, Ba	no dilution	-	1000	
	Fe	no dilution	-	500	
	Co, Mn, Ni, V, Zn	no dilution	-	250	
	Cu, Ag	no dilution	-	125	
	Cr	no dilution	-	100	
	Be	no dilution	-	25	
K-MET QCP-CICV-2	Sb	no dilution	-	500	
K-MET QCP-CICV-3	As, Pb, Se, Tl	no dilution	-	500	
	Cd	no dilution	-	250	

* Denotes volume of mixed stock standard.
** Denotes 10,000 ppm individual stock standards.

ALS Standard Operating Procedure

DOCUMENT TITLE:	DETERMINATION OF METALS AND TRACE ELEMENTS BY INDUCTIVELY COUPLED PLASMA-MASS SPECTROMETRY (METHOD 6020)
REFERENCED METHOD:	EPA 6020, 6020A
SOP ID:	MET-6020
REVISION NUMBER:	17
EFFECTIVE DATE:	1/15/2016

ALS-Kelso SOP Annual Review Statement

SOP Code: MET-6020
Revision: 17

An annual review of the SOP listed was completed on (date): **2/23/17**

☒ The SOP reflects current practices and requires no procedural changes.

Supervisor: RRM Date: 3/3/17

☐ Revision of the SOP is needed to reflect current practices. Draft revisions are listed below.

SOP Section Number	**Description of Revision Needed**	**Date Procedure Change Implemented**	**Supervisor Initials** Indicating Approval of Revision

DETERMINATION OF METALS AND TRACE ELEMENTS BY INDUCTIVELY COUPLED PLASMA-MASS SPECTROMETRY (METHOD 6020; 6020A)

)

ALS-KELSO

SOP ID:	MET-6020	Rev. Number:	17	Effective Date:	1/15/2016

Approved By: _____ Date: 1/8/16
Department Manager/Technical Director – Jeff Coronado

Approved By: _____ Date: 1/8/16
QA Manager – Carl Degner

Approved By: _____ Date: 1/8/16
Laboratory Director – Jeff Grindstaff

Issue Date: _____ Doc Control ID#: _____ Issued To: _____

ANNUAL REVIEW

SIGNATURES BELOW INDICATE NO PROCEDURAL CHANGES HAVE BEEN MADE TO THE SOP SINCE THE APPROVAL DATE ABOVE. THIS SOP IS VALID FOR TWELVE ADDITIONAL MONTHS FROM DATE OF THE LAST SIGNATURE UNLESS INACTIVATED OR REPLACED BY SUBSEQUENT REVISIONS.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

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DETERMINATION OF METALS AND TRACE ELEMENTS BY INDUCTIVELY COUPLED PLASMA-MASS SPECTROMETRY (METHOD 6020)

1. SCOPE AND APPLICATION

1.1. This procedure is used to determine the concentrations of certain elements in water, soil, tissues, aqueous and non-aqueous wastes, and sediment samples using EPA Method 6020 or 6020A. Table 1 indicates analytes that are typically determined by this procedure and lists the standard Method Reporting Limits (MRLs) for each analyte in water and soil. Project-specific MRLs may apply, and if lower than standard MRLs, it is demonstrated through method detection limit determinations and analysis of MRL standards that the MRL is achievable. Method Detection Limits (MDLs) that have been achieved are listed in Table 1. These may change as new studies are performed.

1.2. The complexity of the technique generally requires outside study of appropriate literature as well as specialized training by a qualified spectroscopist. The scope of this document does not allow for the in-depth descriptions of the relevant spectroscopic principles required for gaining a complete level of competence in this scientific discipline.

1.3. In cases where there is a project-specific quality assurance plan (QAPP), the project manager identifies and communicates the QAPP-specific requirements to the laboratory. In general, project specific QAPP's supersede method specified requirements. An example of this are projects falling under DOD ELAP. QC requirements defined in the SOP *Department of Defense Projects – Laboratory Practices and Project Management (ADM-DOD/ADM-DOD5)* may supersede the requirements defined in this SOP.

2. METHOD SUMMARY

2.1. Prior to analysis, samples must be digested using appropriate sample preparation methods. The digestate is analyzed for the elements of interest using ICP-mass spectrometry (ICP-MS).

2.2. Methods 6020 and 6020A describe the multi-elemental determination of analytes by ICP-MS. The method measures ions produced by a radio-frequency inductively coupled plasma. Analyte species originating in a liquid are nebulized and the resulting aerosol transported by argon gas into the plasma torch. The ions produced are entrained in the plasma gas and introduced, by means of an interface, into a mass spectrometer. The ions produced in the plasma are sorted according to their mass-to-charge ratios and quantified with a channel electron multiplier. Interferences must be assessed and valid corrections applied or the data flagged to indicate problems. Interference correction must include compensation for background ions contributed by the plasma gas, reagents, and constituents of the sample matrix.

3. DEFINITIONS

3.1. **Batch** - A batch of samples is a group of environmental samples that are prepared and/or analyzed together as a unit with the same process and personnel using the same lot(s) of reagents. It is the basic unit for analytical quality control.

3.1.1. Preparation Batch - A preparation batch is composed of one to twenty field samples, all of the same matrix, and with a maximum time between the start of processing of the first and last samples in the batch to be 24 hours.

3.1.2. Analysis Batch - Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration (initial or continuing verification) followed by sample extracts interspersed with calibration standards (CCBs, CCVs, etc.) The sequence ends when the set of samples has been analyzed or when qualitative and/or quantitative QC criteria indicate an out-of-control situation.

3.2. **Sample**

3.2.1. Field Sample - An environmental sample collected and delivered to the laboratory for analysis; a.k.a., client's sample.

3.2.2. Laboratory Sample - A representative portion, aliquot, or subsample of a field sample upon which laboratory analyses are made and results generated.

3.3. **Quality System Matrix** - The *matrix* of an environmental sample is distinguished by its physical and/or chemical state and by the program for which the results are intended. The following sections describe the matrix distinctions. These matrices shall be used for purpose of batch and quality control requirements.

3.3.1. Aqueous - Any groundwater sample, surface water sample, effluent sample, and TCLP or other extract. Specifically excluded are samples of the drinking water matrix and the saline/estuarine water matrix.

3.3.2. Drinking water - Any aqueous sample that has been designated a potable or potential potable water source.

3.3.3. Saline/Estuarine water - Any aqueous sample from an ocean or estuary or other salt-water source.

3.3.4. Nonaqueous Liquid - Any organic liquid with <15% settleable solids.

3.3.5. Animal tissue - Any tissue sample of an animal, invertebrate, marine organism, or other origin; such as fish tissue/organs, shellfish, worms, or animal material.

3.3.6. Solids - Any solid sample such as soil, sediment, sludge, and other materials with >15% settleable solids.

3.3.7. Chemical waste - Any sample of a product or by-product of an industrial process that results in a matrix not described in one of the matrices in Sections 3.4.1 through 3.4.6. These can be such matrices as non-aqueous liquids, solvents, oil, etc.

3.3.8. Miscellaneous matrices – Samples of any composition not listed in 3.4.1 – 3.4.7. These can be such matrices as plant material, paper/paperboard, wood, auto fluff, mechanical parts, filters, wipes, etc. Such samples shall be batched/grouped according to their specific matrix.

3.4. Matrix Spike/Duplicate Matrix Spike (MS/DMS) Analysis - In the matrix spike analysis, predetermined quantities of target analytes are added to a sample matrix prior to sample preparation and analysis. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the method used for the analysis. Duplicate samples are spiked, and analyzed as a MS/DMS pair. Percent recoveries are calculated for each of the analytes detected. The relative percent difference (RPD) between the duplicate spikes (or samples) is calculated and used to assess analytical precision. The concentration of the spike should be at the midpoint of the calibration range or at levels specified by a project analysis plan.

3.5. Laboratory Duplicates (DUP) – Duplicates are additional replicates of samples that are subjected to the same preparation and analytical scheme as the original sample. The relative percent difference (RPD) between the sample and its duplicate is calculated and used to assess analytical precision.

3.6. Surrogate - Surrogates are organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. The purpose of the surrogates is to evaluate the preparation and analysis of samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to extraction and analysis. Percent recoveries are calculated for each surrogate.

3.7. Method Blank (MB) - The method blank is an artificial sample composed of analyte-free water or solid matrix and is designed to monitor the introduction of artifacts into the analytical process. The method blank is carried through the entire analytical procedure.

3.8. Laboratory Control Samples (LCS) – The LCS is an aliquot of analyte free water or analyte free solid to which known amounts target analytes are added. The LCS is prepared and analyzed in exactly the same manner as the samples. The percent recovery is compared to established limits and assists in determining whether the batch is in control.

3.9. Independent Verification Standard (ICV) - A pre-mixed, purchased, second-source standard analyzed after the calibration curve. This is used to verify the validity of the initial calibration standards

3.10. Continuing Calibration Verification Standard (CCV) - A mid-level standard analyzed at specified intervals. Used to verify that the initial calibration curve is still valid for quantitative purposes.

3.11. Duplicates and Duplicate Matrix Spikes are additional replicates of samples that are subjected to the same preparation and analytical scheme as the original sample. Depending on the method of analysis, either a duplicate analysis (and/or a matrix spiked sample) or a matrix spiked sample and duplicate matrix spiked sample (MS/DMS) are analyzed.

3.12. Standard Reference Material (SRM) – A material with specific certification criteria and is issued with a certificate or certificate of analysis that reports the results of its characterizations and provides information regarding the appropriate use(s) of the material. An SRM is prepared and used for three main purposes: (1) to help develop accurate methods of analysis; (2) to calibrate measurement systems used to facilitate exchange of goods, institute quality control, determine performance characteristics, or measure a property at the state-of-the-art limit; and (3) to ensure the long-term adequacy and integrity of measurement quality assurance programs.

4. INTERFERENCES

4.1. Isobaric elemental interferences in ICP-MS are caused by isotopes of different elements forming atomic ions with the same nominal mass-to-charge ratio (m/z). A data system must be used to correct for these interferences. This involves determining the signal for another isotope of the interfering element and subtracting the appropriate signal from the analyte isotope signal. Attention should be given to circumstances where very high ion currents at adjacent masses may contribute to ion signals at the mass of interest. Matrices exhibiting a significant problem of this type may require resolution improvement, matrix separation, or analysis using another isotope.

4.2. Isobaric molecular and doubly-charged ion interferences in ICP-MS are caused by ions consisting of more than one atom or charge, respectively. Most isobaric interferences that could affect ICP-MS determinations have been identified in the literature. Refer to Method 6020/A for further discussion.

5. SAFETY

5.1. All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this procedure. This includes the use of personnel protective equipment, such as, safety glasses, lab coat and the correct gloves.

5.2. Chemicals, reagents and standards must be handled as described in the ALS safety policies, approved methods and in MSDSs where available. Refer to the ALS Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

5.3. Hydrochloric and/or Nitric Acid are used in this method. These acids are extremely corrosive and care must be taken while handling them. A face shield should be used while pouring acids. And safety glasses should be worn while working with the solutions. Lab coat and gloves should always be worn while working with these solutions.

5.4. High Voltage - The RF generator supplies up to 2000 watts to maintain an ICP. The power is transferred through the load coil located in the torch box. Contact with the load coil while generator is in operation will likely result in death. When performing maintenance on the RF generator, appropriate grounding of all HV capacitors must be performed as per manufacturer.

5.5. UV Light - The plasma is an intense source of UV emission, and must not be viewed with the naked eye. Protective lenses are in place on the instrument. Glasses with special protective lenses are available when direct viewing of the plasma is necessary.

6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION AND STORAGE

6.1. Aqueous samples are typically collected in plastic containers. Aqueous samples are preserved with nitric acid (pH<2), then refrigerated at 4 ± 2°C from receipt until digestion. Soil or solid samples may be collected in plastic or glass jars. Non-aqueous samples are refrigerated at 4 ± 2°C from receipt until digestion.

6.2. Samples are prepared via procedures in SOPs MET-DIG, MET-3020A, or MET-3050 depending on matrix and project specifications.

6.3. Digestates are stored in the appropriate volumetric containers. Following analysis, digestates are stored until all results have been reviewed. Digestates are neutralized prior to disposal through the sewer system, 2 weeks after data is reviewed.

7. STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

7.1. All standards are prepared from NIST traceable standards. The expiration dates are assigned according to the EPA method and the vendor's assigned expiration dates. For example, working ICS solutions are prepared weekly in accordance with Method 6020, Section 5.6.1.

7.1.1. 1000 ppm Single Element Stock Standard Solutions: Each stock standard is store at room temperature on shelves located in room 113 of the metals lab. The manufacturer, lot number, and expiration date of each stock standard is recorded in a bound logbook also located in room 113. Additionally each stock standard is given a unique, identifying name.

7.1.2. Intermediate Standard Solutions: Intermediate mixed stock solutions are made from the individual stock standards described above. The individual component of each mixed solution is recorded in a bound logbook located in the ICP-MS laboratory and mixed solution is given a unique, identifying name. The expiration date for the intermediate standard is the earlier of any one of its stock components.

7.1.3. Calibration Standards: Calibration standards are made fresh daily from the intermediate standard solutions. Each individual intermediate standard used in the calibration standard is recorded in a bound logbook located in the ICP-MS laboratory, and the calibration standard solution is given a unique, identifying name. The calibration standards unique name is used on the raw data to link the data to the subsequent prepared standards and ultimately the original purchased stock standard.

7.2. Standards Preparation

7.2.1. Expiration of all standard solutions defaults to the earliest expiration date of an individual component unless otherwise specified.

7.2.2. Calibration Standards

The calibration standard is prepared from two intermediate stock solutions. These solutions are prepared in acid rinsed 1000 mL Class A volumetric flasks following the formulations laid out on the attached example standard sheet (see Attachments). The working calibration standard is made daily by aliquoting 2.5 mL of each of the intermediate solutions in to a 100 mL Class A volumetric flask and diluting to volume with 1% HNO3. This standard is also used as the Continuing Calibration Verification (CCV).

7.2.3. Initial Calibration Verification (ICV)

7.2.3.1. The ICV intermediate stock solution is prepared in an acid rinsed 100 mL Class A volumetric flask. The solution is prepared by adding 2.0 mL of Inorganic Ventures QCP-CICV-1, 1.0 mL each of QCP-CICV-2 and QCP-CICV-3, 0.5 mL of 1000 ppm Molybdenum stock solution, 0.5 mL of 1000 ppm Uranium stock solution, and 0.5mL of 1000ppm B, Bi, Sr, Ti solution and diluting to volume with 1% HNO3.

7.2.3.2. The working ICV solution is prepared by aliquoting 0.5 mL of the mixed ICV intermediate solution into an acid rinsed 100 mL Class A volumetric flask and diluting to volume with 1% HNO3.

NOTE: The ICV solution is not at the midpoint of the linear range which may be as high as 1000 µg/L for some elements. The ICV solution used is a premixed standard purchased from Inorganic Ventures and contains the elements of interest between 2.5 and 100 µg/L. This solution provides calibration confirmation at more representative levels, given that most ICP-MS analyses are quantifying analytes in the low-ppb to sub-ppb range.

7.2.4. Interference Check Solutions (ICSA and ICSAB)

7.2.4.1. The ICSA is prepared in an acid rinsed 50 mL Class B volumetric flask by aliquoting 1.0 mL of Elements ICSAm (CS-CAK02) and 0.250 mL of 10 ppm molybdenum solutions and diluting to volume with 1% HNO3.

7.2.4.2. The ICSAB is prepared in an acid rinsed 50 mL Class B volumetric flask by aliquoting 1.0 mL of Elements ICSAm (CS-CAK02), 0.125 mL of Inorganic Ventures 6020ICS-9B, and 0.250 mL of 10 ppm Molybdenum solutions and diluting to volume with 1% HNO3.

7.2.5. Post-digestion spikes are performed by adding appropriate amounts of the calibration intermediate solutions to aliquots of the sample digestate. The volumes of each standard used vary based on the native concentrations found in the field samples. Refer to the post-digestion spike in Section 12 for details.

7.2.6. Refer to the appropriate digestion SOP for details of LCSW and matrix spike solution composition and preparation.

7.2.7. Tuning / Mass Calibration Solution

7.2.7.1. A 1ppm intermediate solution containing Be, Bi, Ce, Co, In, Li, Pb, Mg, and U is prepared by adding 1.0 mL of each from 1000 ppm stock standards to an acid rinsed 1000 mL volumetric flask and diluting to volume with 1% nitric acid. The expiration date for the intermediate solution is the earliest of any one of its stock components.

7.2.7.2. The working solution is prepared in three ways:

- For the Agilent: a 1.0 ppb tune/mass calibration solution is prepared by adding 1.0 mL of intermediate solution to an acid rinsed 1000 mL volumetric flask and diluting to volume with 1% nitric acid.

- For the X-Series (K-ICP-MS-03) instrument a 5.0 ppb tune/mass calibration solution is prepared by adding 5.0 mL of intermediate solution to an acid rinsed 1000 mL volumetric flask and diluting to volume with 1% nitric acid.
- For the NexION (K-ICP-MS-04) instrument a 2.0 ppb tune/mass calibration solution is prepared by adding 2.0 mL of intermediate solution to an acid rinsed 1000 mL volumetric flask and diluting to volume with 1% nitric acid.
- The expiration date for this solution is taken from the intermediate stock above.

7.3. Internal Standards Stock Solution – Prepare solutions by adding appropriate amounts of each 1000 ppm single element stock solution to a acid rinsed 1000 mL volumetric flask and diluting to volume with 1% nitric. Use this solution for addition to blanks, calibration standards and samples at a ratio of 0.5 mL of internal standard to 100 mL of solution, or dilute by an appropriate amount using 1% (v/v) nitric acid, if the internal standards are being added by peristaltic pump. The typical solutions are:

- XSeries instrument: 50ppb Li; 25ppb Sc, Ga, Y; 10ppb Rh, In, Lu, Tm, Th.
- Agilent instrument: 2ppm Li, Sc, Y, Ga, Ge, Ce, Tm, In, Lu, Th
- NexION instrument: 30ppb In, Tm, Lu, Th; 60ppb Li, Rh, Au; 75ppb Sc; 100ppb Ga,Y; 500ppb Ge

7.4. Additional Reagents

7.4.1. Reagent water, ASTM Type II.

7.4.2. All stocks, working solutions and sample dilutions should be prepared using deionized water (DI) conforming to ASTM Type I or ASTM Type II reagent water. For more information on reagent water generation, refer to the related SOP, Operation and Maintenance of Laboratory Reagent Water Systems.

7.4.3. "OmniTrace Ultra" Concentrated Nitric Acid (EM Science # NX0408-2).

7.4.4. Argon (Airgas Industrial Grade – 99.999% pure, bulk delivered).

8. APPARATUS AND EQUIPMENT

8.1. ICP/MS instruments:

8.1.1.

Instrument:	Thermo Electron X-Series
Nebulizer:	Conikal
Spray Chamber:	VG Peltier-cooled
Cones:	Nickel Sampler (1.0 mm orifice)
	Nickel Skimmer (0.75 mm orifice)

8.1.2.

Instrument:	NexION 300D
Nebulizer:	PFA-ST MIcroflow
Spray Chamber:	Cyclonic, Peltier-cooled
Cones:	Nickel Sampler (1.0 mm orifice)
	Nickel Skimmer (0.75 mm orifice)

8.1.3. Instrument: Agilent 7700
Nebulizer: MicroMist
Spray Chamber: Double Pass quartz spray chamber
Cones: Nickel Sampler (1.0 mm orifice)
Nickel Skimmer (0.75 mm orifice)

9. PREVENTIVE MAINTENANCE

9.1. All maintenance is documented in the instrument logbook. ALS/Kelso maintains a service contract with the instrument manufacturer that allows for an unlimited number of service calls and full reimbursement of all parts and labor.

9.2. Most routine maintenance and troubleshooting is performed by ALS staff. Preventive maintenance activities listed below should be performed when needed as determined by instrument performance (i.e. stability, sensitivity, etc.) or by visual inspection. Other maintenance or repairs may, or may not require factory service, depending on the nature of the task.

- cone removal and cleaning
- removal and cleaning of ICP glassware and fittings
- checking and cleaning RF contact strips
- checking air filters and cleaning if necessary
- checking the oil mist filters and cleaning if necessary
- checking the rotary pump oil and adding or changing if necessary
- removal and cleaning of extraction lens
- removal and cleaning of ion lens stack
- replace the electron multiplier as necessary

10. RESPONSIBILITIES

10.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10.2. It is the responsibility of the department supervisor/manager to document analyst training. Documenting method proficiency, as described in the SOP for Documentation of Training, is also the responsibility of the department supervisor/manager.

11. PROCEDURE

11.1. Refer to method 6020 (or 6020A) and the instrument manuals for detailed instruction on implementation of the following daily procedures preceding an analytical run.

11.2. The following parameters are monitored to assure awareness of changes in the instrumentation that serve as signals that optimum performance is not being achieved, or as indicators of the physical condition of certain consumable components (i.e. EMT and cones).

11.2.1. Multiplier Voltages

11.2.2. Gas Flows - Coolant Ar

11.2.3. The nebulizer and auxiliary flows are adjusted later as part of the optimizing procedure.

11.3. Optimization

11.3.1. Gas Flows

11.3.1.1. Allow a period of not less than 30 minutes for the instrument to warm up.

11.3.1.2. Aspirate a mixed tune solution into the plasma and monitor the instrument output signal at mass 115 on the ratemeter. Adjust the nebulizer and auxiliary flows to obtain maximum signal. Adjust the tension screw on the peristaltic pump to obtain minimum noise in the analytical signal. Record flow rates and note any large variances.

Note: Significant differences in flow rates will be observed for different torches and cones.

11.3.2. Tuning

11.3.2.1. Ion Lens Setting - While monitoring the output signal of a mixed tune solution at mass 115 on the ratemeter, adjust the ion lenses to obtain maximum sensitivity. Refer to the instrument manual for details on performing the adjustments.

11.3.2.2. Mass Calibration - Aspirate the tune / mass calibration solution described in section 7.2 and perform the mass calibration using the instrument's Mass Calibration program. (Refer to the instrument manual for details pertaining to the mass calibration procedure.) The acceptance criteria for the mass calibration is <0.1 amu from the true value. If the mass calibration fails criteria re-tune the instrument and perform the mass calibration procedure again.

11.3.2.3. Resolution Check - Using the spectra created during the mass calibration procedure; perform the resolution check to assure the resolution is less than 0.9 AMU at 5% peak height. If the resolution does not pass criteria adjust the instrument's resolution settings, run a new scan of the mass calibration solution and recheck.

11.3.2.4. Stability Check - Using the tune / mass calibration solution, perform a short-term stability check as per EPA Method 6020 or 6020A. The relative standard standard deviations of five scans for each element in the tune solution must

be < 5%. If the test does not pass criteria determine the cause (i.e. dirty cones, improper tune, etc.) correct the problem and re-run the test.

11.4. Analytical Run

11.4.1. Calibrate the instrument using a calibration blank (Standard 0), composed of reagent water and 1% nitric acid, and the working calibration standard (8.2.2). The masses typically monitored and those used for quantification are listed in Table 2. These masses are set as defaults in the instrument's analytical procedures. To begin select the correct method. Nebulize Standard 0 (Blank) into the plasma. Allow 1-2 minutes for system to equilibrate prior to establishing baseline. Follow directions on computer screen to perform standardization. Nebulize the working calibration standard into the plasma. The operator must sign and date the first page of standardization.

11.4.2. After the first CCB and before the ICS standards a CRA (MRL / LLICV / LLCCV) standard is analyzed. Method 6020 requires the detection to be > the MDL but < 2x the MRL. For 6020A, the criteria are 70-130% recovery. For DoD projects, the CRA criteria are 80-120%.

Note: For 6020A the LLCCV must also be analyzed at the end on the analytical run sequence.

11.4.3. Perform the analysis in the order listed below. A daily run log of all samples analyzed is maintained.

Initial Calibration Verification (ICV)
Continuing Calibration Verification (CCV)
Initial Calibration Blank (ICB)
Continuing Calibration Blank (CCB)
CRA (MRL / LLICV / LLCCV)
ICSA
ICSAB
Analyze 10 Samples
CCV
CCB
Analyze 10 Samples
CCV
CCB

Repeat sequence as required to complete analytical run, analyzing CCVs/CCBs every 10 analyses and at the end of the run.

12. QA/QC REQUIREMENTS

12.1. Initial Precision and Recovery Validation

The accuracy and precision of the procedure must be validated before analysis of samples begins, or whenever significant changes to the procedures have been made. To do this, four LCS aliquots are prepared and analyzed. The average percent recovery of for each analyte must be 85-115% (for water, and within the LCS limits for soils) and the RSD <20%.

12.2. Method Detection Limits

12.2.1. A method detection limit (MDL) study must be undertaken before analysis of samples can begin. To establish detection limits that are precise and accurate, the analyst must perform the following procedure. Spike a minimum of seven blank matrices at a level near or below the MRL. Follow the procedures starting in Section 11 to analyze the samples. Refer to CE-QA011, *Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantification* details of performing the MDL study.

12.2.2. Calculate the average concentration found (x) and the standard deviation of the concentrations for each analyte. Calculate the MDL for each analyte using the correct T value for the number of replicates. MDL's must be verified annually or whenever there is a significant change in the background or instrument response.

12.3. For method 6020A, an LLQC sample (a CRA that is carried through the digestion) must be analyzed to verify accuracy at the MRL. The recovery must be 70-130%.

12.4. Instrument Detection Limits (IDLs) and linear ranges studies are performed quarterly. These will be calculated and made available to the ICP-MS operator. Linear range studies determine the Linear Dynamic Range (LDR) of the each instrument by analysis of a high concentration standard with results with ± 10% of the expected value. For non-DoD projects samples may be quantified between the MRL and 90% of the LDR without flagging. The Linear Calibration Range (LCR) is established by the highest calibration standard.

- **Note:** IDLs must be < LOD for DOD projects. DOD project samples with concentrations above the calibration standard must be diluted to bring results within the quantitation range. The LOQ and cal standard establish the quantitation range. The lab may report a sample result above quantitation range if the lab runs and passes a CCV that is > sample result.

12.5. The Initial Calibration Verification (ICV) standard is analyzed immediately after calibration. The results of the ICV must agree within ±10% of the expected value. If the control limits are exceeded, the problem will be identified and the instrument recalibrated.

12.6. A Continuing Calibration Verification (CCV) and Continuing Calibration Blank (CCB) are analyzed after calibration then every 10 samples thereafter with a final CCV/CCB closing the final samples of the analytical run.

12.6.1. The results of the CCV must agree within ±10% of the expected value.

12.6.2. The CCB measured values must be less than the MRL / LOQ for each element for standard applications. Other project-specific criteria may apply (for DoD QSM projects CCB can have no analytes > the LOD).

12.6.3. If the control limits are exceeded, the problem will be identified and corrective action taken. The instrument recalibrated. The previous 10 samples must be reanalyzed.

12.7. The ICSA and ICSAB solutions are analyzed after calibration and before any field samples. The solutions are then reanalyzed every 12 hours. Results of the ICSA are used by the

analyst to identify the impact of potential interferences on the quality of the data. Based on these results appropriate action should be taken when interferences are suspected in an field sample including, but not limited to, selecting and alternative isotope for quantification, quantification, manual correction of the data, elevating the MRL, selection of an alternative method (e.g. optical ICP, GFAA) or flagging the result as estimated when no other action is possible. Results for the spiked analytes in the ICSAB solution must agree with ± 20% of the expected value.

INTERFERENCE CHECK SAMPLE COMPONENTS AND CONCENTRATIONS

	Solution A Concentrations (mg/L)	**Solution B** Concentrations (mg/L)
Al	20.0	20.0
Ca	60.0	60.0
Fe	50.0	50.0
Mg	20.0	20.0
Na	50.0	50.0
P	20.0	20.0
K	20.0	20.0
S	20.0	20.0
C	40.0	40.0
Cl	424	424
Mo	0.05	0.05
Ti	0.40	0.40
As	0.0	0.025
Cd	0.0	0.025
Cr	0.0	0.050
Co	0.0	0.050
Cu	0.0	0.050
Mn	0.0	0.050
Ni	0.0	0.050
Se	0.0	0.025
Ag	0.0	0.0125
V	0.0	0.050
Zn	0.0	0.025

NOTE: The concentration of interfering elements in the ICSA and ICSAB solutions are spiked at levels 5 times lower than recommended in Table 1 of Method 6020A. Running the full strength solutions as described in 6020A introduces too much material approximately 0.35 % dissolved solids into the ICP-MS system when trying to conduct low level analysis. Since the ICP-MS instrumentation is able to handle a maximum of 0.2% solids, the 6020A ICSA solution is higher in interfering components than any sample that would run through the instrument. However, the ICS solutions will be analyzed at levels that will provide approximately 0.1% dissolved solids.

12.8. Internal standards are used to correct for physical interferences. Masses used as internal standards include; ^{71}Ga, ^{115}In, ^{6}Li, ^{175}Lu, ^{103}Rh ^{45}Sc, and ^{89}Y. These internal standards are used

in combination to cover the appropriate mass ranges. Internal standard correction is applied applied to the analytical isotopes via interpolation of the responses from nearest internal standard isotopes (Thermo instruments) or direct correlation of analyte to IS (Agilent), (NexION). This function is performed in real-time by the instruments operating system. Internal standards must be run within 50 AMU of the masses that are analyzed. Internal standard recoveries must fall between 30% and 120% when running method 6020, or 70-125% when running method 6020A Revision 1. Internal standard recoveries for DOD QSM 5.0 5.0 must fall between 30% and 120%. As previously referenced, project specific QAPP's such as DOD QSM 5.0, supersede method specific requirements. If not, then the sample must be reanalyzed after a fivefold or greater dilution has been performed.

12.9. A method blank is digested and analyzed with every batch of 20 (or fewer) samples to demonstrate that there are no method interferences. If the method blank shows any hits above the MRL for standard applications, or >½ the MRL for DOD projects or > 1/10 the sample result, corrective action must be taken. The MB can only be rerun once. Corrective action includes recalculation, reanalysis, system cleaning, or re-extraction and re-analysis.

12.10. Laboratory Control Samples are analyzed at a frequency of 5% or one per batch, whichever is greater. Refer to the current ALS-Kelso DQO spreadsheets for the LCS limits. For method 6020A, the LCS recovery limits are 80-120%. If statistical in-house limits are used, they must fall within the 80-120% range. Project, QAPP, or client-specific control limits may supersede the limits listed, but laboratory limits should be consistent with specified limits in order to establish that the specified limits can be achieved. If the control limits are exceeded, the associated batch of samples will be re-digested and reanalyzed.

12.11. A digested duplicate and matrix spike are analyzed at a frequency of 5% or one per batch, whichever is greater. Refer to the current ALS-Kelso DQO spreadsheets for the matrix spike limits. The matrix spike recovery and relative percent difference will be calculated while analysis is in progress. Project, QAPP, or client-specific control limits may supersede the limits listed. If the control limits are exceeded, the samples will be re-digested and reanalyzed, unless matrix interference or sample non-homogeneity is established as cause. In these instances, the data and the report will be flagged accordingly.

12.12. A Matrix Spike sample is digested one per batch, or per 20 samples (i.e. 5%). Default spike concentrations are listed in the sample digestion SOPs. Spike concentrations may be adjusted to meet project requirements. The matrix spike recovery will be calculated while the job is in progress. Where specified by project requirements, a matrix spike duplicate may be required. Matrix spike recovery criteria are derived from lab data. For method 6020A, the recovery limits are 75-125%. If statistical in-house limits are used, they must fall within the 75-125% range. In some cases, project-specific QC limits may be required. Unless specified otherwise, for DoD QSM projects the project LCS criteria will be used for evaluation of matrix spikes. If an analyte recovery is outside acceptance limits proceed with the additional quality control tests described in sections 12.13 and 12.14. Based on results of these tests, the physical nature of the sample (e.g. homogeneity), and any specific project requirements, a determination can then be made as to appropriate corrective action (e.g. re-digestion, reporting with a qualifier, alternative methodologies, etc.). If the analyte concentration is >4x the spike level the spike control limit is no longer applicable and no action is required. For specifics on the preparation and composition of matrix spike solutions refer to the appropriate digestion SOP.

