UPPER COLUMBIA RIVER

FINAL
Quality Assurance Project Plan for the Macroinvertebrate Tissue Study

Prepared for

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

Prepared by

15375 SE 30th Place, Suite 250
Bellevue, WA 98007

1 International Boulevard, 10th Floor, Suite 1000
Mahwah, NJ 07495

In Association and Consultation with

Parametrix, Inc.
Cardwell Consulting, LLC
Windward Environmental LLC
And
Ramboll Environ

April 2016

SECTION A: PROJECT MANAGEMENT

A1 TITLE AND APPROVAL SHEET

QUALITY ASSURANCE PROJECT PLAN FOR THE MACROINVERTEBRATE TISSUE STUDY

Role	Name	Date
EPA Project Manager	Laura Buelow	Date 4/18/16
EPA Project Manager	Dustan Bott	Date 4/18/16
TAI Project Coordinator	Kris McCaig	Date 4/14/16
EPA Region 10 Quality Assurance Manager	Donald Brown	Date 4/18/16
TAI Technical Coordinator	John Toll	Date 4/14/16
Principal Investigator	Berit Bergquist	Date 4/14/16
Senior Technical Advisor	Rick Cardwell	Date 4/14/2016
Senior Technical Advisor	Dina Johnson	Date 4/14/2016
Senior Technical Advisor	Nicholas Gard	Date 04/14/16
Task QA Coordinator	Rock Vitale	Date 4/14/2016
Analytical Chemistry Laboratory Coordinator	Dave Enos	Date 04/14/16
Chemistry Laboratory Project Manager (ALS)	Jeff Christian	Date 04/14/2016
Chemistry Laboratory QA Manager (ALS)	Lee Wolf (for LW)	Date 04/14/2016
Chemistry Laboratory Project Manager (Vista)	Martha Maier	Date 4/14/2016
Chemistry Laboratory QA Manager (Vista)	Bahar Amiri	Date 4/14/2016
Database Administrator	Randy O'Boyle	Date 4/14/2016

A2 CONTENTS

SECTION A: PROJECT MANAGEMENT iii
A1 TITLE AND APPROVAL SHEET iii
A2 CONTENTS v
LIST OF APPENDICES vii
LIST OF FIGURES ix
LIST OF MAPS xi
LIST OF TABLES xiii
ACRONYMS AND ABBREVIATIONS xv
UNITS OF MEASURE xvii
A3 DISTRIBUTION LIST xix
A4 INTRODUCTION AND TASK ORGANIZATION A-1
A4.1 Introduction A-1
A4.2 Task Organization A-2
A4.2.1 EPA Organization and Responsibilities A-2
A4.2.2 TAI Organization and Responsibilities A-2
A4.2.3 Key Task Personnel A-2
A4.2.4 Laboratories A-4
A5 PROBLEM DEFINITION AND BACKGROUND A-5
A6 DATA NEEDS A-6
A7 DATA QUALITY OBJECTIVES, CRITERIA, AND DESIGN RATIONALE A-7
A7.1 Step 1—State the Problem A-7
A7.1.1 Team Members and Roles A-7
A7.1.2 Schedule A-7
A7.2 Step 2—Identify the Goal of the Study A-8
A7.3 Step 3—Identify Information Inputs A-8
A7.3.1 Sampling Areas and Number of Samples A-8
A7.3.2 Chemical Analyses A-10
A7.3.3 Species A-11
A7.4 Step 4—Define the Boundaries of the Study A-12
A7.4.1 Target Populations for Risk Evaluation A-12
A7.4.2 Geographic Boundaries of the Site A-12
A7.4.3 Temporal Considerations A-13
A7.5 Step 5—Define the Statistics and Types of Inferences A-14

A7.6 Step 6—Specify Performance or Acceptance Criteria A-15
A7.6.1 Sampling Completeness A-15
A7.6.2 Data Quality A-15
A7.7 Step 7—Develop the Plan for Collecting Data A-17
A8 SPECIAL TRAINING/CERTIFICATES A-17
A9 DOCUMENTATION AND RECORDS A-17
A9.1 Field Documentation A-18
A9.2 Chemistry Laboratories A-18
A9.3 Data Quality Documentation A-20
SECTION B: DATA GENERATION AND ACQUISITION B-1
B1 SAMPLING PROCESS DESIGN AND RATIONALE B-1
B1.1 Target Sample Locations and Rationale B-1
B2 SAMPLING METHODS B-4
B3 SAMPLE HANDLING AND CUSTODY B-4
B4 SAMPLE PROCESSING AND ANALYTICAL METHODS B-5
B5 QUALITY CONTROL B-6
B5.1 Analytical Laboratory Quality Control B-6
B5.2 Data Quality Indicators B-7
B6 INSTRUMENT/EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE B-10
B7 INSTRUMENT/EQUIPMENT CALIBRATION AND FREQUENCY B-10
B8 INSPECTION/ACCEPTANCE OF SUPPLIES AND CONSUMABLES B-11
B9 DATA MANAGEMENT B-11
B9.1 Field Data B-12
B9.2 Analytical Laboratory Data B-13
SECTION C: ASSESSMENT AND OVERSIGHT C-1
C1 ASSESSMENTS AND RESPONSE ACTIONS C-1
C2 REPORTS TO MANAGEMENT C-2
SECTION D: DATA VALIDATION AND USABILITY D-1
D1 DATA REVIEW, VERIFICATION, AND VALIDATION D-1
D2 VERIFICATION AND VALIDATION METHODS D-2
D3 RECONCILIATION WITH USER REQUIREMENTS D-3
SECTION E: REFERENCES E-1

LIST OF APPENDICES

Appendix A. Field Sampling Plan for the Macroinvertebrate Tissue Study

Appendix B. Overview of the U.S. Fish and Wildlife Service Crayfish and Mussel Surveys

Appendix C. Quality Assurance Manuals, SOPs, and Sample Benchsheets for ALS Environmental and Vista Analytical

Appendix D. Cultural Resources Coordination Plan

LIST OF FIGURES

Figure A5-1. Site-wide Conceptual Site Model

Figure A7-1. Lake Roosevelt Water Elevation at Grand Coulee Dam

LIST OF MAPS

Map A7-1. Proposed Mussel and Crayfish Sampling Locations

LIST OF TABLES

Table A4-1. Technical Team Task Member Information
Table A7-1. Sampling Areas
Table A7-2 Target Number of Samples to be Collected
Table A7-3. Methods and Sample Mass Requirements and Prioritization
Table A7-4. Macroinvertebrate Tissue Target Analyte List and Analytical Concentration Goals
Table A7-5. Quantitation Limits for PCB Congeners and Dioxins/Furans
Table B5-1. Standard Reference Materials by Analyte
Table B5-2. Measurement Quality Objectives for Macroinvertebrate Tissue

ACRONYMS AND ABBREVIATIONS

95th UCL	95th percentile upper confidence limit on the mean
Agreement	June 2, 2006, Settlement Agreement
ACG	analytical concentration goal
ALS	ALS Environmental
BERA	baseline ecological risk assessment
BSAF	biota sediment accumulation factor
BW	body weight
CCT	Confederated Tribes of the Colville Reservation
CLP	Contract Laboratory Program
COC	chain-of-custody
COI	chemical of interest
CSM	conceptual site model
DL	detection limit
DQO	data quality objective
DMP	data management plan
Ecology	Washington State Department of Ecology
EDD	electronic data deliverable
EDL	estimated detection limit
EMPC	estimated maximum possible concentration
EPA	U.S. Environmental Protection Agency
ESI	Environmental Services, Inc.
Exponent	Exponent, Inc.
FIR	food ingestion rate
FSP	field sampling plan
GIS	geographic information system
HHRA	human health risk assessment
HOC	hydrophobic organic compound
LCS	laboratory control sample
LPIL	lowest practical identification level
MDL	method detection limit
MQO	measurement quality objective
MRL	method reporting limit

MS	matrix spike
MSD	matrix spike duplicate
NIST	National Institute of Standards and Technology
NOAEL	no observed adverse effect level
NRCC	National Research Council Canada
PARCC	precision, accuracy or bias, representativeness, completeness, and comparability
PCB	polychlorinated biphenyl
QA	quality assurance
QA/QC	quality assurance and quality control
QAPP	quality assurance project plan
QC	quality control
QL	quantitation limit
RBC	risk-based concentrations
RI/FS	remedial investigation and feasibility study
RPD	relative percent difference
RSD	relative standard deviation
SHSP	site health and safety plan
Site	Upper Columbia River site
SLERA	screening level ecological risk assessment
SOP	standard operating procedure
SRM	standard reference material
STI	Spokane Tribe of Indians
TAI	Teck American Incorporated
TAL	target analyte list
TCDD	tetrachlorodibenzodioxin
TEF	toxic equivalency factor
TEQ	toxic equivalent
TRV	toxicity reference value
UCR	Upper Columbia River
USFWS	U.S. Fish and Wildlife Service
Vista	Vista Analytical
Windward	Windward Environmental LLC

UNITS OF MEASURE

°C	degree(s) Celsius
in.	inch(es)
g	gram(s)
g/day	gram(s) per day
kg	kilogram(s)
kg/d-wet	kilogram per day wet weight
mg/kg	milligram(s) per kilogram
mg/kg-day	milligram(s) per kilogram per day
ng/kg	nanogram(s) per kilogram
µg/kg	microgram(s) per kilogram

A3 DISTRIBUTION LIST

EPA Project Manager	Laura Buelow
EPA Project Manager	Dustan Bott
EPA Region 10 QA Manager	Donald Brown
TAI Project Coordinator	Kris McCaig
TAI Technical Team Coordinator	John Toll
Principal Investigator	Berit Bergquist
Task QA Coordinator	Rock Vitale
Senior Technical Advisor	Rick Cardwell
Senior Technical Advisor	Dina Johnson
Senior Technical Advisor	Nicholas Gard
Field Supervisor	TBD
Database Administrator	Randy O'Boyle
Analytical Chemistry Laboratory Coordinator	Dave Enos
Chemistry Laboratory Project Manager (ALS)	Jeff Christian
Chemistry Laboratory QA Manager (ALS)	Lee Wolf
Chemistry Laboratory Project Manager (Vista)	Martha Maier
Chemistry Laboratory QA Manager (Vista)	Bahar Amiri

A4 INTRODUCTION AND TASK ORGANIZATION

A4.1 Introduction

This document presents the quality assurance project plan (QAPP) for the benthic macroinvertebrate tissue study (herein referred to as 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 ecological risk assessment (BERA) by Teck American Incorporated (TAI), under U.S. Environmental Protection Agency (EPA) oversight, and the human health risk assessment (HHRA) being completed by EPA. The objective of the RI/FS is to investigate the nature and extent of contamination and unacceptable risk at the Site to humans and the environment.

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 chemicals in the tissues of macroinvertebrates sampled from the Site. Mussels and crayfish were selected as representative macroinvertebrates because they are commonly found in the UCR and are consumed by both people and wildlife. Data collection efforts will focus on obtaining information that will inform the exposure assessments for humans and wildlife receptors that consume these organisms from the Site. Chemistry data for mussels (soft body tissue) and crayfish (the whole body and shell, minus the carapace and stomach)[2] will be used in the HHRA to evaluate exposures of people who consume shellfish. Chemistry data for mussels (soft body tissue) and crayfish (all body parts, including the shell) will be used in the in the BERA to evaluate the exposure of aquatic-dependent, invertebrate-feeding fish and wildlife[3] that forage for mussels and crayfish in nearshore waters.

---

[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.

[2] Includes the head, claws, abdomen, tail, and shell around the claws and tail.

[3] Wildlife is defined to include amphibians, reptiles, birds, and mammals.

EPA's level of effort technical memorandum (USEPA 2013) was used to guide the development of the requirements and design rationale for data collection activities presented 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 the following parties: U.S. Department of the Interior, Washington State Department of Ecology (Ecology), and local tribes (i.e., the Confederated Tribes of the Colville Reservation [CCT] and the Spokane Tribe of Indians [STI]). In addition EPA, under Section 106 of the National Historic Preservation Act, has the primary responsibility for consulting with interested parties. EPA's project managers, Dr. Laura Buelow and Dustan Bott, 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 lab oversight as requested/necessary (i.e., data validation or lab 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). Dr. John Toll (Windward Environmental LLC [Windward]), TAI technical team coordinator, will be responsible for overseeing technical aspects of this task, coordinating with EPA, and managing the overall task schedule.

A4.2.3 Key Task Personnel

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

Technical Team Coordinator—Dr. Toll (Windward) will oversee task activities, review QA reports, and ensure that required activities are completed in sequence. Dr. Toll will work closely with TAI's project coordinator, principal investigator, and task QA coordinator to ensure that all requirements are met and study objectives achieved.

Principal Investigator— Berit Bergquist (Windward) 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. Ms. Bergquist will provide onsite supervision as needed and ensure that proper organism collection, preservation, storage, transport, and chain-of-custody (COC) procedures are followed. She will inform the technical team coordinator when problems occur and will communicate and document corrective actions taken.

Senior Technical Advisors—Dr. Rick Cardwell (Cardwell Consulting, LLC), Dr. Nicholas Gard (Exponent, Inc. [Exponent]), and Ms. Dina Johnson (Ramboll Environ) will serve as senior technical advisors for the study. They are responsible for providing technical oversight in the design, implementation, and data interpretation.

Task QA Coordinator—Rock Vitale (Environmental Services, 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—Dave Enos (TAI) is the analytical chemistry laboratory coordinator. He is responsible for ensuring that laboratory method selection and/or development is satisfactorily completed prior to the analysis of samples; coordinating with the testing laboratories and tracking the laboratories' progress; verifying that the laboratories have implemented the requirements of this QAPP; addressing QA issues related to the laboratories’ analyses; ensuring that the laboratories' capacities are sufficient to undertake the required analyses in a timely manner; and addressing scheduling issues related to the laboratories’ analyses. Mr. Enos will report directly to TAI’s project coordinator and will work closely with the technical team coordinator.

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 laboratories.

Field Supervisor—The field supervisor (to be determined) is responsible for overseeing the planning and coordination of the sampling efforts and for all aspects of mussel and crayfish collection activities to ensure that appropriate sampling, quality assurance, 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 representative(s) in the field, and TAI's project coordinator according to the established lines of communication among the TAI technical team, TAI, and EPA.

A4.2.4 Laboratories

The following responsibilities apply to respective project and QA managers at the analytical laboratories. ALS Environmental (ALS) will perform the sample processing and analyses for metals and conventional parameters, and Vista Analytical (Vista) will conduct the analyses for polychlorinated biphenyls (PCBs) and dioxins/furans. The analytical laboratories will have the following staff available for this project.

Analytical Chemistry Laboratory Project Managers—Jeff Christian (ALS) and Martha Maier (Vista) are 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, 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 laboratory standard operating procedures (SOPs)
- Apprising the laboratory coordinator of the schedule and status of sample analyses and data package preparation
- Notifying the 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 Managers—Lee Wolf (ALS) and Bahar Amiri (Vista) are 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 each 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 managers.

A5 PROBLEM DEFINITION AND BACKGROUND

Chemicals present in mussel and crayfish tissue may have the potential to adversely affect both people and ecological receptors through the ingestion exposure pathway. The preliminary conceptual site model (CSM) provides a framework for considering the relationships among chemical sources, transport and uptake mechanisms into mussel and crayfish tissue, and exposure pathways from mussels and crayfish to people and ecological receptors (Figure A5-1).[4]

A recent survey of dietary and food consumption habits of the CCT (Westat 2012; EI 2012) conducted in the vicinity of the Site has identified the vicinity of the Sanpoil River as a local source of mussels and crayfish; however, Rebecca Lake, Buffalo Lake and other nearby lakes in the vicinity of the Site were reported most frequently as the local source of crayfish (Westat 2012; EI 2012). The consumption of shellfish by recreational visitors to

[4] This CSM is as presented in the BERA work plan (Parametrix et al. 2011) and is subject to change. EPA has retained the lead for conducting the HHRA. This CSM represents the human health exposure pathways identified by EPA for primary and secondary media at the time the HHRA Work Plan (SRC, Inc. 2009) was prepared. Potentially exposed human populations identified by EPA in the work plan included recreational children and adults, adult workers, subsistence children and adults, and residential children and adults. Not all of the potentially complete exposure pathways are represented in this figure, and those that are represented do not necessarily apply to all potentially exposed human populations. As indicated in the HHRA work plan, EPA is planning to refine the exposure scenarios for each receptor population in the baseline HHRA using information provided by the site-specific recreational and tribal-use surveys that were completed in 2011.

the Site is expected to be rare and was therefore not evaluated in the recent recreational fish survey (IEC 2010; IEC 2013).

Benthic invertebrates, such as mussels and crayfish, may be an important food source for aquatic-dependent wildlife using the Site. In particular, ecological receptors such as river otter and mink are likely to consume both mussels and crayfish as a portion of their diet. In addition, invertebrate-eating (invertivorous) fish such as white sturgeon are known to consume crayfish and bivalves (Muir et al. 1986; Semakula 1963).

The U.S. Fish and Wildlife Service (USFWS) conducted mussel and crayfish surveys throughout the Site in 2012 and 2013 (USFSW, Unpublished). Crayfish tissue samples collected by USFWS in 2012 were analyzed for arsenic, cadmium, copper, mercury, lead, and zinc. The results of these studies have not been published, although the data have been made available to TAI. No other studies providing crayfish or mussel tissue chemistry data from Site samples have been conducted. Therefore, additional data are needed to evaluate the potential risks associated with ingestion of mussels and crayfish by people and ecological receptors.

A6 DATA NEEDS

Several published studies were used during the preparation of the screening-level ecological risk assessment (SLERA) to derive biota sediment accumulation factor (BSAF) values for estimating benthic mussel and crayfish tissue concentrations (Hamilton and Buhl 2002; Sample et al. 1998; SLERA Table E-9 [TAI 2010]), because the USFWS data for crayfish (USFWS, Unpublished) were not available at the time the SLERA was prepared. It is not known if the USFWS data would be acceptable for use in the BERA because the crayfish tissue data set from USFWS provides no information about organic chemicals and covers only a subset of target analyte list (TAL)[5] metals, and because neither the QAPP nor the data report were available for review. No tissue data are available for mussels. Although literature-derived BSAFs may provide some insights into mussel tissue concentrations, they do not provide an adequate basis for estimating unacceptable risks to ecological and human receptors consuming mussels from the UCR, because site- and temporal-specific differences in taxa and in the uptake, detoxification, and storage of metals and organic chemicals can significantly influence their concentrations and

[5] TAL metals include aluminum, antimony, arsenic, barium, beryllium, cadmium, calcium, chromium, cobalt, copper, iron, lead, magnesium, manganese, mercury, nickel, potassium, selenium, silver, sodium, thallium, vanadium, and zinc (USEPA 2015) (see http://www.epa.gov/superfund/programs/clp/ismtarget.htm).

therefore the derived risk values. Thus, mussel and crayfish tissues (described herein) will be collected to fill these data gaps.

Data generated through mussel and crayfish sampling and tissue analysis can and will be used, as appropriate and applicable, in the evaluation of unacceptable risks to people, fish, and wildlife (Figure A5-1).

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 mussel and crayfish 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 and ecological receptors from the ingestion of mussels and crayfish from the Site. Accordingly, this study will characterize chemical concentrations in mussel and crayfish tissue in target Site areas. Data collected during this study will be used to inform the BERA (e.g., aquatic-dependent wildlife exposure modeling) and 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

It is anticipated that the field-collection portion of work will begin in late April and be completed in late May to early June 2016. This time period is estimated to be optimal for collecting mussel tissue because the reservoir is typically at its lowest annual level in early May (Figure A7-1); crayfish sampling will be conducted in conjunction with mussel sampling during this time period. Preliminary analytical chemistry results will be available approximately 60 days after ALS has been informed of the compositing plan so that sample processing can begin. 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 the all field sampling activities as required in the Settlement Agreement (USEPA 2006a).

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

Consistent with EPA's level of effort technical memorandum (USEPA 2013), the primary goal of this study is to collect data to delineate and characterize the levels of chemicals in tissues of representative mussel and crayfish taxa from the Site. The data will be used in the evaluation of potential risk to humans and aquatic-dependent, invertivorous wildlife. Specific DQOs to be addressed are to:

- Determine if total concentrations of TAL metals, methylmercury, inorganic arsenic, PCBs, and dioxins/furans in mussel and crayfish tissues pose unacceptable risk to human consumers
- Determine if chemical concentrations of TAL metals in mussel and crayfish tissues pose unacceptable risk to invertivorous fish and wildlife species

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

The following subsections describe the sampling areas and numbers of samples, the chemicals that will be analyzed, and the mussel and crayfish species expected to be collected.

A7.3.1 Sampling Areas and Number of Samples

Evaluation of potential risks to ecological receptors and human consumers requires that representative data be collected within areas appropriate for assessing potential risks to each of these groups. The specific uses of these data will be determined in the baseline risk assessments. For ecological receptors, potential risk will be evaluated by CSM unit and, potentially, collectively. For human consumers, potential risk will be evaluated for areas where mussels and crayfish are known to be captured and consumed by people.[6] Based on data from the UCR tribal consumption and resource use survey, reaches of the

---

[6] In addition, during a conference call with TAI on March 23, 2016, EPA determined that the HHRA will also evaluate sampling areas where total organic carbon exceeds one percent in sediment data previously collected from locations where crayfish and mussel may be present and would be most likely to be harvested by people. The statistical treatment of the data for the different HHRA sampling areas will be determined in the baseline risk assessment.

UCR were seldom identified as the source of locally caught mussels and crayfish consumed by survey respondents (EI 2012). Four percent of respondents identified Reach 6, from the mouth of the Sanpoil River to the Grand Coulee Dam as a principal source of crayfish, while Reaches 1 through 5 accounted for less than 0.03 percent each of the sources from which local crayfish were consumed (EI 2012). None of the UCR reaches were reported as principal sources for consumption of locally harvested mussels (EI 2012). In contrast, the mouth of the Sanpoil River, upstream to Louie Creek or nearby creeks was most frequently identified in the tribal survey as the principal local source of mussels and the second most frequent source of crayfish, contributing 54 and 38 percent of the local mussel and crayfish sources, respectively. The most frequently identified local source areas for crayfish were identified as "Rebecca Lake, Buffalo Lake, or nearby" and represented 68 percent of the locally consumed crayfish sources (EI 2012). Based on anecdotal information provided via email to EPA by a representative of the Spokane Tribe (Knudson 2015), historical collection of mussels along the east side of the UCR, north of the confluence with the Spokane River and extending upriver to approximately River Mile 647 has been reported. Crayfish have been identified along the same stretch.

One area within each of the six Site river reaches will be sampled (Table A7-1). In Reach 6 (Map A7-1), the area along the mouth of Sanpoil River was selected for sampling primarily based on potential exposure of human consumers, although it will also be used to represent exposure of ecological receptors. Likewise, in Reach 5 (Map A7-1), the area north of the Spokane River mouth was selected for sampling primarily based on potential exposure of human consumers although it will also be used to represent exposure of ecological receptors. One area was selected for sampling in each of the remaining four reaches (Map A7-1) to represent exposure of ecological receptors in those areas. However, after the sampling areas were selected, and prior to finalization of this QAPP, it was determined that the area in Reach 2 will also be used to represent exposure of human consumers based on sediment TOC concentrations greater than 1 percent at some locations, as well as the potential for human use at beaches in this area (Toll 2016). It was determined that TOC concentrations were low enough in sediment in sampling areas within Reaches 1, 3, and 4 that these areas would not be targeted for evaluation in the HHRA. In addition to the six Site sampling areas, two reference areas will be sampled: the upper Sanpoil River and Rebecca Lake (Map A7-1). Section B1.1 provides more detailed rationale for the selection of each of these sampling areas.

Within each sampling area, six composite samples of crayfish and six composite samples of mussels will be created from the organisms collected, assuming that a sufficient number of organisms can be collected to provide enough material for chemical analyses of each

composite sample (Table A7-2). To provide sufficient volume for each composite sample and to capture the variability in concentrations in individual organisms, the targeted number of organisms estimated for each composite is five, based on the following rationale:

- **Mussels** – For mussels, available literature (e.g., Bura et al. 2011) suggests that the soft tissue portion of the mussels likely to be encountered in the UCR would be approximately 50 percent of the total weight of the individual mussel. This percentage will be used by the field crew to determine if additional mussels are needed from a given area in order to meet analytical mass requirements. As described in the FSP (and SOP-3 regarding mussel collection), additional mussels may be collected to meet the target analytical mass.
- **Crayfish –** For crayfish, limited information is available regarding the mass of the stomach/carapace relative to the whole body. Using best professional judgment, it is estimated that crayfish collected will be 3.5 to 4 in. in length or larger and that the stomach/carapace of the crayfish is approximately 50 percent of the whole body mass. Using this estimate, five crayfish should provide 75 to 113 g ww of sample mass (see Section 2.2.4 of Appendix A), more than enough tissue to meet sample analysis needs.

Although the targeted number of organisms is five, it will not be limited to five if more organisms can be collected at a specific sampling location within a sampling area. Where field or EPA split samples are required, additional mussels will be collected to reach the target mass (e.g., double the mass will be needed for a split sample). For crayfish, it is estimated that five individuals should provide sufficient mass for both an original and a split sample.

A7.3.2 Chemical Analyses

Analyses of mussel and crayfish[7] samples for TAL metals, methylmercury, inorganic arsenic, PCB congeners, and dioxins/furans will be conducted to support the HHRA. For samples used only in the BERA, the analyte list will be limited to TAL metals. Therefore, at locations where mussel and crayfish tissue samples will be collected to support both the BERA and the HHRA, the analyte list will also include TAL metals, methylmercury, inorganic arsenic, PCB congeners, and dioxins/furans.[8] EPA methods for chemical

[7] Whole body minus the stomach and carapace.

[8] For the risk assessments, toxic equivalents (TEQs) will be calculated using the dioxin/furan congener data, dioxin-like PCB congener data, and toxic equivalency factors (TEFs) from Van den Berg et al. (2006). Mammalian or avian TEFs will be used depending upon the receptor of concern.

analyses of tissue are listed in Table A7-3. All results will be reported on a wet weight basis (with percent moisture reported for all samples and percent lipids also reported for samples analyzed for PCBs and dioxins/furans). Performance acceptance criteria and analyses required to evaluate data quality are discussed in Section A.7.6.1.

For all mussel samples, the soft tissue of the mussel (including any liquid inside the shell) will be analyzed. For crayfish samples, the whole body minus the carapace and stomach[9] represents the tissue most likely to be consumed by people, therefore this portion of the sample will be analyzed separately from the carapace and stomach for locations relevant to both the HHRA and the BERA;[10] the whole body will be analyzed for locations relevant to the BERA only. Minimum tissue mass required for chemical analyses are 30 g ww for the full set of HHRA and BERA analytes, and 4.5 g ww for analytes only intended for the BERA; this mass does not include any needed for split samples (Table A7-3). If less than the minimum sample mass of tissue is recovered, available tissue will be allocated to chemical analyses according to the prioritization listed in Table A7-3.

A7.3.3 Species

All species of mussels and crayfish will be targeted for sampling, although it is not expected that all species will be present at all sampling stations. Neither wildlife nor human consumption are restricted by species;[11] therefore, sampling will not be restricted to specific taxonomic subgroups. The decision regarding whether composite samples should be species-specific or contain a mix of taxonomic subgroups will be determined as part of the compositing plan. Based on the results from the USFWS mussel surveys (Appendix B), the most common UCR mussel species were western/Oregon floaters (*Anodonta* clade 2). Winged/California floaters (*Anodonta* clade 1), a state-listed species of concern, were infrequently observed. Other mussels that may be encountered during sampling include western pearlshell (*Margaritifera falcata*[12]) and the western ridged mussel (*Gonidea angulata*). The presence of Yukon floaters (*Anodonta beringiana*) in the UCR has

---

[9] Whole body minus the stomach and carapace leaves the head, claws, abdomen, tail, and shell around the claws and tail.

[10] Analysis of the stomach and carapace will be limited to TAL metals; this will support estimation of the whole body concentration for use in the BERA using the concentrations and relative weights of each sample type.

[11] However, there is a daily limit of 10 lbs in shell for native crayfish species as stated in the WA sport fishing rules (effective July 1, 2015 to June 30, 2016). Based on the USFWS survey (as summarized in Appendix B), few native species are expected to be found at the Site.

[12] The western pearlshell mussel is found in the Kettle River but has not been found in recent years in the main stem of the UCR. If found, these would, in at least some cases, be preferentially collected for consumption by UCR residents.

never been confirmed. As requested by EPA in the level of effort technical memorandum (USEPA 2013), mussel identification methods will be based on *Freshwater Mussels of the Pacific Northwest (*Nedeau et al. 2009), with the recognition that shell morphology may not be reliable to determine species, but may be reliable for clades (particularly for *Anodonta* sp.). Two species of crayfish were collected during the USFWS survey: the non-native, invasive northern/virile crayfish (*Orconectes virilis*) and the native signal crayfish (*Pacifastacus leniusculus*). Only five native crayfish were collected during the USFWS survey (Appendix B). All collected species will be identified to the lowest practical identification level (LPIL). As requested, if western pearlshell mussels or signal crayfish are found in the reservoir or riverine segments of the UCR, TAI will report the location and consult with EPA.

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

Target populations of primary interest are crayfish and freshwater mussels that live in or on Site sediment. Data collected on these target populations will be used to assess potential risks to ecological receptors and humans as identified within the conceptual site model (see Figure A5-1). Potential ecological wildlife receptors for the BERA that might consume mussels or crayfish as part of their diet, as identified in the SLERA and/or BERA work plan (TAI 2010; 2011), include lesser scaup, raccoon, river otter, and mink. In addition, white sturgeon is an invertivorous ecological fish receptor that could consume mussels or crayfish. As discussed for human exposures in Section A5, it is possible that the Site is also a source of mussels and crayfish for consumption by people.

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. Mussel and crayfish tissue will be collected from representative locations throughout the Site, including some of the locations previously sampled for mussels and crayfish by the USFWS (see Section B7 for additional details on USFWS locations and rationale for selection of sampling locations).

The lower Sanpoil River reference area is located approximately 7 miles upstream from the mouth of the Sanpoil River and the Rebecca Lake reference area is located approximately 8 miles northeast of the Grand Coulee Dam (Map A7-1).

A7.4.3 Temporal Considerations

The most important temporal consideration is timing of reservoir water level drawdown, because mussels will be most accessible for sampling when the reservoir is drawn down. Therefore, sampling in the lower three reaches (4 through 6) is targeted to occur between late April and the middle of May, the window when the reservoir water level is typically at its lowest (Figure A7-1). It is possible that the predicted reservoir draw-down level of ≤ 1,255 ft for 2016 may not be low enough for the successful collection of mussels, based on information obtained from the previous USFWS surveys conducted in 2012 and 2013. In 2012, when the water level ranged from 1,237 to 1.239 ft during surveying, USFWS was able to collect mussels from Reaches 5 and 6, but mussels were found infrequently in 2013 when the water level was higher, ranging from 1,255 to 1,258 ft during surveying. Another temporal consideration is the annual increase in discharge in the upper reaches due to spring runoff, which typically occurs in early to mid-May. Sampling in Reaches 1 and 2 will be targeted to occur before the spring runoff period to decrease the chances of losing crayfish traps as a result of high river flows. If the targeted number of organisms is not collected in spring 2016, EPA may request and coordinate additional sampling using divers to collect mussels in the summer. If flows are too high to keep crayfish traps in place during the proposed sampling period, additional sampling may be considered for the summer.

Concentrations of organic chemicals in mussel tissue may be higher prior to spawning because of higher lipid content and faster uptake kinetics for highly hydrophobic compounds (Bruner et al. 1994). It is presumed that fertilization of mussels in the Pacific Northwest occurs in the summer (Nedeau et al. 2009). Females carry their eggs from late summer through the following spring, and larvae are released from late fall to spring (Nedeau et al. 2009). The timing of breeding and spawning varies by species and habitat; specific spawning times of common species in the UCR were not presented in *Freshwater Mussels of the Pacific Northwest* (Nedeau et al. 2009), and thus it may be difficult to time sampling in association with spawning. Information was not found in the literature regarding observed changes in concentrations of organic chemicals in crayfish tissue related to spawning. For crayfish, egg production and fertilization occur during autumn. In two species of crayfish found in Lake Roosevelt, the native signal crayfish *Pacifastacus leniusculus* and the non-native northern or virile crayfish *Orconectes virilis*, mating occurs

in autumn and the eggs hatch in spring. The fertilized eggs are attached under the female's tail, on the swimmerets, where they stay until ready to hatch. The young are brooded into summer, through one or more molts.

It is possible that molting could affect whole-body chemical concentrations in crayfish. However, Finerty et al. (1990) found that with the exceptions of copper and iron, concentrations of metals in both wild and pond-raised crayfish from southern Louisiana were not significantly different over three seasons. Uncertainties in tissue chemical concentrations associated with the time of sampling, which could result in higher or lower exposure estimates, will be discussed in the risk assessments.

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 mussel and crayfish tissue chemical concentrations and associated unacceptable risks to wildlife and human receptors. 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 BERA and HHRA.

As discussed in Section A.7.3.1, the chemicals to be analyzed in tissue samples collected for the purpose of conducting the HHRA are TAL metals, methylmercury, inorganic arsenic, PCB congeners, and dioxins/furans; the chemicals to be analyzed in tissue samples collected for the BERA are TAL metals only. EPA methods for chemical analyses of tissue are listed in Table A7-3, and the analytical concentration goals are presented in Table A7-4. 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.

Within each sampling area, including reference areas (Map A7-1), six composite tissue samples are targeted for collection (Table A7-2). A single composite will consist of a targeted minimum of five organisms. The ALS laboratory methods for tissue processing and compositing are discussed in ALS SOPs in Appendix C. From the six composites per area, the upper 95th percentile confidence limit on the mean UCLs (95th UCL) for each area, chemical, and tissue type will be used as the exposure point concentrations for both the HHRA and the BERA. In the event that fewer than three composite samples of a tissue

type are collected in a sampling area, the maximum tissue concentration will be used as the exposure point concentration.

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 2006c). 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

Previous mussel and crayfish surveys conducted within the UCR demonstrate that collection success or sample size of target taxa cannot be reliably determined *a priori*. Initial results from USFWS sampling of mussels and crayfish in the UCR indicate that individuals are irregularly distributed within habitats in UCR reaches. In addition to their patchy distribution, unsuccessful collections may occur in some areas due to unforeseen physical challenges, such as the presence of uninhabitable substrate (e.g., bedrock) or an inaccessible sampling station (due to insufficient reservoir drawdown or other physical impediments). To mitigate such potential challenges, relatively large (approximately 5 miles long) sampling areas have been designated, rather than specific locations. In the areas sampled for both the HHRA and the BERA, locations where people are known to collect mussels and crayfish will be given priority. Reconnaissance of all sampling areas will be conducted, as deemed necessary, immediately prior to sampling within an area to identify the most promising sampling locations based on potential habitat for mussels and crayfish.

If the field sampling team cannot collect the targeted number of organisms within the level of effort defined in the field sampling plan (see Section 2.2 of Appendix A), TAI will consult with EPA or their designee as to the benefit of continuing to attempt to collect organisms at a sampling area or a specific location within a sampling area. 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 mussels and crayfish for chemical analyses with the required detection limits (see Table A7-3 and Table A7-4). Determination of sufficiency of mass will be based on field observations at the time of collection. Because of the importance of achieving detection limits below levels of concern for the receptors, chemicals for tissue analysis will be prioritized to ensure sufficient mass

for each chemical for which the sample is analyzed. Precision will be determined by repeatability of chemical measures in 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 A7-4 and 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 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.

Field split tissue samples (i.e., process duplicates) will be prepared to assess the homogeneity of composite samples and the precision of the sample preparation procedures. Field split tissue samples will be prepared by ALS after sample compositing and homogenization for 5 percent of the samples, or for one sample per analytical batch (whichever is greater); however, the full suite of analytes will be prioritized over the preparation of field splits.

EPA split samples (i.e., inter-laboratory splits) will also be prepared from the sample homogenate generated in the lab. Sufficient samples will be collected to analyze EPA splits at a frequency of 15%, if possible. Field replicates may also be required in order to evaluate the heterogeneity of field-collected samples at specific locations within a sampling area. Sufficient samples will be collected to analyze field replicates at a targeted frequency of 20%, if possible. There is no lower limit on the acceptable replicate frequency. Whether or not to analyze field replicates along with the final number of samples to be analyzed will be determined in the compositing plan. The preparation of EPA splits and field replicates 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.

Method detection limits (MDLs) and method reporting limits (MRLs) for metals, and quantitation limits (QLs) for PCBs and dioxins/furans, are listed in Table A7-4 and Table A7-5. Estimated detection limits (EDLs) for PCBs and dioxins/furans are congener and sample specific and will be reported by the laboratory after analysis. In place of an EDL, an estimated maximum possible concentration (EMPC) may be reported if a chromatographic peak does not meet the method identification criteria detailed in Vista's SOPs (Appendix C).

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 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. A cultural resources coordination plan (Appendix D) 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/CERTIFICATES

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 manuals (Appendix C).

Sampling personnel will be familiar with the Site cultural resources coordination plan (Appendix D). 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 web tool database. 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
- Photo 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 Laboratories

Analyses for metals and conventional parameters will be conducted by ALS and analyses for PCB congeners and dioxins/furans will be conducted by Vista. 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 and Vista 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 manuals (Appendix C) 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 and percent lipids
- 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, field split samples (i.e., process duplicates), 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 chromatograms and 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 coordinators 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 each 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 laboratories and during data validation.

Data validation reports will be prepared and provided to the laboratory QA managers. 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 mussel and crayfish tissue study that will result in a data set that supports assessing potential risk to ecological and human receptors.

B1.1 Target Sample Locations and Rationale

Sampling areas within the Site were selected based on the following considerations:

1) Inclusion of one sampling area within each of the six river reaches to provide widespread spatial coverage (six composite samples from different locations within each sampling area will be collected, if sufficient numbers of organisms are available) ,
2) Inclusion of locations where people collect mussels and crayfish based on data from the CCT tribal survey (Westat 2012; EI 2012) and other locations identified by a Spokane Tribe representative,
3) Locations where mussels and crayfish may be most likely found based on the 2012 and 2013 USFWS mussel and crayfish survey results (see Appendix B for details), and
4) Fish sampling areas from the 2013 sampling event (TAI 2009).

Rather than select specific sampling locations, sampling areas approximately 5 river miles in length were identified (Table A7-1; Map A7-1). Specific locations within these areas will be determined in the field based on a reconnaissance of each area conducted during the sampling event before conducting the sampling (as described in Section 2.2 of Appendix A). The rationales for selecting each of the reaches is as follows:

- **Reach 6 (Map A9 of the FSP).** The tribal survey indicated that crayfish are frequently caught at the mouth of the Sanpoil River and upstream areas and, to a more limited extent, in the UCR between the Sanpoil River mouth and the Grand Coulee Dam (EI 2012). This would be the innermost section of Sanpoil Arm. The sampling area was based on potential access points determined from a review of the satellite photographs. No USFWS crayfish survey locations were located in this proposed sampling area but numerous crayfish were collected from Keller Ferry on the opposite side of the UCR (Map A9 in Appendix A). Mussels were reported in moderate numbers at most of the locations surveyed in this reach in

2012 (when the water level was unusually low, ranging from 1,237 to 1,239 ft during surveying) but not in 2013 (when the water level was typical, ranging from 1,255 to 1,258 ft during surveying); all reported mussels were dead (Appendix B).

- **Reach 5 (Map A8 of the FSP).** Information from the Spokane Tribe (Knudson 2015) indicates that mussel collection along the east side of the UCR north of the Spokane River mouth and extending to approximately River Mile 647 has occurred. Crayfish have also been identified in this same stretch. The upstream extent of this area was determined based on the potential human access points along the UCR. No USFWS crayfish or mussel survey locations were located in this proposed sampling area (Map A8 in Appendix A). At locations upstream and downstream of this proposed sampling area, USFWS had moderate success in collecting mussels and crayfish. All reported mussels were dead, and were found in greater numbers in 2012 when water levels were low (Appendix B).
- **Reach 4 (Map A7 of the FSP).** This area overlaps with the 2009, Reach 4, fish sampling area, and is located near the towns of Daisy and Gifford. During the USFWS survey, crayfish were successfully collected in traps at the Daisy, Cloverdale, and Gifford sampling locations. Mussels were collected at most of the survey locations in this area; all were reported to be dead (Appendix B).
- **Reach 3 (Map A6 of the FSP).** This area overlaps with the 2009 Reach 3 fish sampling area, and is located in the Kettle Falls area. During the USFWS survey, crayfish were collected at some locations, most at Kettle Falls. Mussels collected in this area (and in the entire UCR) were reported in the greatest number from the Hayes Island location, which is only exposed during more extreme draw-down years. All mussels reported in survey locations from Reach 3 were reported to be dead (Appendix B).
- **Reach 2 (Map A5 of the FSP).** This area overlaps with the 2009 Reach 2 targeted fish sampling area, and is located near the China Bend boat launch. During the USFWS survey, only two crayfish were collected from the survey locations in this area. Mussels were collected from all survey locations in this area. The location near the China Bend boat launch was the only survey location where more than a few live mussels were found; approximately half of the mussels were reported to be living (Appendix B).
- **Reach 1 (Map A4 of the FSP).** This area overlaps with the 2009 Reach 1 targeted fish sampling area, and is located near Northport. During the USFWS survey, no

crayfish were collected in this area, but mussels were. However, most were dead specimens (Appendix B).

Six composite samples each of mussels and crayfish, ideally representing unique locations within each sampling area, will be targeted for collection: a total of 36 mussel samples and 36 crayfish samples at Site locations (Table A7-2).

In addition to the Site locations, two reference locations were selected: one location upstream in the Sanpoil River and one location at Rebecca Lake. Both of these locations were identified as sources of mussels and crayfish in the tribal survey (EI Ltd 2012). Chemical concentrations in reference area samples will be used for comparison to Site concentrations in the HHRA. Six composite samples each of mussels and crayfish will be targeted for collection within each reference area, for a total of 12 mussel samples and 12 crayfish samples (Table A7-2).

Each composite sample will consist of five organisms (or potentially more for mussels); this number was determined based on an analysis of the sizes of mussels and crayfish that are anticipated to be collected and the analytical mass required for each sample (Section A.7.3.1). If sufficient organisms cannot be collected for a composite sample, and the sample mass is insufficient for all analyses, the priority for analyses specified in Table A7-3 will be followed. For native (i.e., signal) crayfish, Washington State fishing regulations restrict the collection of individuals to those greater than 3.25 in. long. Therefore, any native crayfish collected at locations used for both the HHRA and the BERA will be greater than or equal to 3.25 in. long. At the BERA-only locations, native crayfish of any size may be collected (using a Washington State Department of Fish and Wildlife research permit). Non-native crayfish (i.e., northern/virile crayfish) do not have a size restriction and therefore individual less than 3.25 in. in length may be collected. The specific compositing plan for crayfish (i.e., which specific samples will go into which composite) will be determined in consultation with EPA following the completion of sampling and will be determined based on the number of crayfish collected, their size (i.e., sample mass available for analysis), and locations where they were collected. The specific compositing plan for mussels will also be determined in consultation with EPA, and will be based on the number of mussels collected, the specific sampling locations (within the sampling areas) where they were collected, and the elevations at which they were collected. The compositing plan must be finalized and approved by EPA so that chemical analyses can be completed within the sample holding times (six months for inorganic chemicals and one year for organic compounds).

B2 SAMPLING METHODS

Field sampling methods for collection of mussels and crayfish, are described in the FSP (Appendix A). The FSP includes the following topics:

- Station positioning (Section 2.2.2)
- Field equipment and supplies (Section 2.2.3)
- Sampling methods (Section 2.2.3.1)
- Sample containers and labels (sample labels, sample identifier custody seals, sample custody/tracking procedures) (Section 2.5)
- Field documentation and procedures (field logbooks, photo documentation, COC forms) (Section 3).

SOPs for each sampling method are provided in Attachment 2 of the FSP (Appendix A).

Samples collected will be approved by a cultural resource observer and an EPA observer or its representative before they are retained. 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 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 log, 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 TAI technical team coordinator, TAI project coordinator, and EPA (and EPA's representative[s] 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 laboratories are outlined in the FSP (Appendix A).

After mussels and crayfish are collected in the field, they will be temporarily placed on wet ice in coolers. Upon return from the field, the organisms will be euthanized by transferring them to coolers with dry ice or placing them in a freezer. Frozen organisms will be packed in coolers with dry ice (as detailed in Section B4) and shipped overnight to ALS. After processing, ALS will ship aliquots of samples for PCB and dioxin/furan analyses to Vista. Upon receipt of samples, the laboratories will check the physical integrity of the containers and custody seals, measure the cooler temperatures, and inventory the samples by comparing sample labels to those on the COC forms. The laboratories 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 laboratory coordinator within 24 hours of receipt of the samples. Specific laboratory QA plans are provided in Appendix C. Laboratory project managers will ensure that a sample-tracking record is maintained that follows each sample through all stages of sample processing at the laboratories.

Storage specifications for mussel and crayfish tissue samples are in Table A7-3. The laboratories will maintain COC documentation and documentation of proper storage conditions for the entire time that the samples are in their possession. The laboratories will not dispose of the samples for this task until authorized to do so by EPA. After authorization is obtained, the laboratories 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, individual mussel and crayfish samples will be wrapped in foil (food-grade heavy duty, with dull side in contact with organism), enclosed in re-sealable plastic bags, labeled, and stored in coolers with wet ice. Samples will be frozen and shipped overnight on dry ice to ALS, where they will be stored at -20°C until processing. At ALS, tissues will be composited, freeze-dried, and homogenized according to general ALS SOPs, as well as project-specific SOPs (Appendix C). The metals analyses will be conducted by ALS. Sub-aliquots of freeze-dried tissues will be prepared by ALS and shipped to Vista for analyses of PCBs, dioxins/furans, and percent lipids using the sample handling and custody procedures described in Section B3. There are pros and cons to using freeze-dried tissue for the analyses of organic compounds. The primary concern is possible loss of some lighter PCB congeners. 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. Therefore, as approved by EPA during a conference call with TAI on March 23, 2016, freeze-dried tissue will be used for analyses of both inorganic and organic chemicals. Data validation reports for this study will discuss the potential uncertainties in the PCB congener data as a result of freeze-drying the tissue. Table A7-3 summarizes the preparation and analytical methods for each analyte.

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 laboratories, 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 or field splits (i.e., process duplicates), and method blanks will be 1 for every 20 samples or 1 per extraction or analysis batch, whichever is more frequent. Note that field splits will be prepared and analyzed only as sample mass allows. Names and control limits of applicable SRMs are included in Table B5-1. Because EPA Methods 1668A and 1613B use isotope dilution techniques, analyses of LCS and MS samples are not necessary for PCBs and dioxins/furans. 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 2008a), performance-based control limits have been established by the laboratories. These and all other control limits specified in the method descriptions will be used by the laboratories 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), and for relative percent differences (RPDs) of MS/MSDs and field splits, are provided in the analytical laboratories' QA manuals (Appendix C).

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 field splits (i.e., process duplicates). 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

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 laboratories 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
- Field splits (i.e., process duplicates)
- 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 C).

Analytical concentration goals (ACGs) provide the target concentration required for the chemical analysis. Methods selected for this study are expected to provide sufficient sensitivity to yield ACGs that are below the lowest reference value for this study.

For metals, ALS will determine a MDL for each analyte, as required by EPA (USEPA 2004). 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. For PCBs and dioxins/furans, Vista will determine an EDL for each congener. The EDL is sample specific and provides an estimate of the sample concentration needed to produce a signal that is at least 2.5 times the level of background noise (USEPA 2011). In place of an EDL, an EMPC may be reported if a chromatographic peak does not meet the method identification criteria detailed in Vista's SOPs (Appendix C). ALS will have established MRLs at levels above the MDLs, and Vista will have established QLs at levels above the EDLs. The MRLs and QLs are based on the laboratories' 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.

The laboratories will quantify analytes at concentrations above the MRL/QL. Analytes detected at concentrations between the MDL/EDL and MRL/QL 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/EDL and less than the MRL/QL). Analytes that are not detected will be reported as the MDL/EDL and will be flagged with a “U” qualifier. MDLs/EDLs and MRLs/QLs will be adjusted by the laboratories as necessary to reflect sample dilution or matrix interference. All results will be reported on a wet weight basis (with percent moisture reported for all samples and percent lipids also reported for samples analyzed for PCBs and dioxins/furans). The laboratory will use the sample-specific freeze-dried percent moisture to correct dry weight results to wet weight results according to the following calculation: wet weight result (mg/kg) = dry weight result (mg/kg) x % solids/100.

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 laboratories, 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/EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE

Analytical instrument testing, inspection, maintenance, setup, and calibration will be conducted by the laboratories 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 laboratories' maintenance logs or record books.

B7 INSTRUMENT/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 laboratories’ QA plans.

Calibration standards will be obtained from a commercial vendor, and the laboratories will maintain traceability back to the National Institute of Standards and Technology (NIST). 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.

B8 INSPECTION/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 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 laboratories are provided in the laboratory SOPs and QA plans (Appendix C). 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 laboratories. 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 managers.
- **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 mussel and crayfish tissue survey 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, including field taxonomy of collected mussels and crayfish. 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. Field taxonomy data will be developed and independently reviewed in the field by qualified members of the sampling crew.

B9.2 Analytical Laboratory Data

A variety of manually entered and electronic instrument data will be generated at the laboratories. 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 laboratories. 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 plans (Appendix C). 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 laboratories 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 appraised 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 subcontractor(s), 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 laboratories, 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 coordinator.

The laboratories 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 plans (Appendix C).

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 laboratories, as appropriate. These audits may consist of on-site 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 managers.

Any task team member who discovers or suspects a non-conformance is responsible for reporting the non-conformance to the co-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 technical team coordinator and to EPA. In addition, during corrective actions, communication among the field personnel and the laboratories relative to the accuracy and completeness of the COC documents will follow the procedures for corrective action.

C2 REPORTS TO MANAGEMENT

The laboratories will keep the appropriate technical team coordinator(s) and QA manager(s) apprised of their progress on a regular basis. The laboratories 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 coordinators and QA managers will provide this information to the task QA coordinator who, in turn, will provide this information to the TAI technical team coordinator.

The laboratories will be required to have implemented routine systems of reporting non-conformance issues and their resolutions. These procedures are described in the laboratory QA manuals (Appendix C). 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 laboratories 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 laboratories will be verified and validated according to criteria and procedures described in this section. Data quality and usability will be evaluated, and a discussion 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)
- US EPA Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review (USEPA 2014a)
- USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review (USEPA 2014b)
- USEPA Contract Laboratory Program National Functional Guidelines for Chlorinated Dibenzo-p-Dioxins (CDDs) and Chlorinated Dibenzofurans (CDFs) (USEPA 2011)

Data for analytes not covered by the National Functional Guidelines (i.e., inorganic arsenic, methyl mercury, and low-level total mercury) will be validated in accordance with the applicable analytical method and the requirements detailed in this QAPP. Data will be qualified as estimated, as necessary, if results for applicable method QC (e.g., LCSs, MS/MSDs, field split samples (i.e., process duplicates), 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 field replicates will not be used to qualify the data. Results for other QC procedures will be qualified if they do not meet control limits outlined in EPA’s functional guidelines and SOPs for data validation (USEPA 2009, 2011, 2014a, 2014b). Data will be qualified as undetected based on concentrations of target analytes detected in laboratory or field blanks, according to EPA’s

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 laboratories as required for the selected methods. Current values will be provided in the laboratory QA plans, as applicable.

Equipment rinsate blank concentrations will be expressed on a mass basis calculated from the volume of water used to rinse and the average mass of tissue processed by the laboratory 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, described in the functional guidelines for data review (USEPA 2004). Data will be rejected if control limits for acceptance of data are not met, as described in the functional guidelines for inorganic data review (USEPA 2014a).

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 database is released for use.

Approximately 10 percent of the chemistry data or all of the data from at least 12 samples, whichever is greater, 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 functional guidelines and SOPs for data validation (USEPA 2004) 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 to 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 laboratories and 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 A7-4. 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 TAI technical team coordinator 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.

SECTION E: REFERENCES

Ankley, G.T., W.J. Berry, D.M. DiToro, D.J. Hansen, R.A. Hoke, D.R. Mount, M.C. Reiley, R.C. Swartz, and C.S. Zarba. 1996. Use of equilibrium partitioning to establish sediment quality criteria for nonionic chemicals: A reply to Iannuzzi et al. *Environmental Toxicology and Chemistry*, 15(7):1019-10 14.

Aquametrix Research, Ltd. 1994. Columbia River integrated environmental monitoring program (CRIEMP). 1991-1993 Interpretive Report, CRIEMP, Castlegar, B.C. Canada: 148 pp.

Bruner, K.A., S.W. Fisher, and P.F. Landrum. 1994. The role of the zebra mussel, Dreissena polymorpha, in contaminant cycling: I. The effect of body size and lipid content on the bioconcentration of PCBs and PAHs. *J. Great Lakes. Res.* 20(4):725-734.

Ecology (Washington State Department of Ecology). 2006. Water Quality Standards for surface waters of the State of Washington, Chapter 173-201A. Amended November 20, 2006. Publication No. 06-10-091. Washington State Department of Ecology, Olympia, WA.

EI. 2012. Food questionnaire data report. Upper Columbia River resources survey. Prepared for the Confederated Tribes of the Colville Reservation. Environment International, Ltd., Seattle, WA.