Note: For DOD projects a MS/MSD is required with every extraction batch. The %RSD should be < 20%.

12.13. Post Digestion Spike Test: When analysis is conducted via 6020 a post digestion spike must be performed for each matrix and each batch of sample. The prepared sample or its dilution is spiked for each element of interest at a concentration sufficiently high to be observed. Typically 20 µL of 10,000 ppb intermediate stock is added to a 10 mL aliquot of sample. If analyte concentrations are elevated in the sample, spiking at a higher concentration may be required. For 6020, the post spike should be recovered to within 75-125% of the known value or within the laboratory derived acceptance criteria. When analysis is conducted via 6020A, the post digestion spike test is performed whenever matrix spike or replicate criteria are exceeded. An analyte spike is added to a portion of a prepared sample, or its dilution, and should be recovered to within 80% to 120% of the known value. If this spike fails, then the dilution test should be run on this sample. If both the matrix spike and the post digestion spike fail, then matrix effects are confirmed. For DOD QSM 5.0 the post digestion spike shall be recovered to within 80-120% of the known value.

12.14. Dilution Test: When analysis is conducted via 6020, a serial dilution test must be performed for each matrix and each batch of sample. For sample concentrations that are sufficiently high (minimally, a factor of greater than 100 times the MDL), the analysis of a fivefold (1+4) dilution must agree within ± 10% of the original determination. When analysis is conducted via 6020A, the dilution test is performed whenever matrix spike or replicate criteria and post digestion spike criteria are exceeded. If the dilution test fails then a chemical or physical effect should be suspected. Corrective action can include additional dilution of the sample, the use of alternate methodologies, etc. or the data can be flagged and reported. The exact course of action will be dependent on the nature of the samples and project requirements and should be discussed with the project manager.

12.15. Instrument blanks should be evaluated for potential carryover and rinse times need to bring the analyte signal to within the CCB criteria. Results from instrument blanks run after standards or control samples should be used to establish levels at which carryover in samples may occur. Samples exhibiting similar effects of carryover should be reanalyzed.

12.16. Refer to the Quality Control section of EPA Methods 6020 and 6020A for additional information describing required QA/QC. Note that the nomenclature of certain QC samples in the method differs from that of the CLP SOW, but the function of those samples is equivalent in both cases.

13. DATA REDUCTION AND REPORTING

13.1. Calculations

Calculate sample results using the data system printouts and digestion information. the digestion and dilution information is entered into the data system. The data system then uses the calculations below to generate a sample result.

Aqueous samples are reported in µg/L:

µg/L (Sample) = C* x Digestion Dilution Factor x Post Digestion Dilution Factor.

C*= Concentration of analyte as measured at the instrument in ug/L (in digestate).

Solid samples are reported in mg/Kg:

$$mg/Kg\ (Sample) = C^{*}\ x\ Post\ Digestion\ Dilution\ Factor\ x \frac{Digestion\ Vol.(ml)}{Sample\ wt.(g)}\ x \frac{1mg}{1000ug}\ x \frac{1L}{1000ml}\ x \frac{1000g}{1Kg}$$

C*= Concentration of analyte as measured at the instrument in ug/L (in digestate).

NOTE: If results are to be reported on a dry weight basis, determine the dry weight of a separate aliquot of the sample, using the SOP for Total Solids.

13.2. Common isobaric interferences are corrected using equations equivalent to those listed in EPA Methods 6020, 6020A, and 200.8. Monitoring of multiple isotopes for a single element provides a mechanism for identifying isobaric interferences. Refer to the Interferences section of EPA methods for additional descriptions of possible interferences and the mechanisms required for adequately compensating for their effects.

13.3. Data Review and Reporting

13.3.1. The ICP-MS operator reviews the MS data and signs and dates the Data Review Form. A qualified senior staff spectroscopist performs a secondary review of the data and the Data Review Form is signed and dated. The data is then delivered to the report generation area where it is filed in the service request file. Once all of the data for the service request is complete, a CAR is generated.

13.3.2. The data is saved on the local hard drive and is also copied to the appropriate directory on the network. The data directories are located at r:\icp\wip\data. The data is kept on the local directory for 1 month. The network files are periodically backed up on disc or network tape.

13.3.3. For “non-production” work (such as method development or research/development studies) the analyses are performed under the direction of a senior spectroscopist. All associated data is scrutinized by the senior spectroscopist. Original raw data and associated records are archived in the analytical project file.

13.3.4. The final review and approval of all data is performed by qualified spectroscopists.

14. CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

14.1. Refer to the SOP for *Nonconformity and Corrective Action* (CE-QA008) for procedures for corrective action. Personnel at all levels and positions in the laboratory are to be alert to identifying problems and nonconformities when errors, deficiencies, or out-of-control situations are detected.

14.2. Handling out-of-control or unacceptable data

14.2.1. On-the-spot corrective actions that are routinely made by analysts and result in acceptable analyses should be documented as normal operating procedures, and no

specific documentation need be made other than notations in laboratory maintenance logbooks, runlogs, for example.

14.2.2. Some examples when documentation of a nonconformity is required using a Nonconformity and Corrective Action Report (NCAR):

- Quality control results outside acceptance limits for accuracy and precision.
- Method blanks or continuing calibration blanks (CCBs) with target analytes above acceptable levels.
- Sample holding time missed due to laboratory error or operations.
- Deviations from SOPs or project requirements.
- Laboratory analysis errors impacting sample or QC results.
- Miscellaneous laboratory errors (spilled sample, incorrect spiking, etc.).
- Sample preservation or handling discrepancies due to laboratory or operations error.

15. METHOD PERFORMANCE

15.1. This method was validated through single laboratory studies of accuracy and precision. Refer to the reference method for additional available method performance data.

15.2. The method detection limit (MDL), limit of detection (LOD) and limit of quantitation (LOQ) are established using procedures described in CE-QA011, *Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantification.* Method Reporting Limits are established for this method based on MDL studies and as specified in the ALS, Kelso Quality Assurance Manual.

16. POLLUTION PREVENTION AND WASTE MANAGEMENT

16.1. It is the laboratory's practice to minimize the amount of solvents, acids, and reagents used to perform this method wherever feasibly possible. Standards are prepared in volumes consistent with methodology and only the amount needed for routine laboratory use is kept on site. The threat to the environment from solvents and/or reagents used in this method can be minimized when recycled or disposed of properly.

16.2. The laboratory will comply with all Federal, State, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the ALS Environmental Health and Safety Manual.

16.3. This method uses acid. Waste acid is hazardous to the sewer system and to the environment. All acid waste must be neutralized to a pH of 5-9 prior to disposal down the drain. The neutralization step is considered hazardous waste treatment and must be documented on the treatment by generator record. See the ALS *EH&S Manual* for details.

17. TRAINING

17.1. Refer to the SOP ADM-TRAIN, *ALS-Kelso Training Procedure* for standard procedures.

17.2. A minimum of two senior level spectroscopists are to be maintained on staff at all times. Senior spectroscopists are defined as individuals with a minimum of ten years combined

education and experience in, or related to atomic spectroscopy. Of those ten years, a minimum of two years of ICP-MS experience is required.

17.3. All technical staff is encouraged to attend one technical seminar per year. In addition to the technical seminars, senior spectroscopists are required to complete a one week training session offered by the instrument manufacturer.

17.4. On-the-job-training occurs daily with the senior spectroscopists providing direction to new operators. The physical operation of the equipment is relatively simple. The data reduction and troubleshooting requires extensive experience that can only be gained by hands-on operation of the instrument and assisted evaluation of raw data.

17.5. Training outline

17.5.1. Review literature (see references section). Read and understand the SOP. Also review the applicable MSDS for all reagents and standards used. Following these reviews, observe the procedure as performed by an experienced analyst at least three times.

17.5.2. The next training step is to assist in the procedure under the guidance of an experienced analyst. During this period, the analyst is expected to transition from a role of assisting, to performing the procedure with minimal oversight from an experienced analyst.

17.5.3. Perform initial precision and recovery (IPR) study as described above for water or soil samples. Summaries of the IPR are reviewed and signed by the supervisor. Copies may be forwarded to the employee's training file. For applicable tests, IPR studies should be performed in order to be equivalent to NELAC's Initial Demonstration of Capability.

17.6. Training and proficiency is documented in accordance with the SOP ADM-TRAIN.

18. METHOD MODIFICATIONS

18.1. There are no known modifications in this laboratory standard operating procedure from the reference method.

19. REFERENCES

19.1. USEPA, Test Methods for Evaluating Solid Waste, SW-846, 3rd Edition, Update III Method 6020, Revision 0, September 1994.

19.2. USEPA, Test Methods for Evaluating Solid Waste, SW-846, Update IV, Method 6020A, Revision 1, February 2007.

19.3. Agilent and Thermo Elemental Instrument Manuals

19.4. Department of Defense (DoD) Quality Systems Manual (QSM) for Environmental Laboratories, Version 5.0.

20. CHANGES SINCE THE LAST REVISION

20.1. Section 7.2.4.1: Added 0.250 mL of 10 ppm Molybdenum solution to the ICSA solution.
20.2. Section 12.8: Added Agilent to the direct correlation of analyte to IS statement.
20.3. Section 12.8: Added DoD 5.0 internal standard limits.
20.4. Section 19: Added DOD 5.0 to the Reference Section.
20.5. Section 13: Restored missing information Aqueous (µg/L (Sample) lost in transfer between document versions.
20.6. Added standard DOD standard paragraph in Section 1.
20.7. Added standard paragraph for ASTM I and ASTM II water quality in Section 7.
20.8. Section 12.13: Adjusted post spike recovery criteria, per procedural change form dated 12/29/15.
20.9. Table 2: Added comment: Se78 is the default isotope on the NexION and Agilent instruments.
20.10. Section 11: Former Section 11.2 referring to an obsolete instrument log was removed in its entirety.

TABLE 1
TARGET ANALYTES, MDLs, and MRLs

METHOD	PREP METHOD	ANALYTE	MATRIX	MDL	MRL mg/kg
6020A	EPA 3050B	Aluminum	Soil	0.6	2
6020A	EPA 3050B	Antimony	Soil	0.02	0.05
6020A	EPA 3050B	Arsenic	Soil	0.2	0.5
6020A	EPA 3050B	Barium	Soil	0.02	0.05
6020A	EPA 3050B	Beryllium	Soil	0.005	0.02
6020A	EPA 3050B	Bismuth	Soil	0.02	0.05
6020A	EPA 3050B	Boron	Soil	0.05	0.5
6020A	EPA 3050B	Cadmium	Soil	0.009	0.02
6020A	EPA 3050B	Chromium	Soil	0.07	0.2
6020A	EPA 3050B	Cobalt	Soil	0.009	0.02
6020A	EPA 3050B	Copper	Soil	0.04	0.1
6020A	EPA 3050B	Lead	Soil	0.02	0.05
6020A	EPA 3050B	Manganese	Soil	0.02	0.05
6020A	EPA 3050B	Molybdenum	Soil	0.02	0.05
6020A	EPA 3050B	Nickel	Soil	0.04	0.2
6020A	EPA 3050B	Selenium	Soil	0.2	1
6020A	EPA 3050B	Silver	Soil	0.005	0.02
6020A	EPA 3050B	Thallium	Soil	0.002	0.02
6020A	EPA 3050B	Tin	Soil	0.02	0.1
6020A	EPA 3050B	Uranium	Soil	0.003	0.02
6020A	EPA 3050B	Vanadium	Soil	0.08	0.2
6020A	EPA 3050B	Zinc	Soil	0.2	0.5

TABLE 1 – continued

METHOD	PREP METHOD	ANALYTE	MATRIX	MDL	MRL ug/L
6020A	MET-DIG (CLP)	Aluminum	Water	0.2	2
6020A	MET-DIG (CLP)	Antimony	Water	0.01	0.05
6020A	MET-DIG (CLP)	Arsenic	Water	0.05	0.5
6020A	MET-DIG (CLP)	Barium	Water	0.006	0.05
6020A	MET-DIG (CLP)	Beryllium	Water	0.008	0.02
6020A	MET-DIG (CLP)	Bismuth	Water	0.005	0.05
6020A	MET-DIG (CLP)	Boron	Water	0.07	0.5
6020A	MET-DIG (CLP)	Cadmium	Water	0.005	0.02
6020A	MET-DIG (CLP)	Chromium	Water	0.02	0.2
6020A	MET-DIG (CLP)	Cobalt	Water	0.006	0.02
6020A	MET-DIG (CLP)	Copper	Water	0.03	0.1
6020A	MET-DIG (CLP)	Iron	Water	0.3	1
6020A	MET-DIG (CLP)	Lead	Water	0.004	0.02
6020A	MET-DIG (CLP)	Manganese	Water	0.006	0.05
6020A	MET-DIG (CLP)	Molybdenum	Water	0.008	0.05
6020A	MET-DIG (CLP)	Nickel	Water	0.04	0.2
6020A	MET-DIG (CLP)	Selenium	Water	0.4	1
6020A	MET-DIG (CLP)	Silver	Water	0.005	0.02
6020A	MET-DIG (CLP)	Thallium	Water	0.005	0.02
6020A	MET-DIG (CLP)	Tin	Water	0.01	0.05
6020A	MET-DIG (CLP)	Uranium	Water	0.003	0.02
6020A	MET-DIG (CLP)	Vanadium	Water	0.05	0.2
6020A	MET-DIG (CLP)	Zinc	Water	0.09	0.5

TABLE 1 – continued

METHOD	PREP METHOD	ANALYTE	MATRIX	MDL	MRL mg/kg
6020A	PSEP TISSUE	Aluminum	Tissue	0.2	2
6020A	PSEP TISSUE	Antimony	Tissue	0.002	0.05
6020A	PSEP TISSUE	Arsenic	Tissue	0.02	0.5
6020A	PSEP TISSUE	Barium	Tissue	0.005	0.05
6020A	PSEP TISSUE	Beryllium	Tissue	0.003	0.02
6020A	PSEP TISSUE	Bismuth	Tissue	0.003	0.05
6020A	PSEP TISSUE	Boron	Tissue	0.2	2
6020A	PSEP TISSUE	Cadmium	Tissue	0.002	0.02
6020A	PSEP TISSUE	Chromium	Tissue	0.02	0.2
6020A	PSEP TISSUE	Cobalt	Tissue	0.003	0.02
6020A	PSEP TISSUE	Copper	Tissue	0.02	0.1
6020A	PSEP TISSUE	Iron	Tissue	0.2	1
6020A	PSEP TISSUE	Lead	Tissue	0.0005	0.02
6020A	PSEP TISSUE	Manganese	Tissue	0.008	0.05
6020A	PSEP TISSUE	Molybdenum	Tissue	0.008	0.05
6020A	PSEP TISSUE	Nickel	Tissue	0.02	0.2
6020A	PSEP TISSUE	Selenium	Tissue	0.2	1
6020A	PSEP TISSUE	Silver	Tissue	0.006	0.02
6020A	PSEP TISSUE	Thallium	Tissue	0.0009	0.02
6020A	PSEP TISSUE	Tin	Tissue	0.003	0.05
6020A	PSEP TISSUE	Uranium	Tissue	0.0008	0.02
6020A	PSEP TISSUE	Vanadium	Tissue	0.007	0.2
6020A	PSEP TISSUE	Zinc	Tissue	0.06	0.5

Table 2
Target Element Masses

ANALYTE	ISOTOPES ANALYZED	ISOTOPE REPORTED
Aluminum	27	27
Antimony	121,123	123
Arsenic	75	75
Barium	135,137,138	137
Beryllium	9	9
Cadmium	111,112,114	111
Chromium	52,53	52
Cobalt	59	59
Copper	63,65	65
Lead	206,207,208	208
Manganese	55	55
Molybdenum	95,97,98	98
Nickel	60,61,62	60
Selenium*	77,78,82	82
Silver	107,109	107
Thallium	203,205	205
Uranium	238	238
Vanadium	51	51
Zinc	66,67,68	66

*Se 78 Is the default isotope on the NexION and The Agilent instruments.

ATTACHMENT A
Example Standard Sheets

SOLUTION: ICP-MS, 200.8 INTERMEDIATE STOCK
MATRIX: 2% HNO3

ELEMENT		ALIQUOT OF 1000 ppm Std./1000ml	CONCENTRATION (µg/L)
HNO3		50.0 ml.	5%
Al		1.0 ml.	1000
Sb		1.0 ml.	1000
As		1.0 ml.	1000
Ba		1.0 ml.	1000
Be		1.0 ml.	1000
Cd		1.0 ml.	1000
Cr		1.0 ml.	1000
Co		1.0 ml.	1000
Cu		1.0 ml.	1000
Fe		1.0 ml.	1000
Pb		1.0 ml.	1000
Mn		1.0 ml.	1000
Mo		1.0 ml.	1000
Ni		1.0 ml.	1000
Se		1.0 ml.	1000
Tl		1.0 ml.	1000
V		1.0 ml.	1000
U		1.0 ml.	1000
Zn		1.0 ml.	1000

SOLUTION: ICP-MS, 200.8 SILVER INTERMEDIATE STOCK
MATRIX: 5% HNO3

		ALIQUOT OF	CONCENTRATION
ELEMENT		1000 ppm Std./1000ml	(µg/L)
HNO3		50.0	5%
Ag		1.0	1000

SOLUTION: ICP-MS 25ppb Calibration Standard and CCV
MATRIX: As Required

	ALIQUOT PER	CONCENTRATION
SOURCE	100 ml.	(µg/L)
HNO3 (Ultrex)	As Required	As Required
INTERMEDIATE STOCK	2.5	25.0
SILVER INTERMEDIATE STOCK	2.5	25.0

ATTACHMENT B
Isobaric Interference Corrections

Interference Equations:

Equation Name: Default

?SW82 = I82 * 0.7
?%SE77 = ?SE82 * 0.8484163
?%ARCL77 = I77 - ?%SE77
?%ARCL75 = ?%ARCL77 * 3.0650407
?AS75 = I75 - ?%ARCL75
?%CR53 = I52 * 0.1133652
?%CLO53 = I53 - ?%CR53
?%CLO51 = ?%CLO53 * 3.0650407
?V51 = I51 - ?%CLO51
?PB208 = I208 + I207 + I206

ALS Standard Operating Procedure

DOCUMENT TITLE:	SELENIUM BY BOROHYDRIDE REDUCTION ATOMIC ABSORPTION
REFERENCED METHOD:	EPA 7742
SOP ID:	MET-7742
REVISION NUMBER:	5
EFFECTIVE DATE:	3/03/17

SELENIUM BY BOROHYDRIDE REDUCTION ATOMIC ABSORPTION

ALS-KELSO

SOP ID:	MET-7742	Rev. Number:	5	Effective Date:	3/03/17

Approved By: _____ Date: 2/27/17
Technical Director - Jeff Coronado

Approved By: _____ Date: 2/27/17
QA Manager - Carl Degner

Approved By: _____ Date: 2/28/17
Laboratory Director - Jeff Grindstaff

Issue Date: _____ Doc Control ID#: _____ Issued To: _____

ANNUAL REVIEW

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Signature	Title	Date

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SELENIUM BY BOROHYDRIDE REDUCTION ATOMIC ABSORPTION

1. SCOPE AND APPLICATION

1.1. This Standard Operating Procedure (SOP) describes the procedure used for the analysis of Selenium by borohydride reduction atomic absorption using EPA Method 7742 or Standard Method 3114B. This procedure describes the analysis procedures used to determine the analyte concentration and reporting limits listed. The sample preparation procedures are described in sample preparation SOPs MET-3010A, MET-3050B, and MET-TDIG.

1.2. This procedure is used to determine Selenium in water, soil, and tissue matrices. The procedure may be applied to other miscellaneous sample matrices providing that the analyst demonstrates the ability of the procedure to give data of acceptable quality in that matrix. The Method Reporting Limits (MRLs) are given in Table 1 and Method Detection Limits (MDLs) that have been achieved are documented in the laboratory DQO tables.

1.3. In cases where there is a project-specific quality assurance plan (QAPP), the project manager identifies and communicates the QAPP-specific requirements to the laboratory. In general, project specific QAPP's supersede method specified requirements. An example of this are projects falling under DoD ELAP. QC requirements defined in the SOP *Department of Defense Projects – Laboratory Practices and Project Management* (ADM-DOD/ADM-DOD5) may supersede the requirements defined in this SOP.

2. METHOD SUMMARY

2.1. Samples are prepared according to the nitric acid digestion procedure described in Method 3010 for aqueous and extract samples, the nitric/peroxide/hydrochloric acid digestion procedure described in Method 3050 for sediment, soil, and sludge, and the nitric/closed vessel digestion for tissue. Excess peroxide is removed by evaporating samples to near-dryness at the end of the digestion followed by dilution to volume and degassing the samples upon addition of urea. The selenium is converted to the +4 oxidation state during digestion in HCl. Selenium is then converted to its volatile hydride using hydrogen produced from the reaction of the acidified sample with sodium borohydride in a continuous-flow hydride generator.

2.2. The volatile hydrides are swept into, and decompose in, a heated quartz absorption cell located in the optical path of an atomic absorption spectrophotometer. The resulting absorption of the lamp radiation is proportional to the selenium concentration.

3. DEFINITIONS

3.1. Analysis Sequence - Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration (initial or continuing verification) followed by sample extracts interspersed with calibration standards (CCBs, CCVs, etc...) The sequence ends when the set of samples has been injected

or when qualitative and/or quantitative QC criteria indicate an out-of-control situation.

3.2. Batch - A batch of samples is a group of environmental samples that are prepared and/or analyzed together as a unit with the same process and personnel using the same lot(s) of reagents. It is the basic unit for analytical quality control.

3.2.1. Preparation Batch - A preparation batch is composed of one to twenty field samples, all of the same matrix, meeting the criteria in Section 3.3 and with a maximum time between the start of processing of the first and last samples in the batch to be 24 hours.

3.2.2. Analysis Batch - Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration (initial or continuing verification) followed by sample extracts interspersed with calibration standards (CCBs, CCVs, etc.) The sequence ends when the set of samples has been injected or when qualitative and/or quantitative QC criteria indicate an out-of-control situation.

3.3. Sample

3.3.1. Field Sample - An environmental sample collected and delivered to the laboratory for analysis; a.k.a., client's sample.

3.3.2. Laboratory Sample - A representative portion, aliquot, or subsample of a field sample upon which laboratory analyses are made and results generated.

3.4. Quality System Matrix - The matrix of an environmental sample is distinguished by its physical and/or chemical state and by the program for which the results are intended. The following sections describe the matrix distinctions. These matrices shall be used for purpose of batch and quality control requirements.

3.4.1. Aqueous - Any groundwater sample, surface water sample, effluent sample, and TCLP or other extract. Specifically excluded are samples of the drinking water matrix and the saline/estuarine water matrix.

3.4.2. Drinking water - Any aqueous sample that has been designated a potable or potential potable water source.

3.4.3. Saline/Estuarine water - Any aqueous sample from an ocean or estuary or other salt-water source.

3.4.4. Nonaqueous Liquid - Any organic liquid with <15% settleable solids.

3.4.5. Animal tissue - Any tissue sample of an animal, invertebrate, marine organism, or other origin; such as fish tissue/organs, shellfish, worms, or animal material.

3.4.6. Solids - Any solid sample such as soil, sediment, sludge, and other materials with >15% settleable solids.

3.4.7. Chemical waste - Any sample of a product or by-product of an industrial process that results in a matrix not described in one of the matrices in Sections 3.3.1 through 3.3.6. These can be such matrices as non-aqueous liquids, solvents, oil, etc.

3.4.8. Miscellaneous matrices – Samples of any composition not listed in 3.3.1 – 3.3.7. These can be such matrices as plant material, paper/paperboard, wood, auto fluff, mechanical parts, filters, wipes, etc. Such samples shall be batched/grouped according to their specific matrix.

3.5. Method Blank (MB) - The method blank is an artificial sample composed of analyte-free water or solid matrix and is designed to monitor the introduction of artifacts into the analytical process. The method blank is carried through the entire analytical procedure.

3.6. Laboratory Control Samples (LCS) – The LCS is an aliquot of analyte free water or analyte free solid to which known amounts target analytes are added. The LCS is prepared and analyzed in exactly the same manner as the samples. The percent recovery is compared to established limits and assists in determining whether the batch is in control.

3.7. Matrix Spike/Duplicate Matrix Spike (MS/DMS) Analysis - In the matrix spike analysis, predetermined quantities of target analytes are added to a sample matrix prior to sample preparation and analysis. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the method used for the analysis. Samples are split into duplicates, spiked, and analyzed as a MS/DMS pair. Percent recoveries are calculated for each of the analytes detected. The relative percent difference (RPD) between the duplicate spikes (or samples) is calculated and used to assess analytical precision. The concentration of the spike should be at 3- 5 times the method reporting limit or at levels specified by a project analysis plan.

3.8. Laboratory Duplicates (DUP) – Duplicates are additional replicates of samples that are subjected to the same preparation and analytical scheme as the original sample. The relative percent difference (RPD) between the sample and its duplicate is calculated and used to assess analytical precision.

3.9. Independent Verification Standard (ICV) - A mid-level standard injected into the instrument after the calibration curve and prepared from a different source than the initial calibration standards. This is used to verify the validity of the initial calibration standards

3.10. Continuing Calibration Verification Standard (CCV) - A mid-level standard analyzed at specified intervals. Used to verify that the initial calibration curve is still valid for quantitative purposes.

3.11. Instrument Blank (CCB) - The instrument blank (also called continuing calibration blank) is a volume of clean solvent analyzed on each column and instrument used for sample analysis. The purpose of the instrument blank is to determine the levels of contamination associated with the instrumental analysis itself, particularly with

regard to the carry-over of analytes from standards or highly contaminated samples into subsequent sample analyses.

3.12. Duplicates and Duplicate Matrix Spikes are additional replicates of samples that are subjected to the same preparation and analytical scheme as the original sample. Depending on the method of analysis, either a duplicate analysis (and/or a matrix spiked sample) or a matrix spiked sample and duplicate matrix spiked sample (MS/DMS) are analyzed.

3.13. Standard Reference Material (SRM) – A material with specific certification criteria and is issued with a certificate or certificate of analysis that reports the results of its characterizations and provides information regarding the appropriate use(s) of the material. An SRM is prepared and used for three main purposes: (1) to help develop accurate methods of analysis; (2) to calibrate measurement systems used to facilitate exchange of goods, institute quality control, determine performance characteristics, or measure a property at the state-of-the-art limit; and (3) to ensure the long-term adequacy and integrity of measurement quality assurance programs.

3.14. Standard Curve - A standard curve is a calibration curve that plots concentrations of a known analyte standard versus the instrument response to the analyte. A linear regression calibration model is used. The appropriate criteria for assessing the validity of the calibration curve must be followed prior to quantitation of target analytes in actual sample analyses.

3.15. Method of standard additions (MSA) - The standard addition technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique attempts to compensate for a sample constituent that enhances or depresses the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which cause a baseline shift.

4. INTERFERENCES

4.1. Very high (>1000 mg/L) concentrations of cobalt, copper, iron, mercury, and, nickel can cause analytical interferences through precipitation as reduced metals and associated blockage of transfer lines and fittings.

4.2. Traces of peroxides left following the sample work-up can result in analytical interferences. Peroxides must be removed by evaporating each sample to near-dryness followed by reacting each sample with urea and allowing sufficient time for degassing before analysis.

4.3. Even after acid digestion, flame gases and organic compounds may remain in the sample. Flame gases and organic compounds can absorb at the analytical wavelengths and background correction should be used.

5. SAFETY

5.1. All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this procedure. This includes the use of

personnel protective equipment, such as, safety glasses, lab coat and the correct gloves.

5.2. Chemicals, reagents and standards must be handled as described in the ALS safety policies, approved methods and in SDSs where available. Refer to the ALS Chemical Hygiene Plan and the appropriate SDS prior to beginning this method.

5.3. Hydrochloric and/or Nitric Acid are used in this method. These acids are extremely corrosive and care must be taken while handling them. A face shield should be used while pouring acids. And safety glasses should be worn while working with the solutions. Lab coat and gloves should always be worn while working with these solutions.

6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION AND STORAGE

6.1. Aqueous samples are typically collected in plastic containers. Aqueous samples are preserved with nitric acid (pH<2), then refrigerated at 4 ± 2°C from receipt until analysis.

6.2. Non-aqueous samples may be collected in plastic or glass jars. Non-aqueous samples are refrigerated at 4 ± 2°C from receipt until analysis. Non-aqueous samples should be analyzed as soon as possible following sampling.

6.3. Tissue samples are typically collected in plastic or glass jars. Prepared samples are stored frozen at < 10ºC until preparation. Tissue samples can be held up to one year.

7. STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

7.1. Reagents

7.1.1. Reagent grade chemicals shall be used in all tests. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lowering the accuracy of the determination.

7.1.2. Concentrated Hydrochloric Acid (HCl)

7.1.3. 0.2% Sodium Borohydride ($NaBH_4$). 4g $NaBH_4$ plus 1g of sodium hydroxide dissolved in 2000mL of reagent water.

7.1.4. Urea (H_2NCONH_2)

7.1.5. Reagent and grade

7.2. Standards

7.2.1. Stock standard solutions may be purchased from a number of vendors. All standards purchased from vendors must be traceable to NIST or A2LA certified reference materials. Purchased standards are typically prepared at

a concentration of 1000 ppm, and are prepared in 500 mL plastic bottles. The vendor-assigned expiration date is used.

7.2.2. A 1000 ppb intermediate stock standard is prepared by pipetting 0.100 mL of 1000 ppm stock standard, plus 1.0 mL of concentrated HNO_3, into a 100 mL volumetric and diluting to volume with reagent water.

7.2.3. The ICV is prepared at 1000 ppb as described above. The 1000 ppb intermediate stock used for the ICV is prepared from a source independent of the original 1000 ppm purchased standard.

8. APPARATUS AND EQUIPMENT

8.1. Perkin Elmer AAnylst 200 - Atomic Absorption Spectrophotometer.

8.1.1. FIAS -100 Vapor Generator

8.1.2. Quartz Absorbance Cell

8.1.3. Selenium Hollow Cathode Lamp

8.2. Digestion Hot-Block capable of maintaining 50˚C

8.3. 50 mL Centrifuge Tubes

8.4. Hot Plates capable of maintaining 50˚C

8.5. 50 mL Volumetric Flasks

9. PREVENTIVE MAINTENANCE

9.1. All maintenance activities are recorded in a maintenance logbook kept for each instrument. Pertinent information (serial numbers, instrument I.D., etc.) must be in the logbook. This includes the routine maintenance described in section 9. The entry in the log must include: date of event, the initials of who performed the work, and a reference to analytical control.

9.2. Typical preventive maintenance measures include, but are not limited to, the following items:

- Cleaning the nebulizer and burner head,
- Cleaning the gas-liquid separator,
- Inspection and cleaning of the hollow cathode and deuterium lamp conditions.

10. RESPONSIBILITIES

10.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This

demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10.2. It is the responsibility of the department supervisor/manager to document analyst training. Documenting method proficiency, as described in Methods 7742 and 3114B, is also the responsibility of the department supervisor/manager.