Exponent. 2010. Upper Columbia River data management plan, Amendment No. 1. Prepared for Teck American Incorporated. Exponent, Bellevue, WA.

Hamilton, S.J. and K.J. Buhl. 2002. Selenium and other trace elements in water, sediment, aquatic plants, aquatic invertebrates, and fish from streams in southeaster Idaho near phosphate mining operations: June 2000. U.S. Geological Survey, Columbia Environmental Research Center.

IEC. 2010. Recreational consumption and resource use survey sampling and analysis plan for the Upper Columbia River site human health risk assessment and remedial investigation/feasibility study. Industrial Economics, Inc., Cambridge, MA.

IEC. 2013. Recreational consumption and resource use survey for the Upper Columbia River site human health risk assessment and remedial investigation/feasibility study: data summary report. Final report. Industrial Economics, Inc., Cambridge, MA. Finerty, M.W., J.D. Madden, S.E. Feagley, and R.M. Grodner. 1990. Effects of environs and seasonality on metal residues in tissues of wild and pond-raised crayfish in Southern Louisiana. *Arch. Environ. Contam. Toxicol.*, 19: 94-100.

Johnsen, S.I. and Taugbøl, T. (2010): NOBANIS – Invasive Alien Species Fact Sheet – *Pacifastacus leniusculus*. From: Online Database of the European Network on Invasive Alien Species – NOBANIS. www.nobanis.org, Date of access 1/8/2016.

Knudson, T. 2015. Personal communication (e-mail correspondence with Dustan Bott, USEPA, regarding locations on the Columbia River where people are known to gather mussels or crayfish). Spokane Tribe. December 2, 2015.

Lewis, S.C. 1997. Life history, population dynamics, and management of signal crayfish in Lake Billy Chinook, Oregon. Masters thesis, Oregon State University.

MacDonald, D.D., C.G. Ingersoll, and T.A. Berger. 2000. Development and evaluation of consensus-based sediment quality guidelines for freshwater ecosystems. Arch. *Environ. Contam. Toxicol*. 39:20–31.

MESL (MacDonald Environmental Sciences Ltd.). 1997. Lower Columbia River from Birchbank to the international border: Water quality assessment and recommended objectives. Technical report. Available at: http://www.env.gov.bc.ca/wat/wq/objectives/birchbank/Birchbanktech.pdf. Prepared for Environment Canada and British Columbia Ministry of Environment, Lands and Parks.

Muir WD, Emmett RL, McConnell RJ. 1986. Diet of juvenile and subadult white sturgeon in the Lower Columbia River and its estuary. National Oceanic and Atmospheric Administration, Seattle, WA.

Nedeau, E.J., A.K. Smith, J. Stone, and S. Jepsen. 2009. Freshwater Mussels of the Pacific Northwest. Second Edition. http://www.xerces.org/wp-content/uploads/2009/06/pnw_mussel_guide_2nd_edition.pdf.

Parametrix, Exponent, HydroQual I. 2011. Upper Columbia River Baseline Ecological Risk Assessment Work Plan. (Appendices A-H missing). Prepared for: Teck American Incorporated, Spokane, WA.

Sample, B.E., J.J. Beauchamp, R.A. Efroymson, G.W. Suter, and T.L. Ashwood. 1998. Development and Validation of Bioaccumulation Models for Earthworms, ES/ER/TM-220, Oak Ridge National Laboratory, Oak Ridge, Tennessee.

Sample B.E., Opresko D.M., Suter G.W. 1996. Toxicological benchmarks for wildlife. 1996 revision. ES/ERM-86/R3. Office of Environmental Management, US Department of Energy, Washington, DC.

Semakula, S.N. 1963. The age and growth of the white sturgeon (*Acipenser transmontanus* Richardson) of the Fraser River, British Columbia, Canada. Master of Science Thesis, University of British Columbia.

SRC. 2009. Human health risk assessment work plan for the Upper Columbia River Site remedial investigation and feasibility study. Prepared by Syracuse Research Corporation for U.S. Environmental Protection Agency, Region 10.

TAI. 2009. Upper Columbia River final quality assurance project plan for the 2009 fish tissue study. Prepared by Parametrix, Integral Consulting, Inc., and Exponent for Teck American Incorporated, Spokane, WA.TAI. 2010. Upper Columbia River screening-level ecological risk assessment (SLERA). Prepared by Parametrix, Inc., Exponent, and Integral Consulting, Inc. for Teck American Incorporated, Spokane, WA.

TAI. 2011. Upper Columbia River baseline ecological risk assessment work plan (BERA work plan). Prepared by Parametrix, Inc., Exponent, and Integral Consulting, Inc. for Teck American Incorporated, Spokane, WA.

TAI. 2013a. Upper Columbia River final quality assurance project plan for the Phase 2 sediment study. Prepared by Exponent and HDR HydroQual for Teck American Incorporated, Spokane, WA.

TAI. 2013b. Upper Columbia River final fish tissue data summary and data gap report. Prepared by Exponent, Parametrix, Inc., and Integral Consulting, Inc. for Teck American Incorporated, Spokane, WA.

TCAI. 2007. Upper Columbia River draft general site health and safety plan for the remedial investigation and feasibility study. Prepared for Teck Cominco American Incorporated. December 27, 2007. Integral Consulting Inc., Mercer Island, WA, and Parametrix, Bellevue, WA.

Tielens, J.T. 1994. Bioavailability of trace metals in Franklin D. Roosevelt Lake, Washington, sediments. Master’s thesis submitted to Program in Environmental Science and Regional Planning, Washington State University.

Toll, J. 2016. Personal communication (e-mail correspondence with Marc Stifelman, USEPA, and Cameron Irvine, USEPA, regarding the resolution of general comment #3 on the draft final macroinvertebrate tissue QAPP). Windward Environmental. March 23, 2016.

USEPA. 1988. Guidance for conducting remedial investigations and feasibility studies under CERCLA. Interim final. EPA/540/G-89/004, OSWER Directive 9355.3. Office of Emergency and Remedial Response, US Environmental Protection Agency, Washington, DC.

USEPA (United States Environmental Protection Agency). 1995. SOP for the validation of Method 1668 toxic, dioxin-like PCB data. U.S. Environmental Protection Agency Region 10, Environmental Services Division, Seattle, WA.

USEPA. 2000. Bioaccumulation testing and interpretation for the purpose of sediment quality assessment. EPA-823-R-00-001.

USEPA. 2002a. Guidance for quality assurance project plans. EPA QA/G-5. EPA/240/R-02/009. U.S. Environmental Protection Agency, Office of Environmental Information, Washington, D.C.

USEPA. 2002b. Guidance on environmental data verification and validation. EPA QA/G-8. U.S. Environmental Protection Agency, Office of Environmental Information, Washington, D.C.

USEPA. 2010. USEPA Contract Laboratory Program national functional guidelines for inorganic Superfund data review. U.S. Environmental Protection Agency, Office of Emergency and Remedial Response, Washington, DC. EPA 540-R-10-011. January 2010.

USEPA. 2005. Procedures for the derivation of equilibrium partitioning sediment benchmarks (ESBs) for the protection of benthic organisms: metal mixtures (cadmium, copper, lead, nickel, silver, and zinc). EPA-600-R-02-011. U.S. Environmental Protection Agency, Office of Research and Development. Washington, D.C.

USEPA. 2006a. Settlement agreement for implementation of remedial investigation and feasibility study at the Upper Columbia River Site. June 2, 2006. U.S. Environmental Protection Agency, Region 10, Seattle, WA.

USEPA. 2006b. Guidance for the data quality objectives process. EPA QA/G-4. EPA/600/R-96/055. U.S. Environmental Protection Agency, Office of Environmental Information, Washington, D.C.

USEPA. 2006c. National recommended water quality criteria. Available at http://www.epa.gov/waterscience/criteria/wqcriteria.html. U.S. Environmental Protection Agency, Office of Water and Office of Science and Technology, Washington, D.C.

USEPA. 2007. Framework for metals risk assessment. EPA 120/R-071/001. U.S. Environmental Protection Agency, Office of the Science Advisor Risk Assessment Forum, Washington, D.C.

USEPA. 2008a. SW-846 on-line, test methods for evaluating solid waste physical/ chemical methods. Available at: http://www.epa.gov/epaoswer/hazwaste/test/main.htm. Accessed June 25, 2008. U.S. Environmental Protection Agency, Seattle, WA.

USEPA. 2009. Guidance for labeling externally validated laboratory analytical data for Superfund use. EPA 540-R-08-005. U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response, Washington, D.C. January 2009.

USEPA. 2010. EPA technical team level of effort (LOE) for investigations designed to evaluate risks of contaminants to benthic invertebrate communities in the upper Columbia river (sediment toxicity LOE). Developed by the USEPA Technical Team, with consideration of comments provided by the Participating Parties. February 10, 2010.

USEPA. 2011. USEPA contract laboratory program national functional guidelines for chlorinated dibenzo-p-dioxins (CDDs) and chlorinated dibenzofurans (CDFs) data review. OSWER 9240.1-53. EPA 540-R-11-016. Office of Superfund Remediation and Technology, US Environmental Protection Agency, Washington, DC.

USEPA. 2013. Expected level of effort for sampling and analysis of benthic macroinvertebrate tissue—Upper Columbia River. Developed by the USEPA Technical Team, with consideration of comments provided by the Participating Parties. July 16, 2013.

USEPA. 2014a. National functional guidelines for inorganic Superfund data review. OSWER 9355.0-131. U.S. Environmental Protection Agency, Office of Superfund Remediation and Technology Innovation (OSRTI), Washington, D.C. August 2014.

USEPA, 2014b. National functional guidelines for Superfund organic methods data review. OSWER 9355.0-132. U.S. Environmental Protection Agency, Office of Superfund Remediation and Technology Innovation (OSRTI), Washington, D.C. August 2014.

USFWS. Unpublished. Data files containing results from 2012 and 2013 surveys of mussels and crayfish in the Upper Columbia River Site. Sent to John Toll, Windward Environmental, by Dustan Bott, EPA Region 10, on Dec 15, 2015.

Van den Berg M., Birnbaum L.S., Denison M., De Vito M., Farland W., Feeley M., Fiedler H., Hakansson H., Hanberg A., Haws L., Rose M., Safe S., Schrenk D., Tohyama C., Tritscher A., Tuomisto J., Tysklind M., Walker N., Peterson R.E. 2006. The 2005 World Health Organization reevaluation of human and mammalian toxic equivalency factors for dioxins and dioxin-like compounds. Toxicol Sci 93(2):223-241.

Westat. 2012. Upper Columbia River Site Remedial Investigation and Feasibility Study Tribal Consumption and Resource Use Survey. Final Report. Prepared for the USEPA, Region 10. June 22, 2012.

FIGURES

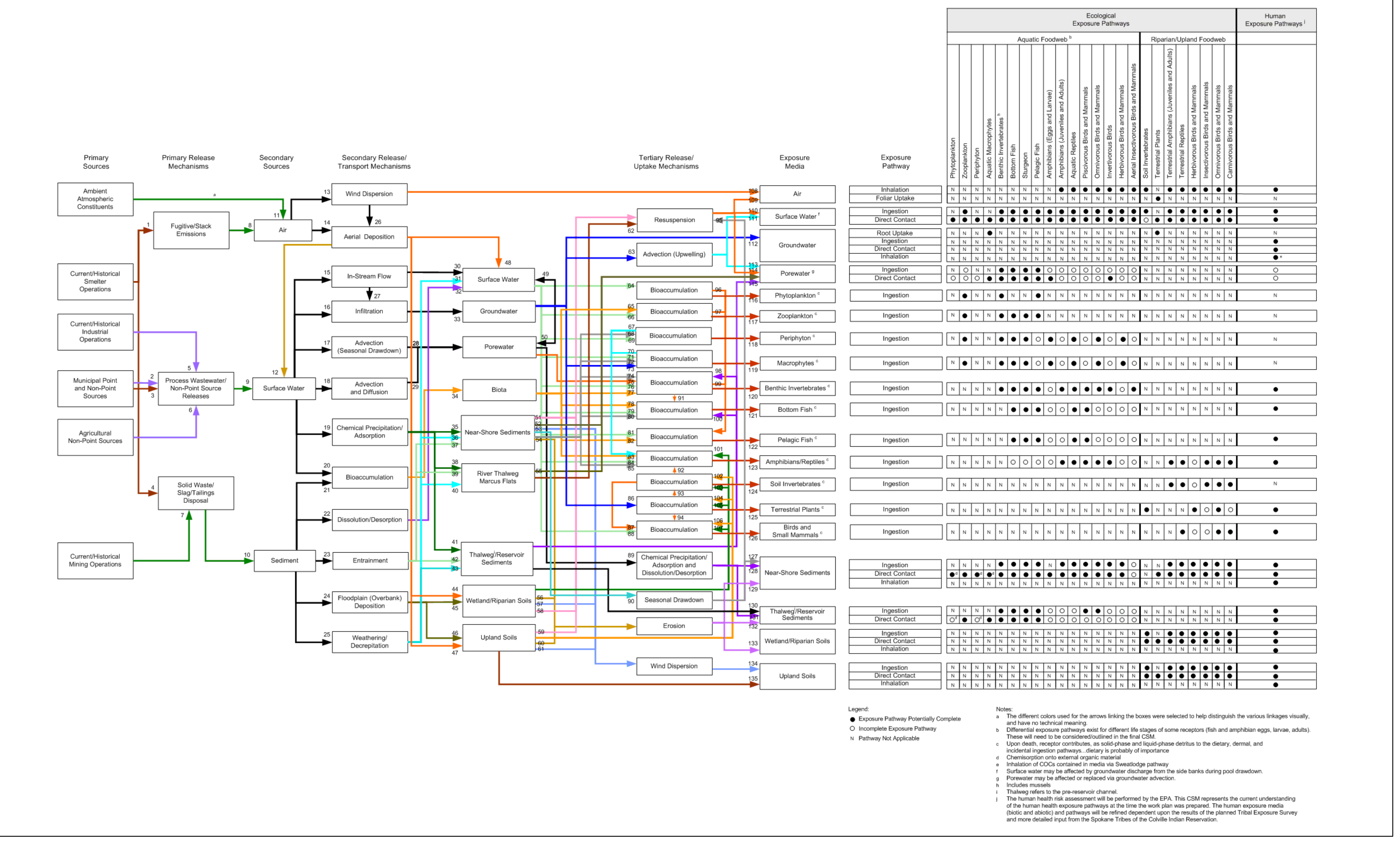


Figure A5-1. Site-wide Conceptual Site Model

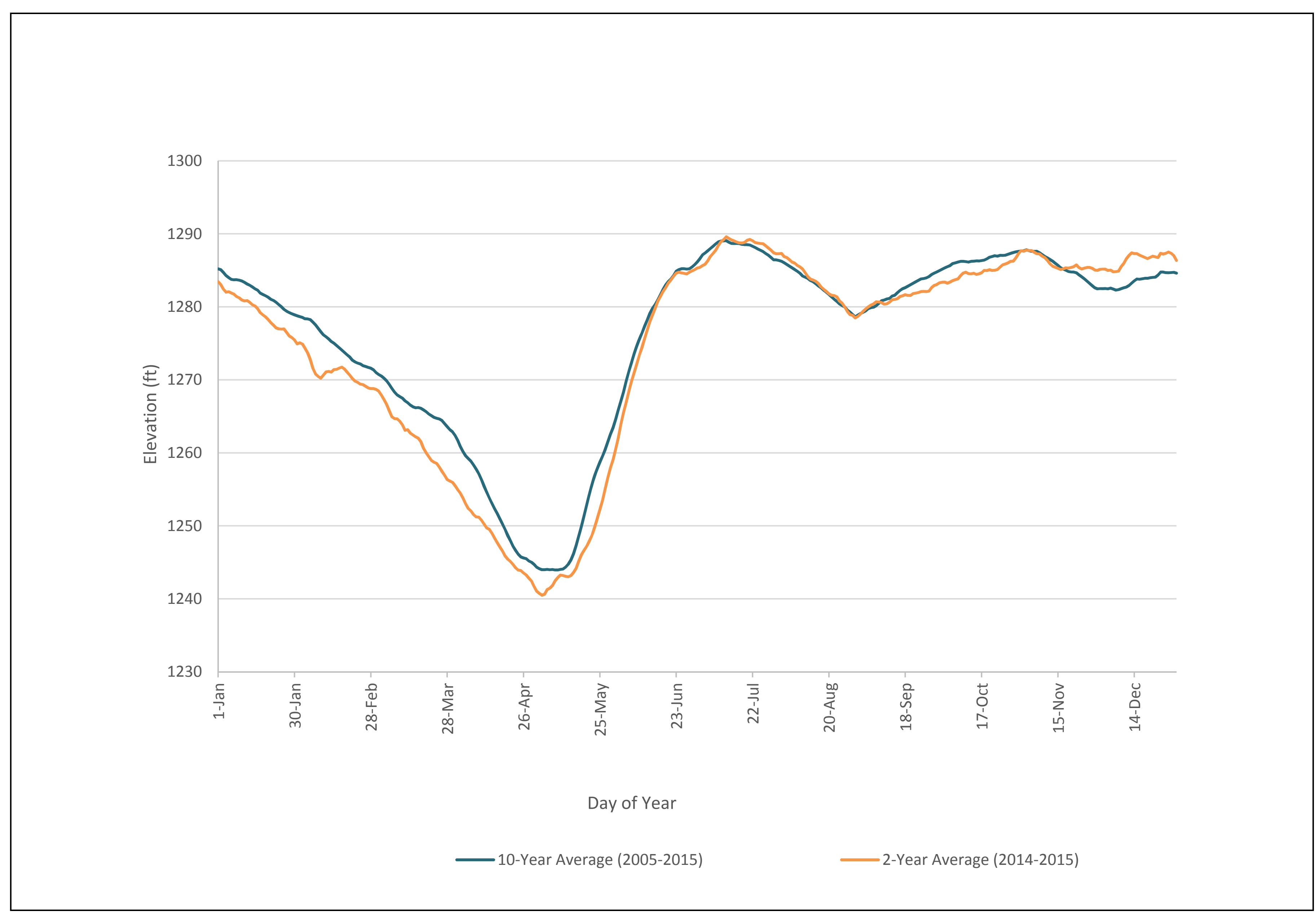
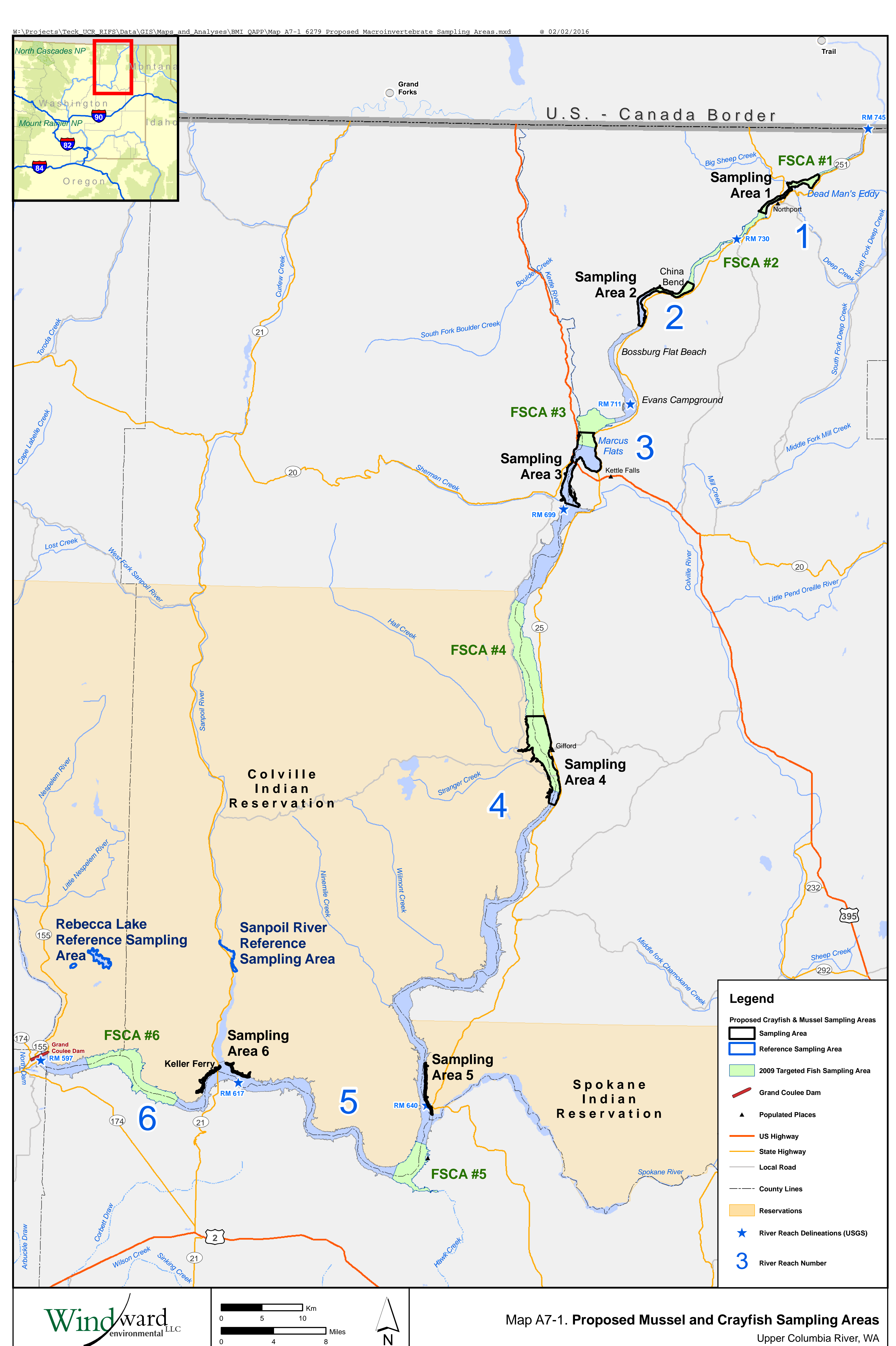


Figure A7-1. Lake Roosevelt Water Elevation at Grand Coulee Dam

MAP

W:\Projects\Teck_UCR_RIFS\Data\GIS\Maps_and_Analyses\BMI_QAPP\Map A7-1 6279 Proposed Macroinvertebrate Sampling Areas.mxd © 02/02/2016



Map A7-1. **Proposed Mussel and Crayfish Sampling Areas**

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
U.S. Environmental Protection Agency			
Laura Buelow	EPA Project Manager	(509) 376-5466	buelow.laura@epa.gov
Dustan Bott	EPA Project Manager	(206)-553-5502	bott.dustan@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			
John Toll	TAI Technical Team Coordinator	(206) 812-5433	johnt@windwardenv.com
Berit Bergquist	Prinicipal Investigator	(206) 812-5403	beritb@windwardenv.com
Rick Cardwell	Senior Technical Advisor	(541) 207-2002	cardwellconsultingllc@comcast.net
Nicholas Gard	Senior Technical Advisor	(425) 519-8728	gardn@exponent.com
Dina Johnson	Senior Technical Advisor	(206) 336-1662	DLJohnson@ramboll.com
Dave Enos	Analytical Chemistry Laboratory Coordinator	(509) 944-3807	dave.enos@teck.com
Rock Vitale	Task QA Manager	(610) 935-5577	rvitale@envstd.com
Randy O'Boyle	Database Administrator	(425) 519-8727	roboyle@exponent.com
Laboratories			
Jeff Christian	Analytical Chemistry Laboratory Project Manager - ALS	(360) 501-3316	Jeff.Christian@alsglobal.com
Lee Wolf	Analytical Chemistry Laboratory QA Manager - ALS	(360) 501-3371	Lee.Wolf@alsglobal.com
Martha Maier	Analytical Chemistry Laboratory Project Manager - Vista	(916) 673-1520 x101	mmaier@vista-analytical.com
Bahar Amiri	Analytical Chemistry Laboratory QA Manager - Vista	(916) 673-1520 x103	bamiri@vista-analytical.com

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

Table A7-1. Sampling Areas

Sampling Area	Abbreviation	Location Type	HHRA	BERA	Approximate River Miles	Description of Location	Rationale for Sampling Area
Area 1	A1	Site		X	733 to 739	Near Northport	Location provides spatial coverage of the UCR for the BERA; overlaps with targeted fish sampling area for Reach 1 in 2009. In the USFWS survey, no crayfish were collected at survey locations in this area. Mussels were reported at some of the USFWS survey locations; with the exception of four mussels at one location, all were reported to be dead.
Area 2	A2	Site	X	X	718 to 725	Stretch of UCR from China Bend boat launch to North Gorge	Location provides spatial coverage of the UCR for the BERA; overlaps with targeted fish sampling area for Reach 2 in 2009. During the USFWS survey, only two crayfish were collected from the survey locations in this area. Mussels were collected from all survey locations in this area; dead mussels were reported at all locations other than the China Bend boat launch, where approximately half of the mussels were reported to be living.
Area 3	A3	Site		X	700 to 706	Near Kettle Falls	Location provides spatial coverage of the UCR for the BERA; overlaps with targeted fish sampling area for Reach 3 in 2009. During the USFWS survey, Crayfish were successfully collected at some locations; the most successful of which was the Kettle Falls location. The highest number of mussels in the entire UCR was reported at the Hayes Island location, which is only exposed during more extreme draw-down years. All mussels reported in survey locations from Reach 3 were reported to be dead.
Area 4	A4	Site		X	671 to 678	Stretch of UCR from Daisy to Gifford	Location provides spatial coverage of the UCR for the BERA; overlaps with targeted fish sampling area for Reach 4 in 2009. During the USFWS survey, crayfish were successfully collected in traps at the Daisy, Cloverdale, and Gifford sampling locations. Mussels were collected at most of the survey locations in this area; all were reported to be dead.
Area 5	A5	Site	X	X	639 to 643	Upstream of the mouth of the Spokane River (east side of river only)	Location reported as a possible source of mussels and crayfish by Spokane Tribe. Provides spatial coverage of the UCR for the BERA. Located just upstream of targeted fish sampling area for Reach 5 in 2009. Extent of the area is based on potential human beach access locations north of the mouth of the Spokane River.
Area 6	A6	Site	X	X	613 to 618	Near mouth of the Sanpoil River (north side of river only)	Location reported as a source of crayfish in the tribal survey (Westat 2012). Provides spatial coverage of UCR for the BERA. Located just upstream of Reach 6 fish sampling area. Extent of the area is based on potential human beach access locations near the mouth of the Sanpoil River.
Rebecca Lake	RL	Reference	X	X	NA	Lake located north of the Grand Coulee Dam	Location noted to be a popular crayfish collection location in the tribal survey (Westat 2012); some individuals also reported collecting mussels from this location.
Sanpoil River	SR	Reference	X	X	NA	Near the Sanpoil Campground and mouth of Silver Creek.	Location noted to be a popular mussel collection location in the tribal survey (Westat 2012); the Sanpoil River was also noted as a popular crayfish collection area.

Notes:

If sample collection at Rebecca Lake is not successful, nearby Buffalo Lake may also be used as a reference location.

BERA - baseline ecological risk assessment

HHRA - human health risk assessment

USFWS - U.S. Fish and Wildlife Service

NA - not applicable

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

Sampling Area	Location Type	HHRA[a]	BERA[b]	Target Number of Composite Samples: Mussels: Soft Tissue	Crayfish: Whole Body Minus Stomach and Carapace	Crayfish: Stomach and Carapace Only	Crayfish: Whole Body (All Parts)	Total
Area 1	Site		X	6[c]			6[c]	12
Area 2	Site	X	X	6	6	6	calculated[d]	18
Area 3	Site		X	6[c]			6[c]	12
Area 4	Site		X	6[c]			6[c]	12
Area 5	Site	X	X	6	6	6	calculated[d]	18
Area 6	Site	X	X	6	6	6	calculated[d]	18
Rebecca Lake	Reference	X		6	6			12
Sanpoil River	Reference	X		6	6			12
Total				**48**	**30**	**18**	**18**	**114**
			EPA Splits [e]	***7***	***5***	***3***	***3***	***18***
			Field Splits [e]	***2***	***2***	***1***	***1***	***6***
			Field Replicates [e]	***10***	***6***	***4***	***4***	***24***

Notes:

For each composite, a target of five mussels and five crayfish was developed. Thus a target of 30 individuals each of mussels and crayfish will be collected from each sampling area.

[a] For crayfish, the HHRA will only inlcude samples for whole body minus stomach and carapace.

[b] For crayfish, the BERA will include data representing whole body samples, which, if not analyzed as whole body samples, will be calculated as described in footnote d of this table.

[c] Sample types that will only be used in the BERA (i.e., all mussel and crayfish samples from Areas 1, 3, and 4) will be analyzed for TAL metals, total mercury, and percent moisture. Analysis of methylmercury, inorganic arsenic, PCB congeners, dioxins/furans, and percent lipids will not be conducted for these samples because these analytes are only of concern for the HHRA.

[d] Whole body tissue concentrations will be calculated based on a weighted sum of whole body minus stomach and carapace, and stomach and carapace only samples. Crayfish samples for whole body minus stomach/carapace and stomach/carapace only samples will be comprised of the same individuals.

[e] EPA splits, field splits, and field replicates are targeted for analysis on a subset of 15%, 5%, and 20% of the total number of samples, respectively. Each sample analyzed as a split or replicate will need twice the targeted sample mass indicated in Table A7-3.

BERA - baseline ecological risk assessment

HHRA - human health risk assessment

Table A7-3. Methods and Sample Mass Requirements and Prioritization

	Sample Preparation		Quantitative Analysis			Target Sample Mass[b] (g)		Analysis Priority	
Analyte	Protocol	Procedure	Protocol	Procedure	Holding Time[a]	BERA[c]	BERA and HHRA[d]	BERA[c]	BERA and HHRA[d]
TAL metals[e]	ALS SOP MET-TDIG (PSEP)	Acid digestion	EPA 6010C, EPA 6020A[f]	ICP-MS, ICP-AES	180 days at -20°C	2	2	1	1
Total mercury	ALS SOP MET 1631	Method (acid) digestion	EPA 1631E	CVAFS	1 year at -20°C	1.5	1.5	2	2
Methyl mercury	ALS SOP MET 1630T	Method (alcohol) digestion	EPA 1630M			NA	1.5	NA	3
Arsenic - inorganic	ALS SOP MET 1632	Method (acid) digestion	EPA 1632A	HG-QFAAS		NA	3	NA	4
PCB congeners	EPA 1668A	Method extraction	EPA 1668A	HRGC/HRMS		NA	10	NA	5
Dioxins/furans	EPA 1613B	Method extraction	EPA 1613B			NA	10	NA	6
Percent moisture	ALS SOP MET-TISP	Freeze-dry/ gravimetric	ALS SOP MET-TISP	Freeze-dry/ gravimetric		NA[g]	NA[g]	1	1
Percent lipids	EPA 1668A		EPA 1668A			NA	NA[h]	NA	5
Total Target Sample Mass[i] (g)						4.5[j]	30[k]		

Notes:

Crayfish and mussel samples will be wrapped in aluminum foil and double bagged in resealable plastic bags. All samples will be frozen and shipped overnight to the analytical laboratory. Upon receipt, the analytical laboratory will store the samples at -20°C until processing. Samples will be processed and extracted for analysis within the holding times listed.

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 typically uses a holding time of two years from the time of freeze-drying for total metals (per EPA comments dated March 11, 2016 on the February 2016 draft final version of this QAPP).

[a] Holding time based on applicable standard operating procedure (see Appendix C).

[b] Based on wet weight. The expected mass of a crayfish composite sample containing 5 organisms (each 3.5 to 4 in long) is 75 to 113 g ww (see Section 2.2.4 of Appendix A).

[c] Crayfish and mussels collected at locations for baseline ecological risk assessment (BERA).

[d] Crayfish and mussels collected at locations for BERA and human health risk assessment (HHRA).

[e] Except mercury.

[f] Recommended method for analysis of calcium, iron, magnesium, potassium, and sodium is EPA 6010C. Recommended method for analysis of aluminum, antimony, arsenic, barium, beryllium, cadmium, chromium, cobalt, copper, lead, manganese, nickel, selenium, silver, thallium, vanadium, and zinc is EPA 6020A. Either EPA 6010C or 6020A may be used for TAL metals (except mercury) as long as the method reporting limits listed in Table A7-4 are achieved for non-detects.

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

[h] Percent lipids will be analyzed with PCB congeners; no additional sample mass required.

[i] The target sample size listed achieves the reporting limits in Table A7-4 and the lowest quantitation limits in Table A7-5.

[j] Includes an additional 1 g to account for possible sample loss during sample processing.

[k] Includes an additional 2 g to account for possible sample loss during sample processing

ALS - ALS Environmental
CVAFS - cold vapor atomic fluorescence spectrometry
HG-QFAAS - hydride generation - quartz furnace atomic adsorption spectrometry
HRGC/HRMS - high resolution gas chromatography/high resolution mass spectrometry
ICP-AES - inductively-coupled plasma - atomic emission spectrometry
ICP-MS - inductively-coupled plasma - mass spectrometry
NA - not applicable
PCB - polychlorinated biphenyl
PSEP - Puget Sound Estuary Program
SOP - standard operating procedure
TAL - target analyte list

Table A7-4. Macroinvertebrate Tissue Target Analyte List and Analytical Concentration Goals

	Risk Based Concentrations (RBCs)			Laboratory		
Analyte	Human Health[a,d]	Wildlife[b]	Wildlife RBC/5[d]	MRL[c]	MDL[c]	ACG[d]
Conventional Parameters						
Percent moisture	na	na	na	na	na	na
Percent lipids	na	na	na	na	na	na
Metals/Metalloids (mg/kg-wet weight)						
Aluminum	17	11.5	2.3	0.4	0.04	2.3
Antimony	0.0069	0.35	0.070	0.01	0.0004	0.01
Arsenic - total	0.0024	2.8	0.56	0.02	0.004	0.02
Arsenic - inorganic	0.0024	na	na	0.004	0.0014	0.004
Barium	3.4	259	52	0.01	0.001	3.4
Beryllium	0.034	3.2	0.63	0.004	0.0006	0.034
Cadmium	0.017	1.8	0.37	0.004	0.0004	0.017
Calcium	NA	na	na	0.8	0.4	0.8
Chromium	26	3.3	0.66	0.04	0.004	0.66
Cobalt	0.0051	9.5	1.9	0.004	0.0006	0.0051
Copper	0.69	5.0	1.0	0.02	0.004	0.69
Iron	12	na	na	0.4	0.2	12
Lead	NA	2.0	0.41	0.004	0.0001	0.41
Magnesium	NA	na	na	0.4	0.12	0.4
Manganese	2.4	222	44	0.01	0.0016	2.4
Mercury	NA	na	na	0.0002	0.000016	0.0002
Methyl mercury	0.0017	0.010	0.0020	0.002	0.0006	0.002
Nickel	0.34	8.3	1.7	0.04	0.004	0.34
Potassium	NA	na	na	4	1.8	4
Selenium	0.086	0.36	0.072	0.2	0.04	0.2
Silver	0.086	2.5	0.50	0.004	0.0012	0.086
Sodium	NA	na	na	4	0.4	4
Thallium	0.00017	0.044	0.009	0.004	0.00018	0.004
Vanadium	0.086	0.43	0.085	0.04	0.002	0.085
Zinc	5.1	82	16	0.1	0.02	5.1
PCBs (µg/kg-wet weight)[e]						
PCB TEQ	2.4E-06[f]	NA	NA	1.5E-08 - 5.0E-05[g]	TBD	MRL
Total PCBs[h]	NA	NA	NA	0.0005-0.002	TBD	MRL
Dioxins/Furans (ng/kg-wet weight)[e]						
Dioxin/furan TEQ	0.0024[i]	NA	NA	0.0015 - 2.5[j]	TBD	MRL

Notes:

The lowest RBC for each chemical is shaded.

[a] Lowest fish risk-based concentrations (RBCs) for human health are based on exposure assumptions provided by EPA in the HHRA work plan (SRC 2009, Table 5-11). RBCs assume an adult shellfish ingestion rate of 175 g/day and the child shellfish ingestion rate is assumed to be half adult rate (i.e. 87.5 g/day) per EPA (SRC 2009, Table 5-11, note 7). Toxicity values reflect 2016 EPA Regional Screening Level values.

[b] Wildlife RBCs were derived from the exposure factors and toxicity reference values (TRVs) provided in the draft SLERA (TAI 2010). TRVs for aluminum and thallium were obtained from Sample et al. (1996). The no observed adverse effect level (NOAEL) was used as the TRV and the body weight (BW) and food ingestion rate (FIR) for the most sensitive mammalian (i.e., mink) or avian (i.e., spotted sandpiper) receptor were used to calculate the RBC, according to the following equation:

Wildlife RBC (mg/kg-wet weight) = (TRV x BW) / (FIR)

Where: TRV - Toxicity reference value (mg/kg-day)
BW - Body weight (kg)
FIR - Food ingestion rate (kg/d-wet)

[c] Method reporting limits (MRLs) and method detection limits (MDLs) for metals were obtained from ALS. MRLs for PCBs and dioxins/furans were obtained from Vista Analytical. All MRLs and MDLs are based on wet weight.

[d] Analytical concentration goals (ACGs) represent the lowest RBC value for human health or 1/5th of the wildlife RBC. If the lowest RBC is lower than the MRL, the MRL will be used as the ACG. The lowest RBC is shaded.

[e] Values in the MRL column represent the range for quantitation limits (QL) based on analysis of 10 grams of sample. QLs by congener are listed in Table A7-5. Data will be reported to the sample-specific estimated detection limit, which is typically 2 to 5 times lower than the QL.

[f] Polychlorinated biphenyl (PCB) toxic equivalent (TEQ) calculated as the sum of dioxin-like PCB congeners adjusted using mammalian toxic equivalency factors (TEFs) (Van den Berg et al. 2006).

[g] PCB TEQ MRLs represent the range of QLs for dioxin-like PCB congeners adjusted using mammalian TEFs (Van den Berg et al. 2006)

[h] Total PCBs will be calculated as the sum of 209 congeners, including non-detects at one-half the detection limit (DL). If no constituents are detected, the total concentration will be flagged as non-detected and represented by the highest DL.

[i] Dioxin/furan TEQ is calculated as the sum of congeners adjusted using mammalian TEFs (Van den Berg et al. 2006). TEQs will be calculated using the sum of the weighted detects and the weighted non-detects at one-half the DL.

[j] Dioxin/furan TEQ MRLs represent the range of QLs for congeners adjusted using mammalian TEFs (Van den Berg et al. 2006).

na - not available
NA - not applicable
TBD - to be determined (upon analysis)

Table A7-5. Quantitation Limits for PCB Congeners and Dioxins/Furans

Analyte	Congener Number	QL by Mass of Sample		
		1 g	5 g	10 g
PCBs (µg/kg-wet weight)				
2-Mono-CB	1	0.005	0.001	0.0005
3-Mono-CB	2	0.005	0.001	0.0005
4-Mono-CB	3	0.005	0.001	0.0005
2,2'-Di-CB 2,6-Di-CB	4/10	0.02	0.004	0.002
2,3’-Di-CB	6	0.01	0.002	0.001
2,3-Di-CB 2,4'-Di-CB	5/8	0.02	0.004	0.002
2,4-Di-CB 2,5-Di-CB	7/9	0.02	0.004	0.002
3,3’-Di-CB	11	0.01	0.002	0.001
3,4-Di-CB 3,4'-Di-CB	12/13	0.02	0.004	0.002
3,5-Di-CB	14	0.01	0.002	0.001
4,4’-Di-CB	15	0.01	0.002	0.001
2,2',3-Tri-CB 2,2',3-Tri-CB	16/32	0.01	0.002	0.001
2,2’,4-Tri-CB	17	0.005	0.001	0.0005
2,2’,5-Tri-CB	18	0.005	0.001	0.0005
2,2’,6-Tri-CB	19	0.005	0.001	0.0005
2,3,3'-Tri-CB 2,3,4-Tri-CB 2',3,4-Tri-CB	20/21/33	0.015	0.003	0.0015
2,3,4’-Tri-CB	22	0.005	0.001	0.0005
2,3,5-Tri-CB	23	0.005	0.001	0.0005
2,3,6-Tri-CB 2,3',6-Tri-CB	24/27	0.01	0.002	0.001
2,3’,4-Tri-CB	25	0.005	0.001	0.0005
2,3’,5-Tri-CB	26	0.005	0.001	0.0005
2,4,4’-Tri-CB	28	0.005	0.001	0.0005
2,4,5-Tri-CB	29	0.005	0.001	0.0005
2,4,6-Tri-CB	30	0.005	0.001	0.0005
2,4’,5-Tri-CB	31	0.005	0.001	0.0005
2,3’,5’-Tri-CB	34	0.005	0.001	0.0005
3,3’,4-Tri-CB	35	0.005	0.001	0.0005
3,3’,5-Tri-CB	36	0.005	0.001	0.0005
3,4,4’-Tri-CB	37	0.005	0.001	0.0005
3,4,5-Tri-CB	38	0.005	0.001	0.0005
3,4’,5-Tri-CB	39	0.005	0.001	0.0005
2,2’,3,3’-Tetra-CB	40	0.005	0.001	0.0005
2,2',3,4-Tetra-CB 2,3,4',6-Tetra-CB 2,3',4',6-Tetra-CB 2,3',5,5'-Tetra-CB	41/64/71/72	0.02	0.004	0.002

Table A7-5. Quantitation Limits for PCB Congeners and Dioxins/Furans

Analyte	Congener Number	QL by Mass of Sample		
		1 g	5 g	10 g
PCBs (µg/kg-wet weight) (continued)				
2,2',3,4'-Tetra-CB 2,3,3',6-Tetra-CB	42/59	0.01	0.002	0.001
2,2',3,5-Tetra-CB 2,2',4,5'-Tetra-CB	43/49	0.01	0.002	0.001
2,2',3,5'-Tetra-CB	44	0.005	0.001	0.0005
2,2',3,6-Tetra-CB	45	0.005	0.001	0.0005
2,2',3,6'-Tetra-CB	46	0.005	0.001	0.0005
2,2',4,4'-Tetra-CB	47	0.005	0.001	0.0005
2,2',4,5-Tetra-CB 2,4,4',6-Tetra-CB	48/75	0.01	0.002	0.001
2,2',4,6-Tetra-CB	50	0.005	0.001	0.0005
2,2',4,6'-Tetra-CB	51	0.005	0.001	0.0005
2,2',5,5'-Tetra-CB 2,3',4,6-Tetra-CB	52/69	0.01	0.002	0.001
2,2',5,6'-Tetra-CB	53	0.005	0.001	0.0005
2,2',6,6'-Tetra-CB	54	0.005	0.001	0.0005
2,3,3',4-Tetra-CB	55	0.005	0.001	0.0005
2,3,3',4'-Tetra-CB 2,3,4,4'-Tetra-CB	56/60	0.01	0.002	0.001
2,3,3',5-Tetra-CB	57	0.005	0.001	0.0005
2,3,3',5'-Tetra-CB	58	0.005	0.001	0.0005
2,3,4,5-Tetra-CB 2,3',4',5-Tetra-CB	61/70	0.01	0.002	0.001
2,3,4,6-Tetra-CB	62	0.005	0.001	0.0005
2,3,4',5-Tetra-CB	63	0.005	0.001	0.0005
2,3,5,6-Tetra-CB	65	0.005	0.001	0.0005
2,3',4,5-Tetra-CB	67	0.005	0.001	0.0005
2,3',4,5'-Tetra-CB	68	0.005	0.001	0.0005
2,3',4',5-Tetra-CB	70	0.005	0.001	0.0005
2,3',5',6-Tetra-CB	73	0.005	0.001	0.0005
2,4,4',5-Tetra-CB	74	0.005	0.001	0.0005
2',3,4,5-Tetra-CB 2,3',4,4'-Tetra-CB	76/66	0.01	0.002	0.001
3,3',4,4'-Tetra-CB	77	0.005	0.001	0.0005
3,3',4,5-Tetra-CB	78	0.005	0.001	0.0005
3,3',4,5'-Tetra-CB	79	0.005	0.001	0.0005
3,3',5,5'-Tetra-CB	80	0.005	0.001	0.0005
3,4,4',5-Tetra-CB	81	0.005	0.001	0.0005
2,2',3,3',4-Penta-CB	82	0.005	0.001	0.0005
2,2',3,3',5-Penta-CB	83	0.005	0.001	0.0005
2,2',3,3',6-Penta-CB 2,2',3,5,5'-Penta-CB	84/95	0.01	0.002	0.001
2,2',3,4,4'-Penta-CB 2,3,4,5,6-Penta-CB	85/116	0.01	0.002	0.001
2,2',3,4,5-Penta-CB	86	0.005	0.001	0.0005

Table A7-5. Quantitation Limits for PCB Congeners and Dioxins/Furans

Analyte	Congener Number	QL by Mass of Sample		
		1 g	5 g	10 g
PCBs (µg/kg-wet weight) (continued)				
2,2',3,4,5'-Penta-CB 2,3,4',5,6-Penta-CB 2',3,4,5,6'-Penta-CB	87/117/125	0.015	0.003	0.0015
2,2',3,4,6-Penta-CB 2,2',3,4',6-Penta-CB	88/91	0.01	0.002	0.001
2,2',3,4,6'-Penta-CB	89	0.005	0.001	0.0005
2,2',3,4',5-Penta-CB 2,2',4,5,5'-Penta-CB	90/101	0.01	0.002	0.001
2,2',3,5,6-Penta-CB	93	0.005	0.001	0.0005
2,2',3,5,6'-Penta-CB	94	0.005	0.001	0.0005
2,2',3,5',6-Penta-CB 2,2',3',4,6-Penta-CB 2,2',4,5,6'-Penta-CB	95/98/102	0.015	0.003	0.0015
2,2',3,6,6'-Penta-CB	96	0.005	0.001	0.0005
2,2',3,4',5-Penta-CB	97	0.005	0.001	0.0005
2,2',4,4',5-Penta-CB	99	0.005	0.001	0.0005
2,2',4,4',6-Penta-CB	100	0.005	0.001	0.0005
2,2',4,5',6-Penta-CB	103	0.005	0.001	0.0005
2,2',4,4,6'-Penta-CB	104	0.005	0.001	0.0005
2,3,3',4,4'-Penta-CB	105	0.005	0.001	0.0005
2,3',4,4',5-Penta-CB 2,3,3',4,5-Penta-CB	118/106	0.01	0.002	0.001
2,3,3',4',5-Penta-CB 2,3,3',4,6-Penta-CB	107/109	0.01	0.002	0.001
2,3,3',4,5'-Penta-CB 2,3,3',5,6-Penta-CB	108/112	0.01	0.002	0.001
2,3,3',4',6-Penta-CB	110	0.005	0.001	0.0005
2,3,3',5,5'-Penta-CB 2,3,4,4',6-Penta-CB	111/115	0.01	0.002	0.001
2,3,3',5',6-Penta-CB	113	0.005	0.001	0.0005
2,3,4,4',5-Penta-CB	114	0.005	0.001	0.0005
2,3',4,4',6-Penta-CB	119	0.005	0.001	0.0005
2,3',4,5,5'-Penta-CB	120	0.005	0.001	0.0005
2,3',4,5',6-Penta-CB	121	0.005	0.001	0.0005
2,3,3',4',5'-Penta-CB	122	0.005	0.001	0.0005
2,3',4,4',5'-Penta-CB	123	0.005	0.001	0.0005
2,3',4',5,5'-Penta-CB	124	0.005	0.001	0.0005
3,3'4,4',5-Penta-CB	126	0.005	0.001	0.0005
3,3',4,5,5'-Penta-CB	127	0.005	0.001	0.0005
2,2',3,3',4,4'-Hexa-CB 2,3,3',4',5,5'-Hexa-CB	128/162	0.01	0.002	0.001
2,2',3,3',4,5-Hexa-CB	129	0.005	0.001	0.0005
2,2',3,3',4,5'-Hexa-CB	130	0.005	0.001	0.0005
2,2',3,3',4,6-Hexa-CB	131	0.005	0.001	0.0005

Table A7-5. Quantitation Limits for PCB Congeners and Dioxins/Furans

Analyte	Congener Number	QL by Mass of Sample 1 g	5 g	10 g
PCBs (µg/kg-wet weight) (continued)				
2,2',3,3',4,6'-Hexa-CB 2,3,3',4,5',6-Hexa-CB	132/161	0.01	0.002	0.001
2,2',3,3',5,5'-Hexa-CB 2,2',3,4,5,6-Hexa-CB	133/142	0.01	0.002	0.001
2,2',3,3',5,6-Hexa-CB 2,2',3,4,5,6'-Hexa-CB	134/143	0.01	0.002	0.001
2,2',3,3',5,6'-Hexa-CB	135	0.005	0.001	0.0005
2,2',3,3',6,6'-Hexa-CB	136	0.005	0.001	0.0005
2,2',3,4,4',5-Hexa-CB	137	0.005	0.001	0.0005
2,2',3,4,4',5'-Hexa-CB 2,3,3',4',5,6-Hexa-CB 2,3,3',4',5',6-Hexa-CB	138/163/164	0.015	0.003	0.0015
2,2',3,4,4',6-Hexa-CB 2,2',3,4',5',6-Hexa-CB	139/149	0.01	0.002	0.001
2,2',3,4,4',6'-Hexa-CB	140	0.005	0.001	0.0005
2,2',3,4,5,5'-Hexa-CB	141	0.005	0.001	0.0005
2,2',3,4,5',6-Hexa-CB	144	0.005	0.001	0.0005
2,2',3,4,6,6'-Hexa-CB	145	0.005	0.001	0.0005
2,2',3,4',5,5'-Hexa-CB 2,3,3',5,5',6-Hexa-CB	146/165	0.01	0.002	0.001
2,2',3,4',5,6-Hexa-CB	147	0.005	0.001	0.0005
2,2',3,4',5,6'-Hexa-CB	148	0.005	0.001	0.0005
2,2',3,4',6,6'-Hexa-CB	150	0.005	0.001	0.0005
2,2',3,5,5',6-Hexa-CB	151	0.005	0.001	0.0005
2,2',3,5,6,6'-Hexa-CB	152	0.005	0.001	0.0005
2,2',4,4',5,5'-Hexa-CB	153	0.005	0.001	0.0005
2,2',4,4',5,6'-Hexa-CB	154	0.005	0.001	0.0005
2,2',4,4',6,6'-Hexa-CB	155	0.005	0.001	0.0005
2,3,3',4,4',5-Hexa-CB	156	0.005	0.001	0.0005
2,3,3',4,4',5'-Hexa-CB	157	0.005	0.001	0.0005
2,3,3',4,4',6-Hexa-CB 2,3,3',4,5,6-Hexa-CB	158/160	0.01	0.002	0.001
2,3,3',4,5,5'-Hexa-CB	159	0.005	0.001	0.0005
2,3,4,4',5,6-Hexa-CB	166	0.005	0.001	0.0005
2,3',4,4',5,5'-Hexa-CB	167	0.005	0.001	0.0005
2,3',4,4',5',6-Hexa-CB	168	0.005	0.001	0.0005
3,3',4,4',5,5'-Hexa-CB	169	0.005	0.001	0.0005
2,2',3,3',4,4',5-Hepta-CB	170	0.005	0.001	0.0005
2,2',3,3',4,4',6-Hepta-CB	171	0.005	0.001	0.0005
2,2',3,3',4,5,5'-Hepta-CB	172	0.005	0.001	0.0005
2,2',3,3',4,5,6-Hepta-CB	173	0.005	0.001	0.0005
2,2',3,3',4,5,6'-Hepta-CB	174	0.005	0.001	0.0005
2,2',3,3',4,5',6-Hepta-CB	175	0.005	0.001	0.0005
2,2',3,3',4,6,6'-Hepta-CB	176	0.005	0.001	0.0005
2,2',3,3',4',5,6-Hepta-CB	177	0.005	0.001	0.0005

Table A7-5. Quantitation Limits for PCB Congeners and Dioxins/Furans

Analyte	Congener Number	QL by Mass of Sample		
		1 g	5 g	10 g
PCBs (µg/kg-wet weight) (continued)				
2,2’,3,3’,5,5’,6-Hepta-CB	178	0.005	0.001	0.0005
2,2’,3,3’,5,6,6’-Hepta-CB	179	0.005	0.001	0.0005
2,2’,3,4,4’,5,5’-Hepta-CB	180	0.005	0.001	0.0005
2,2’,3,4,4’,5,6-Hepta-CB	181	0.005	0.001	0.0005
2,2',3,4,4',5,6'-Hepta-CB 2,2',3,4',5,5',6-Hepta-CB	182/187	0.01	0.002	0.001
2,2’,3,4,4’,5’,6-Hepta-CB	183	0.005	0.001	0.0005
2,2’,3,4,4’,6,6’-Hepta-CB	184	0.005	0.001	0.0005
2,2’,3,4,5,5’,6-Hepta-CB	185	0.005	0.001	0.0005
2,2’,3,4,5,6,6-’Hepta-CB	186	0.005	0.001	0.0005
2,2’,3,4’,5,6,6’-Hepta-CB	188	0.005	0.001	0.0005
2,3,3’,4,4’,5,5’-Hepta-CB	189	0.005	0.001	0.0005
2,3,3’,4,4’,5,6-Hepta-CB	190	0.005	0.001	0.0005
2,3,3’,4,4’,5’,6-Hepta-CB	191	0.005	0.001	0.0005
2,3,3’,4,5,5’,6-Hepta-CB	192	0.005	0.001	0.0005
2,3,3’,4’,5,5’,6-Hepta-CB	193	0.005	0.001	0.0005
2,2’,3,3’,4,4’,5,5’-Octa-CB	194	0.005	0.001	0.0005
2,2’,3,3’,4,4’,5,6-Octa-CB	195	0.005	0.001	0.0005
2,2',3,3',4,4',5,6'-Octa-CB 2,2',3,4,4',5,5',6-Octa-CB	196/203	0.01	0.002	0.001
2,2’,3,3’,4,4’,6,6’-Octa-CB	197	0.005	0.001	0.0005
2,2’,3,3’,4,5,5’,6-Octa-CB	198	0.005	0.001	0.0005
2,2’,3,3’,4,5,5’,6’-Octa-CB	199	0.005	0.001	0.0005
2,2’,3,3’,4,5,6,6’-Octa-CB	200	0.005	0.001	0.0005
2,2’,3,3’,4,5’,6,6’-Octa-CB	201	0.005	0.001	0.0005
2,2’,3,3’5,5’,6,6’-Octa-CB	202	0.005	0.001	0.0005
2,2’,3,4,4’,5,6,6’-Octa-CB	204	0.005	0.001	0.0005
2,3,3’,4,4’,5,5’,6-Octa-CB	205	0.005	0.001	0.0005
2,2’,3,3’,4,4’,5,5’,6-Nona-CB	206	0.005	0.001	0.0005
2,2’,3,3’,4,4’,5,6,6’-Nona-CB	207	0.005	0.001	0.0005
2,2’,3,3’,4,5,5’,6,6’-Nona-CB	208	0.005	0.001	0.0005
Deca-CB	209	0.005	0.001	0.0005
Dioxins/Furans (ng/kg-wet weight)				
1,2,3,4,6,7,8-Heptachlorodibenzodioxin	NA	25	5	2.5
1,2,3,4,6,7,8-Heptachlorodibenzofuran	NA	25	5	2.5
1,2,3,4,7,8,9-Heptachlorodibenzofuran	NA	25	5	2.5
1,2,3,4,7,8-Hexachlorodibenzodioxin	NA	25	5	2.5
1,2,3,4,7,8-Hexachlorodibenzofuran	NA	25	5	2.5
1,2,3,6,7,8-Hexachlorodibenzodioxin	NA	25	5	2.5
1,2,3,6,7,8-Hexachlorodibenzofuran	NA	25	5	2.5
1,2,3,7,8,9-Hexachlorodibenzodioxin	NA	25	5	2.5
1,2,3,7,8,9-Hexachlorodibenzofuran	NA	25	5	2.5
1,2,3,7,8-Pentachlorodibenzofuran	NA	25	5	2.5
1,2,3,7,8-Pentachlorodibenzo-p-dioxin	NA	25	5	2.5

Table A7-5. Quantitation Limits for PCB Congeners and Dioxins/Furans

Analyte	Congener Number	QL by Mass of Sample		
		1 g	5 g	10 g
Dioxins/Furans (ng/kg-wet weight) (continued)				
2,3,4,6,7,8-Hexachlorodibenzofuran	NA	25	5	2.5
2,3,4,7,8-Pentachlorodibenzofuran	NA	25	5	2.5
2,3,7,8-Tetrachlorodibenzodioxin	NA	5	1	0.5
2,3,7,8-Tetrachlorodibenzofuran	NA	5	1	0.5
Octachlorodibenzodioxin	NA	50	10	5
Octachlorodibenzofuran	NA	50	10	5

Notes:

Quantitation limits (QLs) were obtained from Vista Analytical and are equivalent to reporting limits.