11. PROCEDURE

11.1. Sample Preparation

11.1.1. Water samples are first prepared by EPA Method 3010A (ALS SOP; MET-3010A), soil samples by EPA Method 3050B (ALS SOP; MET-3050) with modifications noted below, and tissue samples by closed vessel digestion (ALS SOP; MET-TDIG).

Note: The 3050B soil digestion is modified as follows: After the final peroxide addition (i.e. before the final reduction stage) add 5.0mL of concentrated hydrochloric acid and reduce the digestate volume to less than 5.0mL, but not to dryness. After cooling, dilute the digestate to 100mL with reagent water.

11.2. Digestate Preparation for hydride analysis

11.2.1. Waters – 15 mL of the water digestate is aliquoted to a 50 mL centrifuge tube and 12 mL of concentrated HCl is added. The centrifuge cap is loosely placed on the tube and the sample is heated in a digestion hot-block at 50°C for 30 minutes. After cooling the sample is diluted to 30 mL as needed resulting in a two-fold dilution of the original digestate.

11.2.2. Soil/Sediment - 25 mL of the soil/sediment digestate is aliquoted to a 50 mL centrifuge tube. 0.5 g of Urea is added followed by 20 mL of concentrated HCl. The centrifuge cap is loosely placed on the tube and the sample is heated in a digestion hot-block at 50°C for 30 minutes. After cooling the sample is diluted to 50 mL as needed resulting in a two fold dilution of the original digestate.

11.2.3. Tissues – 8.0 mL of the tissue digestate is aliquoted to a 50 mL centrifuge tube. 0.4 g of Urea is added followed by 16 mL of concentrated HCl. The centrifuge cap is loosely placed on the tube and the sample is heated in a digestion hot-block at 50°C for 30 minutes. After cooling the sample is diluted to 40 mL as needed resulting in a five-fold dilution of the original digestate.

11.3. Standard Preparation

11.3.1. Working standards are prepared in 50 mL volumetric flasks by aliquoting 0, 0.025, 0.05, 0.25, 0.375, and 0.5 mL of 1000 µg/L intermediate standard into 50 mL centrifuge tube containing approximately 15 mL of reagent water. When analyzing tissues, an additional low point using 0.01 mL of the

1000ug/L intermediate is prepared. The appropriate amounts of concentrated HCl and/or HNO_3 are added in order to replicate the matrix of the initial 3010A, 3050B, or tissue digests. An additional 20 mL of concentrated HCl is added and the centrifuge tubes are heated on a hotblock at 50°C of 30 minutes. After cooling, the standards are quantitatively transferred to 50 mL volumetric flask and diluted to volume with reagent water.

11.3.2. An Independent Calibration Verification (ICV) standard is prepared by aliquoting 0.375 mL of a 1000 µg/L intermediate standard prepared from a different source than the intermediate used for the calibration standards. This standard is then processed as described above.

11.4. Method of Standard Additions (MSA):

11.4.1. For Soil and Tissue digestions the single point MSA technique is utilized by default. The matrix of these digests has been shown to produce a low bias of varying degrees when analyzed using the Perkin Elmer hydride system. This low bias is corrected for using the MSA procedure described in section 9.10.1 of EPA method 7000B Revision 2. After the sample digestate is prepared as described above in section 11.1 of this SOP, two 10 mL aliquots are placed in separate autosampler tubes. To the MSA aliquot 0.05 mL of 1000 ppb standard is added. After the two sample aliquots are analyzed the solution concentration (C_x) can be calculated using the following equation:

$$C_x = \frac{S_B * V_S * C_S}{(S_A - S_B)\, V_x}$$

Where:

S_B = analytical signal of parent sample

S_A = analytical signal of MSA sample

V_S = volume of standard added to the MSA sample

V_X = volume of sample aliquots

C_S = concentration of spike solution

Alternately, C_x can be calculated with solution concentrations using the following equation:

$$C_x = \frac{C_P * V_S * C_S}{(C_M - C_P)\, V_x}$$

Where:

C_P = measured concentration of the parent sample

C_M = measured concentration of the MSA sample

V_S = volume of standard added to the MSA sample

V_X = volume of sample aliquots

C_S = concentration of spike solution

Once C_X is determined the final sample concentration is calculated by the appropriate reporting software based on the initial sample aliquot and subsequent digestion and instrumental dilutions, and total solids if applicable.

Note: Since the change of volume from spiking the MSA aliquot is only 0.5%, which is insignificant relative to the accuracy and precision of the final result, this dilution is ignored in the calculations above.

11.5. Instrument Setup

A Perkin Elmer AA200 is used in conjunction with the FIAS-100 hydride generator, and a burner head mounted quartz cell. The sample and carrier (10% HCl) flows are set at 4.2 mL/min and the borohydride (0.2% Sodium Borohydride) flow at 2.1 mL/min. The instrument is optimized to wavelength of 196.0 nm, with a slit width 1.0. The burner is ignited and the system is allowed to warm up for 10 minutes prior to starting the analysis.

CAUTION: The hydride of selenium is very toxic. Precautions must be taken to avoid inhaling the gas.

11.6. Calibration and Analysis

11.6.1. The sampling tube is placed into the calibration blank, allowed to come to equilibrium, and then analyzed. The remaining calibration standards are analyzed similarly in ascending order. After the calibration curve is complete the r value is calculated. If the r value is not $\geq$ 0.995 then the calibration is rejected and must be re-analyzed.

11.6.2. Immediately following the calibration the ICV solution is analyzed followed in order by an Initial Calibration Blank (ICB), CRA, CCV, Continuing Calibration Blank (CCB), and the MRL check standard (i.e. the low calibration standard). ICV and CCV recoveries must fall within ± 10% of their true value, and ICB and CCB results must be less than 3x MDL. The CRA must fall within ±30% of the true value.

11.6.3. Once the above calibration and QC check standards have been successfully run the samples are ready to be analyzed. After a maximum of 10 samples have been analyzed an additional set of CCV and CCB standard must be analyzed using the criteria described above. If one or both of these standards are out of control the problem must be corrected, the instrument recalibrated, the initial QC check standards analyzed, and the samples following the last compliant CCV/CCB check re-analyzed.

11.7. List any matrix modifiers or reducing agents used in the analysis on the associated raw data.

12. QA/QC REQUIREMENTS

12.1. Initial Precision and Recovery Validation

12.1.1. The accuracy and precision of the procedure must be validated before analyses of samples begin, or whenever significant changes to the procedures have been made. To do this, four water samples are spiked with the LCS spike solution, then prepared and analyzed using the applicable method.

12.2. Method Detection Limits and Method Reporting Limits

12.2.1. A method detection limit (MDL) study must be undertaken before analysis of samples can begin. To establish detection limits that are precise and accurate, the analyst must perform the following procedure. Spike seven blank matrix (reagent water) samples with MDL spiking solution at a level below the MRL. Follow the analysis procedures in Section 11 to analyze the samples.

12.2.2. Calculate the average concentration found (x) in µg/mL, and the standard deviation of the concentrations (s) in µg/mL for each analyte. Calculate the MDL for each analyte. Refer to the SOP *Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantification* (CE-QA011). The MDL study must be verified annually.

12.3. Limits of Quantification (LOQ)

12.3.1. Method 7000B requires the laboratory establish a LOQ for each analyte as the lowest reliable laboratory reporting concentration or in most cases the lowest point in the calibration curve which is less than or equal to the desired regulatory action levels, based on the stated project requirements. Analysis of a standard prepared at the LOQ concentration levels or use of the LOQs as the lowest point calibration standard provides confirmation of the established sensitivity of the method. The LOQ recoveries must be within **30%** of the true values to verify the data reporting limit. Refer to *Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantification* (CE-QA011).

12.3.2. The Method Reporting Limits (MRLs) used at ALS are the routinely reported lower limits of quantitation which take into account day-to-day fluctuations in instrument sensitivity as well as other factors. These MRLs are the levels to which ALS routinely reports results in order to minimize false positive or false negative results. The MRL is normally two to ten times the method detection limit.

12.4. Ongoing QC Samples required are described in the ALS-Kelso Quality Assurance Manual and in the SOP for Sample Batches. Additional QC Samples may be required in project specific quality assurance plans (QAPP). For example projects managed

under the DoD ELAP must follow requirements defined in the DoD *Quality Systems Manual for Environmental Laboratories.* General QA requirements for DoD QSM are defined in the laboratory SOP, Department *of Defense Projects – Laboratory Practices and Project Management* (ADM-DOD). General QC Samples and their default control criteria are:

12.4.1. Method Blank

12.4.1.1. A method blank is extracted and analyzed with every batch of 20 (or fewer) samples to demonstrate that there are no method interferences. If the method blank shows any hits above the MRL for standard applications, or ½ the MRL for DoD projects, corrective action must be taken. Corrective action includes recalculation, reanalysis, system cleaning, or re-extraction and reanalysis. Reanalysis may only be done once.

12.4.2. Lab Control Sample (LCS)

12.4.2.1. The laboratory control sample for water samples is composed of analyte-free water into which Selenium is spike. The laboratory control sample for soils and tissues consists of an appropriate reference material (e.g. ERA DO65-540 for soils, NRCC TORT-3 for tissues). The LCS is designed to monitor the accuracy of the procedure. The concentration of the spike in the LCS matrix should be at 5 to 10 times the MRL or at levels specified by a project analysis plan.

12.4.2.2. A lab control sample (LCS) must be prepared and analyzed with every batch of 20 (or fewer) samples. Calculate the LCS recovery as follows:

$$\%R = X/TV \times 100$$

Where X = Concentration of the analyte recovered
TV = True value of amount spiked

The acceptance criterion is **80-120%.** Acceptance limits for other reference materials is specific to the material used. Also, other project-specific limits may be required. If the LCS fails acceptance criteria, corrective action must be taken. Corrective action includes recalculation, reanalysis, or re-extraction and reanalysis.

12.4.3. Matrix Spike

12.4.3.1. A matrix spike (MS) must be prepared and analyzed with every batch of 20 (or fewer) samples. The MS is prepared by adding a known volume of the matrix spike solution to the sample and determining the spiked sample concentration. Calculate percent recovery (%R) as:

$$\%R = \frac{X - X1}{TV} \times 100$$

Where X = Concentration of the analyte recovered
X1 = Concentration of unspiked analyte
TV = True value of amount spiked

12.4.3.2. The acceptance limits for the MS is **75-125%** for all matrices. Also, other project-specific limits may be required. If the MS recovery is out of acceptance limits for reasons other than matrix effects, corrective action must be taken. Corrective action includes recalculation, reanalysis, or re-preparation and reanalysis.

12.4.4. A Duplicate sample is prepared and analyzed one per batch, or per 20 samples. Calculate Relative Percent Difference (RPD) as:

$$RPD = \frac{|R1 - R2|}{(R1 + R2)/2} \times 100$$

Where R1= result for the sample
R2= result for the sample duplicate

The RPD criterion is **20%**. If outside the limit, redigest the sample batch. Determine if the sample is non-homogenous and redigest if it is homogenous.

12.5. Recovery test (post-digestion spike)

12.5.1. The recovery test must be done on all water samples within a digestion batch were that batch's MS fails.

12.5.2. The same sample from which the MS/MSD aliquots were prepared (assuming the MS/MSD recoveries are unacceptable) should also be spiked with a post digestion spike. Otherwise another sample from the same preparation should be used as an alternative. An analyte spike is added to a portion of a prepared sample, or its dilution, and should be recovered to within **80% to 120%** of the known value. The spike addition should produce a minimum level of 10 times and a maximum of 100 times the lower limit of quantitation. If both the MS/MSD and the post digestion spike fail, then matrix effects are confirmed.

12.6. Prior to preparation of samples, blanks should be analyzed to determine possible interferences from sample handling steps, reagents, or glassware. If the blanks show contamination, the source of the contamination should be isolated and minimized.

13. DATA REDUCTION AND REPORTING

13.1. The concentration of the analyte(s) in the sample digest (Cex) is calculated using the calibration curve. The concentration of analytes in the original samples is computed using the following equations:

Aqueous Samples:

$$Concentration\ (\mu g / L) = \frac{(Cex)\ (Vf)\ (D)}{(Vs)}$$

Where Cex = Concentration in digestate in µg/mL
Vf = Final volume of extract in mL
D = Dilution factor
Vs = Volume of sample digested, liters

Nonaqueous Samples:

$$Concentration\ (mg / Kg) = \frac{(Cex)\ (Vf)\ (D)}{(W)}$$

Where Cex = Concentration in digestate in µg/mL
Vf = Final volume of extract in mL
D = Dilution factor
W = Weight of sample digested in grams. The wet or dry weight may be used, depending upon the specific client requirements.

13.2. Sample concentrations are reported when all QC criteria for the analysis has been met. Reported results not meeting QC criteria must be qualified with a standard ALS footnote.

13.3. Data Review

Following primary data interpretation and calculations, all data is reviewed by a secondary analyst. Following generation of the report, the report is also reviewed. Refer to ADM-DREV, *Laboratory Data Review Process* for details.

13.4. Reporting

13.4.1. Reports are generated in the MARRS or Harold reporting software by compiling the SMO login, sample prep database, instrument date, and client-specified report requirements (when specified). The compiled data is also used to create EDDs.

13.4.2. As an alternative, reports are generated using Excel© templates located in R:\ICP\FORMS. The analyst should choose the appropriate form and QC pages to correspond to required tier level and deliverables requirements. The results are then transferred, by hand or electronically, to the templates.

14. CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

14.1. Refer to the SOP for *Non Conformance and Corrective Action* (CE-QA008) for procedures for corrective action. Personnel at all levels and positions in the laboratory are to be alert to identifying problems and nonconformities when errors, deficiencies, or out-of-control situations are detected.

14.2. Handling out-of-control or unacceptable data

14.2.1. On-the-spot corrective actions that are routinely made by analysts and result in acceptable analyses should be documented as normal operating procedures, and no specific documentation need be made other than notations in laboratory maintenance logbooks, run-logs, for example.

14.2.2. Some examples when documentation of a nonconformity is required using a Nonconformity and Corrective Action Report (NCAR):

- Quality control results outside acceptance limits for accuracy and precision.
- Method blanks or continuing calibration blanks (CCBs) with target analytes above acceptable levels.
- Sample holding time missed due to laboratory error or operations.
- Deviations from SOPs or project requirements.
- Laboratory analysis errors impacting sample or QC results.
- Miscellaneous laboratory errors (spilled sample, incorrect spiking, etc.).
- Sample preservation or handling discrepancies due to laboratory or operations error.

15. METHOD PERFORMANCE

15.1. Available method performance data is given in the reference method. In addition, this procedure was validated through single laboratory studies of accuracy and precision as in the determinative procedure. The method detection limit(s) and method reporting limit(s) are established for the determinative procedure. See CE-QA011, *Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation* (CE-QA011).

16. POLLUTION PREVENTION AND WASTE MANAGEMENT

16.1. It is the laboratory's practice to minimize the amount of solvents, acids, and reagents used to perform this method wherever feasibly possible. Standards are prepared in volumes consistent with methodology and only the amount needed for routine laboratory use is kept on site. The threat to the environment from solvents and/or reagents used in this method can be minimized when recycled or disposed of properly.

16.2. The laboratory will comply with all Federal, State, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the ALS Lab Waste Management Plan.

16.3. This method uses acid. Waste acid is hazardous to the sewer system and to the environment. All acid waste must be neutralized to a pH of 2.5-12 prior to disposal down the drain. The neutralization step is considered hazardous waste treatment and must be documented on the treatment by generator record. See the ALS Lab Waste Management Plan for details.

17. TRAINING

17.1. Training outline

17.1.1. Review literature (see references section). Read and understand the SOP. Also review the applicable SDS for all reagents and standards used. Following these reviews, observe the procedure performed by an experienced analyst at least three times.

17.1.2. The next training step is to assist in the procedure under the guidance of an experienced analyst. During this period, the analyst is expected to transition from a role of assisting, to performing the procedure with minimal oversight from an experienced analyst.

17.1.3. Perform initial precision and recovery (IPR) study as described above for water samples. Summaries of the IPR are reviewed and signed by the supervisor. Copies may be forwarded to the employee's training file. For applicable tests, IPR studies should be performed in order to be equivalent to NELAC's Initial Demonstration of Capability.

17.2. Training is documented following the *ALS-Kelso Training Procedure* (ADM-TRAIN).

17.2.1. When the analyst training is documented by the supervisor on internal training documentation forms, the supervisor is acknowledging that the analyst has read and understands this SOP and that adequate training has been given to the analyst to competently perform the analysis independently.

18. METHOD MODIFICATIONS

18.1. There are no known differences from the reference method.

19. REFERENCES

19.1. EPA Method 7742, Revision 0, 1994.
19.2. EPA Method 7000B, Revision 2, 1998.
19.3. Standard Methods 3114 B-2009.

20. CHANGES SINCE THE LAST REVISION

20.1. Updated to current ALS Format.
20.2. Signature Page – updated QA Manager name.
20.3. Various typographical, grammar and formatting revisions..
20.4. Section 11.2.1 – revised volumes to reflect current practice.

20.5. Section 11.6.2 – removed extra CCV.
20.6. Section 12.3.2 – New.
20.7. Section 17 – Revised to current format.
20.8. MDLs were removed from Table 1 and a reference to the DQO Table is inserted in section 1.

Table 1

Selenium MRLs

Analyte	Method Reporting Limit
Water	1.0 ug/L
Soil	0.1 mg/Kg (dry)
Tissue	0.1 mg/Kg (dry)

ALS Standard Operating Procedure

DOCUMENT TITLE:	WASTE EXTRACTION (WET) PROCEDURE (STLC) FOR NON-VOLATILE AND SEMI-VOLATILE PARAMETERS
REFERENCED METHOD:	CALIFORNIA CODE OF REGULATIONS, TITLE 22
SOP ID:	MET-STLC
REVISION NUMBER:	3
EFFECTIVE DATE:	1/15/16

ALS-Kelso SOP Annual Review Statement

SOP Code: MET-STLC
Revision: 3

An annual review of the SOP listed was completed on (date): **1-10-17**

☒ The SOP reflects current practices and requires no procedural changes.

Supervisor: L.J. Date: 1-10-17

☐ Revision of the SOP is needed to reflect current practices. Draft revisions are listed below.

SOP Section Number	**Description of Revision Needed**	**Date Procedure Change Implemented**	**Supervisor Initials** Indicating Approval of Revision

WASTE EXTRACTION (WET) PROCEDURE (STLC) FOR NON-VOLATILE AND SEMI-VOLATILE PARAMETERS EPA 7196A, CALIFORNIA CODE OF REGULATIONS, TITLE 22

ALS-KELSO

SOP ID:	MET-STLC	Rev. Number:	3	Effective Date:	1/15/16

Approved By: _____ Date: 12/17/15
Department Supervisor - Jeff Coronado

Approved By: _____ Date: 12/17/15
QA Manager - Carl Degner

Approved By: _____ Date: 12/18/15
Laboratory Director - Jeff Grindstaff

Issue Date: _____ Doc Control ID#: _____ Issued To: _____

ANNUAL REVIEW

SIGNATURES BELOW INDICATE NO PROCEDURAL CHANGES HAVE BEEN MADE TO THE SOP SINCE THE APPROVAL DATE ABOVE. THIS SOP IS VALID FOR TWELVE ADDITIONAL MONTHS FROM DATE OF THE LAST SIGNATURE UNLESS INACTIVATED OR REPLACED BY SUBSEQUENT REVISIONS.

Signature	Title	Date

TABLE OF CONTENTS

WASTE EXTRACTION (WET) PROCEDURE (STLC) FOR NON-VOLATILE AND SEMI-VOLATILE PARAMETERSS

1. SCOPE AND APPLICATION

1.1. The Waste Extraction Test (WET) Procedure (also known as the STLC procedure) is designed to determine the amount of extractable substances in solid and multiphasic wastes. The method involves extracting the waste with a citrate buffer or reagent water under controlled conditions and analyzing the extract for specific, regulated constituents (See Table 1). This information is then used to determine whether a solid waste is classified as a hazardous waste.

1.2. If a total analysis of the waste demonstrates that individual constituents are not present in the waste, or that they are present but at such low concentrations that the appropriate regulatory levels could not possibly be exceeded, the WET procedure need not be performed.

1.3. In cases where there is a project-specific quality assurance plan (QAPP), the project manager identifies and communicates the QAPP-specific requirements to the laboratory. In general, project specific QAPP's supersede method specified requirements. An example of this are projects falling under DoD ELAP. QC requirements defined in the SOP *Department of Defense Projects – Laboratory Practices and Project Management (ADM-DOD/ADM-DOD5)* may supersede the requirements defined in this SOP.

2. METHOD SUMMARY

2.1. This waste sample is first separated into its liquid and solid phases by filtering the waste through a 0.45 µm glass fiber filter. The liquid phase (if any) is stored for later analysis. The particle size of the solid phase is reduced, if necessary, and is then extracted with an amount of extraction fluid equal to 10 times the weight of the solid phase. The extraction fluid employed is a function of the particular analyses that will be performed on the extract. The sample is placed in a rotary extractor and extracted for 48 hours. Following extraction, the liquid extract is separated from the solid phase by filtration through a 0.45 µm glass fiber filter. The liquid extract and the liquid phase are then analyzed separately, or combined (if miscible) and analyzed together.

3. DEFINITIONS

3.1. Batch - A batch of samples is a group of environmental samples that are prepared and/or analyzed together as a unit with the same process and personnel using the same lot(s) of reagents. It is the basic unit for analytical quality control.

3.2. Preparation Batch - A preparation batch is composed of one to twenty field samples, all of the same matrix, meeting the criteria in Section 3.3 and with a maximum time between the start of processing of the first and last samples in the batch to be 24 hours.

3.3. Sample

3.3.1. Field Sample - An environmental sample collected and delivered to the laboratory for analysis; a.k.a., client's sample.

3.3.2. Laboratory Sample - A representative portion, aliquot, or subsample of a field sample upon which laboratory analyses are made and results generated.

3.4. Quality System Matrix - The matrix of an environmental sample is distinguished by its physical and/or chemical state and by the program for which the results are intended. The following sections describe the matrix distinctions. These matrices shall be used for purpose of batch and quality control requirements.

3.4.1. Aqueous - Any groundwater sample, surface water sample, effluent sample, and TCLP or other extract. Specifically excluded are samples of the drinking water matrix and the saline/estuarine water matrix.

3.4.2. Drinking water - Any aqueous sample that has been designated a potable or potential potable water source.

3.4.3. Saline/Estuarine water - Any aqueous sample from an ocean or estuary or other salt-water source.

3.5. Method Blank (MB) - The method blank is an artificial sample composed of analyte-free water or solid matrix and is designed to monitor the introduction of artifacts into the analytical process. The method blank is carried through the entire analytical procedure.

3.6. Laboratory Control Samples (LCS) – The LCS is an aliquot of analyte free water or analyte free solid to which known amounts target analytes are added. The LCS is prepared and analyzed in exactly the same manner as the samples. The percent recovery is compared to established limits and assists in determining whether the batch is in control.

3.7. Matrix Spike/Duplicate Matrix Spike (MS/DMS) Analysis - In the matrix spike analysis, predetermined quantities of target analytes are added to a sample matrix prior to sample preparation and analysis. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the method used for the analysis. Samples are split into duplicates, spiked, and analyzed as a MS/DMS pair. Percent recoveries are calculated for each of the analytes detected. The relative percent difference (RPD) between the duplicate spikes (or samples) is calculated and used to assess analytical precision. The concentration of the spike should be at 3- 5 times the method reporting limit or at levels specified by a project analysis plan.

3.8. Laboratory Duplicates (DUP) – Duplicates are additional replicates of samples that are subjected to the same preparation and analytical scheme as the original sample. The relative percent difference (RPD) between the sample and its duplicate is calculated and used to assess analytical precision.

4. INTERFERENCES

4.1. Potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

5. SAFETY

5.1. All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this procedure. This includes the use of personnel protective equipment, such as, safety glasses, lab coat and the correct gloves.

5.2. Chemicals, reagents and standards must be handled as described in the ALS safety policies, approved methods and in MSDSs where available. Refer to the ALS Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

5.3. Sodium Hydroxide (NaOH) is a strong caustic and a severe health and contact hazard. Use nitrile or latex gloves while handling pellets or preparing solutions.

6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION AND STORAGE

6.1. Samples which will undergo extraction for organic constituents must be collected in glass containers with Teflon lined lids while samples which will undergo extraction for inorganic constituents may be collected in either polyethylene or glass containers.

6.2. All samples should be placed in coolers after collection and stored at 4°C $\pm$ 2°C prior to filtration and extraction. No preservatives are added to the initial sample waste collected for STLC filtration and extraction.

6.3. A minimum of 50g of sample will be needed to perform the STLC extraction. Additional sample volume will be needed if the samples are multiphasic

6.4. The following table indicates the various holding times allowed, from sample collection to the final sample extract analysis:

Analysis Type	Days from Sample Collection To STLC Extraction	Days from STLC Extraction To Preparative Extraction	Days from Preparative Extraction to Analysis
Semi-volatiles	14	7	40
Metals (except Hg)	180	NA	180
Mercury	28	NA	28

7. STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

7.1. Reagent grade chemicals shall be used in all tests. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lowering the accuracy of the determination. The preparation for all laboratory prepared reagents and solutions must be documented in a laboratory logbook. Refer to the SOP *Reagent/Standards Login and Tracking (*ADM-RTL*)* for the complete procedure and documentation requirements.

7.2. All stocks, working solutions and sample dilutions should be prepared using deionized water (DI) conforming to ASTM Type I or ASTM Type II reagent water. For more information on reagent water generation, refer to the related SOP, Operation and Maintenance of Laboratory Reagent Water Systems).

7.3. Sodium Hydroxide (NaOH), 10N.

7.4. Citric Acid monohydrate, granular.

7.5. Nitrogen gas, high purity (99.99%), (used for filtration).

7.6. Extraction fluid # 1 (Sodium Citrate Buffer): Add 168g citric acid monohydrate to 4.0L of reagent water. Adjust the pH with 10N NaOH to 5.0 + 0.1.

7.7. Extraction fluid # 2 (DI water): Use laboratory reagent water as the extraction fluid.

7.8. NOTE: This fluid is used for the determination of hexavalent chromium and other organic/inorganic analyses when requested by the client.

8. APPARATUS AND EQUIPMENT

8.1. Rotary Extractor apparatus: The rotary extractor apparatus must be capable of rotating the extraction vessel in an end-over-end fashion (see Figure 1) at 30 + 2 rpm.

8.2. Extraction Vessel.

8.3. Filtration Device.

8.4. Filter Media: 142 mm glass fiber filters, 0.45 [illegible].Pall Versapor 450

8.5. pH Meter.

8.6. Balance capable of weighing to + 0.01g.

8.7. Magnetic Stirrer w/Teflon coated stirring bar.

8.8. Assorted Laboratory Glassware

9. PREVENTIVE MAINTENANCE

9.1. Maintenance activities are recorded in a maintenance logbook kept for each instrument. Pertinent information (serial numbers, instrument I.D., etc.) must be in the logbook. The entry in the log must include: date of event, the initials of who performed the work, and a reference to analytical control.

10. RESPONSIBILITIES

10.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10.2. It is the responsibility of the department supervisor/manager to document analyst training. Documenting method proficiency is also the responsibility of the department supervisor/manager.

11. PROCEDURE

11.1. Preliminary Evaluation

11.1.1. The sample matrix must first be classified to determine the proper treatment of the sample prior to sample preparation. The sample is classified as either Type I, II, III, or IV as follows:

11.1.2. Type I: If the waste or other material is a mill-able solid, the sample shall be passed through a No. 10 (2 mm) standard sieve prior to sample preparation. If the sample contains any solid particles that will not pass through a No. 10 sieve (i.e. rocks, pebbles, wood or plant debris), they shall be removed and discarded. The solids that remain in the waste or other material shall be milled, if necessary, and passed through a No. 10 sieve. The sample is then extracted.

11.1.3. Type II: If the waste or other material is a filterable mixture of liquid and solids in which the solids constitute 0.5% or greater (by weight) of the entire sample, the liquid and solids shall be separated by filtration through a 0.45 micron filter. The filtrate obtained shall be collected and set aside. The separated solids shall then be passed through a No. 10 standard sieve as described. The solids are then extracted using a ratio of 10 mL of extraction solution per 1 gram of solids. After completion of the solids extraction, the sample extract is then combined with the filtrate previously obtained prior to sample digestion and analysis.

11.1.4. Type III: If the waste or other material is a non-filterable and non-mill-able sludge, slurry, or oily material, no further treatment of the sample is necessary and the sample is extracted. However, if the waste contains any solid particles as described, they shall be removed and discarded prior to extraction.

11.1.5. Type IV: If the waste or other material is a liquid containing less than 0.5% by weight of undissolved solids, the sample shall not be subject to the extraction procedure specified, and will be digested and analyzed as is.

11.2. Extraction Fluid Determination

11.2.1. Extraction Fluid #1 (Sodium Citrate Buffer) is used for most inorganic analyses, with the exception of hexavalent chromium.

11.2.2. Extraction Fluid #2 (DI water) is used for hexavalent chromium and other miscellaneous inorganic analyses (pH, TDS, conductivity) when requested. Most organic analyses also use DI water for the extraction fluid when specified by the client.

11.2.3. Extraction Fluid used for Organic analyses *must be purged with nitrogen* prior to use.

11.3. STLC Extraction

11.3.1. Once the sample has been classified and the proper treatment of the sample has been completed, the sample is the extracted as follows:

11.3.2. 50 grams of sample is placed in a clean polyethylene (for inorganic analytes) or Teflon container (for organic analytes) suitable for use in a rotary extractor. If the sample was classified as Type II, the separated solids shall be extracted using all of the solids available. A similar container shall also be set aside for use as an extraction blank and will be carried through the same procedure as the sample.

11.3.3. In order to provide enough sample extract when performing the DI extraction for organic analyses, additional sample will be needed. Calculate the additional sample weight by determining the total sample extract volume from table below and back calculating using the 10:1 extraction solution: sample weight ratio to obtain the sample weight.

DI WATER EXTRACTION

ANALYSIS	SAMPLE EXTRACT VOLUME (mL)	MS&DUP	MS/MSD
Hexavalent Chromium	50	100	

Semi-Volatiles	1000		2000
Miscellaneous tests (pH, TDS, conductivity)	200	400	

11.3.4. The extraction solution is then added to each container, including the extraction blank, using a volume of 1000 mL for a 100g sample. If the sample was classified as Type II, a ratio of 10 mL of extraction solution per 1 gram of solids is used. The extraction solution will consist of either of sodium citrate buffer or DI water.

11.3.5. The containers will be sealed with tightly fitted caps and agitated using the rotary extractor at 30 $\pm$ 2 rpm for 48 hours with the temperature maintained at 20 - 40°C throughout the extraction process.

11.3.6. After 48 hours has elapsed, the contents of the sample container are then filtered using 0.45 micron membrane filter. Pressure filtration is used as an acceptable alternative to vacuum filtration.

11.4. Sample Analysis

11.4.1. After extraction of the sample matrix is completed, the sample extract is then processed as follows:

11.4.2. Extracts that are to be analyzed for Metals, the sample(s) shall be acidified with nitric acid to five percent by volume after filtration and before the digestion. For extracts requiring the analysis for metal elements (except hexavalent chromium), the sample(s) is digested using Method 3010A (ICP) or 3020A (ICPMS). 10 mL of extract to 50 mL of reagent water before digestion.

11.4.3. For extracts requiring the analysis for Hexavalent Chromium, the sample is analyzed directly using Method 7196A.

11.4.4. For extracts requiring the analysis for inorganic constituents (i.e. TDS, conductivity, pH, etc.), the sample is processed according to the particular analysis method.