Data will be reported to the sample-specific estimated detection limit, which is typically 2 to 5 times lower than the QL.

CB - chlorobiphenyl

NA - not applicable

PCB - polychlorinated biphenyl

Table B5-1. Standard Reference Materials by Analyte

Analyte	SRM 1	Control Limits for SRM 1 (percent recovery)	SRM 2	Control Limits for SRM 2 (percent recovery)
Conventional Parameters				
Percent moisture	NA	NA	NA	NA
Percent lipids	NA	NA	NA	NA
Metals/Metalloids[a]				
Aluminum	NA	NA	NA	NA
Antimony	NA	NA	NA	NA
Arsenic - total	TORT-3	75-128	DORM-4	72-131
Arsenic - inorganic[b]	NA	NA	NA	NA
Barium	NA	NA	NA	NA
Beryllium	NA	NA	NA	NA
Cadmium	TORT-3	77-125	DORM-4	76-126
Calcium	NA	NA	NA	NA
Chromium	TORT-3	70-135	DORM-4	73-130
Cobalt	NA	NA	NA	NA
Copper	TORT-3	76-125	DORM-4	75-127
Iron	TORT-3	77-125	DORM-4	74-130
Lead	TORT-3	74-130	DORM-4	70-135
Magnesium	NA	NA	NA	NA
Manganese	TORT-3	71-128	NA	NA
Mercury	TORT-3	70-130	NA	NA
Methyl mercury	TORT-3	67-133	NA	NA
Nickel	TORT-3	76-125	DORM-4	67-140
Potassium	NA	NA	NA	NA
Selenium	TORT-3	72-131	DORM-4	72-131
Silver	NA	NA	NA	NA
Sodium	NA	NA	NA	NA
Thallium	NA	NA	NA	NA
Vanadium	TORT-3	78-127	NA	NA
Zinc	TORT-3	76-125	DORM-4	75-127
PCBs[c]				
209 congeners	NA	NA	NA	NA
Dioxins/Furans[c]				
17 congeners	NA	NA	NA	NA

Notes:

[a] Standard reference materials (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.

[b] No SRM is available for inorganic arsenic. Matrix spikes and blank spikes will be used to evaluate extraction efficiency and method performance.

[c] No SRMs are available for the high resolution organics methods. These analytical methods use isotope dilution techniques and each sample will be spiked with an internal standard.

DORM-4 - National Research Council Canada (NRCC) fish protein reference material for trace metals

NA - not applicable

PCB - polychlorinated biphenyl

TORT-3 - NRCC lobster hepatopancreas reference material for trace metals

Table B5-2. Measurement Quality Objectives for Macroinvertebrate Tissue

Analysis	Bias[a] (percent)	Precision[b] (RPD)	Completeness (percent)
Conventional parameters[c]	NA	40	90
TAL metals except mercury	75-125	40	90
Total mercury	70-130	40	90
Methyl mercury	65-135	40	90
Arsenic - inorganic	50-150	40	90
PCB congeners	NA	40[d]	90
Dioxins/furans	NA	40[d]	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.

[c] Includes percent lipids and percent moisture.

[d] Applies to each congener.

NA - not applicable

PCB - polychlorinated biphenyl

APPENDIX A

FIELD SAMPLING PLAN FOR THE MACROINVERTEBRATE TISSUE STUDY

CONTENTS

LIST OF MAPS .... **A-iv**
LIST OF TABLES .... **A-iv**
LIST OF FIGURES .... **A-iv**
ACRONYMS AND ABBREVIATIONS .... **A-v**
UNITS OF MEASURE .... **A-vi**

1 INTRODUCTION **A-1-1**
1.1 OVERVIEW A-1-1

2 SAMPLE COLLECTION AND PROCESSING **A-2-1**
2.1 SAMPLING AREAS A-2-1
2.2 FIELD SURVEY AND SAMPLING METHODS A-2-2
2.2.1 Task Schedule A-2-3
2.2.2 Sampling Location Positioning A-2-3
2.2.3 Field Equipment and Supplies A-2-3
2.2.4 Sample Collection Methods A-2-4
2.2.5 Sampling Contingencies A-2-10
2.2.6 Sample Mass Requirements A-2-10
2.2.7 Sample Acceptability and Quality Assurance A-2-11
2.2.8 Quality Control Samples A-2-11
2.2.9 Location IDs A-2-11
2.2.10 Sample IDs for Individual Organisms A-2-11
2.2.11 Sample IDs for Composite Samples A-2-12
2.2.12 Equipment Decontamination Procedures A-2-13
2.3 SAMPLE HANDLING A-2-13
2.4 CULTURAL RESOURCES A-2-15
2.5 SAMPLE PACKAGING AND TRANSPORT A-2-16
2.6 STUDY-DERIVED WASTE A-2-17
2.7 PROCEDURES FOR AQUATIC INVASIVE SPECIES CONTROL A-2-17

3 FIELD DOCUMENTATION **A-3-1**
3.1 FIELD LOGBOOK A-3-1
3.2 CHAIN-OF-CUSTODY PROCEDURES A-3-3

4 REFERENCES **A-4-1**

Attachment A1. General Site Health and Safety Plan Addendum
Attachment A2. Standard Operating Procedures
Attachment A3. Examples of Various Field Forms
Attachment A4. Archaeological Monitoring Protocol

LIST OF FIGURES

Figure A1. Diagram of Measurements for Mussels

Figure A2. Diagram of Measurements for Crayfish

LIST OF MAPS

Map A1. Proposed Mussel and Crayfish Sampling Areas

Map A2. USFWS Mussel Survey Locations

Map A3. USFWS Crayfish Survey Locations

Map A4. Detail for Sampling Area 1

Map A5. Detail for Sampling Area 2

Map A6. Detail for Sampling Area 3

Map A7. Detail for Sampling Area 4

Map A8. Detail for Sampling Area 5

Map A9. Detail for Sampling Area 6

Map A10. Detail for Rebecca Lake Reference Sampling Area

Map A11. Detail for Sanpoil River Reference Sampling Area

LIST OF TABLES

Table A1. Sampling Areas

Table A2. Methods and Sample Mass Requirements and Prioritization

Table A3. Target Number of Samples to be Collected

ACRONYMS AND ABBREVIATIONS

BERA	baseline ecological risk assessment
COC	chain-of-custody
DGPS	differential global positioning system
EPA	U.S. Environmental Protection Agency
FSP	field sampling plan
GPS	global positioning system
HHRA	human health risk assessment
ID	identification number
LPIL	lowest practical identification level
QA/QC	quality assurance and quality control
QAPP	quality assurance project plan
QC	quality control
RI/FS	remedial investigation and feasibility study
SHSP	site health and safety plan
Site	Upper Columbia River site
SOP	standard operating procedure
TAI	Teck American Incorporated
TAL	Target Analyte List
UCR	Upper Columbia River
USFWS	U.S. Fish and Wildlife Service
UTM	universal transverse mercator
WGS84	World Geodetic System of 1984

UNITS OF MEASURE

°C	degree(s) Celsius
ft	feet
g	gram(s)
mm	millimeter(s)
ww	wet weight

1 INTRODUCTION

This document presents the field sampling plan (FSP) for the macroinvertebrate tissue study (herein 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 baseline ecological risk assessment (BERA) being completed by Teck American Incorporated (TAI) under U.S. Environmental Protection Agency (EPA) oversight and the human health risk assessment (HHRA) being completed by EPA. The objective of the RI/FS is to investigate the nature and extent of contamination and unacceptable risk at the Site to people and the environment.

The primary objective of this study is to characterize the concentrations of the chemicals of interest in tissues of mussels and crayfish sampled from the Site. Mussels and crayfish were selected as representative macroinvertebrates because they are commonly found in the UCR and are consumed by both people and wildlife. Data collection efforts will focus on obtaining information that will inform the exposure assessments for humans, fish, and wildlife that consume benthic invertebrates from the Site. Chemistry data for collected mussels (soft body tissue) and crayfish (the whole body and shell, minus the carapace and stomach)[2] will be used in the HHRA to evaluate exposures of people who consume shellfish. Chemistry data for mussels (soft body tissue) and crayfish (all body parts, including the shell) will be used in the BERA for fish and aquatic-dependent wildlife[3] that forage for those invertebrates in shallow, nearshore waters. This FSP describes how and where mussel and crayfish tissues will be collected for chemical analyses.

EPA's level of effort technical memorandum (USEPA 2013) was used to guide the development of the requirements and design rationale for data collection activities presented in this FSP.

1.1 OVERVIEW

Mussels and crayfish reside in and on the sediments of the Site (Bortleson et al. 2001), and comprise a key part of the aquatic food webs because they consume zooplankton, plants

[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.

[2] Includes the head, claws, abdomen, tail, and shell around the claws and tail.

[3] Wildlife is defined to include amphibians, reptiles, birds, and mammals.

(e.g., phytoplankton, algae, and macrophytes), and detritus. In turn, they constitute prey for fish and wildlife, and are also consumed by humans. Mussels and crayfish in the Site are primarily exposed to chemicals via the sediments and associated porewater, the overlying water column, and through consumption of food items and sediments.

Data on chemical concentrations in the tissues of crayfish from the Site are limited to those from a recent study conducted by the U.S. Fish and Wildlife Service (USFWS), in which analyses were conducted for a subset of Target Analyte List (TAL) metals (USFWS unpublished). Mussel tissue chemistry data have not been collected from the Site. Thus, mussel and crayfish tissues (described herein) will be collected to fill these data gaps. Specific factors to be addressed with this work include:

- Determine if total concentrations of TAL metals, methylmercury, inorganic arsenic, PCBs, and dioxins/furans in mussels and crayfish tissues pose unacceptable risk to human consumers.
- Determine if chemical concentrations of TAL metals in mussel and crayfish tissues pose unacceptable risk to invertivorous fish and wildlife species.

This FSP describes field methods that will be used to collect mussel and crayfish tissues for the study. Section 2 of this FSP describes field 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.
- **Attachment A2—Standard Operating Procedures (SOPs).** Detailed field procedures to be used include:
  - SOP-1 – Recording Macroinvertebrate Tissue Sample Collection Locations
  - SOP-2 – Sample Labeling
  - SOP-3 – Mussel Tissue Sample Collection
  - SOP-4 – Crayfish Tissue Sample Collection
  - SOP-5 – Decontamination of Sampling Equipment
  - SOP-6 – Field Documentation
  - SOP-7 – Sample Packaging and Shipping
  - SOP-8 – Sample Custody
  - SOP-9 – Digital Camera Use and Documentation Procedures
  - SOP-10 – Handling and Reporting of Cultural Resources.

- **Attachment A3—Examples of Various Field Forms.** Contains examples of various forms that will be used during field sampling (tissue collection, processing, and external examination forms); a chain-of-custody (COC) form; and sample labeling forms.
- **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.

2.1 SAMPLING AREAS

Mussel and crayfish will be collected from a total of six primary sampling areas in the Site and two reference areas that are not part of the Site for use in the HHRA (Map A1 and Table A1). In each of these sampling areas, the collection of sufficient organisms for six composites will be targeted for both mussels and crayfish. Thus, a total of 36 Site composites and 12 reference composites will be targeted for each species during this sampling effort. The following summarizes the factors considered for the selection of these sampling areas:

- Spatial coverage of the Site—One sampling area was identified in each of the six river reaches to provide thorough spatial coverage of the Site and to include a variety of different river habitats (i.e., lacustrine, transitional, and riverine).
- Human use areas—Portions of Reaches 5 and 6 sampling areas (i.e., Sampling Areas 5 and 6) were identified as potential local source areas for mussels and crayfish that may be consumed by people. This selection was based on data provided in the Site tribal consumption and resource use survey data summary reports (Westat 2012; EI Ltd. 2012) and anecdotal information provided by a Spokane Tribe representative.
- USFWS mussel and crayfish sampling areas—Areas were selected to include locations where mussel and/or crayfish collection was attempted and successful during the USFWS survey (Maps A2 and A3; see Appendix B of the quality assurance project plan [QAPP] for details).
- Location of fish sampling areas—When possible, the mussel and crayfish sampling areas were selected to overlap with the fish sampling areas (TAI 2009).
- Reference areas—These areas were defined based on information regarding popular areas where tribal survey respondents reported collecting mussels and crayfish. Reference area samples will be used for comparison to Site concentrations in the HHRA.

As a starting point, each sampling area was defined to be approximately 5 river miles in length, although important characteristics, such as human access points and USFWS sampling areas, influenced the exact area sizes. Maps A4 through A9 provide detailed aerials of these sampling areas, along with other relevant information used to define the sampling areas (i.e., approximate fish sampling area boundaries, USFWS mussel/crayfish collection locations, and possible access points for the human health sampling areas on the Site [Sampling Areas 5 and 6 only]). The reference sampling areas are shown on Maps A10 and A11.

Specific sampling locations within the sampling areas (i.e., beaches for mussel collection and crayfish trap locations) will be determined by the field crew, in consultation with EPA oversight or the agency's authorized representative. A brief reconnaissance will be conducted at the start of sampling activities in a given sampling area to identify beaches that may contain mussels and locations with suitable crayfish habitat. The following characteristics will be used to identify such locations:

- Mussel—Beaches selected for attempted mussel sampling will include flat or gently sloping beaches, preferably with finer sediments (e.g., silt) to improve the chances of finding mussels.
- Crayfish—Crayfish traps will be placed in areas identified by the field crew as providing good crayfish habitat. Such areas will include those with loose cobbles and boulders and tree or plant debris, as well as areas near structures such as docks. In consultation with EPA, structures (e.g., refueling stations or docks) that could influence chemical concentrations will be avoided.

Trap and beach locations will be selected during the reconnaissance to ensure that the best habitat areas are included, and that the sampling locations provide good spatial coverage within a given sampling area when possible, based on the available habitat. Details regarding the number of traps to deploy and the number of beaches to survey are discussed in Sections 2.2.4.1 (mussels) and 2.2.4.2 (crayfish).

2.2 FIELD SURVEY AND SAMPLING METHODS

It is anticipated that sampling will be carried out by one team,[4] and that sampling will be conducted primarily from a boat. Boat-based sampling will facilitate easier transport within each of the sampling areas, access to hard-to-reach areas (for the collection of BERA

[4] Depending on scheduling and water level predictions, two teams may be used to conduct sampling.

samples), and easier deployment of crayfish traps. The collection of mussel samples will be done from the beach (i.e., the boat will drop the team off at each beach).

The boat (or boats) will need to have space large enough to accommodate sampling team members, the vessel's captain (and any additional boat crew), one EPA oversight individual, one cultural resource monitor, and one archaeologist. The vessels also should be able to accommodate the following gear: mussel and crayfish sampling equipment (e.g., crayfish traps and viewing buckets for collecting mussels in wadeable waters), sample processing supplies, coolers, and multiple sampling equipment boxes containing sample containers and other ancillary equipment. Vessels must include navigational lights, anchors, and basic sonar equipment (e.g., fathometer). Vessel operators must be familiar with the area and have the capability to make headway and maneuver in the potentially turbulent, high-velocity waters of the Site. The field team leader, together with the vessel crew, will also oversee shoreline access from the vessel to ensure the safety of the field team and that boat activity has minimal impacts on potential sampling locations.

2.2.1 Task Schedule

Subject to EPA approval, field sampling is expected to be conducted during the spring of 2016 (likely starting in late April and ending by late May), and to take approximately 4 to 5 weeks. 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

Sampling locations within the sampling areas (i.e., the start/end of mussel collection beaches and crayfish trap locations) will be recorded using a hand-held global positioning system (GPS) or a boat-mounted differential global positioning system (DGPS)[5] and an associated navigation system (e.g., Nobeltec™ marine navigation software). The procedure for locating each sampling location by universal transverse mercator (UTM) coordinates is detailed in SOP-1 (Attachment A2). The standard projection method to be used during field activities will be the horizontal datum of World Geodetic System of 1984 (WGS84) .

2.2.3 Field Equipment and Supplies

Field equipment and supplies anticipated for this study include mussel and crayfish sampling equipment (e.g., crayfish traps and viewing buckets for collecting mussels in

[5] If a signal for the DGPS cannot be received, a hand-held GPS unit will be used to locate sampling location coordinates.

wadeable waters), decontamination supplies, sample processing supplies (e.g., resealable bags and heavy-duty food grade aluminum foil), coolers, shipping containers, cameras, field logs and forms (or electronic tablet), personal protective equipment, 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 sampling areas.

Sample containers, distilled/deionized water, coolers, and packaging material for samples will be supplied by the analytical laboratory. Details on required sample mass for the analysis of mussel and crayfish tissues 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-2 (Attachment A2) and an example of a sample label is provided in Attachment A3.

2.2.4 Sample Collection Methods

This section describes the mussel and crayfish tissue sampling methods that will be implemented in the field. These methods are supported by SOPs and summarized in the following subsections.

Mussel Sampling

According to the EPA level of effort technical memorandum (USEPA 2013), five species of mussels may be collected at the Site:

- *Anodonta* clade 2 – Western floater
- *Anodonta* clade 1 – California floater
- *Anodonta beringiana* – Yukon floater (historic populations)
- *Margaritifera falcata* – Western pearlshell (historic populations)
- *Gonidea angulata* – Western ridged mussel

In addition, an invasive clam, *Corbicula fluminea*, has previously been collected from the Site and will be retained for possible analysis if found during sampling. Inclusion of any *Corbicula* will be determined as part of the compositing plan to be developed after sampling is completed.

Mussels will be collected from beaches identified within the sampling area by the field crew based on best professional judgment and using available information regarding previously sampled Site locations and areas where mussels are known to be abundant. Mussels will be handpicked by the sampling crew from the shoreline and/or from wadeable waters; viewing buckets may be used in areas of deeper or turbid waters (see SOP-3). Precise locations of mussel collection (i.e., the start and end points of the sampling beach) will be recorded via a hand-held GPS unit.

A target of five mussels per composite was identified based on the available information regarding the species of mussels present in the Site, the expected mass of the tissue to be analyzed, and the required analytical mass (i.e., 4.5 g ww for BERA-only samples and 30 g ww for BERA and HHRA samples; see Table A2).[6] However, because the average size of mussels that will be encountered by the field crew is unknown, the number of organisms collected from a given beach area will not be limited to five if more can be collected at a specific sampling location within a sampling area. No mussels less than 2 cm in length will be collected. Because the mass of soft tissue that will be analyzed will not be known in the field (i.e., mussel shells will not be removed in the field), the field crew may collect additional individual mussels if they are concerned that the five mussels targeted for a composite will not provide sufficient analytical mass. The following summarizes how mussel weights can be estimated by the field crew to ensure that sufficient mass is collected for analysis:

- Available literature (e.g., Bura et al. 2011) suggests that the soft tissue portion of the mussels likely to be encountered at the Site would be approximately 50 percent of the total weight of the mussel.
- Based on this estimate (i.e., that half of the total weight is the shell), the field crew will target the collection of at least 9 g ww for BERA-only samples and at least 60 g ww for BERA and HHRA samples.

Additional individuals that are located after a sufficient number of mussels have been collected to achieve the targeted analytical mass will also be collected, and may be composited with the other individuals to create sufficient mass for a field split sample and/or EPA split sample.

[6] The target analytical mass for BERA-only and BERA and HHRA samples takes into account sample loss due to sample processing (e.g., during homogenization and freeze-drying).

A total of six composite samples is targeted for collection in each sampling area; ideally each composite sample will be collected from a separate beach area, although this may change depending on the availability of mussels in the sampling area. At a given sampling beach, the field crew will search a maximum of 150 m (approximately 500 ft) of shoreline, from approximately 10 m (approximately 33 ft) above the waterline to the maximum wadeable water depth. Specific procedures for how the beach areas will be searched are discussed in SOP-3. Water level fluctuations in the riverine portion of the Site due to hydropower generation will be taken into account during sampling. If no mussels are found after a reasonable effort to locate them, the field crew will document the area as searched and move on to another beach. In each sampling area, the field crew will attempt to collect mussels until sufficient tissue for six composite samples has been collected, or until a maximum of 12 beaches have been searched, whichever condition is met first. Any decision to end sampling before this level of effort is met will be made in consultation with EPA.

Field teams will collect any of the mussel species present at the sampling areas (along with any *Corbicula*) and identify to the lowest practical identification level (LPIL, genus or species). Mussel identification methods will be based on *Freshwater Mussels of the Pacific Northwest (*Nedeau et al. 2009), with the recognition that shell morphology may not be reliable to determine species, but may be reliable for clades (particularly for *Anodonta* sp.). As requested by EPA, TAI will report the location and consult with EPA if western pearlshell mussels are found in the reservoir or riverine segments of the Site. It should be noted that although western pearlshell mussels are preferred by people for consumption, this species has only recently been found in the Kettle River, and has not been found in the main stem of the UCR.

Individual mussel (and clam) characteristics to be recorded in the field form include taxonomic identification; shell length, width, and breadth (as shown on Figure A1); total weight; and presence of holes in shells, or other evidence of parasitism. An example of a field form used to enter data is shown in Attachment A3. Mussels collected from a given beach 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 station location, date, and time of sample (SOP-9). All mussels and clams will be individually wrapped in heavy-duty food grade aluminum foil (dull side toward the organisms) and placed in a resealable plastic bag. Bagged mussels and the associated label will be placed in a second resealable plastic bag and then placed in a cooler with ice, as described in SOP-3. Soft tissue from mussels will be removed from the shell at the analytical laboratory prior to analysis (see SOP-3); any liquid inside the shell will be included with the sample during

homogenization. Each mussel sample will be evaluated for the following acceptance criteria:

- Proper identification
- Appropriate sample handling and decontamination process (SOP-5).

It should be noted that although live mussels will be preferentially collected, dead mussels will not be rejected from the sample if sufficient numbers of live samples for chemical analyses are not found. Information from the 2012 and 2013 USFWS surveys indicated that the majority of mussels found were dead, and consisted of only shells or of dead mussels with some tissue remaining (USFWS unpublished); see Appendix B of the QAPP for details). The exclusion of dead mussels could complicate efforts to collect sufficient mussels for analysis. Furthermore, it is likely that the fish and wildlife that consume mussels would consume the tissue of dead mussels when encountered. If both dead and live mussels are retained at a given location, the dead and live mussels will be bagged separately, and inclusion of the dead mussels will be determined as part of the compositing plan to be prepared in consultation with EPA after sampling is completed.

Crayfish Sampling

Two species of crayfish have been trapped in the Site—northern crayfish (*Orconectes virilis*) and signal crayfish (*Pacifastacus leniusculus*). Crayfish identification will be determined based on the use of applicable field guides and field crew knowledge of these species; photographs will be taken of all crayfish collected at the site, as discussed below and in SOP-3. As requested by EPA, TAI will report the location and consult with EPA if signal crayfish are encountered in the Site; non-target species (i.e., species other than crayfish) captured in crayfish traps will be released at point of capture and noted on the non-target species form (see Attachment A3).

Crayfish will be sampled via the deployment of baited traps with escape guards (e.g., minnow traps) that will be placed in areas with potential crayfish habitat. Crayfish habitat includes areas with loose cobbles and boulders, areas with tree or plant debris, and areas near structures such as docks (see SOP-4).[7] Bait will be placed in a claw-proof bag (e.g., nylon or cheesecloth) or bait canister to ensure that crayfish do not consume the bait, which could affect sampling results; broken bait bags/canisters will be noted on the field forms. Within each sampling area, the specific locations where traps will be set will be determined by the field crew based on best professional judgment. The field crew will

[7] Traps will not be placed in the vicinity of structures such as refueling stations, which could influence chemical concentrations.

target areas that provide good habitat and, when possible, good spatial coverage of the sampling area. Locations where the USFWS survey (USFWS unpublished) found crayfish will also be used to inform the selection of specific sampling locations within the sampling areas (see Appendix B of the QAPP for details).