12. QA/QC REQUIREMENTS

12.1. This method shall operate under the formal Quality Assurance Program established at ALS and must maintain records that define the quality of data that is generated. Data shall be compared to established criteria in order to determine if the results of the analyses meet the performance characteristics of the method. It is required that an initial demonstration of capability and periodic analysis of laboratory reagent blanks, laboratory fortified blanks, and other QC solutions as a continuing check on performance. The accuracy and precision of the procedure must be validated *before* analyses of samples begin, or whenever significant changes to the procedures have been made.

12.2. Method Preparation Blank - This is STLC extraction fluid processed in exactly the same way as samples. This sample is generated during the analytical process, after extraction and filtration has been performed. A method preparation blank must be processed with each analytical batch or with every 20 samples, whichever is more frequent. It must not contain the target analytes at or above the laboratory reporting limit.

12.3. Matrix Spike - Analyze at least one matrix spike and duplicate sample with each analytical batch or with every 20 samples of similar matrix, whichever is more frequent. A matrix spike and duplicate shall be performed for each waste type (soil, sludge, liquid waste, etc.).The spiked samples must be subjected to the entire sample preparation and analytical process. Matrix spikes are to be added after extraction and filtration.

12.4. Laboratory Control Sample (LCS) - This is STLC extraction fluid which has been spiked with the compounds of interest. This sample is generated during the analytical process, after extraction and filtration has been performed. It must go through the same preparation and analytical steps as samples. At least one LCS must be analyzed with each analytical batch or with every 20 samples, whichever is more frequent.

12.5. Duplicate Analyses (Inorganic analyses only) - At least one set of duplicate samples must be analyzed with each analytical batch or with every 20 samples, whichever is more frequent. The duplicate samples must be subjected to the entire sample preparation and analytical process. The relative percent difference between the duplicate results cannot exceed 20% unless the concentration found in at least one of the duplicates is less than five times the reporting limit.

13. DATA REDUCTION AND REPORTING

13.1. See the analytical method SOPS.

14. CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

14.1. Refer to the SOP for *Non Conformance and Corrective Action* (CE-QA008) for procedures for corrective action. Personnel at all levels and positions in the laboratory are to be alert to identifying problems and nonconformities when errors, deficiencies, or out-of-control situations are detected.

14.2. Handling out-of-control or unacceptable data

14.2.1. On-the-spot corrective actions that are routinely made by analysts and result in acceptable analyses should be documented as normal operating procedures, and no specific documentation need be made other than notations in laboratory maintenance logbooks, run-logs, for example.

14.2.2. Some examples when documentation of a nonconformity is required using a Nonconformity and Corrective Action Report (NCAR):

- Quality control results outside acceptance limits for accuracy and precision.
- Method blanks or continuing calibration blanks (CCBs) with target analytes above acceptable levels.
- Sample holding time missed due to laboratory error or operations.
- Deviations from SOPs or project requirements.
- Laboratory analysis errors impacting sample or QC results.
- Miscellaneous laboratory errors (spilled sample, incorrect spiking, etc.).
- Sample preservation or handling discrepancies due to laboratory or operations error.

15. METHOD PERFORMANCE

15.1. Available method performance data is given in the reference method. In addition, this procedure was validated through single laboratory studies of accuracy and precision as in the determinative procedure. The method detection limit(s) and method reporting limit(s) are established for the determinative procedure. See CE-QA011, *Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation.*

16. POLLUTION PREVENTION AND WASTE MANAGEMENT

16.1. It is the laboratory's practice to minimize the amount of solvents, acids, and reagents used to perform this method wherever feasibly possible. Standards are prepared in volumes consistent with methodology and only the amount needed for routine laboratory use is kept on site. The threat to the environment from solvents and/or reagents used in this method can be minimized when recycled or disposed of properly.

16.2. The laboratory will comply with all Federal, State, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the ALS Environmental Health and Safety Manual.

16.3. This method uses acid/bases. Waste acid/base is hazardous to the sewer system and to the environment. All waste must be neutralized to a pH of 2.5-12 prior to disposal down the drain. The neutralization step is considered hazardous waste treatment and must be documented on the treatment by generator record. See the ALS EH&S Manual for details.

17. TRAINING

17.1. Training outline

17.1.1. Review literature (see references section). Read and understand the SOP. Also review the applicable MSDS for all reagents and standards used. Following these reviews, observe the procedure performed by an experienced analyst at least three times.

17.1.2. The next training step is to assist in the procedure under the guidance of an experienced analyst. During this period, the analyst is expected to transition from a role of assisting, to performing the procedure with minimal oversight from an experienced analyst.

17.1.3. Perform initial precision and recovery (IPR) study as described above for water samples. Summaries of the IPR are reviewed and signed by the supervisor. Copies may be forwarded to the employee's training file. For applicable tests, IPR studies should be performed in order to be equivalent to NELAC's Initial Demonstration of Capability.

17.2. Training is documented following the ADM-TRAIN, *ALS-Kelso Training Procedure*.

17.2.1. When the analyst training is documented by the supervisor on internal training documentation forms, the supervisor is acknowledging that the analyst has read and understands this SOP and that adequate training has been given to the analyst to competently perform the analysis independently.

18. METHOD MODIFICATIONS

18.1. Extracts intended for organics analyses are refrigerated until preparative extraction.

18.2. Teflon extraction containers are used to reduce the potential for breakage. Containers are washed in hot, soapy water and rinsed with acetone: hexane, HCl, and DI water prior to use.

18.3. Extracts are acidified per analytical method requirements.

19. REFERENCES

19.1. California Code of Regulations, Title 22, Section 66261.126 Appendix II, May 2005.

19.2. SW-846 3rd Edition, Update III, December 1996.

19.3. Table 1: List of Inorganic and Organic Persistent and Bioaccumulative Toxic Substances and Their Soluble Threshold limit Concentration (STLC).

19.4. Figure 1: Rotary Extractor apparatus.

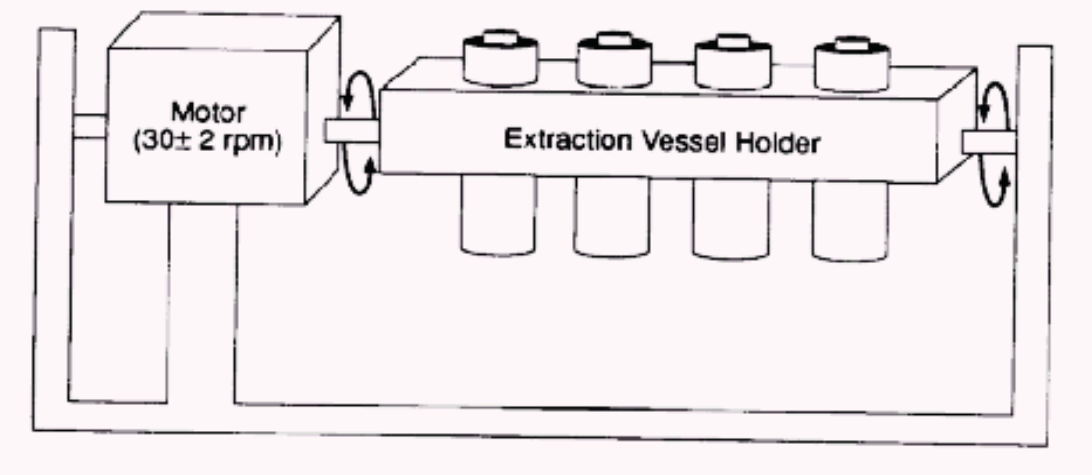
20. CHANGES SINCE THE LAST REVISION

20.1. Minor formatting corrections to enhance readability.
20.2. Section 1.4: Added section on DOD/ QAPP and Project Management.
20.3. Section 7: Included water quality paragraph.
20.4. Section 11.4.2: Added change request dated 12/14/15.
20.5. Section 11.3.2: Sample amount change request dated 12/14/15.
20.6. Section 3: Updated Definitions Section.

Table 1 - List of Inorganic and Organic Persistent and Bioaccumulative Toxic Substances and Their Soluble Threshold limit Concentration (STLC)

Constituents	Regulator level (mg/L)
Antimony and/or antimony compounds	15
Arsenic and/or arsenic compounds	5.0
Barium and/or barium compounds	100.0
Beryllium and/or beryllium compounds	0.75
Cadmium and/or cadmium compounds	1.0
Chromium (VI) compounds	5.0
Chromium and/or chromium (III) compounds	560
Cobalt and/or cobalt compounds	80
Copper and/or copper compounds	25
Fluoride salts	180
Lead and/or lead compounds	5.0
Mercury and/or mercury compounds	0.2
Molybdenum and/or molybdenum compounds	350
Nickel and/or nickel compounds	20
Selenium and/or selenium compounds	1.0
Silver and/or silver compounds	5.0
Thallium and/or thallium compounds	7.0
Vanadium and/or vanadium compounds	24
Zinc and/or zinc compounds	250
Aldrin	0.14
Chlordane	0.25
DDT,DDE,DDD	0.1
2,4-Dichlorophenoxyacetic acid (2,4-D)	10
Dieldrin	0.8
Dioxin (2,3,7,8-TCDD)	0.001
Endrin	0.02
Heptachlor	0.47
Kepone	2.1
Lindane (gamma-BHC)	0.4
Methoxychlor	10.0
Mirex	2.1
Pentachlorophenol	1.7
Polychlorinated biphenyls (PCBs)	5.0
Toxaphene	0.5
Trichloroethylene	204
2,4,5-Trichlorophenoxypropionic acid (2,4,5-TP) (Silvex)	1.0

Table 1



ALS Standard Operating Procedure

DOCUMENT TITLE:	SAMPLE PREPARATION OF BIOLOGICAL TISSUE FOR METALS ANALYSIS BY ICP-OES, AND ICP-MS
REFERENCED METHOD:	
SOP ID:	MET-TDIG
REVISION NUMBER:	5
EFFECTIVE DATE:	5/29/16

ALS-Kelso SOP Annual Review Statement

SOP Code: MET-TDIG
Revision: 5

An annual review of the SOP listed was completed on (date): _____

☐ The SOP reflects current practices and requires no procedural changes.

Supervisor: Date:

☒ Revision of the SOP is needed to reflect current practices. Draft revisions are listed below.

SOP Section Number	**Description of Revision Needed**	**Date Procedure Change Implemented**	**Supervisor Initials** Indicating Approval of Revision
13.6.1	Change weighed to the nearest 0.1 mg. Weighed to the nearest 1.00 mg.	L.J.	10-25-17

ALS-Kelso SOP Annual Review Statement

SOP Code: MET-TDIG
Revision: 5

An annual review of the SOP listed was completed on (date): **5-11-17**

☒ The SOP reflects current practices and requires no procedural changes.

Supervisor: L.J. Date: 5-11-17

☐ Revision of the SOP is needed to reflect current practices. Draft revisions are listed below.

SOP Section Number	**Description of Revision Needed**	**Date Procedure Change Implemented**	**Supervisor Initials** Indicating Approval of Revision

SAMPLE PREPARATION OF BIOLOGICAL TISSUE FOR METALS ANALYSIS BY CVAA, ICP-OES, AND ICP-MS

ALS-KELSO

SOP ID:	MET-TDIG	Rev. Number:	5	Effective Date:	5/29/2016

Approved By: _____ Date: 5/16/16
Department Supervisor/Technical Director – Jeff Coronado

Approved By: _____ Date: 5/16/16
QA Manager – Carl Degner

Approved By: _____ Date: 5/17/16
Laboratory Director – Jeff Grindstaff

Issue Date: _____	Doc Control ID#: _____	Issued To: _____

ANNUAL REVIEW

SIGNATURES BELOW INDICATE NO PROCEDURAL CHANGES HAVE BEEN MADE TO THE SOP SINCE THE APPROVAL DATE ABOVE. THIS SOP IS VALID FOR TWELVE ADDITIONAL MONTHS FROM DATE OF THE LAST SIGNATURE UNLESS INACTIVATED OR REPLACED BY SUBSEQUENT REVISIONS.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

TABLE OF CONTENTS

SAMPLE PREPARATION OF BIOLOGICAL TISSUE FOR METALS ANALYSIS BY CVAA, ICP-OES, AND ICP-MS

1. SCOPE AND APPLICATION

1.1. This procedure describes techniques used for sample preparation and acid digestion of biological tissue samples. This procedure is applicable to the analysis of biological tissue for heavy metals. The procedure provides a convenient and efficient digestion/dissolution technique which allows for the simultaneous or sequential analysis of the sample for metals. The digestates may be analyzed by Inductively Coupled Plasma-Optical Emission Spectroscopy (ICP-OES), Inductively Coupled Plasma-Mass Spectrometry (ICP-MS), or Cold Vapor Atomic Absorption (CVAA). The procedure includes, but is not restricted to, the metals listed in Table 1.

2. METHOD SUMMARY

2.1. A representative tissue sample is lyophilized, blended, then sub-sampled for conventional oven digestion. Oxidation is brought about by the use of concentrated nitric acid in a Teflon closed vessel. The digestate is then analyzed for metallic constituents by CVAA, ICP-OES, or ICP-MS methods.

3. .DEFINITIONS

3.1. Batch - A batch of samples is a group of environmental samples that are prepared and/or analyzed together as a unit with the same process and personnel using the same lot(s) of reagents. It is the basic unit for analytical quality control.

3.2. Preparation Batch - A preparation batch is composed of one to twenty field samples, all of the same matrix, meeting the criteria in Section 3.3 and with a maximum time between the start of processing of the first and last samples in the batch to be 24 hours.

3.3. Sample

3.3.1. Field Sample - An environmental sample collected and delivered to the laboratory for analysis; a.k.a., client's sample.

3.3.2. Laboratory Sample - A representative portion, aliquot, or subsample of a field sample upon which laboratory analyses are made and results generated.

3.4. Quality System Matrix - The matrix of an environmental sample is distinguished by its physical and/or chemical state and by the program for which the results are intended. The following sections describe the matrix distinctions. These matrices shall be used for purpose of batch and quality control requirements.

3.4.1. Aqueous - Any groundwater sample, surface water sample, effluent sample, and TCLP or other extract. Specifically excluded are samples of the drinking water matrix and the saline/estuarine water matrix.

3.4.2. Drinking water - Any aqueous sample that has been designated a potable or potential potable water source.

3.4.3. Saline/Estuarine water - Any aqueous sample from an ocean or estuary or other salt-water source.

3.5. Method Blank (MB) - The method blank is an artificial sample composed of analyte-free water or solid matrix and is designed to monitor the introduction of artifacts into the analytical process. The method blank is carried through the entire analytical procedure.

3.6. Laboratory Control Samples (LCS) – The LCS is an aliquot of analyte free water or analyte free solid to which known amounts target analytes are added. The LCS is prepared and analyzed in exactly the same manner as the samples. The percent recovery is compared to established limits and assists in determining whether the batch is in control.

3.7. Matrix Spike/Duplicate Matrix Spike (MS/DMS) Analysis - In the matrix spike analysis, predetermined quantities of target analytes are added to a sample matrix prior to sample preparation and analysis. The purpose of the matrix spike is to evaluate the effects of the sample matrix on the method used for the analysis. Samples are split into duplicates, spiked, and analyzed as a MS/DMS pair. Percent recoveries are calculated for each of the analytes detected. The relative percent difference (RPD) between the duplicate spikes (or samples) is calculated and used to assess analytical precision. The concentration of the spike should be at 3- 5 times the method reporting limit or at levels specified by a project analysis plan.

3.8. Laboratory Duplicates (DUP) – Duplicates are additional replicates of samples that are subjected to the same preparation and analytical scheme as the original sample. The relative percent difference (RPD) between the sample and its duplicate is calculated and used to assess analytical precision.

3.9. Standard Reference Material (SRM**)** – A material with specific certification criteria and is issued with a certificate or certificate of analysis that reports the results of its characterizations and provides information regarding the appropriate use(s) of the material. An SRM is prepared and used for three main purposes: (1) to help develop accurate methods of analysis; (2) to calibrate measurement systems used to facilitate exchange of goods, institute quality control, determine performance characteristics, or measure a property at the state-of-the-art limit; and (3) to ensure the long-term adequacy and integrity of measurement quality assurance programs.

4. INTERFERENCES

4.1. Refer to the determinative method for a discussion of interferences.

5. SAFETY

5.1. All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this procedure. This includes the use of personnel protective equipment, such as, safety glasses, lab coat and the correct gloves.

5.2. Chemicals, reagents and standards must be handled as described in the ALS safety policies, approved methods and in MSDSs where available. Refer to the ALS Environmental, Health and Safety Manual and the appropriate MSDS prior to beginning this method.

5.3. Nitric and hydrochloric acids are used in this method. Acids are extremely corrosive and care must be taken while handling. A face shield should be used while pouring acids. And safety glasses should be worn while working with the solutions. Lab coat and gloves should always be worn while working with these solutions.

6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION AND STORAGE

6.1. Samples are typically collected in plastic containers and iced or refrigerated at 4 ± 2ºC from time of collection until preparation or analysis. Sample may be frozen at ≤-10ºC or as specified by project requirements.

7. STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

7.1. Reagent grade chemicals shall be used in all tests. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lowering the accuracy of the determination. The preparation for all laboratory prepared reagents and solutions must be documented in a laboratory logbook. Refer to the SOP *Reagent/Standards Login and Tracking (ADM-RTL)* for the complete procedure and documentation requirements.

7.2. All stocks, working solutions and sample dilutions should be prepared using deionized water (DI) conforming to ASTM Type I or ASTM Type II reagent water. For more information on reagent water generation, refer to the related SOP, Operation and Maintenance of Laboratory Reagent Water Systems.

7.2.1. Reagent water - ASTM Type I or Type II water

7.3. Concentrated nitric acid.

7.4. Standards

7.4.1. Stock standard solutions may be purchased from a number of vendors. All reference standards, where possible, must be traceable to SI units or NIST certified reference materials. The vendor-assigned expiration date is used.

7.4.2. Metals spiking solutions: Five solutions are needed to prepare the matrix spiking standards: SS1, SS2, SS3, SS4, and SS5.

7.4.3. Follow the formulations laid out in Table 2. These solutions are prepared in acid rinsed Class A volumetric flasks using purchased custom mixed standards or 1000 ppm single analyte standards. Aliquots are made using acid rinsed Class A volumetric pipettes of the appropriate size.

7.4.4. SS1 (Al, Ag, Ba, Be, Cd, Co, Cr, Cu, Fe, Pb, Mn, Ni, Sb, V, and Zn): Fill a 1000 mL volumetric flask approximately half full with reagent water, add 50 mL of nitric acid and mix. Next add 100 mL of the custom mixed standard (CAS-CAL-14) purchased from "Inorganic Ventures". In addition add 50 mL of 1000 ppm Antimony. Dilute to volume with reagent water, mix thoroughly and transfer to a 1000 mL Teflon bottle for storage. The solution expiration date is determined by the earliest expiration date of any single component in the solution.

7.4.5. SS2 (As, Cd, Pb, Se, Tl and Cu): Fill a 500 mL volumetric flask approximately half full with reagent water, add 25 mL of nitric acid and mix. Next add 2.0 mL each of 1000 ppm Arsenic, Cadmium, Lead, Selenium, Thallium and Copper. Dilute to volume with reagent water, mix thoroughly and transfer to a 500 mL Teflon bottle for storage. The solution expiration date is determined by the earliest expiration date of any single component in the solution.

7.4.6. SS3 (Hg, As, Se, and Tl): Fill a 500 mL volumetric flask approximately half full with reagent water, add 25 mL of nitric acid and mix. Next add 50 mL each of 1000 ppm Arsenic, Selenium, and Thallium. Dilute to volume with reagent water, mix thoroughly and transfer to a 500 mL Teflon bottle for storage. The solution expiration date is determined by the earliest expiration date of any single component in the solution. For mercury analysis, add 6 mL of 1000 ppm Hg.

7.4.7. SS4 (B, Mo): Fill a 500 mL volumetric flask approximately half full with reagent water, add 25 mL of nitric acid and mix. Next add 50 mL each of 1000 ppm Boron and Molybdenum. Dilute to volume with reagent water, mix thoroughly and transfer to a 500 mL Teflon bottle for storage. The solution's expiration date is determined by the earliest expiration date of any single component in the solution.

7.4.8. SS5 (K, Na, Mg, Ca): Fill a 200 mL volumetric flask approximately half full with reagent water, add 10 mL of nitric acid and mix. Next, add 20 mL of 10000 ppm Potassium, Sodium, Magnesium, Calcium. Dilute to volume with reagent water, mix thoroughly and transfer to a Teflon bottle for storage. The solution expiration date is determined by the earliest expiration date of any single component in the solution.

8. PREVENTIVE MAINTENANCE

8.1. All maintenance activities are recorded in a maintenance logbook kept for the freeze drying instrument. Pertinent information (serial numbers, instrument I.D., etc.) must be in the logbook. Maintenance entries should include date, symptom of problem, corrective actions, and description of maintenance, date, and name. The log should contain a reference to return to analytical control.

8.2. Centrifuge tubes must be thoroughly pre-cleaned with 1:4 HCl, and rinsed with DI water. All laboratory equipment used for trace metals analysis shall be stored in the clean room, and shall not be used for any other purpose.

8.3. Routine cleaning of the sample handling and digestion apparatus is necessary. Refer to the SOP for Metals Laboratory Glassware Cleaning.

9. RESPONSIBILITIES

9.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

9.2. It is the responsibility of the department supervisor/manager to document analyst training. Documenting method proficiency, as described in the SOP: *ALS-Kelso Training Procedure* (ADM-TRAIN), is also the responsibility of the department supervisor/manager.

9.3. When new or unfamiliar situations develop, it is also the responsibility of the employee to immediately notify the section supervisor, project manager, or the laboratory director for quick resolution of all issues. Due to the nature of the work, it is important that all work be documented, department supervisors, project managers and when necessary, the client, be contacted immediately when questions arise.

10. APPARATUS AND EQUIPMENT

10.1. Lyophilizing apparatus, LABCONCO Model 7948040 freeze dryer.

10.2. Conventional laboratory oven capable of precise temperature control at 105°C.

10.3. Analytical balance capable of weighing to 0.1 mg.

10.4. 50 mL graduated poly tubes.

11. PREVENTIVE MAINTENANCE

11.1. All maintenance activities are recorded in a maintenance logbook kept for the freeze drying instrument. Pertinent information (serial numbers, instrument I.D., etc.) must be in the logbook. Maintenance entries should include date, symptom of problem, corrective actions, and description of maintenance, date, and name. The log should contain a reference to return to analytical control.

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11.3. Routine cleaning of the sample handling and digestion apparatus is necessary. Refer to the SOP for Metals Laboratory Glassware Cleaning.

12. RESPONSIBILITIES

12.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

12.2. It is the responsibility of the department supervisor/manager to document analyst training. Documenting method proficiency is also the responsibility of the department supervisor/manager.

13. PROCEDURE

13.1. Obtain a representative tissue sample that will yield ~ 300 mg of freeze-dried solids.

Note: This is designed to be a general guideline. Approximately 300 mg of dry sample is typically required to obtain the desired detection limits that often are necessary for tissue analysis.

13.2. Slice the sample into thin pieces prior to filling the drying vessel. Homogenize the sample before freeze drying.

13.3. Cap the vessel and freeze the sample in a conventional freezer. Note: Using the Labconco Model 7948040 freeze dryer, the samples do not need to be frozen prior to freeze drying.

13.4. When the sample is frozen, remove from freezer and follow the manufacturer's instructions for operation of the freeze *- dryer.

13.5. Blend the dry solids to obtain a homogeneous sample. The sample may be stored dry until digestion.

13.6. Sample Digestion

13.6.1. Transfer 300 mg of dried sample, weighed to the nearest 0.1 mg, to a 50 mL decomposition vessel.

13.6.2. Add 4.5mL concentrated nitric acid to the vessel.

13.6.3. Follow the manufacturer's instructions for the use of the decomposition vessels. Place the vessel in a conventional oven at 105°C for a minimum of 12 hours. If using the conventional oven option, monitor oven temperatures for each batch and record this data onto the appropriate benchsheet.

13.6.4. Cool the vessel, open to relieve pressure and vent the gases. If Antimony, Silver or Tin are target analytes, then add 10 ml of deionized water to the

cooled vessel. Re-cap and tighten the vessel. Return to the oven for an additional hour.

13.6.5. Cool the vessel, open to relieve pressure and vent the gasses. Transfer the sample to a volumetric container and dilute to 30 ml. The sample is ready for analysis.

14. QA/QC REQUIREMENTS

14.1. Initial Precision and Recovery Validation

14.2. The accuracy and precision of the procedure must be validated before analyses of samples begin, or whenever significant changes to the procedures have been made. To do this, four water samples are spiked with the LCS spike solution, then prepared and analyzed using the applicable method.

14.3. Ongoing QC Samples required are described in the ALS-Kelso Quality Assurance Manual and in the SOP for Sample Batches. Additional QC Samples may be required in project specific quality assurance plans (QAPP). General QA requirements for DoD QSM are defined in the laboratory SOP, Department *of Defense Projects – Laboratory Practices and Project Management (ADM-DOD; ADM-DOD5).* General QC Samples are:

14.3.1. Method Blank

14.3.1.1. A method blank is extracted and analyzed with every batch of 20 (or fewer) samples to demonstrate that there are no method interferences. If the method blank shows any hits above the reporting limit, corrective action must be taken. Corrective action includes recalculation, reanalysis, system cleaning, or re-extraction and reanalysis. For some project specific needs, exceptions may be noted and method blank results above the MRL may be reported for common lab contaminants.

14.3.2. Lab Control Sample (LCS)

14.3.2.1. The laboratory control sample is composed of both a SRM and a laboratory created sample. The laboratory created LCS is created by spiking the elements defined in the project plan into a digestion vessel. The LCS is designed to monitor the accuracy of the procedure.

14.3.2.2. Prepare one laboratory control samples (LCS) and a duplicate laboratory control sample (DLCS) with every batch of 20 (or fewer) samples whichever is more frequent.

14.3.2.3. Analyze a standard reference material (SRM) at 5% frequency or one per batch, whichever is more frequent. Standard Reference Material (SRM's) should be representative of the tissue sample being analyzed.

14.3.3. Sample Duplicate

14.3.3.1. Samples analyzed by methods 6010, 6020 or GFAA require one sample duplicate at with every batch of 20 (or fewer) samples. Methods 200.7 and 200.8 require one sample duplicate at with every batch of 10 (or fewer) samples.

14.3.4. Matrix Spike

14.3.4.1. A matrix spike (MS) is prepared and analyzed with every batch of 20 (or fewer) samples if analyzed by methods 6010, 6020 or GFAA. Methods 200.7 and 200.8 require one sample duplicate at with every batch of 10 (or fewer) samples.

14.3.4.2. The MS is prepared by adding a known volume of the matrix spike solution to the sample and determining the spiked sample concentration. Spikes should be added directly to the dry sample. Spike solutions should be multi-element with analyte concentrations high enough to minimize volume added to sample.

15. DATA REDUCTION AND REPORTING

15.1. Digestion data sheets, including weights and volumes used are completed and a batch lot number is assigned and attached to the data sheet. The Manufacturer's lot numbers for the reagents used are added to the digestion data sheet.

15.2. Spiking sheets are completed including all spike data and volumes of spiking solutions used.

15.3. Following primary data interpretation and calculations, all data is reviewed by a secondary analyst. Following generation of the report, the report is also reviewed. Refer to the *SOP for Laboratory Data Review Process* for details. The person responsible for final review of the data report and/or data package should assess the overall validity and quality of the results and provide any appropriate comments and information to the Project Chemist to inclusion in the report narratives.

16. CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

16.1. Refer to the SOP for *Non Conformance and Corrective Action* (CE-QA008) for procedures for corrective action. Personnel at all levels and positions in the laboratory are to be alert to identifying problems and nonconformities when errors, deficiencies, or out-of-control situations are detected.

16.2. Handling out-of-control or unacceptable data

16.2.1. On-the-spot corrective actions that are routinely made by analysts and result in acceptable analyses should be documented as normal operating procedures, and no specific documentation need be made other than notations in laboratory maintenance logbooks, run-logs, for example.

16.2.2. Some examples when documentation of a nonconformity is required using a Nonconformity and Corrective Action Report (NCAR):

- Quality control results outside acceptance limits for accuracy and precision.
- Method blanks or continuing calibration blanks (CCBs) with target analytes above acceptable levels.
- Sample holding time missed due to laboratory error or operations.
- Deviations from SOPs or project requirements.
- Laboratory analysis errors impacting sample or QC results.
- Miscellaneous laboratory errors (spilled sample, incorrect spiking, etc.).
- Sample preservation or handling discrepancies due to laboratory or operations error.

17. METHOD PERFORMANCE

17.1. Refer to the Determinative Methods.

18. POLLUTION PREVENTION AND WASTE MANAGEMENT

18.1. It is the laboratory's practice to minimize the amount of solvents, acids, and reagents used to perform this method wherever feasibly possible. Standards are prepared in volumes consistent with methodology and only the amount needed for routine laboratory use is kept on site. The threat to the environment from solvents and/or reagents used in this method can be minimized when recycled or disposed of properly.

18.2. The laboratory will comply with all Federal, State, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the ALS Environmental Health and Safety Manual.

18.3. This method uses acid. Waste acid is hazardous to the sewer system and to the environment. All acid waste must be neutralized to a pH of 2.5-12 prior to disposal down the drain. The neutralization step is considered hazardous waste treatment and must be documented on the treatment by generator record. See the ALS EH&S Manual for details.

19. TRAINING

19.1. Training outline

19.1.1. Review literature (see references section). Read and understand the SOP. Also review the applicable MSDS for all reagents and standards used. Following these reviews, observe the procedure performed by an experienced analyst at least three times.

19.1.2. The next training step is to assist in the procedure under the guidance of an experienced analyst. During this period, the analyst is expected to transition from a role of assisting, to performing the procedure with minimal oversight from an experienced analyst.

19.1.3. Perform initial precision and recovery (IPR) study as described above for water samples. Summaries of the IPR are reviewed and signed by the supervisor. Copies may be forwarded to the employee's training file. For applicable tests, IPR studies should be performed in order to be equivalent to NELAC's Initial Demonstration of Capability.

19.2. Training is documented following the SOP for Documentation of Training.

19.2.1. When the analyst training is documented by the supervisor on internal training documentation forms, the supervisor is acknowledging that the analyst has read and understands this SOP and that adequate training has been given to the analyst to competently perform the analysis independently.

20. METHOD MODIFICATIONS

20.1. Not applicable.

21. REFERENCES

21.1. Recommended Guidelines for Measuring Metals in Puget Sound Marine Water, Sediment, and Tissue Samples; April 1997.

22. CHANGES SINCE THE LAST REVISION

22.1. Signature page: Updated Quality Assurance Manager.
22.2. Updated SOP references throughout the document.
22.3. Updated SOP to the current ALS SOP format.
22.4. Removed GFAA prep/analysis throughout the text of the document.
22.5. Added Hg prep/analysis throughout the text of the document.
22.6. Removed GFAA and inserted CVAA into Table 1.
22.7. Section 7.4.6: Added Hg.