A total of six composite samples is targeted for collection in each sampling area; each sample will consist of five crayfish (i.e., a total of 30 crayfish in each area). The target of five crayfish per composite was determined based on an analysis of the sizes of crayfish that are anticipated to be collected (3.5 to 4 in. in total length or larger; see SOP-4 for crayfish size requirements) and the required analytical mass needed for each sample (i.e., 4.5 g ww for BERA-only samples and 30 g ww for BERA and HHRA samples; see Table A2).[8] The following summarizes how a composite size of five crayfish was determined:

- Based on the available information, the average crayfish size that the field crew will encounter is estimated to be about 3.5 to 4 in. in total length. Crayfish of this size will generally weigh between 30 and 45 g ww (based on unpublished data), meaning that a composite of five crayfish will weigh between 150 and 225 g ww.
- The carapace and stomach of the crayfish are estimated to comprise approximately 50 percent of the whole-body weight.
- For the BERA and HHRA samples, the total weight would be approximately split in half for analysis (i.e., the whole-body minus carapace and stomach samples, and the carapace- and stomach-only samples). Based on the above information, the weight of each of these samples would be 75 to 113 g ww, more than sufficient to meet the analytical requirements.

If additional individuals are trapped, these will be retained, and may be composited with the other individuals to create sufficient mass for a field split sample and/or EPA split sample. All non-native crayfish (i.e., northern/virile crayfish) will be retained regardless of size. For native (i.e., signal) crayfish, only individuals greater than 3.25 in. in total length will be retained, in accordance with Washington State fishing regulations, for the human health exposure areas (i.e., Sampling Areas 2, 5 and 6 and both reference areas). All crayfish, regardless of size, will be retained in other sampling areas.

The compositing scheme will be determined in consultation with EPA following the completion of sampling; therefore, individual crayfish will be processed separately for shipment to the laboratory. In each sampling area, a total of 30 traps will be deployed (i.e.,

[8] The target analytical mass for BERA-only and BERA and HHRA samples takes into account sample loss due to sample processing (e.g., during homogenization and freeze drying).

one trap per targeted crayfish). The use of 30 traps for a maximum of 3 overnight periods was determined based on the catch-per-unit effort information provided in the USFWS survey (Appendix B of the QAPP). Any decision to end sampling before this level of effort is met will be made in consultation with EPA. Precise locations will be recorded using GPS, and ropes will be inconspicuously marked to minimize vandalism. Traps will be checked twice daily (mornings and evenings) during sampling for a maximum of three nights in a given sampling area. Crayfish from each trap will be removed to a bucket for processing. It is anticipated that crayfish found in traps would still be living. Any dead crayfish recovered in traps may also be retained in consultation with EPA oversight; this information would be noted on the field form. Field teams will identify captured crayfish by species and record it on the specimen collection form. Once the targeted number of crayfish are collected (n = 30) or the maximum period designated for trap deployment has been reached (whichever comes first), all traps and ropes will be removed from the sampling area.

Each crayfish will be photographed, and the photograph ID will be documented on the field form so that the photograph can be subsequently labeled with station location, date, and time of sample collection (SOP-9). Length (both total and carapace, as indicated in Figure A2), total weight, and the presence of lesions, missing limbs, and general condition of collected individual crayfish will be noted on the field form. In addition, the field crew will note on the field forms if any of the following are applicable: 1) crayfish are molting, 2) crayfish are carrying eggs, 3) crayfish were dead upon trap retrieval, or 4) bait bags/canisters are broken. This information will be considered in the development of the compositing plan that will be prepared in consultation with EPA following the completion of the sampling effort. All crayfish will be individually wrapped in heavy-duty food grade aluminum foil (dull side toward the organism), and placed in a resealable plastic bag with the associated label. The label and foil-wrapped/bagged crayfish will be placed in a second clean, resealable plastic outer bag, and then stored in a cooler with ice while in the field, as described in SOP-4.

Each crayfish sample will be evaluated for the following acceptance criteria:

- Proper deployment and functioning of trap
- Adequate retrieval and appropriate sample handling

Crayfish tissue dissection and compositing will be performed by the analytical laboratory. For BERA samples, crayfish will be analyzed as whole-body samples (i.e., no dissection will be needed). For samples that will be used for both the BERA and HHRA, crayfish will be analyzed as two parts: the carapace and stomach will be removed and analyzed as one sample, and to better represent the tissue generally consumed by people, the remaining

parts will be analyzed as the other sample (see the ALS Environmental SOPs in Appendix C of the QAPP for details on tissue dissection procedures). Whole-body chemical concentrations can be calculated for use in the BERA using the weights of the two samples (i.e., the carapace and stomach, and the other remaining tissue) and their chemical concentrations. Target samples and tissue types are summarized in Table A3.

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 QAPP), absence of recoverable mussel and/or crayfish, and/or above-average river flow conditions. Specific sample locations were not identified for this sampling effort to help accommodate such circumstances. The field sampling crew will select specific sampling locations within the sampling areas based on available habitat and physical conditions. If it is necessary to expand the designated sampling area, the field sampling team will consult with EPA oversight or the agency's authorized representative.

If the targeted number of samples for either mussels or crayfish in a given sampling area is not met after the maximum level of effort identified in this FSP has been completed, the number of samples collected in that area will be less than what was targeted. For crayfish, it is also possible that the level of effort may be reduced (i.e., traps may be deployed for fewer than three nights) if crayfish sampling is unsuccessful and suitable habitat is limited. This decision would be made in consultation and agreement with EPA oversight.

2.2.6 Sample Mass Requirements

Sampling for mussels and crayfish will be conducted to inform the risk assessments for invertivorous wildlife and humans that could consume these organisms. To conduct the desired analyses for the refined list of chemicals identified in the QAPP, a target sample mass of 4.5 g ww is needed for BERA-only samples, and a sample mass of 30 g ww is needed for BERA and HHRA samples (Table A2).

Note that these minimum tissue needs are based on analyzable tissues (i.e., only soft tissue for mussels, and the separated crayfish tissues for samples that will be used in both the BERA and HHRA). However, analytes have been prioritized so that samples with less than the minimum sample volume requirement can be appropriately handled. The prioritization sequence is indicated in Table A2.

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 organism that is collected, as described in Section 2.2.4. Field personnel will apply the criteria for mussel and crayfish tissue collection using their experience and best professional judgment.

2.2.8 Quality Control Samples

Laboratory QC samples will be used to assess sample variability and evaluate potential sources of contamination. For mussel and crayfish, given the likelihood that insufficient tissue may be collected at some sites, allocating tissue to chemical analyses will be prioritized. If sufficient tissue is collected, the analytical laboratory will be instructed to analyze a field split sample at a targeted frequency of 5 percent of the total number of samples, or one sample per analytical batch (whichever is greater). EPA split samples may also be created if sufficient tissue is available. Detailed information on quality assurance and quality control (QA/QC) procedures, limits, and reporting is provided in the QAPP.

2.2.9 Location IDs

Mussel collection beaches and crayfish trap locations will be determined by the field crew during field sampling, and will each be assigned a unique identifier. These location IDs will consist of the following parts:

- Two-digit sampling area code—see Table A1 for the sampling area code (e.g., A1 = Sampling Area 1, RL = Rebecca Lake, and SR = Sanpoil River)
- Sample type code—MB for mussel beach and CT for crayfish trap
- Two-digit sequential number.

Examples:

A4-CT03 = the third crayfish trap placed in Sampling Area 4

SR-MB01 = the first mussel beach in the Sanpoil River reference sampling area.

These location IDs will be used to document sampling locations within the sampling areas.

2.2.10 Sample IDs for Individual Organisms

Each individual organism will be assigned a unique identifier. The sample ID will include the location ID (as described in Section 2.2.9), the species code, and the individual number, as shown below.

- Two-digit sampling area code—see Table A1 for the sampling area code (e.g., A1 = Sampling Area 1, RL = Rebecca Lake, and SR = Sanpoil River)

- Sample type code—MB for mussel beach and CT for crayfish trap
- Two-digit sequential number
- Species code—MU for mussels, CL for clam, or CR for crayfish
- Two-digit individual number—sequential number for each individual collected in a given sampling area (e.g., mussel #03 collected in Sampling Area 4). Note that this is important to be able to link analytical chemistry results.

Examples:

A4-MU-03 = Mussel individual #03 collected from Sampling Area 4

RL-CR-01 = Crayfish #01 collected from the Rebecca Lake Reference Sampling Area.

Individual organisms will be wrapped in heavy-duty food grade aluminum foil (dull side towards the organisms) and 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-4).

2.2.11 Sample IDs for Composite Samples

Mussel and crayfish tissue composites will be prepared by the analytical laboratory upon receipt of samples; a compositing scheme (i.e., which individuals will be included in which composite) will be determined in consultation with EPA after sampling is completed. This compositing plan will document which individual organisms will be included in each composite sample. This information will also be documented by the analytical laboratory during processing.

Unique sample identifiers will be assigned to these composites. All mussel samples will be composited as soft tissue (i.e., the shell will be removed; any liquid inside the shell will be retained for analysis); samples will be designated as ST. As noted above, crayfish analysis will be conducted based on composites of whole-body samples for BERA-only samples (designated as WB), or for BERA and HHRA samples, based on composites of both whole-body minus carapace/stomach (designated as PB) and carapace/stomach samples (designated as CS). It is essential that the same individuals are used in each composite so that whole-body tissue concentrations can be mathematically calculated; therefore, there should be complementary tail meat and remaining body results for each composite. The composite ID will consist of the following elements:

- Two-digit sampling area code—see Table A1 for the sampling area code (e.g., A1 = Sampling Area 1, RL = Rebecca Lake, and SR = Sanpoil River)
- Species code—MU for mussels, CL for clam, and CR for crayfish

- Composite number—a four-digit code starting with C, followed by a sequential three-digit number (e.g., C001)
- Tissue code—WB for whole body, PB for whole body minus carapace/stomach, CS for carapace/stomach, and ST for soft tissue.

Examples:

A6-MU-C001-ST = the first composite mussel soft tissue sample consisting of mussels from Sampling Area 6

A4-CR-C002-WB = the second composite whole-body tissue sample consisting of crayfish from Sampling Area 4

RL-CR-C006-CS = the sixth composite carapace and stomach tissue sample consisting of crayfish from the Rebecca Lake Reference Sampling Area.

2.2.12 Equipment Decontamination Procedures

All sampling equipment (other than crayfish traps) coming into direct contact with samples will be decontaminated prior to beginning field work, between sampling stations, and at the conclusion of the field effort as outlined in SOP-5 (Attachment A2). Nitrile or latex gloves will be used for handling samples and will be discarded in between sampling stations. Clean gloves will be worn at each sampling station 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-6; Attachment A2); laboratory procedures are presented in Appendix C (analytical laboratory) 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. The following documentation will be provided with samples:

- A field form that contains information about each sampling location
- Sample coordinates (recorded on the field form and/or recorded electronically)
- Photo documentation (SOP-9)
- 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):

- Sample ID
- Location ID (i.e., the trap or beach location)
- 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.

Individual organisms will be wrapped in heavy-duty food grade aluminum foil (dull side in contact with the organism), and then placed inside a resealable plastic bag. The bagged sample and sample label will then be placed inside a second resealable plastic bag. This process will ensure that when individual organisms are prepared for shipment, the sample label will remain with the correct organism.

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

- Taxonomic identification of collected specimens to LPIL
- Length, width, and breadth of mussel (Figure A1)
- Total weight
- Location collection information (i.e., location ID, date, and time)
- Photograph ID (all organisms will be photographed)
- Presence of holes in shells or evidence of parasitism, and whether the organism is alive or dead.

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

- Taxonomic identification of collected specimens
- Total length and carapace length (Figure A2)
- Total individual weight

- Location collection information (i.e., location ID, date, and time)
- Photograph ID (all organisms will be photographed)
- Presence of broken bait bag/container in the crayfish trap
- Presence of lesions or missing limbs, whether the crayfish is molting or carrying eggs, and other general notes regarding the condition of collected crayfish (including whether the organism is alive or dead).

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 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. 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. 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

After completing each day of sampling, all samples will transferred from the coolers with wet ice into a freezer or cooler with dry ice, there to be held until preparation for shipment to the laboratory. Although there is no specified duration of time after collection that samples must be frozen, this will be done as soon as the field crew returns to the location where samples are being held. A secure area will be available for sample holding and preparing samples for shipment to the laboratory (SOP-7; Attachment A2). The temperature of the cooler/freezer will be recorded in the logbook twice daily (both in the morning and evening). 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.

All samples will be stored frozen in a secure onshore area (i.e., in a freezer or in a cooler with dry ice) while they are awaiting shipping. Prior to shipping to the analytical laboratory, samples will be packed on dry ice and shipped via priority overnight delivery service or courier service so that they arrive at the processing laboratory within 48 hours from the time of sample shipment.

Styrofoam® coolers placed inside of cardboard boxes will be used as shipping containers, and appropriate shipping labels indicating the use of dry ice will be affixed to the boxes. Sufficient 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. 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
- Perishable goods label.

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.

2.7 PROCEDURES FOR AQUATIC INVASIVE SPECIES CONTROL

Aquatic invasive species are a serious ecological and economic threat, and sampling with research vessels has the potential to spread non-native noxious weeds, pathogens, and exotic flora and fauna among water bodies. The sampling vessel captain and crew will need to be familiar with the risks of invasive species and trained on inspection and decontamination procedures. The sampling vessel(s) and sampling equipment that has previously been used at other sites (e.g., crayfish traps) will be thoroughly inspected and cleaned before the field effort to prevent transport of exotic species (e.g., New Zealand mudsnail, quagga and zebra mussels, and milfoil). Because the Site is not an Area of Extreme or Moderate Concern, the sampling vessels do not need to be decontaminated between sampling locations within the Site or after the sampling effort is complete. If invasive species (e.g., zebra mussels or New Zealand mud snails) are collected during sampling, the presence of these species will be immediately reported to the appropriate management agencies (e.g., USFWS).

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 sampling areas will be collected.

3.1 FIELD LOGBOOK

All field activities and observations will be noted in a field log (SOP-6). 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, station designation, sampler, types of sample(s) 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 location(s) within each sampling area
- Task start date and end date
- Weather conditions
- Name of person making entries and other field staff (including EPA oversight and boat crew)
- Onsite visitors, if any
- Sampling vessel
- Date and collection time of each sample
- The sampling location name(s)
- Coordinates and water depths for deployed crayfish traps
- Start and end coordinates for mussel beaches
- Condition of retrieved crayfish traps
- Specific information on each type of sampling activity
- 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 crayfish trap locations and mussel beach locations, as well as the number of individual organisms collected at each location. 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 logbook(s) 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 (i.e., mussel or crayfish tissue)
- Number of sample containers (i.e., coolers) shipped
- Requested analyses for each sample (as shown in Table A2)
- 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. If sample temperatures fall outside the range (i.e., the internal temperature of the cooler is >0°C), the field supervisor will be alerted immediately and field personnel will increase the amount of dry ice 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.

Mussel and crayfish composites will be prepared by the analytical laboratory upon receipt of the compositing plan. This plan, which will specify the individuals to be included in each composite, will be determined in consultation with EPA after sampling is completed. This compositing plan will document which individual organisms will be included in each composite sample. This information will also be documented by the analytical laboratory during processing. Unique sample identifiers will be assigned to these composites.

4 REFERENCES

Bortleson, G.C., S.E. Cox, M.D. Munn, R.J. Schumaker, and E.K. Block. 2001. Sediment-quality assessment of Franklin D. Roosevelt Lake and the upstream reach of the Columbia River, Washington, 1992, Water-Supply Paper 2496, US Geological Survey, Tacoma, WA.

Bura, M., R. Muscalu-Nagy, C. Hevesi, and I. Banatean-Dunea. 2011. Morphometric studies on *Anodonta anatina* bivalve population from the Dognecea Lake. Sci Papers: Anim Sci Biotech 44(2):9-12.

EI Ltd. 2012. Food questionnaire data report: Upper Columbia River resources survey. Prepared for Confederated Tribes of the Colville Reservation, Nespelem, WA.

Nedeau, E.J., A.K. Smith, J. Stone, and S. Jepsen. 2009. Freshwater Mussels of the Pacific Northwest. Second Edition. http://www.xerces.org/wp-content/uploads/2009/06/pnw_mussel_guide_2nd_edition.pdf.

TAI. 2009. Upper Columbia River final quality assurance project plan for the 2009 fish tissue study. Prepared by Parametrix, Integral Consulting, Inc., and Exponent for Teck American Incorporated, Spokane, WA.TAI. 2010. Upper Columbia River screening-level ecological risk assessment (SLERA). Prepared by Parametrix, Inc., Exponent, and Integral Consulting, Inc. for Teck American Incorporated, Spokane, WA.

USEPA (United States Environmental Protection Agency). 2013. Expected level of effort for sampling and analysis of benthic macroinvertebrate tissue—Upper Columbia River. Developed by the USEPA Technical Team, with consideration of comments provided by the Participating Parties. July 16, 2013.

USFWS (U.S. Fish and Wildlife Service). Unpublished. Data files containing results from 2012 and 2013 surveys of mussels and crayfish in the Upper Columbia River Site. Sent to John Toll, Windward Environmental, by Dustan Bott, EPA Region 10, on December 15, 2015.

FIGURES

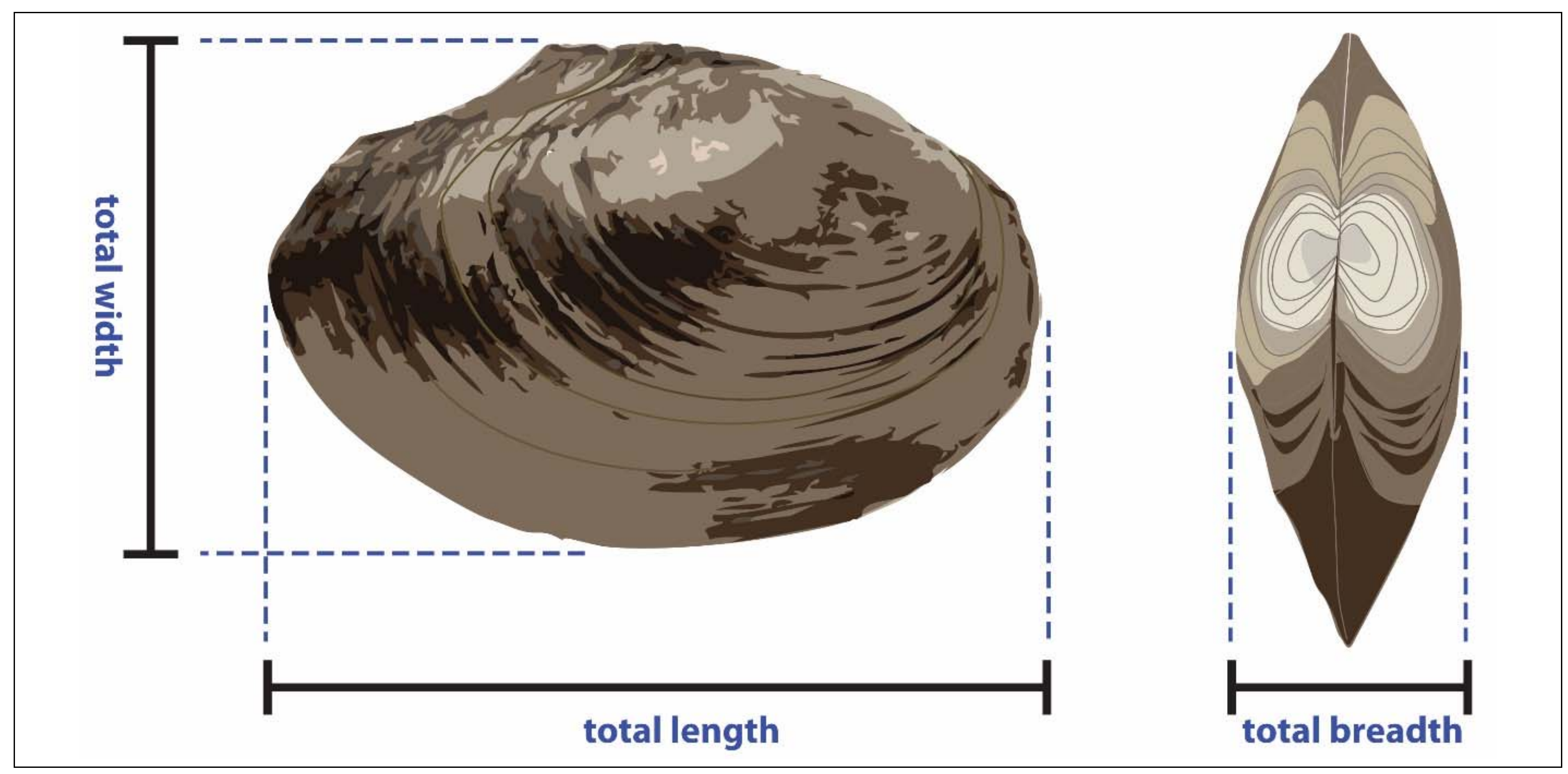


Figure A1. Diagram of Measurements for Mussels

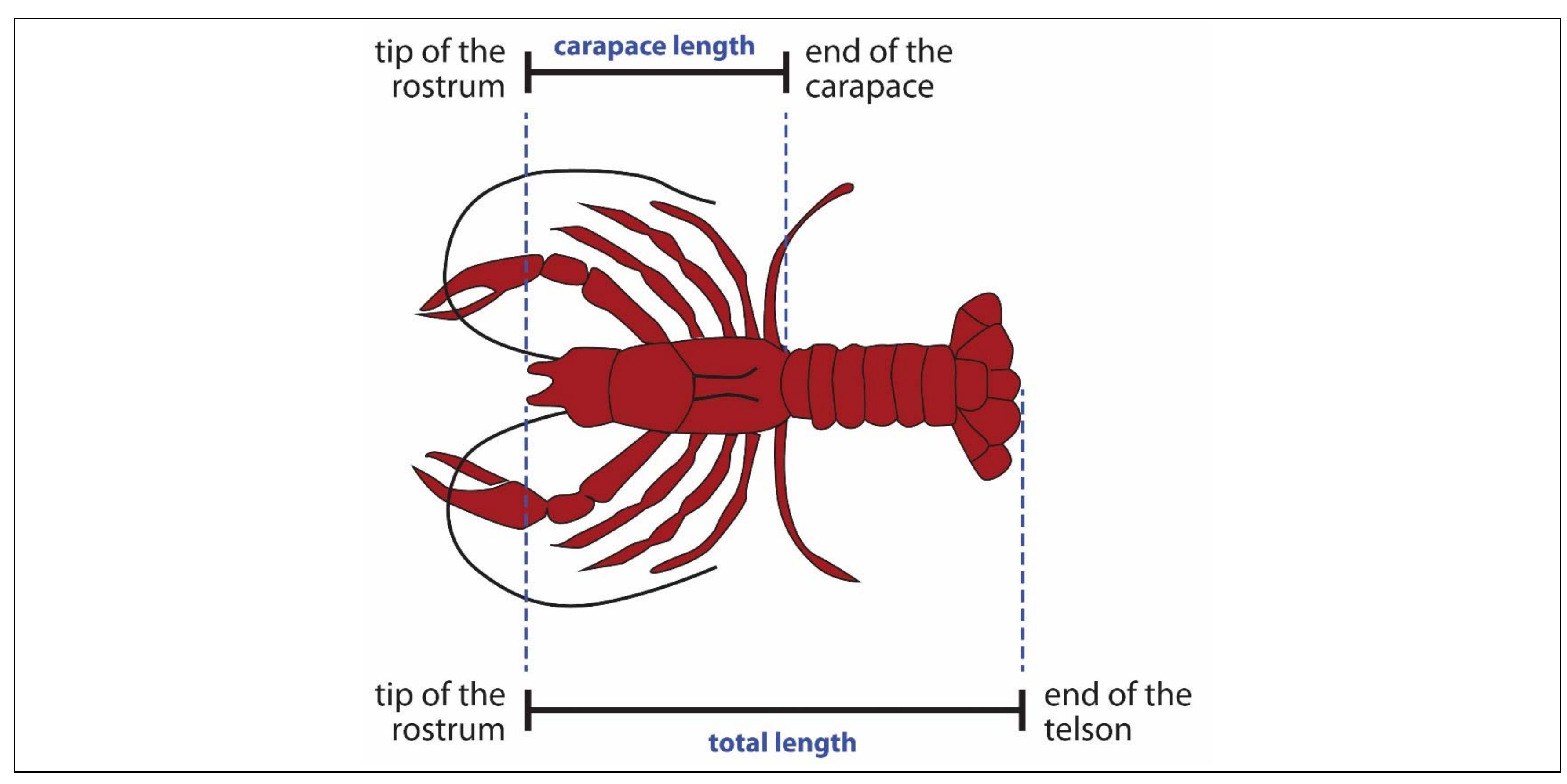
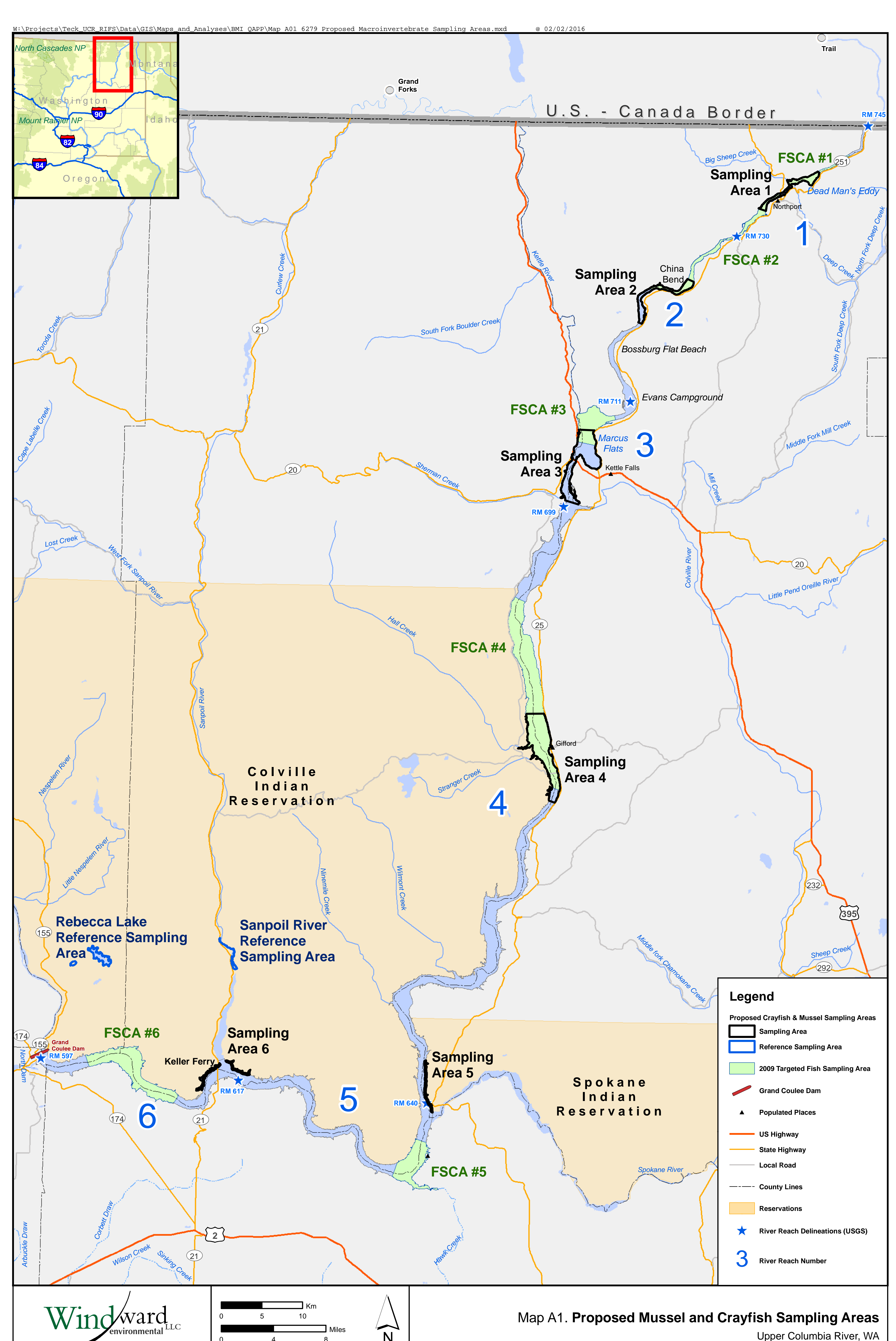