Table 1

Selected Elements and Analysis Procedures for Tissue Samples

Element	CVAA	ICP-OES	ICP-MS
Aluminum		√	√
Antimony		√	√
Arsenic			√
Barium		√	√
Beryllium		√	√
Boron		√	
Cadmium		√	√
Calcium		√	
Chromium		√	√
Cobalt		√	√
Copper		√	√
Iron		√	
Lead		√	√
Lithium		√	
Magnesium		√	
Manganese		√	√
Mercury	√		
Molybdenum		√	√
Nickel		√	√
Potassium		√	
Phosphorus		√	
Selenium			
Silver		√	√
Sodium		√	
Strontium		√	
Thallium		√	√
Tin		√	√
Vanadium		√	
Zinc		√	√

Table 2

METALS SPIKING SOLUTIONS CONCENTRATIONS FORM

Solution Name	Element	mLs of 1000ppm Solution	Final Volume	Solution Conc. mg/L	Concentration in the digest mg/L
SS1	HNO3	50.0	1000ml	-	
	Al	100*	1000ml	200	2
	Ag	100*	1000ml	5	0.05
	Ba	100*	1000ml	200	2
	Be	100*	1000ml	5	0.05
	Cd	100*	1000ml	5	0.05
	Co	100*	1000ml	50	0.5
	Cr	100*	1000ml	20	0.2
	Cu	100*	1000ml	25	0.25
	Fe	100*	1000ml	100	1
	Pb	100*	1000ml	50	0.5
	Mn	100*	1000ml	50	0.5
	Ni	100*	1000ml	50	0.5
	Sb	50	1000ml	50	0.5
	V	100*	1000ml	50	0.5
	Zn	100*	1000ml	50	0.5
SS2 GFAA SPIKE	HNO3	25.0	500ml	-	
	As	2.0	500ml	4	0.04
	Cd	2.0	500ml	4	0.04
	Pb	2.0	500ml	4	0.04
	Se	2.0	500ml	4	0.04
	Tl	2.0	500ml	4	0.04
	Cu	2.0	500ml	4	0.04
SS3	HNO3	25.0	500ml	-	
	As	50.0	500ml	100	1
	Se	50.0	500ml	100	1
	Tl	50.0	500ml	100	1
SS4	HNO3	25	500ml	-	
	B	50	500ml	100	1
	Mo	50	500ml	100	1
SS5	HNO3	10.0	200ml	-	
	K**	20	200ml	1000	10
	Na**	20	200ml	1000	10
	Mg**	20	200ml	1000	10
	Ca**	20	200ml	1000	10

Operation of the Freeze Drier Labconco 7948040

Operation Checklist

The following checklist should be followed after each use of the Stoppering Tray Dryer.

1. Wipe out the interior of the Stoppering Tray Dryer chamber with a paper towel to remove any moisture or debris.
2. Wipe the interior of the collector chamber (base unit)of the freeze dry system with a paper towel to remove any accumulated moisture.
3. Check the collector chamber drain hose on the freeze dry system (base unit) to ensure that the hose is free of moisture and that the drain plug is securely installed.
4. Using a paper towel, wipe the freeze dry system collector chamber (base unit) lid gasket and the Stoppering Tray Dryer door gasket to remove any dirt and contaminants that could cause a vacuum leak. Vacuum grease is not required on the door gasket or collector lid gasket to obtain proper vacuum seal.

Starting the Freeze Dry cycle

1. On the left side of the Stoppering Tray Dryer unit flip the black switch to the on position. On the left side of the base unit flip the black switch to the on position. On the base unit that the condensing coil is located push the manual button. The collector temperature must reach -40˚C before the vacuum pump can be turned on.

2. Place the containers holding the pre-frozen samples onto the trays in the Stoppering Tray Dryer unit on top of the base unit. When closing the door pick up left hand bottom corner of door while closing. Turn the door handle all the way to the right. The door should be covering the entire black gasket attached to the Stoppering tray unit. Make sure that the vacuum release valve on Stoppering Tray Dryer unit is turned to the closed position. Press the mode (see attached diagram) button until the green light is on for Automatic on the Stoppering Tray Dryer unit. Press the run stop button on the Stoppering Tray Dryer unit P1 will appear in the display and the amber led button by the run stop button will light. Once the Stoppering Tray Dryer unit reaches -40˚C it will hold at that temperature for one hour and forty eight minutes. After one hour and forty eight minutes the vacuum pressure in the display should decrease. If the vacuum is not decreasing press the vacuum on the collector unit button and open and close the door lifting up the bottom left hand corner of the door while closing. Turn the door handle all the way to the right. Press the vacuum button to restart the vacuum.

3. When the vacuum pressure (mBar) is the same on the Stoppering tray dryer and the base unit the samples have finished the freeze drying process (typically after 48 hours, depending on mass of tissue). Press the vacuum button on the collector unit to turn the vacuum pump off. Flip the switch on the side of the base unit to off. Flip the switch on the side of the Stoppering Tray Dryer unit to off. Open the chamber by moving the vacuum release control on the front of the Stoppering Tray Dryer unit to the open position. When the sound of air through the back fill port is no longer audible, the chamber door is ready to open.

Alarms

A number of unusual events may occur during a lyophilization procedure that can adversely affect the operation of the Stoppering Tray Dryer. If an event occurs, and the beeper will sound. The beeper will automatically mute itself after one minute. The specific alarm can be identified observing the display. The following "out of specification" conditions will initiate an alarm:

System Temperature Variations
Once the system temperature has stabilized for 20 minutes, if the manual set point temperature or automatic hold temperature varies more than ±2˚C as measured by the system temperature sensor, the Red Alarm indicator and word "TMP" on the display will flash until the end of the run.

Vacuum
Once the system vacuum is low and stabilize at a point where it changes less than 0.020mBar in 5 minutes if the vacuum changes more than 0.500mBar , the red Alarm indicator and the word "VAC" on the display will flash until the end of the run.

System Temperature Set Point
If during a ramp mode the system temperatures stabilize without reaching the set point temperature, the control will enter the next Hold mode. The Red Alarm indicator will flash and the program indicator "Px" on the display will flash until the end of the run.

Power Failure
If a power failure occurs while a run in is progress, the Red Alarm indicator and Run/Stop indicator will flash when the power in restored. Once the power is restored, the process will continue as programmed until completion. Pressing Run/Stop cancels the flashing warning.

Vacuum Pump

The oil in the Vacuum pump should be checked before every use. It must be changed if it is cloudy, shows particles or is discolored. The useful life of the vacuum pump oil can be extended if the vacuum pump is operated for an extended period of time after a freeze dry run.

Changing the Vacuum pump oil:

Pump oil is located in the ICPMS Instrument Laboratory.

1. Make sure the Vacuum pump is turned off
2. Remove the front cover of the bottom unit of the freeze drying unit by pressing up on the metal clasp located on the bottom of the front cover in the middle. Use a small screwdriver.
3. Slide the Pump out far enough to allow a 1 L plastic container to placed under the drain on the front of the Vacuum pump.
4. Remove the Grey cap on top of the Vacuum pump.
5. Remove the Grey plug on the bottom of the Vacuum pump.
6. After all of the used oil has drained out of the pump, pour a small amount of the unused oil into the vacuum pump with the drain plug removed to rinse out the oil reservoir. Place the used oil in the **Used Oil** container located in the ICPMS Instrumentation Laboratory.

Place the drain plug on the vacuum pump (hand tighten) fill the pump up with approx 400 mls of oil the visible oil should be 1 inch from the top mark on the outside of the vacuum pump. Screw the grey cap back onto the top of the vacuum pump (hand tighten)

ALS Standard Operating Procedure

DOCUMENT TITLE:	TISSUE SAMPLE PREPARATION
REFERENCED METHOD:	N/A
SOP ID:	MET-TISP
REVISION NUMBER:	11
EFFECTIVE DATE:	2/23/17

ALS-Kelso SOP Annual Review Statement

SOP Code: MET-TISP
Revision: 11

An annual review of the SOP listed was completed on (date): _____

☐ The SOP reflects current practices and requires no procedural changes.

Supervisor: Date:

☒ Revision of the SOP is needed to reflect current practices. Draft revisions are listed below.

SOP Section Number	**Description of Revision Needed**	**Date Procedure Change Implemented**	**Supervisor Initials** Indicating Approval of Revision
11.2.2.2	Talc free Vinyl gloves remove nitrile.	3-9-17	L.J.
11.2.2.3	Remove A and remove Vinyl glove.	3-9-17	L.J.

TISSUE SAMPLE PREPARATION

ALS-KELSO

SOP ID:	MET-TISP	Rev. Number:	11	Effective Date:	2/23/17

Approved By: _____ Date: 2/14/17
Department Manager/Technical Director – Jeff Coronado

Approved By: _____ Date: 2/14/17
QA Manager - Carl Degner

Approved By: _____ Date: 2/14/17
Laboratory Director – Jeff Grindstaff

Issue Date: _____ Doc Control ID#: _____ Issued To: _____

ANNUAL REVIEW

SIGNATURES BELOW INDICATE NO PROCEDURAL CHANGES HAVE BEEN MADE TO THE SOP SINCE THE APPROVAL DATE ABOVE. THIS SOP IS VALID FOR TWELVE ADDITIONAL MONTHS FROM DATE OF THE LAST SIGNATURE UNLESS INACTIVATED OR REPLACED BY SUBSEQUENT REVISIONS.

Signature	Title	Date
Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

TABLE OF CONTENTS

TISSUE SAMPLE PREPARATION

1. SCOPE AND APPLICATION

1.1. This standard operating procedure describes procedures for the initial preparation of tissue samples prior to sample analysis. Customer-specific contracts or statement of works (SOWs) with alternate procedures will take precedence over this SOP.

1.2. This SOP is intended to provide guidance for the preliminary preparation of tissue samples prior to the sample aliquoting and analytical preparation described in individual analytical SOPs. The procedures described in this SOP also apply to compositing and subsampling of tissue samples for analyses to be subcontracted.

2. METHOD SUMMARY

2.1. Tissue samples are inherently heterogeneous requiring special considerations in order to obtain a truly representative sample aliquot for analysis. This SOP provides guidance for handling tissue samples prior to the sample preparation steps described in analytical SOPs. This SOP applies to samples delivered to the lab in whole body form or in the form of pre-dissected tissues.

2.2. The sample handling strategy must consider:

- what analyses are to be performed (metals, organics, or both, and VOC or non-VOC),
- how much sample is available
- are the analyses to be performed on individual samples or composite homogenates,
- are the analyses to be performed on whole body, edible portions or specific organs, and
- are any of the analyses going to be subcontracted which may require subsampling.

2.3. Proper preparation and handling of tissue samples is required to obtain a representative sample, avoid contamination, and to ensure loss of sample and target constituents is minimized.

3. DEFINITIONS

3.1. Sample: The material presented to the laboratory for analysis or testing.

3.2. Sample Aliquot: A representative part or portion of a sample for analysis which is a fraction of the whole sample. See subsampling also.

3.3. Compositing: The process by which sample aliquots from two or more samples are united to form a combined sample which is subsequently analyzed.

3.4. Subsampling: The process by which a representative portion is obtained from a whole sample.

3.5. Service Request: The service request (SR) is a document prepared at the time of sample receipt and summarizes sample analysis and reporting instructions about a customer's sample(s).

3.6. QAPP: Quality Assurance Project Plan document provided by the client specifically written for their project.

3.7. VOC Analyses: Volatile organic compounds (VOC) analyses, including halogenated and aromatic volatile organic compounds and gasoline range organics (GRO) analyses.

3.8. Non-VOC Analyses: Any analysis other than a VOC analysis.

4. INTERFERENCES

4.1. If precautions are not taken, cross-contamination can occur when handling tissue samples in large quantities. Equipment must be thoroughly cleaned as described in this SOP and related SOPs. Also, the SOP describes the use of homogenization and rinsate blanks to monitor any possible contamination.

4.2. For organics samples, polypropylene and polyethylene (plastic) surfaces, implements, and containers are a potential source of adsorption and contamination and should not be used. Gloves should be talc free and of non-contaminating materials.

5. SAFETY

5.1. All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this procedure. This includes the use of personnel protective equipment, such as, safety glasses, lab coat and the correct gloves.

5.1.1. A Cut and Puncture resistant glove should be worn underneath the talc-free, vinyl glove/nitrile glove on the hand opposite the hand being used for cutting.

5.2. Chemicals, reagents and standards must be handled as described in the ALS safety policies, approved methods and in SDSs where available. Refer to the ALS Chemical Hygiene Plan and the appropriate SDS prior to beginning this method.

6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION AND STORAGE

6.1. Refer to the determinative method.

7. STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

7.1. Not applicable to this procedure.

8. APPARATUS AND EQUIPMENT

8.1. Refer to the Procedure section for specific equipment used based on the determinative analysis to be performed. The use of implements and surfaces may vary depending on the analyses to be performed.

8.2. Hobart Food Chopper, or comparable device.

8.3. Tissumizer.

8.4. Waring blender, or similar device.

8.5. Freeze-drier, Labonco or equivalent.

8.6. Glass or PTFE cutting boards.

8.7. Knives and cutting implements – refer to Procedure section.

8.8. Standard laboratory glassware (beakers, scintillation vials, etc.)

8.9. VOA vial – pre-cleaned, 40ml with Teflon-lined cap.

8.10. Pre-cleaned glass jars with PTFE lined lids, various sizes.

8.11. Gloves

8.11.1. Metals Analysis: talc-free, contamination-free vinyl gloves.

8.11.2. Organics Analysis: talc-free, contamination-free nitrile gloves.

8.11.3. Cut and Puncture resistant glove.

8.12. Heavy duty aluminum foil.

9. PREVENTIVE MAINTENANCE

9.1. No specific maintenance steps are needed other than normal cleaning and inspection of apparatus.

9.2. For organics samples, polypropylene and polyethylene (plastic) surfaces, implements, and containers are a potential source of adsorption and contamination and should not be used. Gloves should be talc free and of non-contaminating materials.

10. RESPONSIBILITIES

10.1. It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. Final review and sign-off of the data is performed by the department supervisor/manager or designee.

10.2. Sample custodians, together with project managers and department supervisors, are responsible for documenting any required sample preparation (including the percent solids or percent lipids determination if required) on the service request. All personnel preparing

tissue samples should be familiar with the contents of this document prior to commencing work.

10.3. Tissue sample preparation is to be performed only by lab analysts instructed in the proper handling techniques outlined in this SOP. It is the responsibility of the analyst to perform this procedure to complete all documentation required for data review.

11. PROCEDURE

11.1. Sample Login

11.1.1. Any special sample handling must be noted on the service request and on a label attached to the sample itself. During sample receiving, a sample custodian must follow the procedures described below.

11.1.2. Tissue Samples with Limited Quantity

11.1.2.1. An assessment of the required sample quantity should be made by the project chemist when taking delivery of the sample. This assessment must take into consideration the tests, the required detection limits and the necessary quality assurance samples. If the quantity of sample given to the laboratory is insufficient for the analyses requested, the sample custodian will, along with the project chemist estimate the total amount of sample available. A "LIMITED SAMPLE VOLUME" tag is attached to the sample on which is recorded the estimated sample quantity.

11.1.2.2. In some cases it may be beneficial to perform sample preparations as described in this SOP prior to estimating the sample amount. In this case, the analyst preparing the sample will provide the project chemist with an estimate of the amount available.

11.1.2.3. The project manager must determine if limited sample quantity exists and set the priorities for the analyses and, if possible, estimate the quantity of sample to be *used for each test*. This information is to be documented and placed in the project file and on the service request to communicate to the laboratory staff. For example, 8081 use 10 g; metals use 1g.

11.1.2.4. NOTE. Samples that are quantity limited and require multiple analyses must be identified as soon as possible. Optimally, this should happen during sample login; however, discovery at any time should trigger appropriate actions.

11.2. Sample Homogenization

11.2.1. This section outlines the steps for preparing homogenous samples of whole fish, edible fish (fillets), edible shellfish, worm composite homogenates, eggs, and plant tissues.

11.2.1.1. Samples for Organics Analyses:

11.2.1.1.1. Equipment used for the processing of tissue samples for organics analyses should be stainless steel, anodized aluminum, glass or polytetrafluorethylene (PTFE). Polypropylene and polyethylene (plastic) surfaces, implements, and containers are a potential source of adsorption and contamination and should not be used. Gloves worn should be talc free, nitrile, consisting of contamination free materials. A Cut and Puncture resistant glove should be worn underneath the talc-free, nitrile glove on the opposite hand not being used for cutting. Filleting should be done on glass or PTFE cutting boards that are cleaned properly between samples or on cutting boards that are covered with heavy duty aluminum foil (hexane rinsed) that is changed between samples. Tissue should be handled with pre-cleaned, high quality, corrosion-resistant stainless steel instruments. Fillets or homogenate should be stored in cleaned glass jars of suitable dimensions with PTFE lined lids. If the sample is to be analyzed for VOCs, the homogenization steps should be performed on sample tissue that is partially frozen or chilled. An aliquot of the homogenate should be placed in a clean 40 mL VOA vial and libeled "FOR VOA ANALYSIS ONLY".

11.2.1.1.2. Prior to handling each sample, utensils, cutting boards and containers should be washed in a detergent hot water solution and rinsed with tap water, hexane, and DI water. Pre-cleaned, certified sample containers may be used without further cleaning. If the sample is to be analyzed for VOCs, methanol is substituted for the rinsing of implements with acetone and the hexane rinsing of the aluminum foil. Exposure to solvent vapors must be minimized.

11.2.2. Samples for Metals Analyses:

11.2.2.1. Equipment used in the processing of samples for metals analyses should be of PTFE, ceramic, polypropylene or polyethylene. Filleting should be performed on PTFE cutting boards which are cleaned after each sample. Knives with titanium or high quality stainless steel blades may be used for tissue resections. Tissue should be stored in glass jars with PTFE lined lids.

11.2.2.2. A Cut and Puncture resistant glove should be worn underneath the talc-free, vinyl or vinyl/nitrile glove on the opposite hand not being used for cutting.

11.2.2.3. Prior to sample handling, utensils, cutting boards and containers should be washed in a detergent hot water solution, rinsed with tap water, 25% HCl (except metal utensils), and DI water. Pre-cleaned, certified sample containers may be used without further cleaning.

11.2.3. Samples for both Metals and Organics Analyses:

11.2.3.1. If the sample is to be prepared for both organics and metals, care must be taken to use equipment and cleaning procedures that are non-contaminating for both. Quartz, ceramic, glass and PTFE are recommended materials for sample processing equipment. Knives with titanium or high quality stainless

steel blades may be used for tissue resections. Glass or PTFE cutting boards should be used. If the sample is to be analyzed for VOC's, the homogenization steps should be performed on sample tissue that is partially frozen or chilled. An aliquot of the homogenate should be placed in a clean 40mL VOA vial and labeled "FOR VOA ANALYSIS ONLY".

11.2.3.2.Prior to handling each sample, utensils, cutting boards and containers should be washed in a detergent hot water solution and rinsed with tap water, acetone, methanol, or hexane (as appropriate), and DI water. Pre-cleaned, certified sample containers may be used without further cleaning. Non-metallic surfaces and utensils should also be rinsed with 25% HCl followed by DI water. If the sample is to be analyzed for VOCs, methanol is substituted for the rinsing of implements with acetone. Exposure to solvent vapors must be minimized.

11.2.3.3.A Cut and Puncture resistant glove should be worn underneath the talc-free, vinyl/nitrile glove on the opposite hand not being used for cutting.

11.2.4. Sample Preparation

11.2.4.1.Each tissue sample may be homogenized in the original glass bottle container if there is sufficient space to allow thorough mixing. If homogenization is not achievable in the original container, place the entire sample contents into a clean glass jar. Generally, liquids contained in the container are to be considered part of the sample. If the sample requires size reduction prior to homogenization, chop the sample into the 1-2" chunks using a titanium or stainless steel bladed knife. Large samples may require the use of industrial food processors such as a Hobart Food Chopper, or comparable device. Size-reduced chunks of tissue are thoroughly homogenized to a paste-like consistency using a Tissumizer, Waring blender, or similar device until it reaches a paste-like consistency. Transfer the sample paste to a glass jar for storage and freeze until ready for sample extraction. The new sample container is labeled with the sample I.D., the word "homogenized", initialed, and dated.

11.2.4.2.Whole Fish Tissue

11.2.4.2.1.Samples may be frozen in the field or in the laboratory. While still partially frozen, rinse the fish with DI water to remove extraneous materials and liquids. Cut the fish into appropriate size chunks and mechanically macerate the sample using cutting tools appropriate for the size of the sample and the analysis type. If necessary, process fish tissue chunks through the Hobart Food Chopper. To ensure thorough mixing, divide the ground sample into quarters, mix opposite quarters and then mix halves. Homogenize sample using a Tissumizer or Waring blender until it reaches a paste-like consistency. Transfer the sample paste to a glass jar for storage and freeze until ready for sample extraction. The new sample container is labeled with the sample I.D., the word "homogenized", initialed, and dated.

11.2.4.3.Edible Fish Tissue

11.2.4.3.1.If the client or QAPP indicates that only edible tissue be analyzed, the fish must be filleted. If the sample arrives pre-filleted, the sample tissue may be frozen before processing. If the sample is not yet filleted, the sample should remain chilled until the filleting is completed. Freezing can result in the contamination of edible tissues from the bursting of internal organs. Fish having ruptured internal organs should be noted on the prep benchsheet and the Project Manager consulted. Rinse the fish with DI water to remove extraneous materials and liquids. Remove scales from scaled fish or skin from non-scaled fish. Rinse the fish again prior to filleting. A separate or clean cutting board should be used for filleting. Gloves should be changed between samples. Carefully remove the fillets from the carcass by following the steps outlined in Appendix A. Care should be taken to avoid contaminating fillet with inadvertent puncture of internal organs. Cut the fillet tissue into appropriate size chunks and mechanically macerate the sample using cutting and grinding tools appropriate for the size of the sample and the analysis type. Proper selection of maceration equipment must consider the potential contaminants, sample size/volume and amount of tissue likely to be lost in using the equipment.

Divide the ground sample into quarters, mix opposite quarters and then mix halves. Again homogenize the sample using an appropriate blending mixer. Continue repeating this process until the sample is truly homogenous and no chunks of tissue remain. Freeze sample until ready for extraction.

11.2.4.4.Shellfish Tissue

11.2.4.4.1.Shellfish should be frozen as soon as possible after receipt by the laboratory unless samples can be prepared within 48 hours of sampling. Edible portions of various shellfish are described below and resection described in Appendix B. Thawing of frozen shellfish samples should be kept to a minimum during tissue removal to avoid loss of liquids. Shellfish should be rinsed with DI water prior to tissue removal to dislodge external debris. When multiple organisms constitute a single sample, the edible tissues are collected, composited and homogenized.

11.2.4.5.Bivalve mollusks (oysters, clams, mussels, and scallops).

11.2.4.5.1.Bivalves are typically prepared by severing the adductor muscle, prying open the shell, and removing all of the soft tissue. The soft tissue includes viscera, meat, and body fluids.

11.2.4.6.Crabs

11.2.4.6.1.Edible tissue includes all leg and claw meat, back shell meat and body cavity meat. Internal organs generally are removed. If the crab is soft shelled, the entire crab is used in the sample.

11.2.4.7.Shrimp and Crayfish - Edible tissue includes the tail meat.

11.2.4.8.Lobster - Edible tissue includes the tail and claw meat.

11.2.4.9.Worms

11.2.4.9.1.Samples are typically supplied to the lab in sample jars containing multiple organisms. Liquid and specimens constitute the entire sample and are blended together typically in the sample container. When a worm sample containing dirt particles or significant amounts of water is encountered, the technician should contact the project manager to seek guidance from the client.

11.2.4.10.Eggs

11.2.4.10.1.Avian eggs are typically removed from the shell and blended. Aquatic eggs are blended including the soft shell.

11.2.4.11.Internal Organs Extraction

11.2.4.11.1.Organs such as livers or kidneys must be identified and removed by an experienced sample technician following clear written resection procedures or other guidance provided by the client.

11.2.4.12.Plant Tissue

11.2.4.12.1.Plant tissue should be handled using the size reduction, homogenization and implement cleaning steps as outlined. Where these procedures are inappropriate, specific written procedures or guidance from the client is recommended.

11.2.4.12.2.If drying is requested by the client or is project-specified, a subsample for mercury analysis is taken from the wet sample, and then the plant tissue is dried at 60˚C prior to homogenization.

11.2.4.13.Small Mammals and Rodents

11.2.4.13.1.There are two primary concerns in working with small mammals and rodents: safety and sample homogenization.

11.2.4.13.2.Small mammals are potential carriers of lethal viruses, such as hantavirus and rabies, and bacteria that can be contracted through inhalation or direct contact. Typically, these organisms are excreted in the feces and distributed on the air as the fecal matter dries. During the sample preparation process, tissue is typically freeze-dried in order to calculate a percent solids value and to analyze for metals. As such, it is possible to increase the potential for dispersion of the bacteria or viruses after the sample is homogenized and processed. Prior to processing, all samples should be stored frozen.

11.2.4.13.3.Prior to sample homogenization, instructions should be received from the client regarding the processing of the hide. For organics, it is recommended that the hides be left on the carcass and the entire sample be homogenized. For metals, there is a potential for

accumulation in the hair. As a non-digestible portion of the rodent, inclusion of the hair may result in a high bias if the data is to be used in estimating bioaccumulation up the food chain. Skinning may be a preferred alternative when metals are the primary chemicals of concern.

11.2.4.13.4. Homogenization should be done while the carcass is still partially frozen.

11.2.4.13.5. If the hide is to be included in the homogenization, snip the feet from the animal using stainless steel scissors.

11.2.4.13.6. The tail should be removed if it will prevent complete homogenization of the sample (e.g., the tail of a mouse or rat may result in incomplete homogenization and should not be included with the sample). Remove seeds, grasses, and mud from the hide.

11.2.4.13.7. If the hide is to be removed from the carcass, make an incision through the skin on the back of the neck (do not cut into the muscle). In most cases, the hide can be removed by pulling the incision horizontally along the back in one direction, and over the ears, head and snout in the opposite direction. The eyes are usually lost during this procedure. Continue to skin the animal by peeling the hide over the hind legs, off the underside of the animal, and around the front legs. The hide is removed at the hind legs and the snout. Care should be taken not to tear the connective tissue under the hide. Fat should be scraped from the hide when possible and included with the sample. Rinse the skinned carcass with DI water to remove any hair or dirt that has accumulated during the skinning procedure.

11.2.4.13.8. Homogenize the sample using a stainless steel Waring blender. Select a blender cup that is sized in accordance with the amount of sample to be homogenized. That is, small samples should be homogenized using small blender cups. This will improve the overall homogenization and recovery of the sample. Continue to mix the sample into a paste like consistency. Make sure no chunks of muscle, hide, or bone are distributed in the sample. Transfer the sample paste to a glass jar for storage and freeze. The new sample container is labeled “homogenized”, initialed, and dated.

11.2.5. <u>General Provision for Handling Large Sample Mass</u>

11.2.5.1. In some cases, large specimens will be received by the laboratory for homogenization prior to chemical analysis. For the purpose of this SOP, ‘large’ is defined as requiring preliminary size reduction to allow sequential processing of the sample. Sub-samples of the whole specimen should be cut to a size appropriate for the blender, mixer, or grinder that will be used. After each individual fraction is processed, the homogenized material is added to a reservoir large enough to hold all fractions as they accumulate. The reservoir will be constructed of a material suitable for the analytical application as defined. For very large specimens (i.e. >20 pounds), high grade stainless steel containers are used (large bowls or small drums).

11.2.5.2. Blending of the combined fractions to achieve a whole homogenous material is achieved via manual mixing. In general, this is accomplished using a high grade stainless steel paddle or spoon of appropriate size (i.e. relative to the whole homogenate). Very large specimens (i.e. >20 pounds) generally require secondary processing through the grinder, particularly when large amounts of skin, bone, and/or cartilage is present. In these cases, the Hobart grinder is generally used.

11.3. Compositing

11.3.1. Each sample is to be logged in and receive a lab code. Additionally, the sample composite also is assigned a lab code. The compositing process is to be performed by trained staff. It is to be performed in an area free of contamination. It is imperative that the samples are treated in a manner consistent with the requirements of the tests to be performed on the composited sample. Compositing of homogenates should be performed according to this SOP or specific instructions provided by the client.

11.3.2. Documentation (use applicable bench sheet)

The analyst preparing the composite will document

- that homogenization was done before removing an aliquot,
- the quantity of each (field or discrete) sample used for the composite,
- the date and time of compositing, and
- any other pertinent observations.

11.3.3. Tissue Samples with Limited Quantity

11.3.3.1. Samples and sample composites that are quantity limited will be handled by the same procedure.

11.3.4. Compositing Procedure

11.3.4.1. Each tissue sample is first homogenized.

11.3.4.2. An equal weight of sample aliquot from each of the homogenized samples is weighed into a clean glass sample bottle. The amount to be weighed of each sample will depend upon the number of analyses to be performed on the composite and if the quantity of any individual sample is limited.

11.3.4.3. The mixture of the individual sample aliquots is thoroughly homogenized in the glass container. The composite sample bottle is labeled with:

- the name of the composite,
- the lab code of the composite,
- the analyst's initials
- the date of composite preparation
- The composite sample and the remaining (individual, discrete) samples are stored frozen until analysis.

11.3.5. Tissue Samples Requiring VOC Analyses

11.3.5.1. A separate aliquot of the composite homogenate should be placed in 40 mL VOA vial container for later analysis by the VOA department. Each container should be labeled with the lab identifier, date, initials, and "FOR VOA ANALYSIS ONLY". To minimize losses of volatile constituents, the sample should be kept as cold as possible, the work should be completed as quickly as possible, and the VOA vial filled to the top to minimize head space.

11.4. Sub-sampling

The sample is first thoroughly homogenized. A sample aliquot is removed and placed into a clean glass container of appropriate size and labeled as follows:

- the name of the sample,
- the lab code of the sample,
- "homogenized" written on the label,
- the purpose of the sub-sample (e.g. "dioxin subsample")
- the analyst's initials
- the date.

11.5. Freeze-Drying

11.5.1. Depending on project specifications, samples may require freeze-drying. Freeze-drying may be performed on a separate portion of sample to determine % Freeze-Dried Solids, or may be done on the analytical subsample for certain tests. The analyst should obtain direction from the supervisor and/or Project Chemist.

11.5.2. Weigh 5-8 g of sample (wet weight) into a scintillation vial. Freeze the sample for at least 2 hours.

11.5.3. Remove the sample from the freezer and place in the freeze drier for at least 24 hours or longer if necessary for the particular sample matrix.

11.5.4. Record the measurements on the applicable bench sheet.

11.5.5. When freeze drying samples that require PFOA/PFOA, aluminum foil must be placed on the container before putting the lid on, after the freeze drying process and before freezing the sample.

12. QA/QC REQUIREMENTS

12.1. A rinsate blank should be prepared to accompany each batch of tissue samples. The blank is comprised of a collection of DI water rinses of cleaned equipment (knives, cutting boards and mixers/grinders) *prior* to the commencement of sample batch preparation. If contamination of the samples is suspected, the rinsate blank is extracted and analyzed for contaminants. The rinsate blank should be labeled with the extraction date and the associated SR numbers and stored at 4˚ C. In the event that contamination is suspected, the rinsate blank can be analyzed to confirm the presence of contaminants in the tissue preparation process.

12.2. A homogenization blank is prepared to determine if the homogenization equipment was effectively cleaned between samples. Unless a project plan specifies otherwise, the laboratory prepares two homogenization blanks with each shift of sample preparation. One is a 500 mL aliquot for Metals testing and the other is a 1000 mL aliquot for Organics testing. Any requirements other than the labs default procedure must be defined in the project plan and communicated to the laboratory.

12.2.1. Some project quality plans may require homogenization blanks between each sample. Following the blending of a tissue sample decontaminate the Hobart mixer (model HCM62) by following these steps:

- Wash the bowl, blade assembly, and lid with soap and hot water.
- Rinse all parts with deionized water.
- Move to fume hood and hexane rinse all parts.
- Allow excess hexane to evaporate.

12.2.2. Reassemble the mixer and make ready for the next sample.

12.2.3. Fill the bowl with deionized water and turn the mixer on for the approximately average time used for the type of samples being processed.

12.2.4. Aliquot the deionized water to bottles appropriate for the testing being conducted and preserve accordingly. If insufficient sample volume is produced for the required testing, repeat the procedure after the next tissue sample is homogenized.