Figure A2. Diagram of Measurements for Crayfish

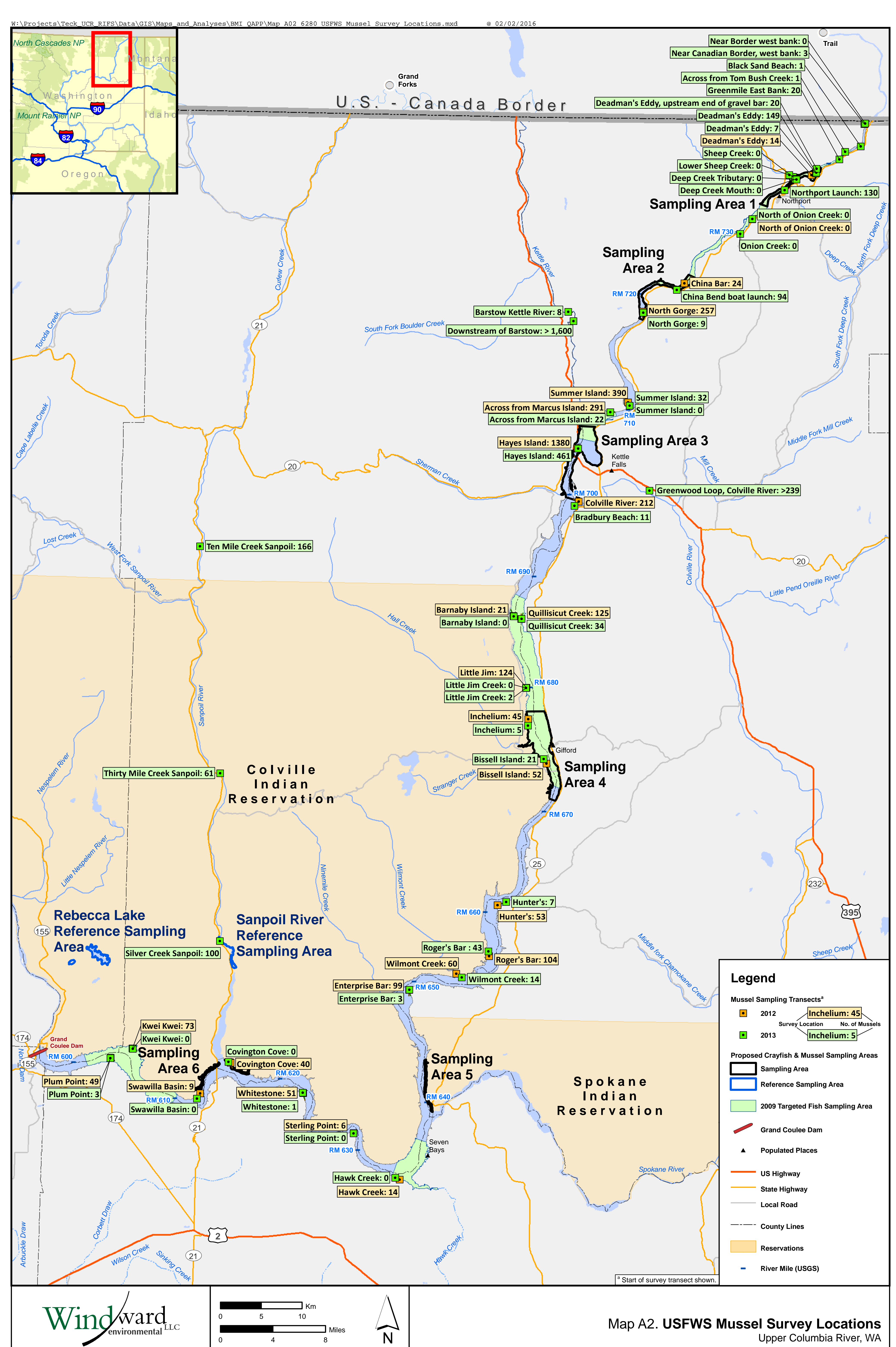
MAPS



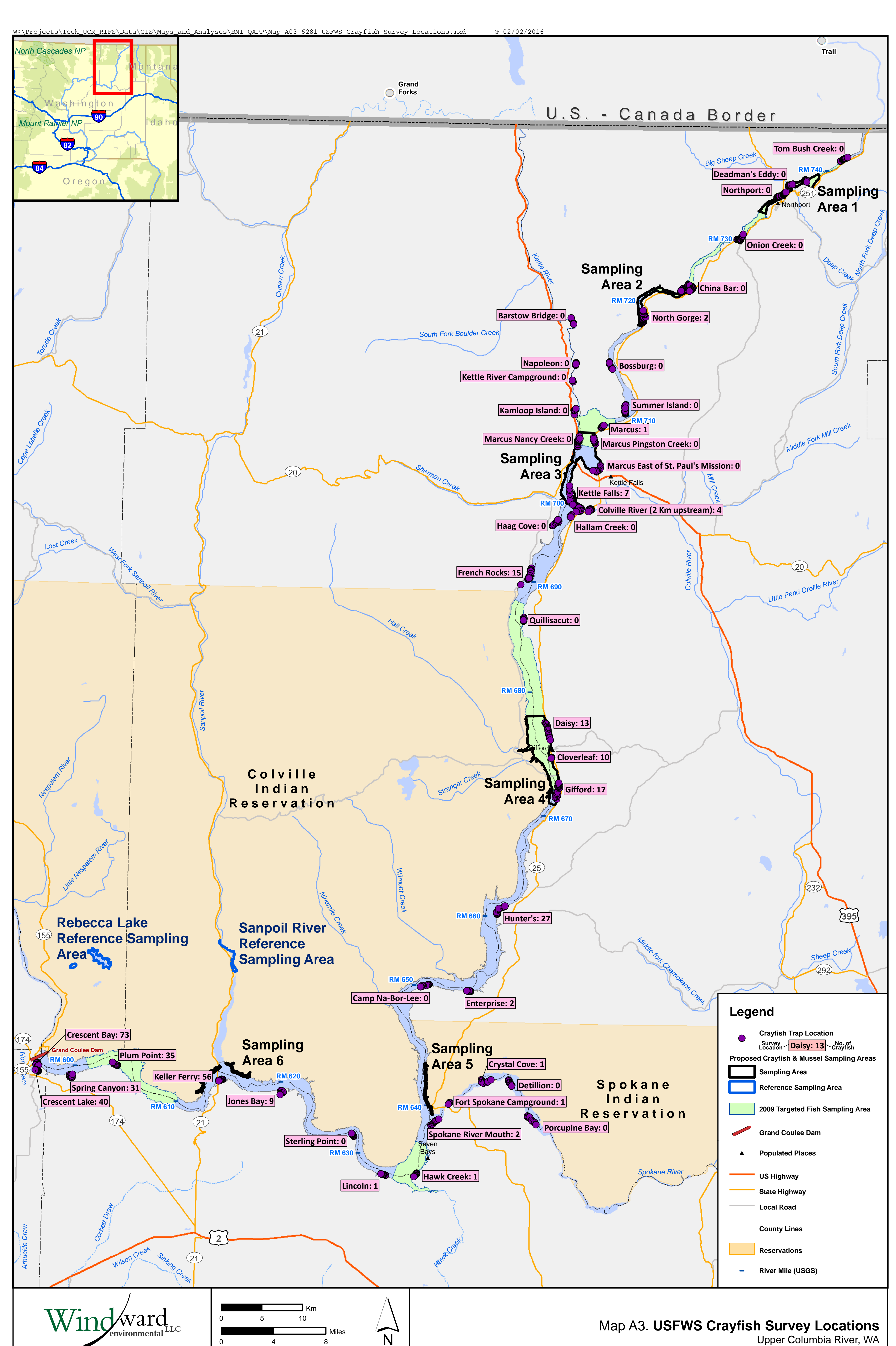
Windward environmental LLC

Map A1. **Proposed Mussel and Crayfish Sampling Areas**

Upper Columbia River, WA

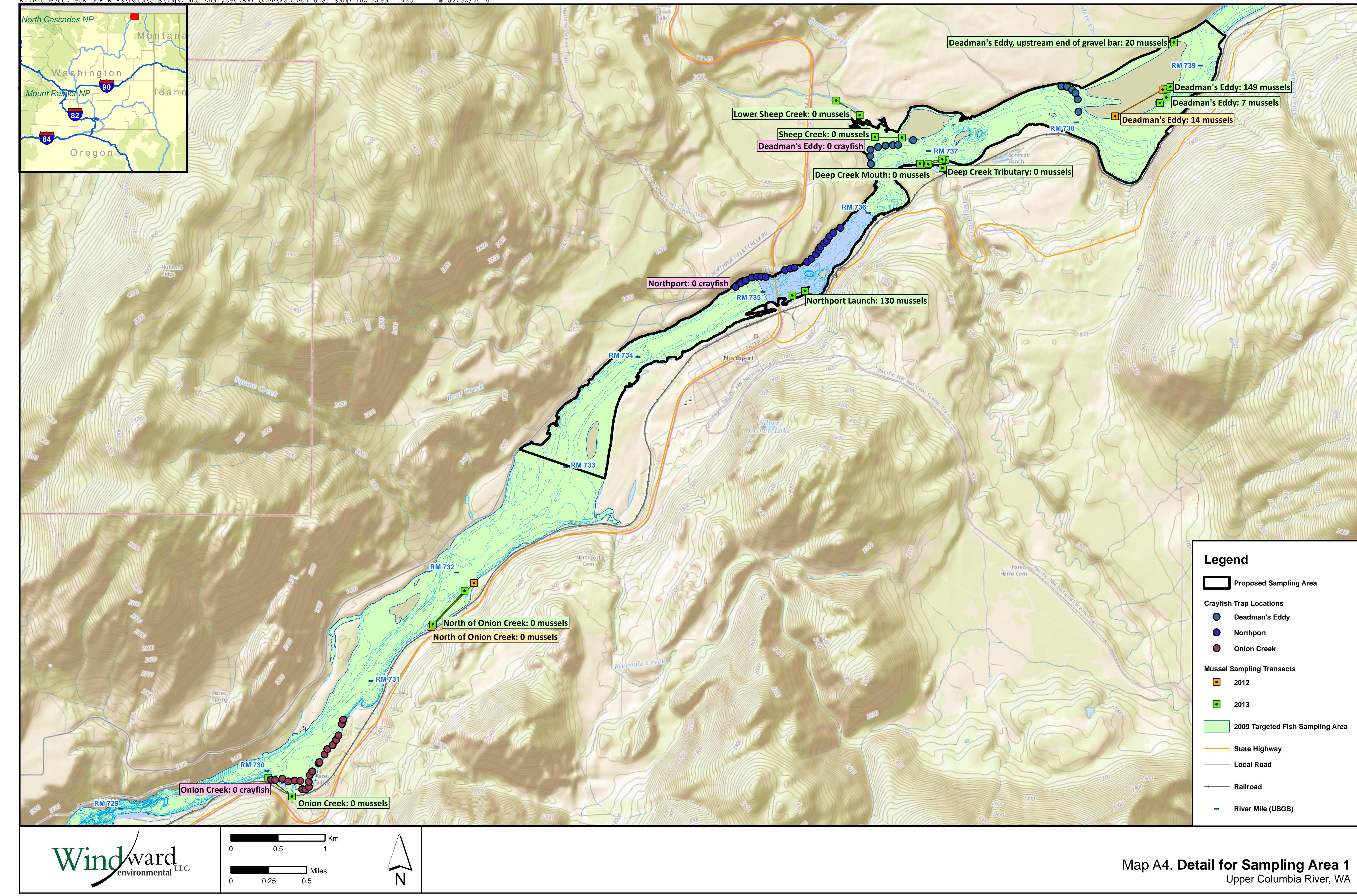


W:\Projects\Teck_UCR_RIFS\Data\GIS\Maps_and_Analyses\BMI_QAPP\Map_A02_6280_USFWS_Mussel_Survey_Locations.mxd © 02/02/2016
North Cascades NP
Montana
Washington
Mount Rainier NP
Idaho
Oregon
90
82
84
U.S. - Canada Border
Grand Forks
Trail
Northport
Kettle Falls
Gifford
Seven Bays
Near Border west bank: 0
Near Canadian Border, west bank: 3
Black Sand Beach: 1
Across from Tom Bush Creek: 1
Greenmile East Bank: 20
Deadman's Eddy, upstream end of gravel bar: 20
Deadman's Eddy: 149
Deadman's Eddy: 7
Deadman's Eddy: 14
Sheep Creek: 0
Lower Sheep Creek: 0
Deep Creek Tributary: 0
Deep Creek Mouth: 0
Northport Launch: 130
Sampling Area 1
North of Onion Creek: 0
North of Onion Creek: 0
Onion Creek: 0
RM 730
Sampling Area 2
China Bar: 24
China Bend boat launch: 94
RM 720
North Gorge: 257
North Gorge: 9
Barstow Kettle River: 8
Downstream of Barstow: > 1,600
Summer Island: 390
Summer Island: 32
Summer Island: 0
Across from Marcus Island: 291
Across from Marcus Island: 22
RM 710
Sampling Area 3
Hayes Island: 1380
Hayes Island: 461
Greenwood Loop, Colville River: >239
RM 700
Colville River: 212
Bradbury Beach: 11
Ten Mile Creek Sanpoil: 166
RM 690
Barnaby Island: 21
Barnaby Island: 0
Quillisicut Creek: 125
Quillisicut Creek: 34
Little Jim: 124
Little Jim Creek: 0
Little Jim Creek: 2
RM 680
Inchelium: 45
Inchelium: 5
Bissell Island: 21
Bissell Island: 52
Sampling Area 4
RM 670
Thirty Mile Creek Sanpoil: 61
Colville Indian Reservation
Hunter's: 7
Hunter's: 53
RM 660
Roger's Bar : 43
Roger's Bar: 104
Wilmont Creek: 60
Wilmont Creek: 14
Enterprise Bar: 99
Enterprise Bar: 3
RM 650
Rebecca Lake Reference Sampling Area
Sanpoil River Reference Sampling Area
Silver Creek Sanpoil: 100
Kwei Kwei: 73
Kwei Kwei: 0
Sampling Area 6
Grand Coulee Dam
RM 600
Plum Point: 49
Plum Point: 3
Swawilla Basin: 9
Swawilla Basin: 0
Covington Cove: 0
Covington Cove: 40
RM 620
RM 610
Whitestone: 51
Whitestone: 1
Sampling Area 5
RM 640
Sterling Point: 6
Sterling Point: 0
RM 630
Hawk Creek: 0
Hawk Creek: 14
Spokane Indian Reservation
Kettle River
Curlew Creek
Toroda Creek
South Fork Boulder Creek
Cape Labelle Creek
Sherman Creek
Lost Creek
West Fork Sanpoil River
Hall Creek
Sanpoil River
Nespelem River
Little Nespelem River
Stranger Creek
Nineile Creek
Wilmont Creek
Colville River
Little Pend Oreille River
Mill Creek
Middle Fork Mill Creek
South Fork Deep Creek
Deep Creek
North Fork Deep Creek
Middle fork Chamokane Creek
Sheep Creek
Spokane River
Corbett Draw
Wilson Creek
Sinking Creek
Hawk Creek
Arbuckle Draw
21
20
25
155
174
2
232
395
Legend
Mussel Sampling Transects[a]
2012
2013
Inchelium: 45
Inchelium: 5
Survey Location
No. of Mussels
Proposed Crayfish & Mussel Sampling Areas
Sampling Area
Reference Sampling Area
2009 Targeted Fish Sampling Area
Grand Coulee Dam
Populated Places
US Highway
State Highway
Local Road
County Lines
Reservations
River Mile (USGS)
[a] Start of survey transect shown.
Windward environmental LLC
Km 0 5 10
Miles 0 4 8
N
Map A2. USFWS Mussel Survey Locations
Upper Columbia River, WA

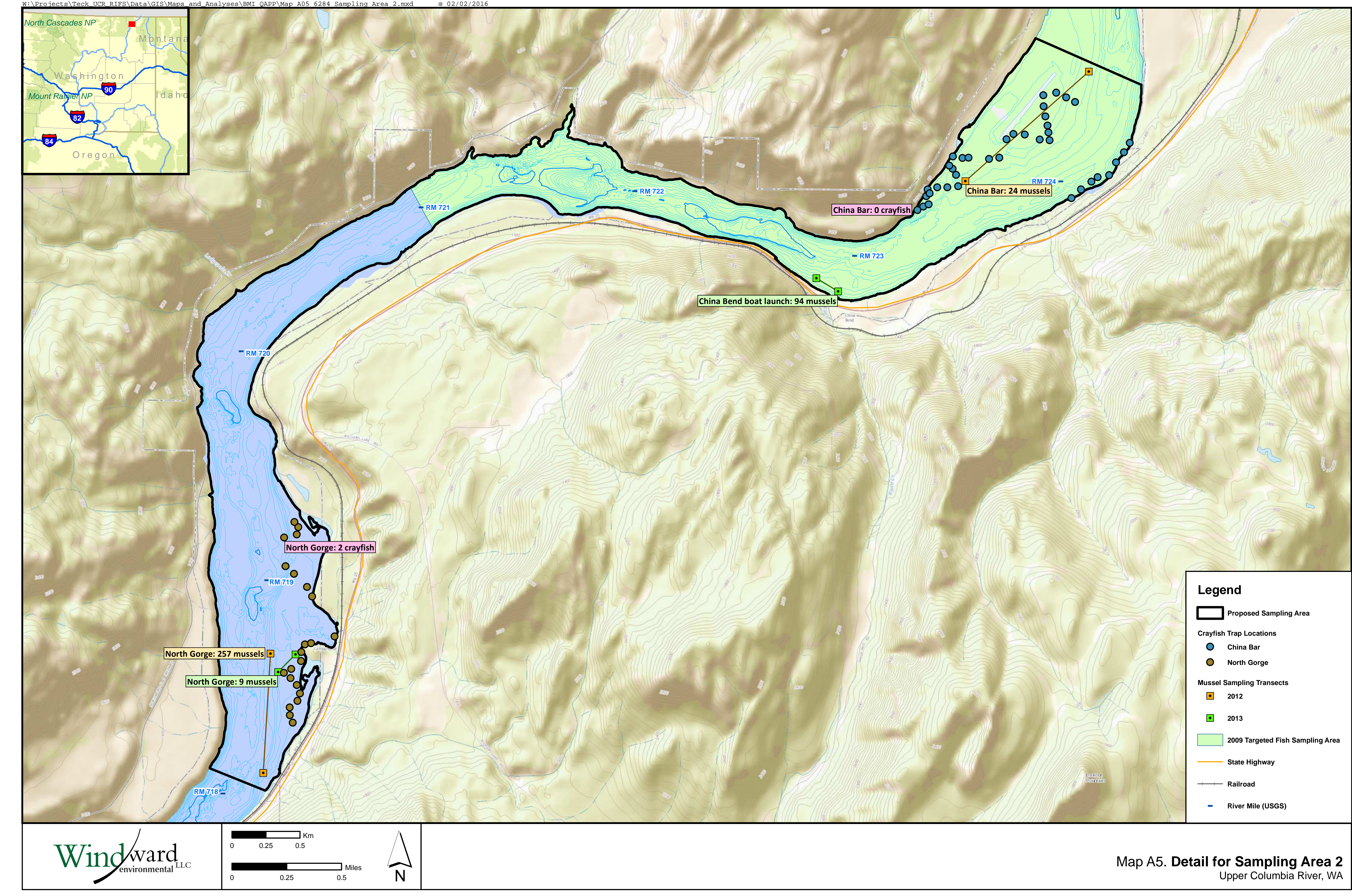


W:\Projects\Teck_UCR_RIFS\Data\GIS\Maps_and_Analyses\BMI_QAPP\Map_A03_6281_USFWS_Crayfish_Survey_Locations.mxd
© 02/02/2016
North Cascades NP
Montana
Washington
Idaho
Mount Rainier NP
Oregon
90
82
84
Grand Forks
Trail
U.S. - Canada Border
Tom Bush Creek: 0
Deadman's Eddy: 0
Northport: 0
Sampling Area 1
Northport
RM 740
RM 730
Onion Creek: 0
Big Sheep Creek
Deep Creek
North Fork Deep Creek
Sampling Area 2
China Bar: 0
RM 720
North Gorge: 2
Barstow Bridge: 0
Kettle River
South Fork Boulder Creek
Napoleon: 0
Kettle River Campground: 0
Bossburg: 0
Kamloop Island: 0
Summer Island: 0
RM 710
Marcus: 1
Marcus Nancy Creek: 0
Marcus Pingston Creek: 0
Sampling Area 3
Marcus East of St. Paul's Mission: 0
Kettle Falls
Kettle Falls: 7
RM 700
Colville River (2 Km upstream): 4
Haag Cove: 0
Hallam Creek: 0
Sherman Creek
Mill Creek
South Fork Deep Creek
Middle Fork Mill Creek
Curlew Creek
Toroda Creek
Cape Labelle Creek
Lost Creek
West Fork Sanpoil River
French Rocks: 15
RM 690
Colville River
Little Pend Oreille River
Quillisacut: 0
Hall Creek
RM 680
Daisy: 13
Gifford
Cloverleaf: 10
Sanpoil River
Nespelem River
Colville Indian Reservation
Stranger Creek
Sampling Area 4
Gifford: 17
RM 670
Little Nespelem River
Wilmont Creek
Ninemile Creek
RM 660
Hunter's: 27
Rebecca Lake Reference Sampling Area
Sanpoil River Reference Sampling Area
Middle fork Chamokane Creek
Sheep Creek
RM 650
Camp Na-Bor-Lee: 0
Enterprise: 2
Crescent Bay: 73
Grand Coulee Dam
RM 600
Plum Point: 35
Sampling Area 6
Keller Ferry: 56
RM 620
Spring Canyon: 31
Jones Bay: 9
Crescent Lake: 40
RM 610
Sampling Area 5
Crystal Cove: 1
Detillion: 0
Spokane Indian Reservation
Fort Spokane Campground: 1
RM 640
Porcupine Bay: 0
Spokane River Mouth: 2
Sterling Point: 0
Seven Bays
RM 630
Hawk Creek: 1
Lincoln: 1
Spokane River
Corbett Draw
Wilson Creek
Sinking Creek
Arbuckle Draw
Hawk Creek
20
21
25
155
174
232
292
395
2
Legend
Crayfish Trap Location
Survey Location
Daisy: 13
No. of Crayfish
Proposed Crayfish & Mussel Sampling Areas
Sampling Area
Reference Sampling Area
2009 Targeted Fish Sampling Area
Grand Coulee Dam
Populated Places
US Highway
State Highway
Local Road
County Lines
Reservations
River Mile (USGS)
Km
0 5 10
Miles
0 4 8
N
Windward environmental LLC
Map A3. USFWS Crayfish Survey Locations
Upper Columbia River, WA

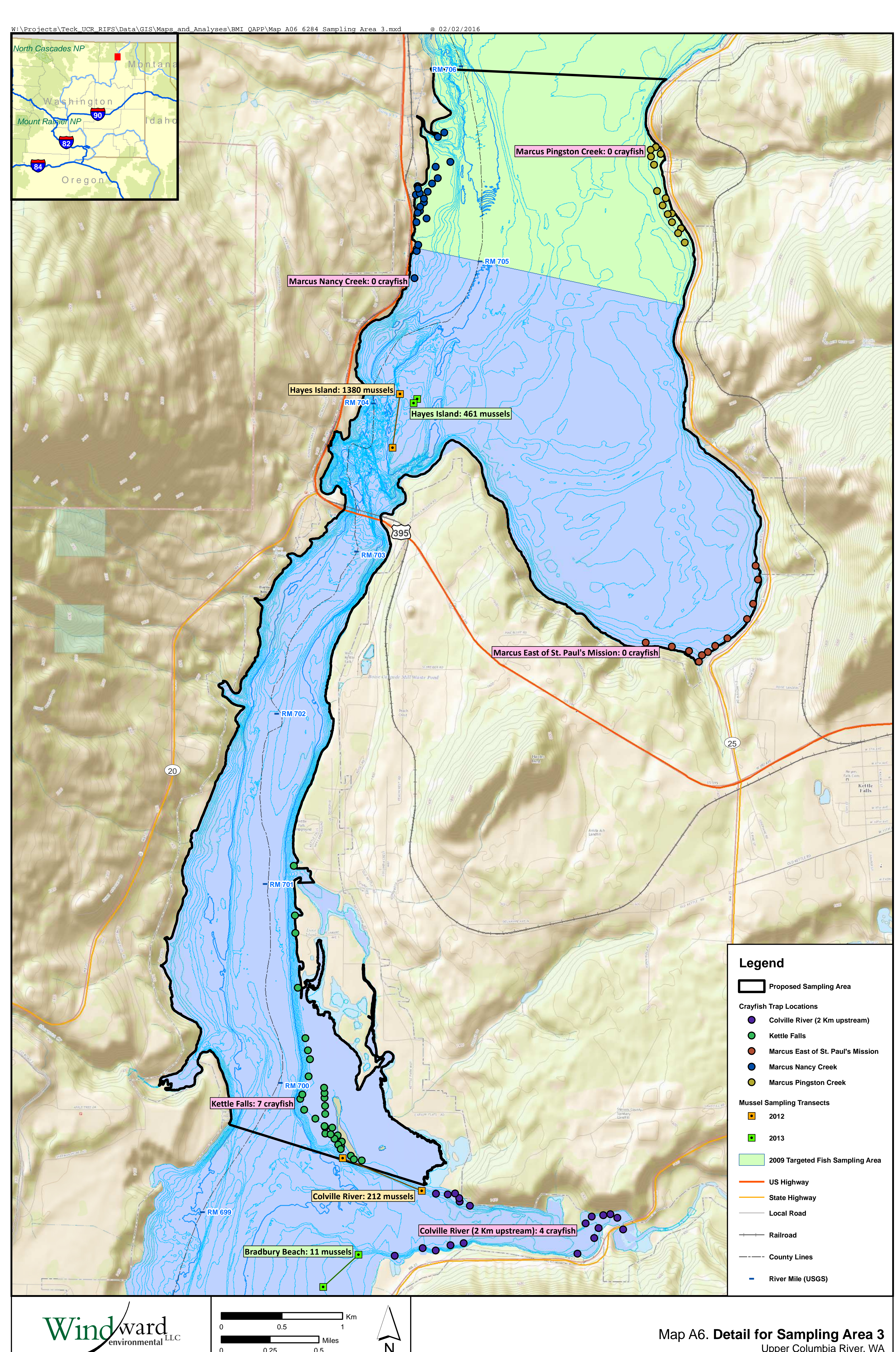
W:\Projects\Teck_UCR_RIFS\Data\GIS\Maps_and_Analyses\BMI_QAPP\Map_A04_6285_Sampling_Area_1.mxd © 02/02/2016