13. DATA REDUCTION AND REPORTING

13.1. Sample handling documentation must include information about sample homogenization (was it done or not), compositing, and sub-sampling. The established and applicable data be
13.2. nch sheets provide a means for recording this information. Completed bench sheets listing the sample handling information are filed in the project file with the raw data.

14. CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

14.1. Refer to the SOP for *Non Conformance and Corrective Action* (CE-QA008) for procedures for corrective action. Personnel at all levels and positions in the laboratory are to be alert to identifying problems and nonconformities when errors, deficiencies, or out-of-control situations are detected.

14.2. Handling out-of-control or unacceptable data

14.2.1. On-the-spot corrective actions that are routinely made by analysts and result in acceptable analyses should be documented as normal operating procedures, and no specific documentation need be made other than notations in laboratory maintenance logbooks, runlogs, for example.

14.2.2. Some examples when documentation of a nonconformity is required using a Nonconformity and Corrective Action Report (NCAR):

- Quality control results outside acceptance limits for accuracy and precision

- Method blanks or continuing calibration blanks (CCBs) with target analytes above acceptable levels
- Sample holding time missed due to laboratory error or operations
- Deviations from SOPs or project requirements
- Laboratory analysis errors impacting sample or QC results
- Miscellaneous laboratory errors (spilled sample, incorrect spiking, etc)
- Sample preservation or handling discrepancies due to laboratory or operations error

15. METHOD PERFORMANCE

15.1. Refer to determinative methods.

16. POLLUTION PREVENTION AND WASTE MANAGEMENT

16.1. It is the laboratory's practice to minimize the amount of solvents, acids, and reagents used to perform this method wherever feasibly possible. Standards are prepared in volumes consistent with methodology and only the amount needed for routine laboratory use is kept on site. The threat to the environment from solvents and/or reagents used in this method can be minimized when recycled or disposed of properly.

16.2. The laboratory will comply with all Federal, State, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the ALS Lab Waste Management Plan.

16.3. This method uses non-halogenated solvents and any waste generated from this solvent must be collected. The solvent will then be added to the hazardous waste storage area and disposed of in accordance with Federal and State regulations.

17. TRAINING

17.1. Training outline – Training Plan

17.1.1. Review literature (see references section). Read and understand the SOP. Also review the applicable MSDS for all reagents and standards used. Following these reviews, observe the procedure as performed by an experienced analyst at least three times.

17.1.2. The next training step is to assist in the procedure under the guidance of an experienced analyst until the supervisor feels the new employee can work independently. During this period, the analyst is expected to transition from a role of assisting, to performing the procedure with minimal oversight from an experienced analyst.

17.2. Training is documented following the *ALS-Kelso Training Procedure* (ADM-TRAIN).

17.3. When the analyst training is documented by the supervisor on internal training documentation forms, the supervisor is acknowledging that the analyst has read and understands this SOP and that adequate training has been given to the analyst to competently perform the analysis independently.

18. METHOD MODIFICATIONS

18.1. This section is not applicable because this procedure is a laboratory developed method.

19. REFERENCES

19.1. Kateman and L. Buydens, *Quality Control in Analytical Chemistry*, Second Edition, John Wiley & Sons, Inc., New York, NY, 1993: Chapter 2 on Sampling and especially sections 2.5 (Sample Quality) and 2.7 (Handling of Samples).

19.2. *Guidance For Assessing Chemical Contaminant Data For Use In Fish Advisories;* Volume 1; Fish Sampling and Analysis, 3rd Edition; USEPA Office of Water; EPA 823-B-00-007; Nov 2000.

19.3. *Recommended Protocols for Measuring Selected Environmental Variables in Puget Sound*; Tetra Tech, Inc.; final report TC-3991-04 Recommended Guidelines for Measuring Organic Compounds in Puget Sound Sediment and Tissue Samples Revision April 1996.

19.4. *PCB's and Mirex In Fish Tissue and Clams* New York State Department of Health Wadsworth Center For Laboratories and Research; Albany, N.Y. 10/6/81

19.5. *Draft Method 1613-Tissue*; Determination of PCDDs and PCDFs in Fish and Other Tissue Using Method 1613; USEPA Office of Water June 1993.

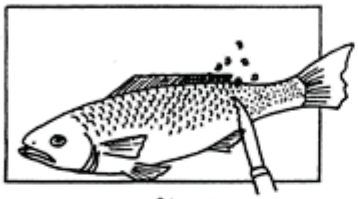
20. CHANGES SINCE THE LAST REVISION

20.1. Various typographical, grammatical, and format revisions.
20.2. Revised to reflect name changes to SDS and Chemical Hygiene Plan.
20.3. Section 5.1: Added use of Cut and Puncture resistant glove into the Safety Section.
20.4. Section 8.11: Added additional glove types to the equipment list.
20.5. Section 11: Inserted the use of Cut and Puncture Resistant gloves throughout the section.

APPENDIX A
Fish Filleting Procedure

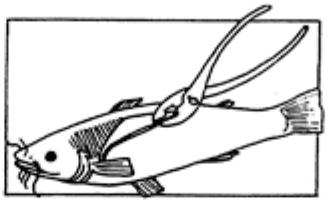
1 Scaled Fish

After removing the scales (by scraping with the edge of a knife) and rinsing the fish:



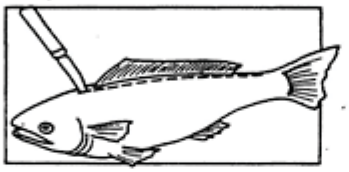
1b Scaleless Fish

Grasp the skin at the base of the head (preferably with pliers) and pull toward the tail.



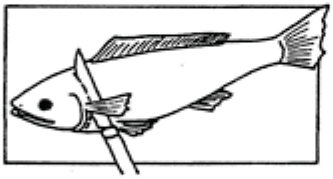
Note: This step applies only for catfish and other scaleless species.

2



Make a shallow cut through the skin (on either side of the dorsal fin) from the top of the head to the base of the tail.

3



Make a cut behind the entire length of the gill cover, cutting through the skin and flesh to the bone.

4

Make a shallow cut along the belly from the base of the pectoral fin to the tail. A single cut is made from behind the gill cover to the anus and then a cut is made on both sides of the anal fin. Do not cut into the gut cavity as this may contaminate fillet tissues.

5

Remove the fillet.

Source: U.S. EPA, 1991d.

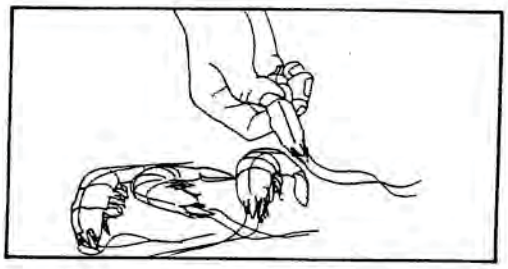
Figure 7-3. Illustration of basic fish filleting procedure.

APPENDIX B
General Procedure for Removing Edible Tissues from Shellfish

Heading, peeling and deveining shrimp

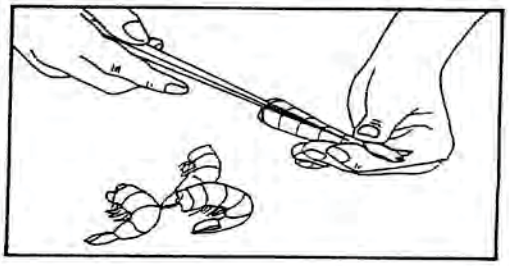
1

To head a shrimp, hold it in one hand. With your thumb behind shrimp head, push head off. Be sure to push just the head off so that you do not lose any meat.



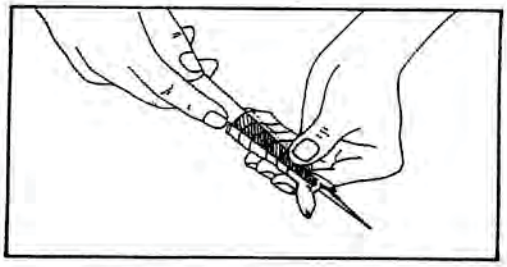
2

If using a deveiner, insert it at head end, just above the vein.



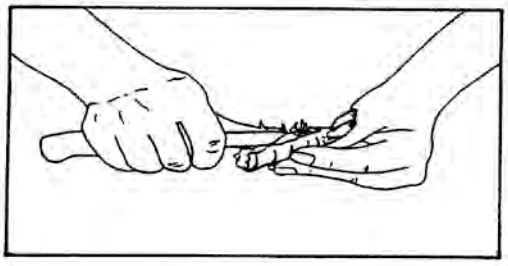
3

Push through shrimp to the tail and split and remove shell. This removes vein at the same time.



4

If you prefer to use a paring knife, shell shrimp with your fingers or knife. Then use knife to gently remove vein.



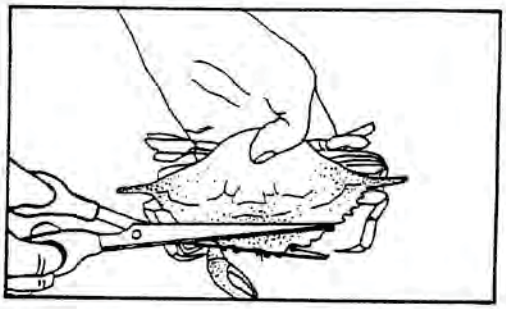
Source: UNC Sea Grant Publication UNC-SG-88-02

G-3

Cleaning soft-shell crabs

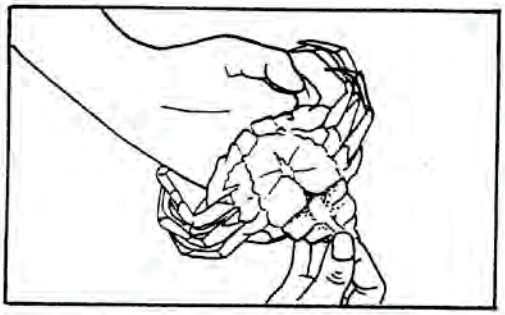
1

Hold crab in one hand and cut across body just behind eyes to remove eyes and mouth.



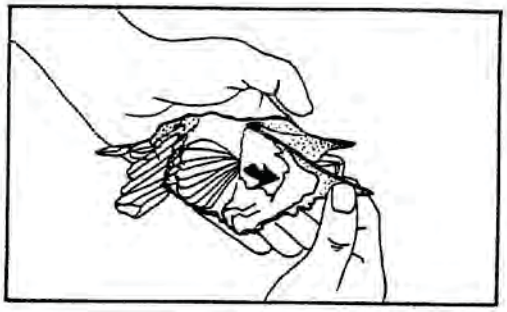
2

Turn crab on its back. Lift and remove apron and vein attached to it.



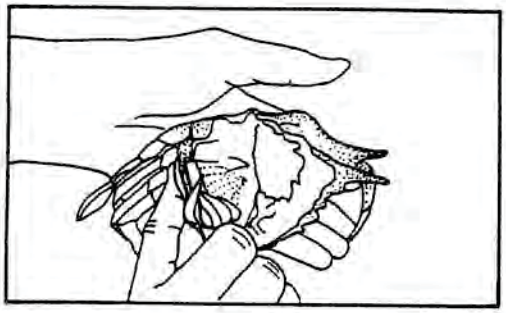
3

Turn crab over and lift one side of top shell.



4

With a small knife, scrape off grayish-feathery gills. Repeat procedure on other side.

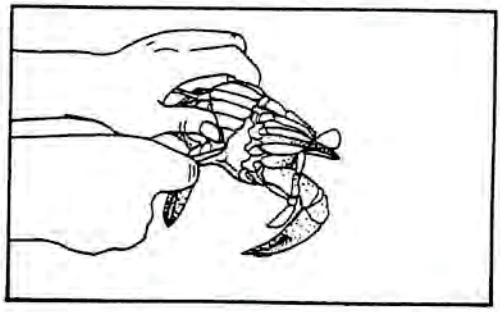


Source: UNC Sea Grant Publication UNC-SG-88-02

Cleaning hard-shell crabs

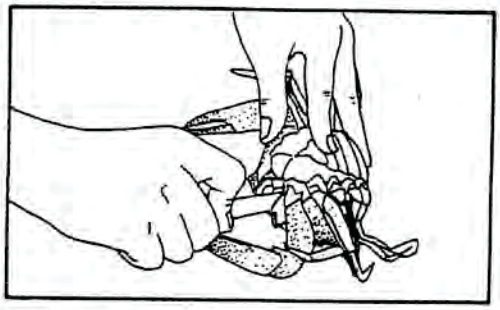
1

Hold crab in one hand. Turn crab over and stab straight down at point of apron with a knife.



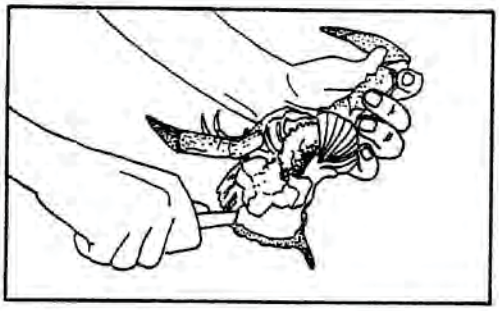
2

Make two cuts from this point to form a V-pattern that will remove mouth.



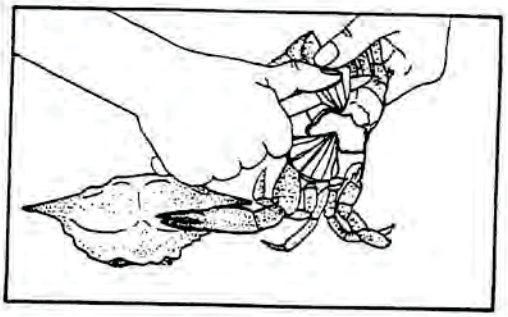
3

Do not remove knife after making second cut. Firmly press crab shell to cutting surface without breaking back shell. With other hand, grasp crab by legs and claws on the side where you are holding knife, and pull up. This should pull crab body free from back shell.



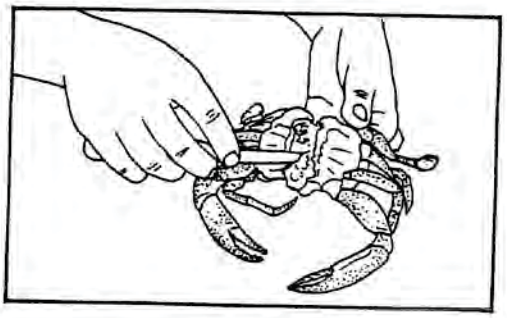
4

Remove gray, feathery gills, which are attached just above legs. Cut and scrape upward to remove gills.



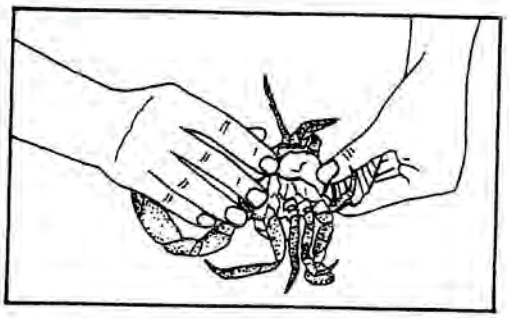
5

Remove all loose material—viscera and eggs—from body cavity.



6

If apron did not come loose with shell, remove it.



Source: UNC Sea Grant Publication UNC-SG-88-02

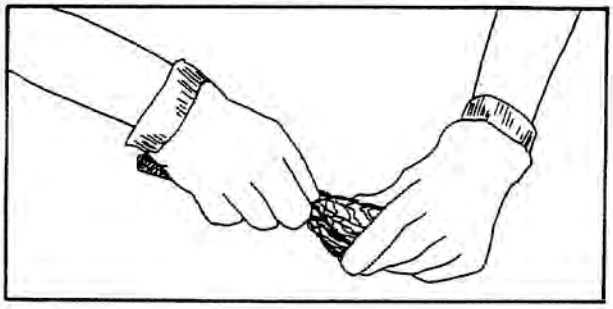
Shucking oysters

1

Oyster shells are especially sharp; be sure to wear gloves to protect your hands. Chip off a small piece of shell from the thin lip of the oyster until there is a small opening.

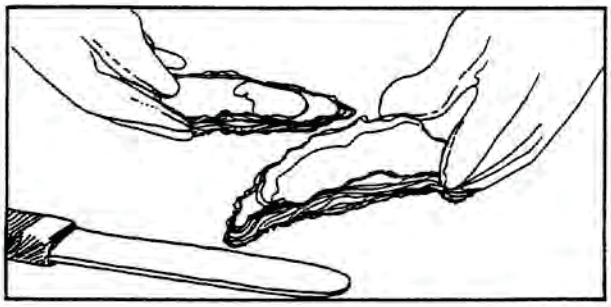
2

Insert knife blade into the opening and cut muscle free from top and bottom shells.



3

Remove oyster meat from the shell.

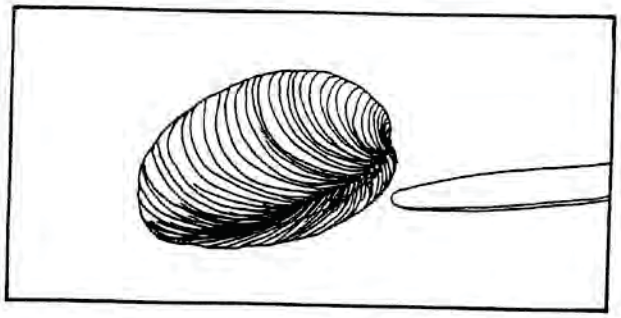


Source: UNC Sea Grant Publication UNC-SG-88-02

Shucking clams

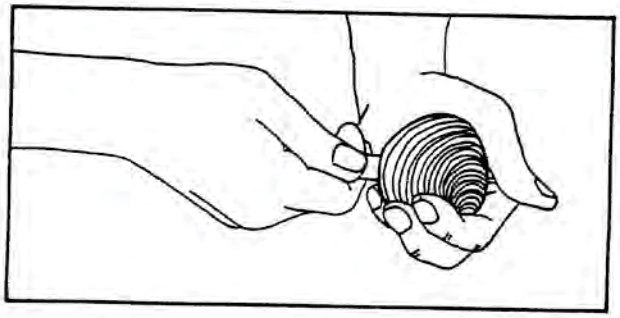
1

In the back of clam near the hinge is a black ligament. Toward the front where ligament ends is a weak spot. Insert your knife at this spot.



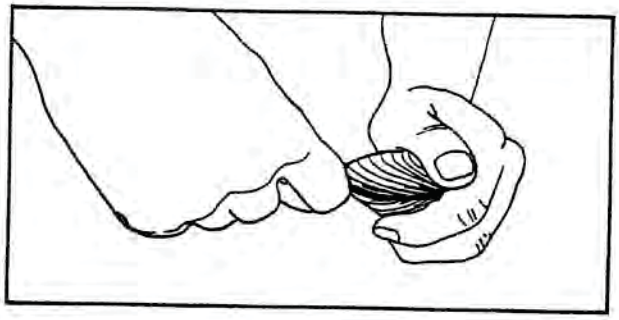
2

Inside are two muscles. Run the knife around the shell to sever both muscles.



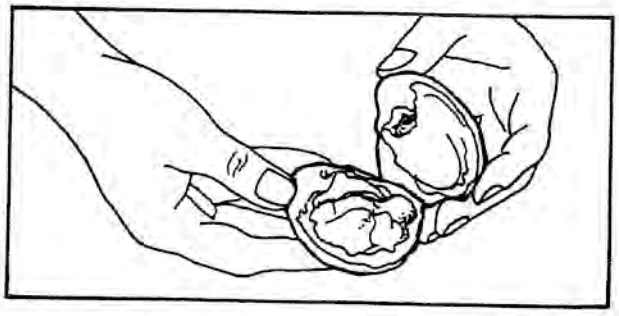
3

Now insert the knife blade into the front of the shell and separate the two shells.



4

Scrape the meat free from the top and bottom shell.



Source: UNC Sea Grant Publication UNC-SG-88-02

Example Benchsheet

Service Request Number(s):

TISSUE HOMOGENIZATION

Laboratory ID	Weight of Sample* (g)	Tare of Jar (g)				

Comments:

* The weight of sample Is after homogenization.

Balance ID:	Date Balance Checked:
Analyst:	Date:
Reviewed:	Date:

APPENDIX C

CULTURAL RESOURCES COORDINATION PLAN

CONTENTS

LIST OF MAPS

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ACRONYMS AND ABBREVIATIONS

ACHP	Advisory Council on Historic Preservation
APE	area of potential effects
ARPA	Archaeological Resources Protection Act of 1979
CCT	Confederated Tribes of the Colville Reservation
CERCLA	Comprehensive Environmental Response, Compensation and Liability Act
CFR	Code of Federal Regulations
CRCP	cultural resources coordination plan
EPA	U.S. Environmental Protection Agency
Lake Roosevelt	Franklin D. Roosevelt Lake
MOA	Memorandum of Agreement
NAGPRA	Native American Graves Protection and Repatriation Act
National Register	National Register of Historic Places
NHPA	National Historic Preservation Act
NPS	National Park Service
QAPP	quality assurance project plan
RCW	Revised Code of Washington
RI/FS	remedial investigation and feasibility study
RM	river mile
SHPO	State Historic Preservation Officer
Site	Upper Columbia River site
STI	Spokane Tribe of Indians
TAI	Teck American Incorporated
THPO	Tribal Historic Preservation Officer
UCR	Upper Columbia River
USBR	U.S. Bureau of Reclamation
USC	United States Code
WAC	Washington Administrative Code

UNITS OF MEASURE

m	meter(s)

1 INTRODUCTION

This document presents the cultural resources coordination plan (CRCP) for the Upper Columbia River (UCR) site (herein the 'Site') remedial investigation and feasibility study (RI/FS) with emphasis placed on sampling activities associated with the plant tissue study.

1.1 BACKGROUND

As specified in the Statement of Work associated with the June 2, 2006 Settlement Agreement (USEPA 2006), "For all RI/FS activities at the Site involving plant tissue collection or ground penetration/disturbance, the Company shall work with the potentially affected parties to assess the effects of the planned work and seek ways to avoid, minimize or mitigate any adverse effects on historic properties." The purpose of this CRCP is to describe known or likely physical impacts of proposed plant tissue and co-located soil sampling, provide relevant background information, define measures for protecting resources, and define procedures for consulting with the appropriate state, federal, and tribal parties with interests in the cultural resources of the Site.

The Site is located wholly within the State of Washington and includes approximately 150 river miles of the Columbia River extending from the U.S.-Canada border to the Grand Coulee Dam, as well as areas in proximity to contamination necessary for implementation of the response actions described in the 2006 Settlement Agreement. The Colville Indian Reservation borders the UCR from approximately river mile (RM) 690 to the Grand Coulee Dam. The Spokane Indian Reservation borders the UCR to the east from approximately RM 650 to RM 640. Franklin D. Roosevelt Lake (Lake Roosevelt) and associated lands are administered by the U.S. Bureau of Reclamation (USBR) and the National Park Service (NPS) of the U.S. Department of the Interior.

The U.S. Environmental Protection Agency (EPA) has responsibilities under the National Historic Preservation Act (NHPA) to consider how its undertakings would affect historic properties. As defined in the NHPA, "historic properties" include archaeological resources, historic-period buildings and structures, and traditional cultural places listed in or determined eligible for listing in the National Register of Historic Places (National Register). To meet the NHPA requirements, EPA must ensure that sampling and other activities would avoid, minimize, or mitigate any adverse effects to any historic properties.

The CRCP is organized into six sections, as follows: 1) this introductory section, which includes summary information on the archaeology, prehistory, Native peoples, and

Euroamerican historical development of the project area; 2) an overview of the relevant federal, state, and tribal laws and regulations, and other appropriate procedures and requirements; 3) a description of the proposed sampling program and its potential physical effects; 4) a plan for coordination and consultation with all affected parties to address known and likely impacts to cultural resources in implementing the proposed work; 5) a list of references; and 6) a glossary of terms.

1.2 CULTURAL SETTING

The broader context of the cultural development of the upper Columbia region[1] provides the critical framework for understanding the importance of the cultural resources in the area. Archaeological and historical resources reflect broad patterns of cultural use and development, just as ongoing traditional use of areas and natural resources represents cultural continuity that can be important to individual and social identities. This section of the CRCP serves as a brief introduction to the cultural history of the upper Columbia region. The primary source of information on the prehistory of the area is Goodal et al. (2004); for Native peoples, the source is Kennedy and Bouchard (1998); and for Euroamerican history, McKay and Renk (2002).

Archaeological research contributes significantly to our understanding of the prehistoric past. In the upper Columbia region, systematic archaeological research began in the late 1930s and has continued to the present. Almost 500 archaeological resources have been recorded in and along Lake Roosevelt, representing prehistoric, protohistoric, ethnohistoric, and historic-period human use and occupation. Research at some of these resources has provided the outlines of prehistoric cultural development in the upper Columbia region. Human presence in the region extends back at least 11,000 years. These first humans lived in small groups and were mobile foragers, hunting and gathering plants. The presence of the Columbia River led to an early focus on the abundance of riverine resources. Beginning about 8,000 years ago, populations appear to have increased and led to a gradual trend to less mobility and more permanent settlements. The growing population also led to use of a greater diversity of resources and increasing reliance on fish.

[1] The phrase "upper Columbia region" herein refers to the drainage of the upper Columbia River from around Grand Coulee to the Arrow Lakes area in British Columbia. The upper Columbia region includes, but is not limited to, the Site as defined in the Settlement Agreement. This distinction is important because general patterns of cultural development in the upper Columbia region as a whole provide the framework for addressing the significance of the cultural resources within the Site boundaries.

Permanent settlements increased in size and became concentrated in the river valleys beginning about 6,000 years ago, probably in response to continued population growth. Use of resources in upland areas expanded to meet the needs of the burgeoning populations and settlements. These trends continued until about 1,000 years ago, when there is evidence for a decline in population size. There were fewer settlements, villages were smaller, and there was less use of upland areas.

Cultural patterns of the late prehistoric period were reflected in the lives of the Native peoples at the time of Euroamerican contact. At the time of contact, the UCR was the homeland of the Lakes, Colville, Spokane, and Sanpoil peoples. The Lakes people occupied the Columbia River valley from the vicinity of modern Northport, Washington, north into the Arrow Lakes area of modern British Columbia. The Colville lived along the river downstream of the Lakes as far as around the mouth of the Spokane River. Downriver of the Colville were the Spokane, in the Spokane River drainage, and the Sanpoil, who lived along the Columbia River from around the mouth of the Spokane River to near the modern location of the Grand Coulee Dam.

All of these groups spoke Interior Salish languages and shared many cultural features. Their cultural differences largely reflected differences in the local environments in which they lived. The social, political, and economic foundation of these groups was historically the winter village. The villages were concentrated in the river valleys, and each village was politically independent. Residents of the villages relied on provisions gathered, dried, and stored during the summer to survive through the winter. With the coming of spring, families began moving out of the winter village and shifting among the warm -season camps near resource locations. Gathering of plants and hunting game in upland areas were important subsistence activities during this season, but salmon constituted the most important food staple. Kettle Falls was a major aboriginal fishery, attracting people from throughout the region.

Native life began to change with the introduction of elements of Euroamerican culture. Horses reached the region in the 1700s and significantly changed Native travel and transportation. European diseases such as smallpox appeared in the late 1700s and had disastrous consequences for Native groups. Populations may have declined as much as 80 percent between the 1780s and 1840s. Direct contact with Euroamericans came in the early 1800s, when fur-trade posts were established on the Spokane River and at Kettle Falls.

When American settlement began in the 1840s, it bypassed the upper Columbia region. The discovery of gold in the region in the 1850s led to a major influx of Americans and growing conflict between the new settlers and Indian groups. A series of treaties with Indian groups

was signed in 1855 but did not include the peoples of the upper Columbia region. As American settlement continued, the federal government responded by Presidential Executive Order creating the Colville Reservation in 1872 for the Colville, Spokane, Methow, Okanogan, Sanpoil, Lakes, Calispel, Coeur d'Alene, and scattering bands. Separate reservations were later set aside for the Spokane, Calispel and Coeur d' Alene Tribes. Both the Colville and Spokane reservations have subsequently lost lands to the allotment process in the late 1800s and early 1900s as well as inundation from the waters of Lake Roosevelt. The Colville Reservation is now the home of the 12 tribes that comprise the Confederated Tribes of the Colville Reservation (CCT); the Spokane Reservation is the home of the Spokane Tribe of Indians (STI).

As noted above, the direct Euroamerican presence in the upper Columbia region began with the establishment of fur-trade posts on the Spokane River and at Kettle Falls. These posts were constructed between 1810 and 1825. The fur traders were followed by Christian missionaries in the 1830s and 1840s. A more substantial Euroamerican presence in the region developed in the 1850s, with the discovery of gold near Fort Colville. Conflicts between miners and Indians led to a military campaign in the Spokane River valley in 1858 and the establishment of an army post (Fort Colville) near Kettle Falls in 1859.

American settlement in the upper Columbia River drainage accelerated in the 1860s, initially spurred by mining. Farmers eventually followed the miners, but agricultural activity was limited until the construction of the Spokane Falls and Northern Railway through the region in 1890. With improved access to markets, farming—especially orchard crops—developed as one of the economic mainstays of the area, although mining has continued to play an important role.

The growing demands for agriculture led to plans to construct a dam at Grand Coulee. The dam would provide water for irrigation and inexpensive hydroelectric power. Construction of the dam began in 1934 and was completed in 1942. More than 82,000 acres above the dam was flooded, resulting in the relocation of 11 towns and about 3,000 residents. Since its creation, Lake Roosevelt has provided a growing number of recreational and tourist activities, which have become increasingly important to local economies.

2 OVERVIEW OF LAWS AND REGULATIONS

Implementation of the RI/FS would occur primarily on federal and tribal lands. Federal and tribal laws and regulations addressing cultural resources will therefore provide the primary legal framework for this coordination plan. It is possible, however, that implementation of the RI/FS may require activities on private or non-federal, non-tribal public lands. This overview therefore includes a brief description of relevant state laws and executive orders. Ferry, Lincoln, and Stevens counties, which border the UCR, do not appear to have any ordinances addressing cultural resources that would be relevant to the Site RI/FS.

Relevant federal, tribal, and state laws and regulations directly addressing cultural resources are briefly outlined below, as well as pertinent executive orders issued by the President of the United States and the Governor of Washington.

2.1 FEDERAL LEGISLATION AND REGULATIONS

An overview of federal legislation and regulations is provided below. There are three key laws relevant to Site RI/FS activities. The NHPA guides all federal agency actions that could affect cultural resources. Implementation of the RI/FS constitutes an "undertaking" as defined in the NHPA; therefore, complying with the NHPA requirements is the responsibility of EPA. The Archaeological Resources Protection Act of 1979 (ARPA) and the Native American Graves Protection and Repatriation Act (NAGPRA) apply to activities that could affect archaeological resources and Indian burials on federal and tribal lands. These laws and their implementing regulations would therefore apply to RI/FS activities conducted on federal and tribal lands.

2.1.1 National Historic Preservation Act of 1966, as Amended through 1992 (16 USC 470-470w)

The NHPA is the centerpiece of federal legislation protecting cultural resources. In the Act, Congress states that the federal government will "provide leadership in the preservation of the prehistoric and historic resources of the United States," including resources that are federally owned, administered, or controlled. For federal agencies, Sections 106 and 110 of the Act provide the foundation for how federal agencies are to manage cultural resources, but other sections provide further guidance. The implementing regulations for the NHPA are in 36 Code of Federal Regulations (CFR) Part 800. These regulations are summarized below.

2.1.1.1 Section 106

Similar to the National Environmental Policy Act of 1969, Section 106 of the NHPA requires federal agencies to take into account the effects of their actions or programs specifically on historic and archaeological properties, prior to implementation. This is accomplished through consultation with the State Historic Preservation Officer (SHPO) and/or the Advisory Council on Historic Preservation (ACHP). On lands held by a tribe with a Tribal Historic Preservation Officer (THPO), the THPO has the same duties and responsibilities as the SHPO. If an undertaking on federal lands may affect properties having historic value to a federally recognized Indian tribe, such tribe shall be afforded the opportunity to participate as interested persons during the consultation process defined in 36 CFR 800. Compliance can also be accomplished using agreed-upon streamlined methods and agreement documents such as programmatic agreements.

The Section 106 process is designed to identify possible conflicts between historic preservation objectives and the proposed activity, and to resolve those conflicts in the public's interest through consultation. Neither the NHPA nor the ACHP regulations require that all historic properties be preserved. Rather, they only require the agency proposing the undertaking to consider the effects of the proposed undertaking prior to implementation.

Failure to take into account the effects of an undertaking on historic or cultural properties can result in formal notification from the ACHP to the head of the federal agency of foreclosure of the ACHP's opportunity to comment on the undertaking pursuant to NHPA. A notice of foreclosure can be used by litigants against the federal agency in a manner that can halt or delay critical activities or programs.

The process for compliance with Section 106 consists of the following steps:

1. **Identification of Historic Properties**— Identification of historic properties located within the area of potential effects (APE) is accomplished through review of existing documentation and/or field surveys.
2. **Property Evaluation**—Evaluation of the identified historic properties is accomplished using National Register of Historic Places criteria (36 CFR Part 63) in consultation with the SHPO and, if necessary, the ACHP. Properties that meet the criteria will be considered "Eligible" for listing in the National Register, and will be subject to further review under Section 106. Properties that do not meet the criteria will be considered "Not Eligible" for listing in the National Register, and will not be subject to further Section 106 review.

3. **Determination of Effect**—An assessment is made of the effects of the proposed project on properties that were determined to meet the National Register criteria, in consultation with the SHPO and, if necessary, the ACHP. One of the following effect findings will be made:

   - No Historic Properties Affected—If no historic properties are found or no effects on historic properties are found, the agency official provides appropriate documentation to the SHPO/THPO and notifies consulting parties. However, the federal agency must proceed to the assessment of adverse effects when it finds that historic properties may be affected or the SHPO/THPO or ACHP objects to a "No Historic Properties Affected" finding. The agency must notify all consulting parties and invite their views.
   - No Historic Properties Adversely Affected—When the Criteria of Adverse Effect are applied (36 CFR 800.5(a)), and it is found that historic properties will not be adversely affected by the undertaking, the agency may make a finding of "No Historic Properties Adversely Affected." This finding is submitted to the SHPO for concurrence. Typically, the ACHP will not review "No Adverse Effect" determinations. However, the ACHP will intervene and review "No Historic Properties Adversely Affected" determinations if it deems it appropriate, or if the SHPO/THPO or another consulting party and the federal agency disagree on the finding and the agency cannot resolve the disagreement. If Indian tribes disagree with the finding, they can request the ACHP's review directly, but this must be done within the 30-day review period. Agencies must retain records of their findings of "No Historic Properties Adversely Affected" and make them available to the public. The public should be given access to the information when they so request, subject to Freedom of Information Act and other statutory limits on disclosure, including the confidentiality provisions in Section 304 of the NHPA. Failure of the agency to carry out the undertaking in accordance with the finding requires the agency official to reopen the Section 106 process and determine whether the altered course of action constitutes an adverse effect.
   - Historic Properties Adversely Affected—Adverse effects occur when an undertaking may directly or indirectly alter characteristics of a historic property that qualify it for inclusion in the Register. Reasonably foreseeable effects caused by the undertaking that may occur later in time, be farther removed in distance, or be cumulative also need to be considered. The finding of "Historic Properties Adversely Affected" is submitted to the SHPO for concurrence. The SHPO/THPO may suggest changes in a project or impose conditions so that adverse effects can be avoided and thus result in a "No Historic Properties Adversely Affected" determination.

4. **Resolution of Adverse Effects/Mitigation**—When adverse effects are found, the consultation must continue among the federal agency, SHPO/THPO, and consulting parties to attempt to resolve them. The agency official must notify the ACHP when adverse effects are found, and should invite the ACHP to participate in the consultation when circumstances exist, as outlined in 36 CFR 800.6(a)(1)(i)(A)-(C). A consulting party may also request the ACHP to join the consultation.

   When resolving adverse effects without the ACHP, the agency official consults with the SHPO/THPO and other consulting parties to develop a Memorandum of Agreement (MOA). The MOA will outline the steps or actions to be taken prior to implementation of the project, in order to mitigate the adverse effects on the historic property. Stipulations included in an MOA may include (but are not limited to) documentation, modification of the project to lessen the adverse effects on the property, efforts to sell or relocate the resource, or step-by-step consultation with interested parties throughout the process to ensure it is carried out according to plan.

   The MOA is executed between the agency official and the SHPO/THPO and filed with required documentation with the ACHP. This filing is the formal conclusion of the Section 106 process and must occur before the undertaking is approved.

In some cases, streamlining of the Section 106 process can be accomplished through the use of programmatic agreements. The ACHP and the agency official may negotiate a programmatic agreement to govern the implementation of a particular program or the resolution of effects from complex projects or multiple undertakings. Programmatic agreements are particularly useful when programs or projects affecting historic properties are similar and repetitive, and have known effects, such as routine maintenance or a series of similar rehabilitation projects.

2.1.1.2 Section 101(d)(2)

This section of the NHPA provides for the assumption by federally recognized Indian tribes of all or any part of the functions of a SHPO with respect to tribal lands (e.g., all lands within the exterior boundaries of any Indian reservation and all dependent Indian communities). Section 101(d)(2) requires federal agencies, in carrying out their Section 106 responsibilities, to consult with federally recognized Indian tribes that attach religious or cultural significance to a historic property. The agency will consult with federally recognized Indian tribes in the Section 106 process to identify, evaluate, and treat historic properties that have religious or cultural importance to those groups.

2.1.1.3 Section 110

Section 110 of the NHPA is intended to ensure that historic preservation is integrated into the ongoing programs of federal agencies. This section of the Act requires agencies to identify, evaluate, and nominate for listing in the National Register, historic properties owned or controlled by the agency; use historic properties to the maximum extent feasible; ensure documentation of historic properties that are to be altered or damaged; carry out programs and projects that further the purpose of the Act; and undertake such planning and actions as may be necessary to minimize harm to any formally designated National Historic Landmark properties.

2.1.1.4 Section 111

Section 111 of the NHPA requires agency officials, to the extent practicable, to establish and implement alternatives for historic properties, including adaptive use, that are not needed for current or projected agency uses or requirements. Further, Section 111 allows the proceeds from any lease to be retained by the agency to defray the cost of administration, maintenance, repair, and related expenses of historic properties.

2.1.1.5 Section 112

Section 112 of the NHPA requires that agency officials who are responsible for protection of historic properties pursuant to the NHPA ensure that all actions taken by employees or contractors meet professional historic preservation standards established by the Secretary of the Interior (Professional Qualifications Standards of the Secretary of the Interior's Standards and Guidelines in Archaeology and Historic Preservation [NPS 1983]).

2.1.1.6 Section 304

Section 304 of the NHPA requires that information about the location, character, or ownership of a historic property be withheld from public disclosure when the federal agency head or other public official determines that disclosure may cause a significant invasion of privacy, risk, and/or harm to the historic property, or impede the use of a traditional religious site by practitioners.

2.1.1.7 Comprehensive Environmental Response, Compensation and Liability Act and the National Historic Preservation Act

EPA's Comprehensive Environmental Response, Compensation and Liability Act (CERCLA) manual, CERCLA Compliance with Other Laws Manual: Part II. Clean Air Act

and Other Environmental Statutes and State Requirements (USEPA 1989), outlines how "substantive compliance" with the NHPA is to be achieved in CERCLA actions.[2] The initial step is determining if cultural resources are known or are likely to be present "in or near the area under study in the RI." This step may require conducting a survey of both the location of the proposed remedial action and any associated actions that would occur off site. The CERCLA manual referenced above defines three stages of a survey: Stage IA, literature search and sensitivity study; Stage IB, field investigation; and Stage II, site definition and evaluation. All studies should include Stage IA but implementation of Stage IB is contingent on the results of Stage IA, and the need for Stage II is contingent on the results of Stage IB. If results of the survey identify significant cultural resources (i.e., resources listed or considered eligible for listing in the National Register), effects of the proposed remedial action and associated actions to the significant resources must be evaluated. Adverse effects to significant resources must be either avoided or mitigated. Any proposed mitigation measures must be incorporated into the remedial design process.

2.1.2 Archaeological Resources Protection Act of 1979 (16 USC 470aa-470ll)

ARPA is essentially an update to the 1906 Antiquities Act. It expands and strengthens the activities prohibited under the Antiquities Act, increases the criminal penalties for violation, establishes civil penalties, and provides further guidelines for the issuance of permits. This Act continues to apply only to federal and Indian lands (the definition of "Indian lands" in ARPA differs very slightly from the definition of "Tribal lands" in the NHPA). Most archaeological excavations and collection of artifacts on these lands are allowed only with an ARPA permit. Trafficking in illegally obtained archaeological resources from federal and Indian lands is also prohibited. Individuals convicted of violating the Act are liable for the value of the archaeological resource itself, and the cost of restoration or repair of the damage caused by illegal excavation or collection.

The implementing regulations are 43 CFR Part 7 (U.S. Department of the Interior), which applies to federal lands that are not within military reservations or national forests. The regulations include detailed definitions of "archaeological resource" and "Indian lands"

[2] As stated in the June 2, 2006 Settlement Agreement (USEPA 2006), "The Parties intend that this RI/FS, while not being carried out under an administrative order or judicial order issued pursuant to the provisions of the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), will be consistent with the National Contingency Plan ('NCP'), 40 CFR Part 300."

(lands held in trust by the United States on behalf of a federally recognized tribe or individual members of a federally recognized tribe).

2.1.3 Native American Graves Protection and Repatriation Act (25 USC 3001-3013)

NAGPRA establishes that Native American human remains and associated funerary objects found on federal or tribal lands belong to the lineal descendants of the Native American. When the lineal descendants cannot be determined, the remains belong to the tribe on whose land the remains were found (when found on tribal lands), or to the Indian tribe with the "closest cultural affiliation."[3] This latter rule also applies to unassociated funerary objects, sacred objects, and objects of cultural patrimony (all defined in the Act). NAGPRA applies to both human remains intentionally excavated (which would require an ARPA permit) and those accidentally discovered.

NAGPRA also requires all federal agencies and museums to inventory their holdings of Native American human remains and funerary objects. Once the inventories are completed, the agencies and museums are to notify the appropriate tribes of the remains and other objects in their collections. The remains and associated funerary objects are to be returned (repatriated) at the request of the lineal descendants or tribe. The same requirement applies to unassociated funerary objects, sacred objects, and objects of cultural patrimony for which a cultural affiliation can be demonstrated. Exceptions to the repatriation requirement are objects that are "indispensable for completion of a specific scientific study, the outcome of which would be of major benefit to the United States."

The implementing regulations are 43 CFR Part 10, which largely expand on the elements of the statute. The regulations detail 1) the process of consultation with Indian tribes to address either intentional excavation of human remains or inadvertent discovery of human remains; 2) how agencies and museums are to inventory their collections; and 3) the repatriation process. When human remains, funerary objects, sacred objects, and objects of cultural patrimony are inadvertently discovered on federal lands, the following steps are to be followed: 1) ongoing activity in the area of the find must cease and a reasonable effort made to protect the find; and 2) the federal land agency (i.e., the federal agency on whose lands the remains or objects have been found) must be immediately notified by telephone, with

[3] Cultural affiliation is defined in the implementing regulations, 43 CFR 10.2(e), and refers to a relationship of shared group identity, which can be reasonably traced historically or prehistorically between a present-day Indian tribe or Native Hawaiian organization and an identifiable earlier group.

written confirmation. The federal land agency must then notify the appropriate tribe(s) and further secure and protect the discovery. The activity may be halted for up to 30 days while an appropriate response to the find is negotiated by the federal agency and the appropriate tribe(s).

2.1.4 American Indian Religious Freedom Act (42 USC 1996)

This Act states that it is the policy of the United States to protect and preserve the rights of American Indians to practice traditional religions. That policy includes rights of access to sacred sites and to the use and possession of sacred objects. There are no implementing regulations.

2.2 PRESIDENTIAL EXECUTIVE ORDERS

Presidential executive orders define policies and procedures for federal agencies to facilitate their execution of laws passed by the U.S. Congress or clarify how specific laws are to be implemented. Presidential executive orders can be considered instructions or directives from the President to federal agencies on how to carry out specific laws. The executive orders listed below are either directly related to cultural resources or define relationships between federal agencies and tribes.

2.2.1 Executive Order 11593. Protection and Enhancement of the Cultural Environment

Issued in 1971, Executive Order 11593 states that the federal government would provide leadership in "preserving, restoring, and maintaining the historic and cultural environment of the Nation." Federal agencies were directed to inventory cultural resources under their jurisdiction and nominate National Register-eligible properties to the National Register. Properties that have been determined eligible are not to be transferred, sold, demolished, or altered without providing the ACHP with an opportunity to comment. Properties to be demolished or substantially altered were to be documented prior to demolition or alteration. National Register properties or National Register-eligible properties under federal control were to be maintained following standards set by the Secretary of the Interior. Executive Order 11593 also assigns specific responsibilities to the Secretary of the Interior, including managing the National Register of Historic Places and assisting and advising other federal agencies in the management of cultural resources.

2.2.2 Executive Order 13007. Indian Sacred Sites

Issued in 1996, Executive Order 13007 directs federal agencies to provide access and ceremonial use of Indian sacred sites, where practicable, legal, and not inconsistent with essential agency functions. Agencies are also directed to avoid adversely affecting sacred sites and maintain the confidentiality of such sites. A "sacred site" as defined by this executive order is a specific location that is sacred because of its religious significance to or ceremonial use in an Indian religion.

2.2.3 Executive Order 13175. Consultation and Coordination with Indian Tribal Governments

Issued in 2000, Executive Order 13175 directs federal agencies to consult with tribal officials in the development of policies and regulations that have "tribal implications" or that preempt tribal law. Executive Order 13175 also emphasizes the importance of government-to-government relationships between the U.S. Government and tribes. Agencies must designate an official responsible for implementing the Executive Order and must document tribal consultation in the development of the relevant policies and regulations.

2.3 TRIBAL LEGISLATION AND REGULATIONS

Tribal laws and regulations addressing cultural resources would apply to lands on the reservations and off-reservation trust lands. The CCT and the STI are the two tribes whose laws and regulations would be potentially applicable to the Site. The legal code of the CCT addresses cultural resources, as summarized below. This code applies to both on- reservation actions and off-reservation actions by federal agencies that could affect cultural resources. STI does not currently have laws that specifically address cultural resources. Both tribes have THPOs, who have the same authority and responsibilities as the SHPO on their respective reservations and on off-reservation trust lands.

2.3.1 Confederated Tribes of the Colville Reservation. Colville Tribal Law and Order Code Chapter 4-4, Cultural Resources Protection

This Colville Tribal Code establishes the Colville Cultural Resources Board, which has the responsibility of developing policies and procedures to protect cultural resources of interest and concern to the Colville Tribes, both on and off the Colville Reservation. The Board reviews proposed federal agency actions off the reservation and is responsible for reviewing all proposed on-reservation actions that could affect significant cultural resources. The code

also establishes a Colville Register of Historic and Archaeological Properties for listing of historic properties on the Colville Reservation.

This code defines the roles and responsibilities of the Colville History and Archaeology Department, which include identifying significant cultural resources on the reservation, nominating properties to the National Register and the Colville Register, and promoting efforts to protect cultural resources on the reservation.

Chapter 4-4 of the Colville Tribal Code prohibits the excavation, disturbance, or other adverse effects to archaeological resources and historic properties on the reservation without a permit issued by the Colville History and Archaeology Department. The code defines the procedure for the issuance of permits and the responsibilities of permittees.

2.4 STATE LEGISLATION AND REGULATIONS

Washington State laws and regulations regarding archaeological and historical resources, as well as the law protecting Indian graves, are not applicable on federal lands or on tribal trust lands. These laws would apply, however, to any RI/FS-related activities that would affect private lands or non-federal or non-tribal public lands.

2.4.1 Revised Code of Washington (RCW) Chapter 27.44, Indian Graves and Records

This legislation prohibits the removal or other disturbance of Indian burials, cairns, and "glyptic or painted records." "Burials" and "graves" are not defined in the statute. Excavation or removal of burials is permitted only under provisions of a permit issued by the Washington Department of Archaeology and Historic Preservation. Procedures for obtaining permits are defined in Washington Administrative Code (WAC) Chapter 25-48.

2.4.2 RCW Chapter 27.53, Archaeological Sites and Resources

This legislation prohibits the excavation or disturbance of archaeological sites on public and private lands in Washington except under provisions of a permit issued by the Washington Department of Archaeology and Historic Preservation. Procedures for obtaining permits are defined in WAC Chapter 25-48.

2.4.3 RCW Chapter 68.60, Abandoned and Historic Cemeteries and Historic Graves

This legislation prohibits the destruction, alteration, or other disturbance of historic al and abandoned cemeteries and historic graves (Indian graves and burials are protected in RCW Chapter 27.44). A historic cemetery is defined in the statute as one established before November 1889. A historic grave is a grave or graves outside of a cemetery placed prior to June 1990.

2.4.4 RCW Chapter 43.21C, State Environmental Policy Act

This legislation directs state and local agencies in Washington to address environmental impacts of proposed projects. The implementing rules (WAC Chapter 197-11) require that impacts to historic and cultural resources are to be addressed in the State Environmental Policy Act process.

3 PROPOSED SAMPLING PROGRAM

A summary of the potential sampling areas is provided in Table C1, with a detailed description of sampling techniques provided in this quality assurance project plan (QAPP). As indicated in the QAPP, plant tissue and co-located soil samples will be collected from up to 16 potential sampling areas within the Site (Map C1). Specific sampling locations within each sampling area will be determined after a reconnaissance of the area has been conducted just prior to sampling. Detailed sampling methods and maps of each sampling area are provided in the field sampling plan (Appendix A of this QAPP).

4 COORDINATION PLAN

The objective of the CRCP is to ensure that implementation of the RI/FS and associated sampling activities does not adversely affect any cultural resources. The plan therefore defines a general process and more specific procedures to meet this objective.

The two main challenges in meeting this objective are 1) the iterative process of remedial investigations; and 2) the high density of cultural resources in the study area. The iterative process is a challenge because there are likely to be several rounds of sampling (and associated actions) that extend over several years. Coordination and consultation must therefore also be an iterative process as methods and locations are defined for each round of sampling.

The high density of cultural resources is a challenge because it is highly likely that every round of intrusive sampling will occur at the identified location of one or more cultural resources. At the same time, the high density is potentially misleading by suggesting that all cultural resources in the UCR have been identified. Most—if not all—of the Lake Roosevelt lands have been surveyed for cultural resources in the past. Few of the surveys conducted prior to about 1975 are likely to have met current regulatory and professional standards. In addition, many of the previous surveys focused on archaeological resources to the exclusion of other types of cultural resources (and older archaeological surveys documented only evidence of prehistoric use or occupation). Finally, it is likely that there are some locations previously surveyed at which burials or buried archaeological resources are present but not evident and therefore not recorded at the time of the survey (many surveys both in the past and in the present rely entirely or primarily on surface evidence of archaeological resources or burials).

This plan therefore defines procedures that address sampling at known locations of cultural resources and locations where no cultural resources are currently recorded.

4.1 GENERAL CONSULTATION FRAMEWORK

Implementation of the RI/FS constitutes an "undertaking" as defined in the NHPA; therefore, complying with the NHPA requirements is the responsibility of EPA. EPA is the lead federal agency for cultural resources consultation and coordination for the UCR site. Any issues or concerns related to cultural resources during the planning and/or implementation of Site work shall be brought to the attention of EPA for consultation with the UCR Cultural Resources Working Group, as appropriate. Successful implementation of

the RI/FS and of this CRCP, given the issues defined above, will require ongoing consultation and coordination with the UCR Cultural Resources Working Group consisting of NPS, the USBR, the CCT, the STI, and the Washington SHPO (i.e., the consulting parties). Other consulting parties (as defined in 36 CFR 800.2(c)) may be recognized in the future whose participation would be important for general consultation or coordination in the RI/FS process or for specific sampling locations. For the purposes of cultural resources coordination activities, the "consulting parties" referred to in this plan are distinguished from other "participating parties" to the RI/FS process.

4.2 CULTURAL RESOURCE PROCEDURES IN THE SAMPLING PROCESS

This section defines general procedures to be followed in the sampling process to minimize the potential for inadvertent disturbance of cultural resources. More specific protocols to respond to discoveries are defined in the following subsections.

In addition, the UCR Cultural Resources Working Group recommended to Teck American Incorporated (TAI) that it provide cultural awareness, avoidance, and sensitivity training/refresher to field personnel, as appropriate, prior to the commencement of field activities.

4.2.1 Archaeological Monitoring in the Sampling Program

To ensure compliance with the NHPA and the applicable requirements, procedures, and standards of the NPS, USBR, CCT, and STI, the following procedures have been developed to address potential discoveries, including inadvertent discoveries, of cultural materials and deposits (including sacred objects, funerary objects, and objects of cultural patrimony as defined in NAGPRA), including Indian burials and human remains (as defined in NAGPRA), during plant tissue and co-located soil sampling and associated activity that could result in ground disturbance.

4.2.1.1 Notification of Planned Plant Sampling

TAI shall notify EPA at least 15 days in advance of any sample collection activity, unless shorter notice is agreed to by EPA. Notification to EPA may be provided by e-mail or by letter. As for all RI/FS activities at the Site involving ground penetration and disturbance, TAI shall work with potentially affected parties to assess the effects of the planned work and seek ways to avoid, minimize, or mitigate any adverse effects on historic properties. Further, plant tissue and co-located soil sampling cannot be performed at the Site without 1) clearance of

proposed sample locations by tribal and federal/state cultural resources coordinators, 2) a cultural monitor present on site with each field crew conducting plant tissue and co-located soil sampling unless otherwise indicated by CCT and; 3) approval by EPA.

The names and contact information for potentially affected parties (i.e., representatives of the federal land-managing agencies and tribes) are provided in Attachment C1 of this plan. TAI will work with EPA to establish a procedure for timely notification of these parties.

4.2.1.2 Professional Archaeologist and/or Tribal Cultural Monitor On Site

An archaeological monitor and/or tribal representative will be present on-site when ground-disturbing sampling or sampling-related activity occurs, unless CCT specifies one is not necessary. The archaeological monitor and/or tribal representative will visually examine all samples to determine if evident or likely artifacts are present or if other deposits are present that are likely to be cultural in origin. The archaeological monitor and/or tribal representative will not make physical contact with the soil in the sample unless artifacts or other cultural deposits are present. If artifacts or likely archaeological deposits are present, the archaeologist or tribal representative will record the location of the materials and photograph the materials in place in such a manner to provide information on provenience. The artifacts and other archaeological materials will then be re-deposited at their original location.

The archaeological monitor and/or tribal representative will document their observations on a daily basis, including field notes and photographs that record the location and character of the sampling or other ground-disturbing activity, any archaeological discoveries made, and any decisions made within the provisions of this plan by the archaeological monitor and tribal representative in response to any archaeological discoveries. A standardized archaeological monitoring form may be substituted for the field notes referenced above.

All archaeological monitors and tribal representatives will be required to have read the applicable health and safety plan and to have complete understanding of the archaeological monitoring provisions of this plan. The archaeological monitors will also be required to meet requirements for personal protective equipment. In addition, all on-site personnel are subject to the directions of the task field supervisor at all times.

4.2.1.3 Discoveries—Archaeological Monitors Present

At the discretion of the archaeological monitor or tribal representative, ground-disturbing sampling or associated activity may be slowed or halted at any time that a suspected archaeological object or archaeological resource is encountered. The objective of this slowing or halting of ground-disturbing activity is to allow the archaeologist to confirm

and/or make a preliminary assessment of the discovery. At the discretion of the archaeological monitor or tribal representative, a specific sample may be relocated from the location of the discovery but at the sampling location. Such relocation will be coordinated with the on-site sampling manager or supervisor.

At the request of the archaeological monitor or tribal representative, the sampling personnel will either

- Assist in securing access to the location of the discovery and take appropriate measures to protect the location of the discovery from rain, stormwater, and other possible disturbances, or
- Assist in moving the artifacts to a protected and secure area of the site away from the immediate sampling area. Removal of artifacts from the discovery location will be undertaken only if leaving the artifacts in place would jeopardize their integrity due to erosion or collection by unauthorized individuals.

The archaeological monitor, tribal representative, or a member of the TAI technical team will remain on site to ensure the security of the find until more extensive efforts can be made to secure the site from further disturbance or a more extensive evaluation and documentation of the discovery can be made.

Notification of any archaeological discoveries must be provided to EPA for further coordination with consulting parties within 24 hours of the discovery. All telephone notification of discoveries must be promptly followed by notification in writing (via e-mail or conventional mail).

4.2.1.4 Discovery of Human Remains

Native peoples in the study area consider the graves of their ancestors to be important in both their cultural identity and in defining their relationship with the land. These graves are therefore considered sacred and should be left undisturbed. Should inadvertent disturbance occur, the remains and associated materials (funerary objects) must be treated with respect and honor. All appropriate federal, tribal, and state laws, regulations, and procedures regarding burials should be rigorously enforced.

In the event that likely or confirmed human remains are encountered, all further sampling or other ground-disturbing activity will cease immediately. To comply with 43 CFR 10.4(b), any discoveries of human remains must be reported to the NPS and USBR immediately by telephone, followed by written notification. Any discoveries within the boundaries of the CCT or the STI reservations must also be reported immediately to the respective tribe.

TAI will notify EPA for further coordination with consulting parties (consisting minimally of the NPS, USBR, CCT, STI, and the Washington SHPO). The TAI technical team will assist the archaeological monitor and tribal representative in securing the location of the discovery.

If no archaeological monitor or tribal representative is present, the TAI technical team will secure the location of the discovery in such a manner that both maintains the physical integrity of the remains and any associated objects and precludes further disturbance, or a member of the TAI technical team will remain on site until an archaeologist or tribal representative can arrive to assess the find.

Other conditions for responses to discoveries of archaeological materials may be defined in the permits issued for the sampling program. Responses to any discoveries of burials must comply with provisions of NAGPRA and its implementing regulations (in addition to those referenced above), as well as the existing protocols of the NPS, USBR, CCT, and STI (copies of these protocols are provided in Attachment C1).

4.2.2 Curation

Artifacts and other cultural materials that may be recovered during the sampling program (with the exception of human remains and associated items subject to NAGPRA) will be curated at a facility that meets the standards of 36 CFR 79. The appropriate facility or facilities will be designated by the NPS and USBR in consultation with the tribes for items recovered from federal lands. The appropriate tribe will designate the curation facility for cultural materials recovered from tribal lands.

4.2.3 Reporting

Within 150 days of completion of each sampling activity that is covered under this plan,[4] a professional archaeologist will prepare a confidential[5] written report that presents the results of the archaeological monitoring and responses to any discoveries of archaeological resources or burials. The report will include 1) copies of field notes, descriptions, and maps of all locations at which sampling-related archaeological monitoring was conducted; 2) descriptions of any discoveries made during such monitoring and the outcome of the discoveries (including the rationale for the decisions for the disposition of any finds); 3) descriptions and maps of all non-monitored locations at which inadvertent discoveries

[4] Sampling or other RI/FS activities that do not require coordination under this plan will not result in generation of this reporting requirement.

[5] Refer to Section 4.3, Confidentiality.

were made and the outcome of those discoveries; and 4) recommendations for any changes in the monitoring protocol or coordination plan that may be appropriate to address results of the monitoring or how well existing coordination procedures worked. A standardized archaeological monitoring form may be substituted for the field notes referenced above.

The draft report will be provided to EPA for review and dissemination to the consulting parties for review and comment.

4.3 CONFIDENTIALITY

TAI shall make its best efforts, in accordance with state and federal law, to ensure that its employees and contractors keep the discovery of any found or suspected human remains, other cultural items, and potential historic properties confidential. Pertinent TAI employees and contractors will be required to read and sign a confidentiality statement that specifies procedures to be followed in response to media and public contacts regarding archaeological and other cultural resources. To the extent permitted by law, prior to any release of information, EPA, TAI, and the other consulting parties shall concur on the amount of information, if any, to be released to the public, any third party, and the media, and the procedures for such a release.

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6 GLOSSARY OF TERMS

Burial—A burial is defined in NAGPRA as "[a]ny natural or prepared physical location, whether originally below, on, or above the surface of the earth, into which as part of the death rite or ceremony of a culture, individual human remains are deposited."

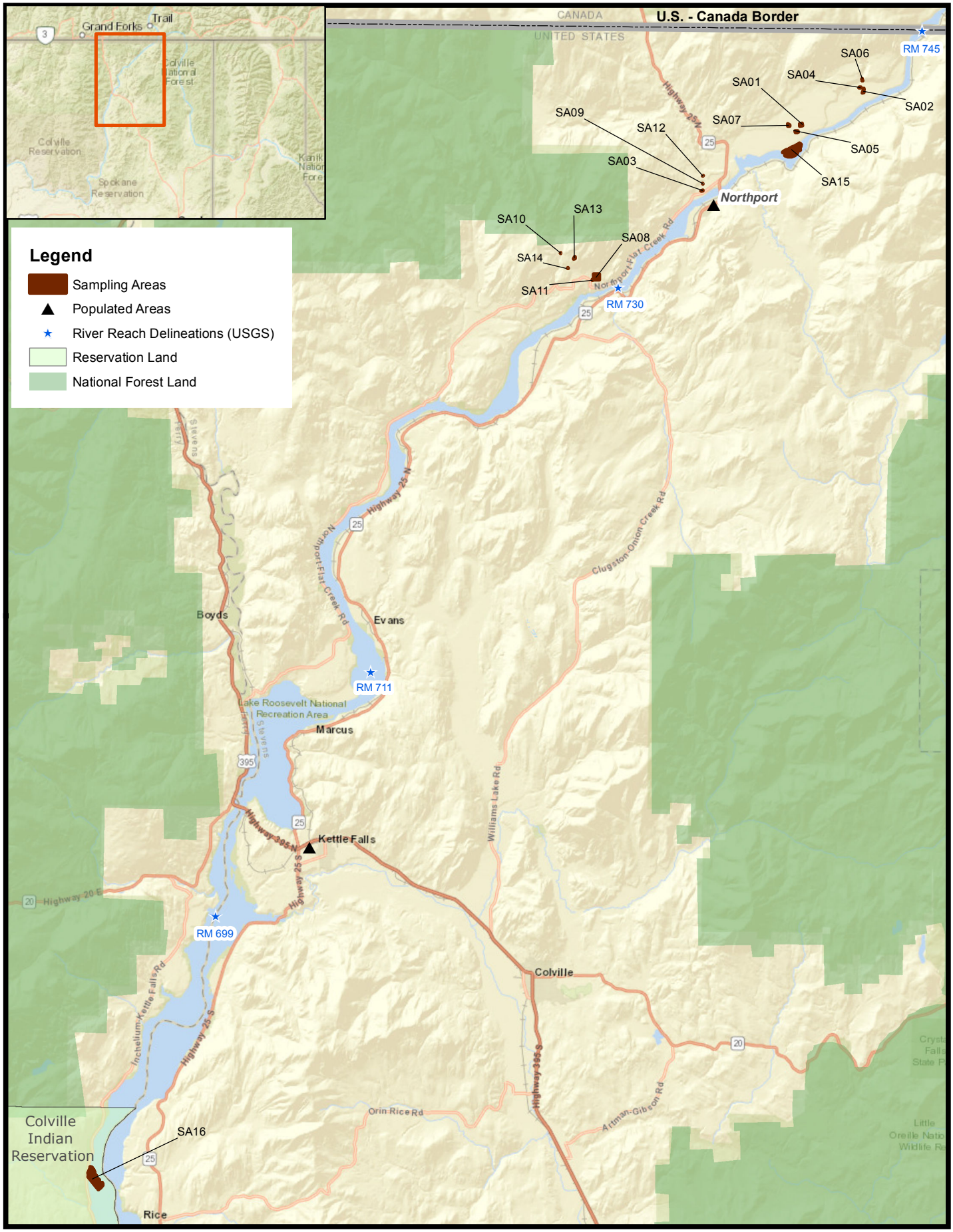
Curation—Long-term storage and preservation of archaeological collections. Archaeological collections from federal lands must be curated at facilities that meet the standards of 36 CFR 79.

Ethnohistoric—Information on Native peoples gathered from historical accounts.

Historic, historic-period, historical—The NHPA uses the term "historic" to refer to properties that are listed or have been determined eligible for listing on the National Register of Historic Places. To avoid confusion with this definition of "historic," "historic-period" or "historical" are used to reference resources, places, events, and people associated with the period since the appearance of Euroamericans and the beginning of written accounts (ca. 1780–1810 in the Pacific Northwest).

Protohistoric—The period of time transitional from prehistory to history. In the Pacific Northwest, protohistoric can be generally defined as from the late 1600s until late 1700s.

Map



RAMBOLL ENVIRON

0 1 2 4 Kilometers

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Map C1. Proposed Plant Study Sampling Areas

Upper Columbia River, WA

TABLE

Table C1. Proposed Sampling Areas

Sampling Area ID	Overlap with Prior UCR RI/FS Sampling Area[a]	Type of Property Ownership/ Management	Rationale for Inclusion	Average IC Soil Lead (mg/kg)[a]	Average IC Soil Arsenic (mg/kg)[a]
SA01	2014R-258	Tribal allotment	High soil lead. Sarvisberry, kinnikinnick, black tree lichen, ponderosa pine, chokecherry, wild rose, camas and wild strawberry recorded during August 2017 field reconnaissance, as well as habitat for bitterroot, lomatium, morel, shaggy mane, spring beauty, and wild caraway.	678	46.8
SA02	2014R-401	Tribal allotment	High soil lead. Sarvisberry, kinnikinnick, ponderosa pine, chokecherry, and hazelnut recorded during August 2017 field reconnaissance, as well as habitat for morels.	1120	80.8
SA03	2014R-441	Tribal allotment	High soil lead. Sarvisberry, kinnikinnick, ponderosa pine, chokecherry, wild rose, hazelnut, and wild strawberry recorded during August 2017 field reconnaissance, as well as habitat for morels.	624	43.6
SA04	2014R-402	Tribal allotment	Sarvisberry, kinnikinnick, ponderosa pine, chokecherry, wild rose, hazelnut, dwarf huckleberry, and wild strawberry recorded during August 2017 field reconnaissance, as well as habitat for bitterroot, lomatium, morel, shaggy mane, and wild caraway.	542	34
SA05	2014R-410	Tribal allotment	Sarvisberry, kinnikinnick, black tree lichen, ponderosa pine, chokecherry, and wild rose recorded during August 2017 field reconnaissance, as well as habitat for bitterroot, lomatium, shaggy mane, and wild caraway.	370	35
SA06	2014R-403	Tribal allotment	Sarvisberry, kinnikinnick, wild rose, and hazelnut recorded during August 2017 field reconnaissance, as well as habitat for shaggy mane.	394	26.1
SA07	2014R-259	Tribal allotment	Sarvisberry, black tree lichen, ponderosa pine, chokecherry, and wild rose recorded during August 2017 field reconnaissance, as well as habitat for bitterroot, lomatium, shaggy mane, and wild caraway.	226	19.7

Table C1. Proposed Sampling Areas

Sampling Area ID	Overlap with Prior UCR RI/FS Sampling Area[a]	Type of Property Ownership/ Management	Rationale for Inclusion	Average IC Soil Lead (mg/kg)[a]	Average IC Soil Arsenic (mg/kg)[a]
SA08	2014U-ADA-023	Tribal allotment	Sarvisberry, black tree lichen, ponderosa pine, chokecherry, wild rose, and hazelnut recorded during August 2017 field reconnaissance, as well as habitat for bitterroot, lomatium, shaggy mane, and wild caraway.	151	18.3
SA09	2014R-442	Tribal allotment	Sarvisberry, ponderosa pine, chokecherry, wild rose, red willow, hazelnut, and wild strawberry recorded during August 2017 field reconnaissance, as well as habitat for shaggy mane, spring beauty, and wild caraway.	243	21.7
SA10	2016R-808-O2	Tribal allotment	Sarvisberry, kinnikinnick, ponderosa pine, puffball, and wild rose recorded during August 2017 field reconnaissance, as well as habitat for bitterroot, lomatium, shaggy mane, and wild caraway.	42.6	6.98
SA11	2016R-804-O1	Tribal allotment	Sarvisberry, black tree lichen, ponderosa pine, wild rose, and hazelnut recorded during August 2017 field reconnaissance, as well as habitat for bitterroot, lomatium, shaggy mane, and wild caraway.	121	7.74
SA12	2014R-440	Tribal allotment	Sarvisberry, black tree lichen, chokecherry, and hazelnut recorded during August 2017 field reconnaissance, as well as habitat for shaggy mane, spring beauty, and wild caraway.	136	9.12
SA13	2016R-801-O3	Tribal allotment	Sarvisberry, chokecherry, and wild rose recorded during August 2017 field reconnaissance, as well as habitat for bitterroot, lomatium, shaggy mane, and wild caraway.	37	15.1
SA14	2016R-805-O2	Tribal allotment	Sarvisberry, black tree lichen, wild rose, hazelnut, wild mint, and tule recorded during August 2017 field reconnaissance, as well as habitat for bitterroot, lomatium, shaggy mane, and wild caraway.	54.1	5.31

Table C1. Proposed Sampling Areas

Sampling Area ID	Overlap with Prior UCR RI/FS Sampling Area[a]	Type of Property Ownership/ Management	Rationale for Inclusion	Average IC Soil Lead (mg/kg)[a]	Average IC Soil Arsenic (mg/kg)[a]
SA15[b]	RFA-001, RFA-002, RFA-003, RFA-004, RFA-005	Washington Department of Natural Resources	Possible presence of willows in a previously sampled area with moderately high concentrations of lead in relict floodplain soil.	389[c]	15.8[c]
SA16[b]	Barnaby Island Campground	National Park Service	Possible presence of willows in a previously sampled area with low concentrations of lead in beach sediment.	46.2[d]	1.99[d]

Notes:

[a] Based on the UCR 2010 Beach Sediment Study (Integral 2014), the UCR 2014 Residential Soil Study (CH2M Hill 2016), the UCR 2014 Upland Soil Study (Windward et al. 2015), or the UCR 2016 Residential Soil Study (Ramboll Environ 2017).

[b] Sampling areas were not visited during the August 2017 field reconnaissance event. Areas are publicly-accessible and are included for potential sampling of willows.

[c] These averages represent the average of pre-averaged replicates for each of the five decision units sampled.

[d] Averages are based on sample results for <63 µm, 63 to 125 µm, and 125 to 250 µm size fraction samples.

IC - incremental composite

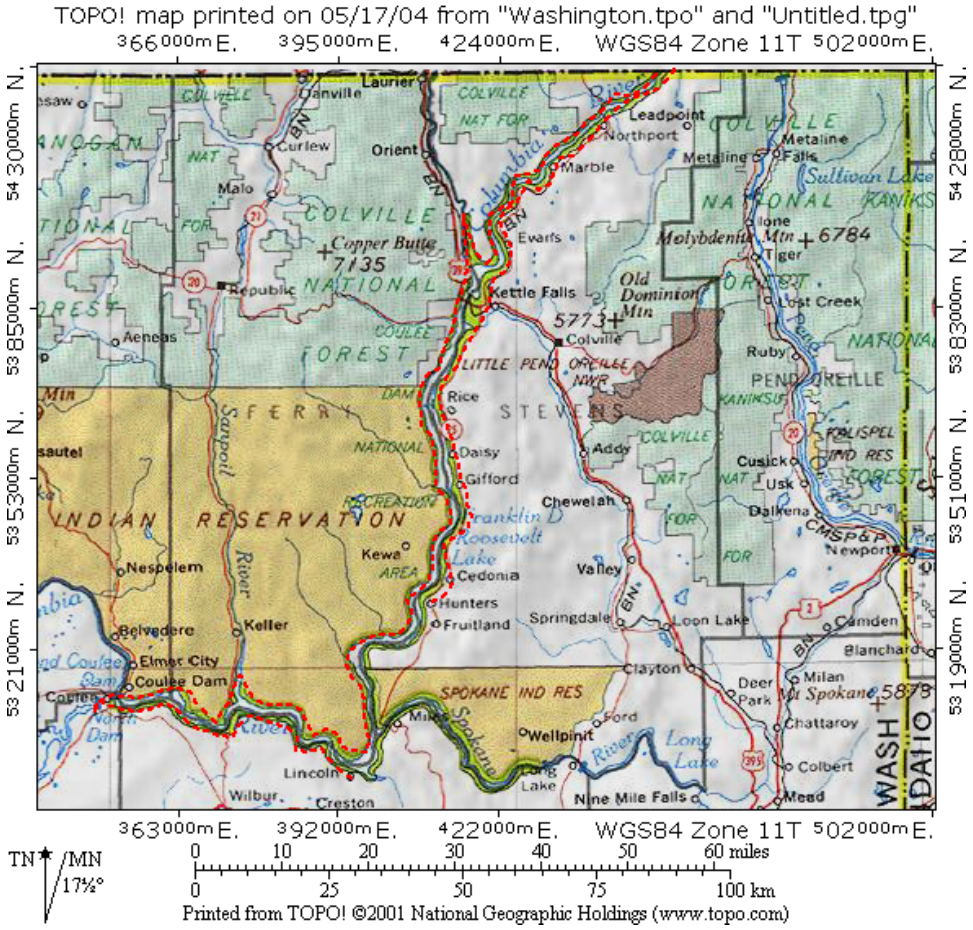
ATTACHMENT C1

PROTOCOLS FOR INADVERTENT DISCOVERIES

NAGPRA Inadvertent Discoveries or Intentional Excavations: Confederated Tribes of the Colville Reservation, National Park Service, and the Bureau of Reclamation

Lake Roosevelt Protocols for Native American Graves Protection and Repatriation Act (NAGPRA) Inadvertent Discoveries or Intentional Excavations: Confederated Tribes of the Colville Reservation, National Park Service, and the Bureau of Reclamation

This protocol is intended to cover NAGPRA items exposed by inadvertent discoveries or intentional excavations within the boundaries of lands managed by the National Park Service (NPS)/Lake Roosevelt National Recreation Area. The term "NAGPRA items" in this document refers to human NAGPRA items, associated funerary objects, and objects of cultural patrimony as they are defined in 25 USC 3001. This document does not address inadvertent discoveries on lands within reservation boundaries or trust land outside of the reservation boundaries of the Confederated Tribes of the Colville Reservation (CCT). Funding of actions is not covered under this protocol.



Map of Lake Roosevelt National Recreation Area

This protocol covers those areas highlighted in red within the recreation area, which is the yellow highlighted portion of the Lake Roosevelt shoreline.

1. If NAGPRA items that are potentially human are encountered, any activity in the vicinity of the discovery shall cease and all reasonable efforts shall be made to protect the NAGPRA items and all appropriate effort shall be made to determine if the NAGPRA items are human. The activity shall resume only when clearance to proceed is received by the CCT Tribal Historic Preservation Officer and the National Park Service's designated official.

2. If the NAGPRA items are determined to be human, the burial or location shall not be disturbed in any way. Any discovered human NAGPRA items and associated artifacts will be treated in a respectful manner.

3. In cases where a potential crime scene exists, *personnel except those necessary to protect the location will leave the immediate vicinity in order to prevent unintentional destruction of crime scene information.* A National Park Service law enforcement officer will be immediately notified.

4. The Colville Tribal Historic Preservation Officer and the archaeologists working for the Colville Tribes and the Park Service (numbers listed below) will also be contacted immediately after law enforcement. For NAGPRA discoveries associated with the Lake Roosevelt shoreline, the Reclamation archaeologist must also be contacted. Live phone contact is required; backup staff are identified if the primary contacts are unavailable. Phone contact will be followed up by written confirmation, e-mail is acceptable. E-mail should not include detailed (site specific information) for security reasons.

5. A professional archaeologist will assist law enforcement in determining if the NAGPRA items are archaeological in origin. If the crime scene is ARPA-related (i.e., there is evidence for intentional disturbance or looting of archaeological materials), an archaeologist shall assist law enforcement as needed in the collection of archeological data to support the ARPA case.

6. Guy Moura, CCT THPO and Program Manager of the CCT History/Archaeology Program is the primary contact for the CCT. Mr. Moura's phone number at the Program is (509) 634-2695 and email is guy.moura@colvilletribes.com. After hours, Mr. Moura can be contacted at (509) 631-1705 (cell). If Mr. Moura cannot be reached, then Brenda Convington, Senior Archaeologist is the alternate contact at (509) 634-2699 (office) or (509) 634-1737 (cell) and at brenda.covington@colvilletribes.com. In the event that neither Mr. Moura or Ms. Covington cannot be contacted, then Arrow Coyote, CCT Senior Archaeologist will be contacted at (509) 634-2736 (office) or (509) 634-1280 (cell) and at arrow.coyote@colvilletribes.com. Ms. Covington or Ms. Coyote shall participate in the NAGPRA consultation process on Mr. Moura's behalf until his return. Jackie Cook, Repatriation Specialist, will also participate in the NAGPRA consultation process. Ms. Cook's contact information is (509) 634-2635 (office) or (509) 631-1176 (cell) and jackie.cook@colvilletribes.com. The CCT shall maintain a presence at the

location of the discovery as needed until all contacts have been made and appropriate treatment of the NAGPRA items has been conducted.

Keith Holliday, NPS Project Manager for the Lake Roosevelt National Recreation Area, is the primary contact for the NPS. Mr. Holliday's phone number is (509) 754-7858, FAX is (509) 738-3108, and e-mail address is keith_holliday@nps.gov.

Justin Eichelberger, NPS Archaeologist, is also a contact person for the NPS. Mr. Eichelberger's phone number is (509) 754-7860 or (509) 631-4191 (cell), and e-mail address is Justin_eichelberger@nps.gov.

Derek Beery, Power Office Archaeologist, is Reclamation's contact. His phone numbers are (509) 633-9233 [desk] and (509) 237-4477 [cell phone], his FAX number is (509) 633-9138, and e-mail address is dbeery@usbr.gov. If Derek Beery is not available, contact Sean Hess, Regional Archaeologist, at (208) 378-5316, FAX (208) 378-5305, or at e-mail address shess@usbr.gov.

7. As soon as the NAGPRA items have been determined to be human, then all effort shall be made in the field to determine whether human NAGPRA items are Native American. If yes, skip steps 8 and 9 below and proceed to step 10.

8. If the NAGPRA items are determined not to be Native American, then Washington State laws apply and shall be followed (Title 68, Chapter 68.50 RCW HUMAN NAGPRA ITEMS).

9. If the NAGPRA items' affiliation cannot be determined in the field, further non-destructive analysis of human NAGPRA items and/or associated cultural materials may be required. The CCT, NPS, and Reclamation shall coordinate regarding the types of non-destructive analysis to be conducted.

10. Provenience information will be collected as specified by the written plan of action. The Reclamation contract language for burials recovered in the shoreline of the National Recreation Area will also apply and should agree with the written plan of action and these protocols.

11. Recording of provenience may include any or all of the following: documenting the location of the burial or scattered NAGPRA items and general site conditions on a site form or on an addendum to an existing form; describing the surface visible NAGPRA items to the degree that can be accomplished without causing additional disturbance to the grave; documenting the location of the burial on a USGS 7.5' topographic sheet and with a GPS unit.

12. If it is possible to rebury or cap the NAGPRA items in place, then that decision shall be documented in the written plan of action (see below).

13. If NAGPRA items must be excavated or removed, procedures will be specified by the written plan of action. The Reclamation contract language for burials recovered in the shoreline of the NRA will also apply and should agree with the written plan of action and these protocols. If NAGPRA items are to be excavated or removed by personnel other than those employed by the CCT or the U.S. government, an ARPA permit will be required from the NPS.

14. Excavation or removal procedures may include any or all of the following: NAGPRA items will be removed using standard professional archaeological practices in a culturally sensitive manner at the direction of a CCT History/Archaeology Department representative. Such practices may include collection of horizontal provenience data referenced to a site datum point; if excavation is required, vertical provenience data shall be tracked through the use of controlled 10-cm levels within a standard grid unit, screening of all excavated fill through 1/8-inch screen mesh, and photographic and to-scale plan map documentation of excavated features. All recovered items shall be listed in the field during collection to minimize handling after recovery.

15. Inadvertent discoveries that result from activities requiring easements or other non-ARPA permits (such as access, construction, etc.) shall be dealt with by the permitting agencies, which may be Reclamation or the NPS. This protocol document will be included with documents issued to permittees.

16. The written plans of action for individual discoveries will detail exact procedures for further implementation of NAGPRA. A sample written plan of action is attached.

Template NAGPRA Plan of Action for Lake Roosevelt

This plan of action shall comply with the requirements of the Native American Graves Protection and Repatriation Act (NAGPRA) (25 USC 3001 et seq.), its implementing regulations (43 CFR Part 10) and the Archaeological Resources Protection Act (ARPA) (16 USC 470 et seq.) with its implementing regulations (43 CFR Part 7).

1. The kinds of objects to be considered as cultural items as defined in Sec. 10.2 (b):
   - ✓ Human remains
   - ✓ Associated funerary objects
   - ✓ Unassociated funerary objects
   - ✓ Objects of cultural patrimony
   - ✓ Sacred objects

   These objects are cultural objects as defined under NAGPRA 43CFR Part 10.2 (d)

2. The specific information used to determine custody pursuant to Sec. 10.6:
   - ✓ Traditional association (this is where tribe's area of interest is cited with reference to Lake Roosevelt)
   - ✓ Cultural affiliation
   - ✓ Evidence: Geographical, archaeological, linguistic, folklore, oral tradition, historical

3. The planned treatment, care, and handling of human remains and other objects as defined in NAGPRA

4. The planned archaeological recording of the human remains and other objects as defined in NAGPRA

5. The kinds of analysis planned for each kind of object

6. Any steps to be followed to contact Indian tribe officials at the time of intentional excavation or inadvertent discovery of specific human remains and other objects as defined in NAGPRA

7. The kind of traditional treatment, if any, to be afforded the human remains and other objects as defined in NAGPRA by members of the Indian tribe

8. The nature of reports to be prepared

9. The planned disposition of human remains, and other objects as defined in NAGPRA.

NAGPRA Inadvertent Discoveries and Intentional Excavations on the Lake Roosevelt National Recreation Area: Spokane Tribe of Indians, National Park Service, and Bureau of Reclamation

Protocols for NAGPRA Inadvertent Discoveries and Intentional Excavations on the Lake Roosevelt National Recreation Area: Spokane Tribe of Indians, National Park Service, and Bureau of Reclamation

This protocol is intended to cover NAGPRA items exposed by inadvertent discoveries and intentional excavations within the boundaries of lands managed by the National Park Service/Lake Roosevelt National Recreation Area (Figure 1), excluding inadvertent discoveries on lands within reservation boundaries of the Spokane Tribe of Indians (STI) (Figure 2). For procedures within STI reservation boundaries (as shown in Figure 2 along the left bank [east side of the Columbia River], from the mouth of the Spokane River and north to the Spokane Reservation boundary) please see the Spokane Tribe's *Procedure for the Inadvertent Disturbance or Discovery of Spokane Human Remains and Cultural Resources*. Funding of actions is not covered under this protocol.

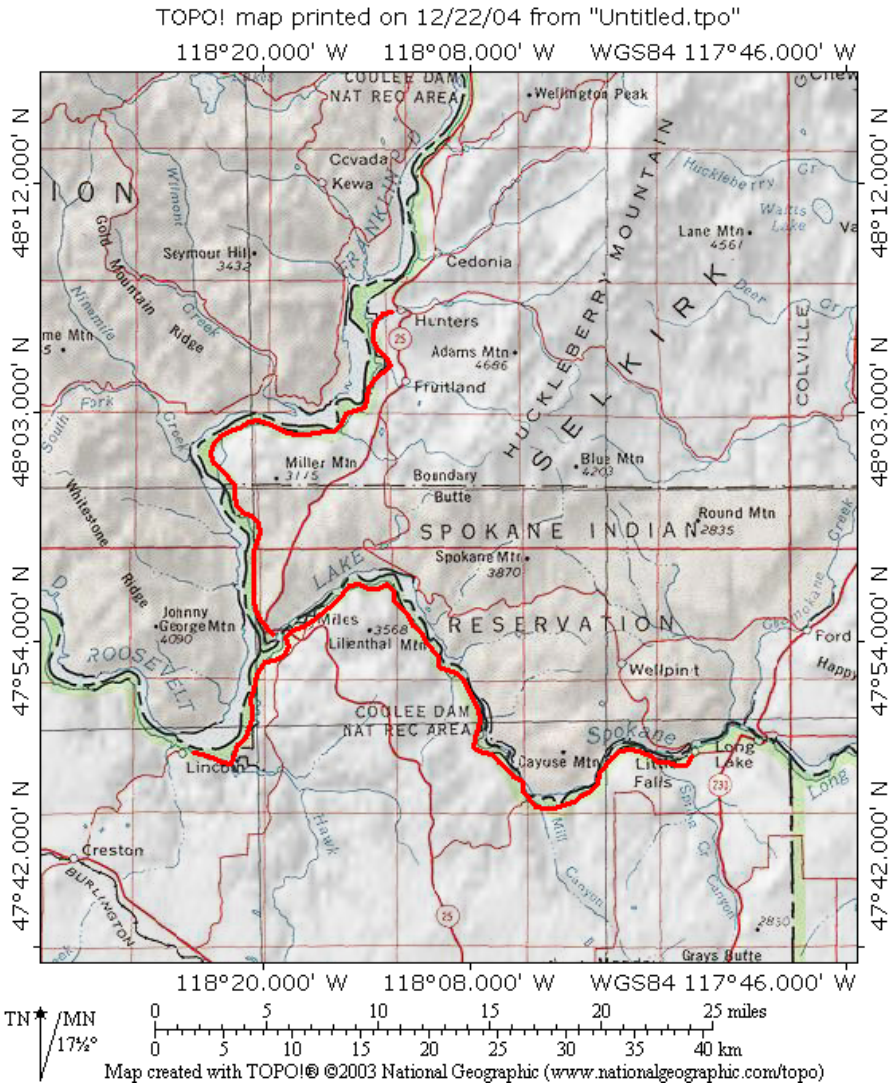


Figure 1. Lake Roosevelt National Recreation Area Shoreline Areas Managed by the National Park Service and Bureau of Reclamation

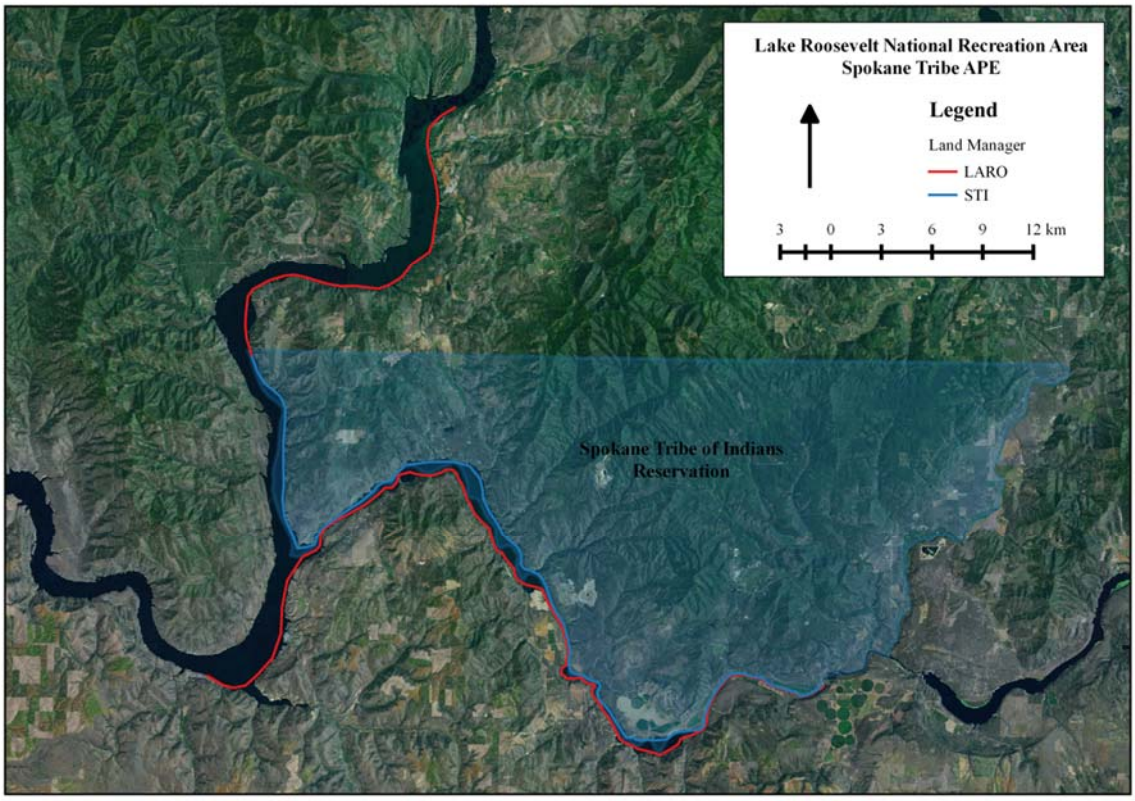


Figure 2. Spokane Tribe of Indians Reservation Land Not Covered by NPS Protocol

1. If remains that are potentially human are encountered, any activity in the vicinity of the discovery shall cease and all appropriate effort shall be made to determine if the remains are human. NAGPRA dictates that the 'stop work' order shall be for 30 days, but this period can be shortened in consultation between affected parties.

2. If the remains are determined to be human, the burial or location shall not be disturbed in any way. Any discovered human remains and associated artifacts will be treated in a respectful manner.

3. The person(s) making the discovery shall immediately notify NPS law enforcement. In cases where a potential crime scene exists, *personnel except those necessary to protect the location will leave the immediate vicinity in order to prevent unintentional destruction of crime scene information.*

4. The person(s) making the discovery shall immediately notify the Spokane Tribal Historic Preservation Officer (STI THPO), the Park Service archaeologist, and the Reclamation archaeologist (numbers are listed below) immediately after law enforcement.

   Live phone contact is required; backup staff are identified if the primary contacts are unavailable. Phone contact will be followed up by written confirmation, e-mail is acceptable.

5. **Notifications:**

   - Randy Abrahamson, STI THPO, is the primary contact for the STI. Mr. Abrahamson's phone number at the Department is (509) 258-4315, FAX (509) 258-6965, and his e-mail address is randya@spokanetribe.com. After work hours, Mr. Abrahamson can generally be reached at (509) 951-0524 (cell). If Mr. Abrahamson cannot be reached, John Matt (Preservation Department Director), James Harrison (Principal Investigator), Jackie Corley(Tribal Archaeologist), Laura McCullough (Project Archaeologist), or Chris Casserino (Project Archaeologist) shall be contacted at (509) 258-4060. If none of the above people can be reached, then the on-site STI crew leader shall be presumed delegated as the primary STI representative and shall participate in the NAGPRA consultation process until Mr. Abrahamson's return. The STI shall maintain a presence at the location of the discovery as needed until all contacts have been made and appropriate treatment of the remains has been conducted.

   - Derek Beery, Power Office Archaeologist, is Reclamation's contact. His phone number is (509) 237-4477 [cell phone], (509) 633-9233 [desk] FAX 633-9138, and e-mail address is "dbeery@usbr.gov." If Derek Beery is not available, contact Sean Hess, Regional Archaeologist (208) 378-5316, FAX (208) 378-5305, and e-mail address is shess@usbr.gov.

   - Keith Holliday, NPS Project Manager for the Lake Roosevelt National Recreation Area, is the primary contact for the NPS. Mr. Holliday's phone number is (509) 754-7858 or (509) 631-0306, and his FAX is (509) 738-3108, and e-mail address is keith_holliday@nps.gov. .

   - Spokane Tribal Law Enforcement can be reached at 1-888-258-6899 and/or 258-7766, and at (509) 633-9441, ext. 123. If Tribal Law Enforcement is not available, the North District Ranger number is (509) 738-6266 ext. 162 or cell (509) 631-4722.

6. A professional archaeologist will assist law enforcement in determining if the remains are archaeological in origin. If the discovery is determined to be a recent crime scene, field personnel shall follow direction from law enforcement officers.

7. If the discovery is determined to be an ARPA crime scene (i.e., there is evidence for intentional disturbance or looting of archaeological materials), an archaeologist shall assist law enforcement as needed in the collection of archeological data to support the ARPA case.

8. If the discovery is determined not to be a crime scene, an attempt will be made to determine whether the remains are human remains.

9. Documentation: If the remains are human, the location of the burial or scattered remains and general site conditions shall be documented. Documentation will include locating the burial on a USGS 7.5' topographic sheet and with a GPS unit, and recording the location on a site form or on an addendum to an existing form. Surface visible remains will be described to the degree that can be accomplished without causing any additional disturbance.

   If NAGPRA applies to the remains, a written plan of action will be drafted by the NPS and Reclamation archaeologists in coordination with the STI THPO. The party responsible for making the NAGPRA determination must document in writing the basis of that determination. Documentation methods will be described in the written plan of action for each discovery.

10. If possible and if agreed upon by all parties, human remains and associated objects shall be protected in place. If it is possible to rebury or cap the remains in place, then further actions under NAGPRA are not required. If the tribe prefers, protective actions can be conducted after locational information is collected.

11. If it is not possible to protect the remains in place, all efforts shall be made to determine in the field whether NAGPRA applies to the human remains. If NAGPRA does not pertain to the discovered remains, then WA state laws apply and shall be followed (Chapter 27.44 RCW: INDIAN GRAVES AND RECORDS, at http://www.oahp.wa.gov/rcw2744.htm).

12. Recovery: Remains or associated items that cannot be protected in place shall be recovered in a culturally sensitive manner according to the written plan of action developed by the STI, the NPS, and Reclamation. If remains are threatened and must be recovered before a written plan of action can be completed, the steps identified below shall be followed, at minimum:

    Collection of horizontal provenience data referenced to a site datum point; if excavation is required, vertical provenience data shall be tracked through the use of controlled 10-cm levels within a standard grid unit, screening of all excavated fill through 1/4-inch screen mesh (1/8-inch if sediments are sand), and (No photography, etc. if NAGPRA) of excavated features. Methods employed shall be designed to document information about burial practices and to recover any associated grave goods.

13. The NPS shall publish Notices of Intent to Make Disposition in local newspapers. The newspapers shall be named in the Written Plan of Action for each discovery.

14. After recovery and during the 30-day waiting period after newspaper notices are published by the NPS, NAGPRA items shall be stored and protected by the STI.

15. The written plans of action for individual discoveries within the Lake Roosevelt National Recreation Area will detail exact procedures for further implementation of NAGPRA.