Map A4. **Detail for Sampling Area 1**
Upper Columbia River, WA

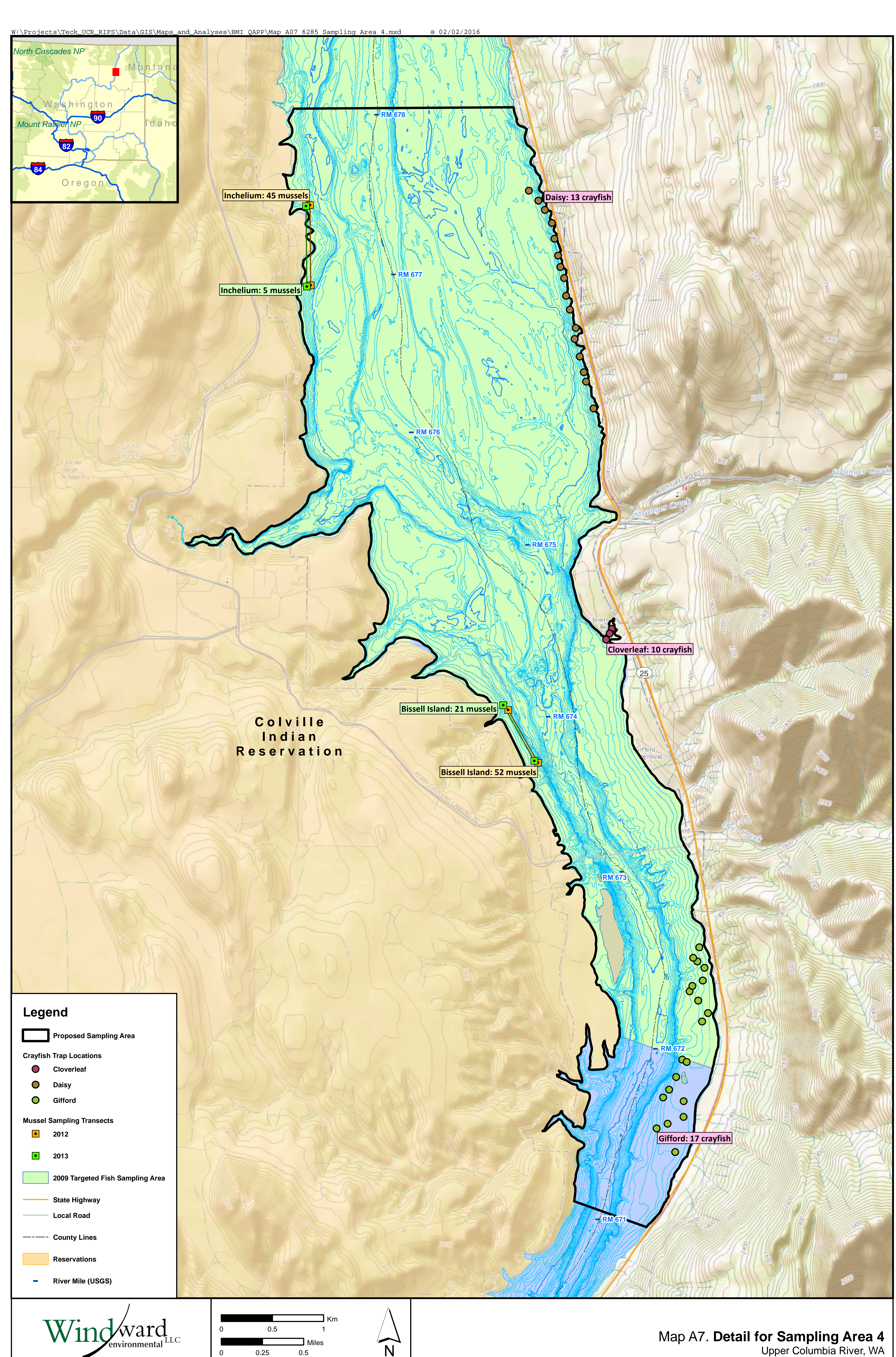


W:\Projects\Teck_UCR_RIFS\Data\GIS\Maps_and_Analyses\BMI_QAPP\Map_A05_6284_Sampling_Area_2.mxd ◎ 02/02/2016
North Cascades NP
Montana
Washington
Mount Rainier NP
Idaho
Oregon
90
82
84
RM 721
RM 722
RM 723
RM 724
RM 720
RM 719
RM 718
China Bar: 0 crayfish
China Bar: 24 mussels
China Bend boat launch: 94 mussels
North Gorge: 2 crayfish
North Gorge: 257 mussels
North Gorge: 9 mussels
Legend
Proposed Sampling Area
Crayfish Trap Locations
China Bar
North Gorge
Mussel Sampling Transects
2012
2013
2009 Targeted Fish Sampling Area
State Highway
Railroad
River Mile (USGS)
Windward environmental LLC
Km
0 0.25 0.5
Miles
0 0.25 0.5
N
Map A5. Detail for Sampling Area 2
Upper Columbia River, WA

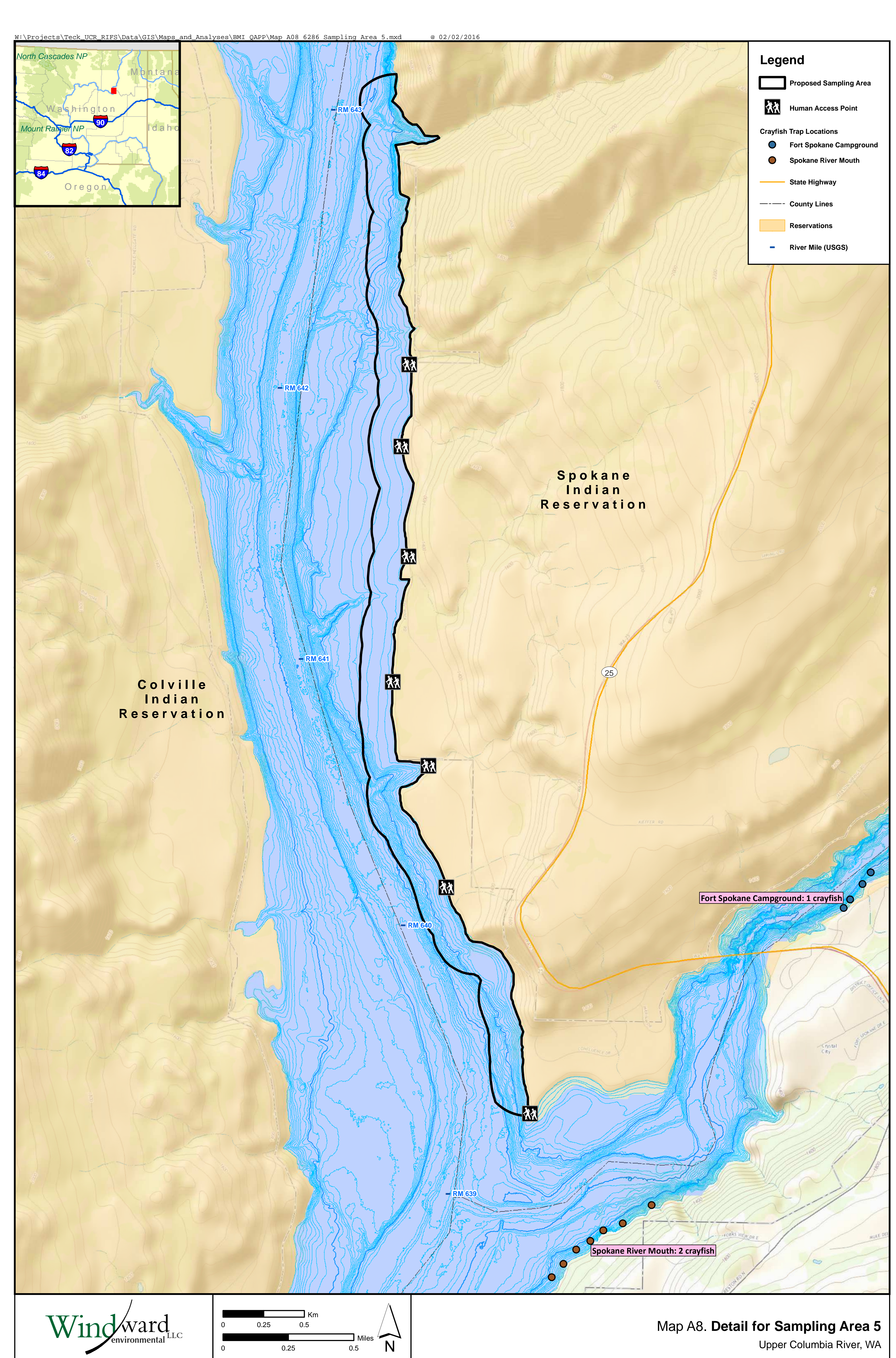


Map A6. **Detail for Sampling Area 3**
Upper Columbia River, WA

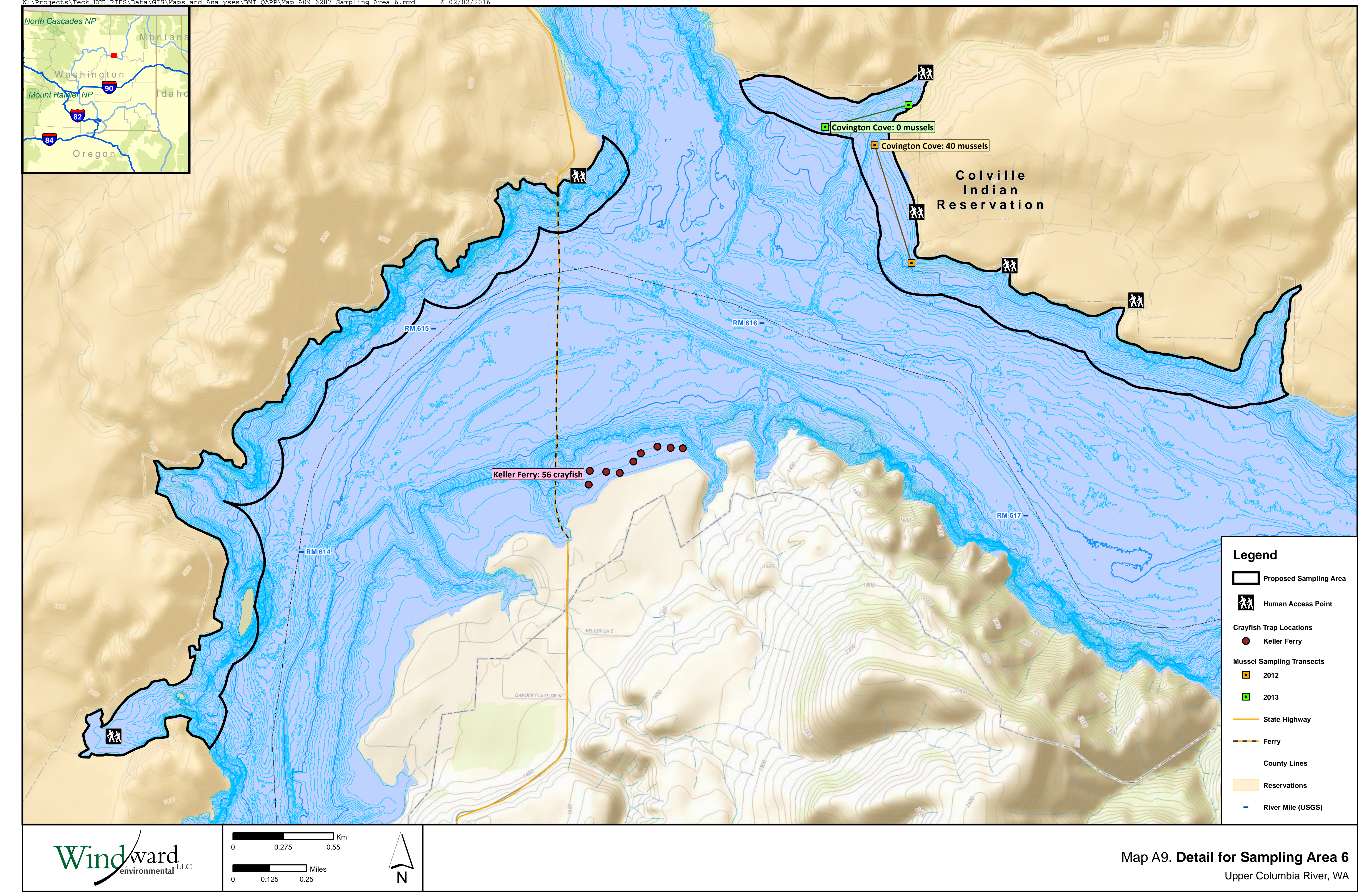
W:\Projects\Teck_UCR_RIFS\Data\GIS\Maps_and_Analyses\BMI_QAPP\Map_A07_6285_Sampling_Area_4.mxd © 02/02/2016



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

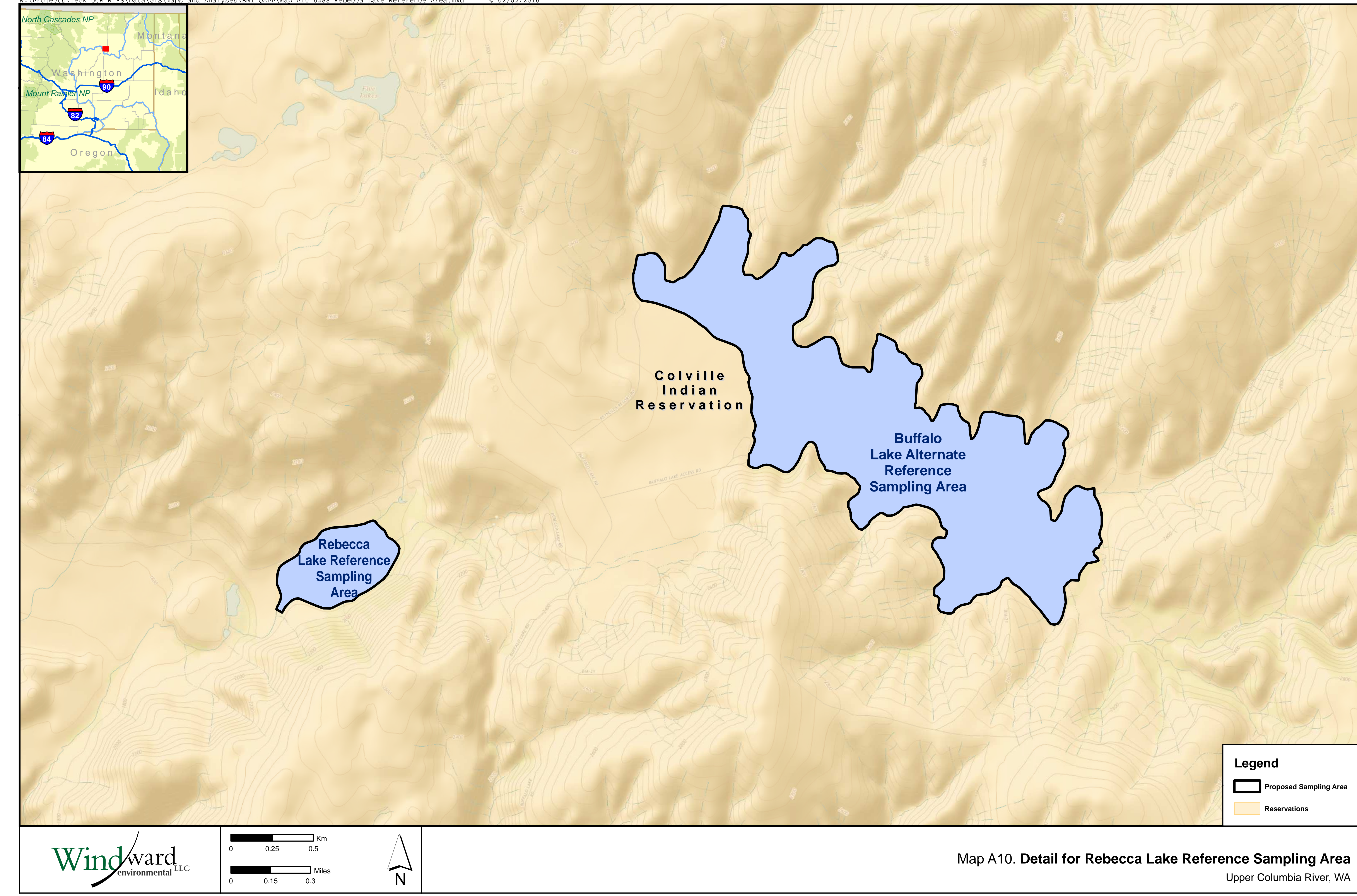

W:\Projects\Teck_UCR_RIFS\Data\GIS\Maps_and_Analyses\BMI_QAPP\Map_A08_6286_Sampling_Area_5.mxd @ 02/02/2016
Legend
Proposed Sampling Area
Human Access Point
Crayfish Trap Locations
Fort Spokane Campground
Spokane River Mouth
State Highway
County Lines
Reservations
River Mile (USGS)
North Cascades NP
Montana
Washington
Mount Rainier NP
Idaho
Oregon
90
82
84
RM 643
RM 642
RM 641
RM 640
RM 639
Spokane Indian Reservation
Colville Indian Reservation
25
Fort Spokane Campground: 1 crayfish
Spokane River Mouth: 2 crayfish
Windward environmental LLC
Km
0 0.25 0.5
Miles
0 0.25 0.5
N
Map A8. Detail for Sampling Area 5
Upper Columbia River, WA

W:\Projects\Teck_UCR_RIFS\Data\GIS\Maps_and_Analyses\BMI_QAPP\Map_A09_6287_Sampling_Area_6.mxd © 02/02/2016



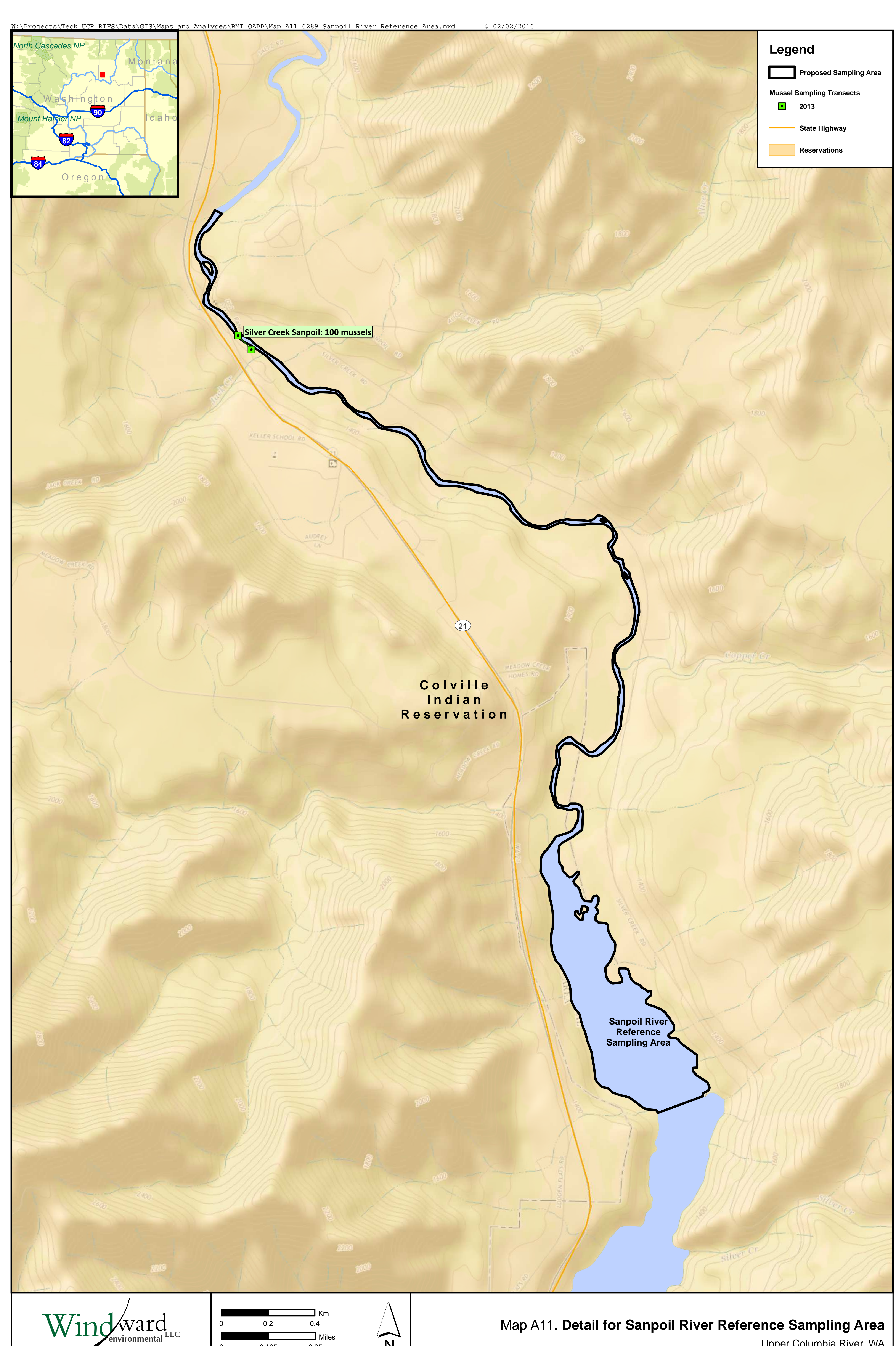
Map A9. **Detail for Sampling Area 6**
Upper Columbia River, WA

W:\Projects\Teck_UCR_RIFS\Data\GIS\Maps_and_Analyses\BMI_QAPP\Map A10 6288 Rebecca Lake Reference Area.mxd © 02/02/2016



Map A10. **Detail for Rebecca Lake Reference Sampling Area**

Upper Columbia River, WA



Map A11. **Detail for Sanpoil River Reference Sampling Area**

TABLES

Table A1. Sampling Areas

Sampling Area	Abbreviation	Location Type	HHRA	BERA	Approximate River Miles	Description of Location	Rationale for Sampling Area
Area 1	A1	Site		X	733 to 739	Near Northport	Location provides spatial coverage of the UCR for the BERA; overlaps with targeted fish sampling area for Reach 1 in 2009. In the USFWS survey, no crayfish were collected at survey locations in this area. Mussels were reported at some of the USFWS survey locations; with the exception of four mussels at one location, all were reported to be dead.
Area 2	A2	Site	X	X	718 to 725	Stretch of UCR from China Bend boat launch to North Gorge	Location provides spatial coverage of the UCR for the BERA; overlaps with targeted fish sampling area for Reach 2 in 2009. During the USFWS survey, only two crayfish were collected from the survey locations in this area. Mussels were collected from all survey locations in this area; dead mussels were reported at all locations other than the China Bend boat launch, where approximately half of the mussels were reported to be living.
Area 3	A3	Site		X	700 to 706	Near Kettle Falls	Location provides spatial coverage of the UCR for the BERA; overlaps with targeted fish sampling area for Reach 3 in 2009. During the USFWS survey, Crayfish were successfully collected at some locations; the most successful of which was the Kettle Falls location. The highest number of mussels in the entire UCR was reported at the Hayes Island location, which is only exposed during more extreme draw-down years. All mussels reported in survey locations from Reach 3 were reported to be dead.
Area 4	A4	Site		X	671 to 678	Stretch of UCR from Daisy to Gifford	Location provides spatial coverage of the UCR for the BERA; overlaps with targeted fish sampling area for Reach 4 in 2009. During the USFWS survey, crayfish were successfully collected in traps at the Daisy, Cloverdale, and Gifford sampling locations. Mussels were collected at most of the survey locations in this area; all were reported to be dead.
Area 5	A5	Site	X	X	639 to 643	Upstream of the mouth of the Spokane River (east side of river only)	Location reported as a possible source of mussels and crayfish by Spokane Tribe. Provides spatial coverage of the UCR for the BERA. Located just upstream of targeted fish sampling area for Reach 5 in 2009. Extent of the area is based on potential human beach access locations north of the mouth of the Spokane River.
Area 6	A6	Site	X	X	613 to 618	Near mouth of the Sanpoil River (north side of river only)	Location reported as a source of crayfish in the tribal survey (Westat 2012). Provides spatial coverage of UCR for the BERA. Located just upstream of Reach 6 fish sampling area. Extent of the area is based on potential human beach access locations near the mouth of the Sanpoil River.
Rebecca Lake	RL	Reference	X	X	NA	Lake located north of the Grand Coulee Dam	Location noted to be a popular crayfish collection location in the tribal survey (Westat 2012); some individuals also reported collecting mussels from this location.
Sanpoil River	SR	Reference	X	X	NA	Near the Sanpoil Campground and mouth of Silver Creek.	Location noted to be a popular mussel collection location in the tribal survey (Westat 2012); the Sanpoil River was also noted as a popular crayfish collection area.

Notes:

If sample collection at Rebecca Lake is not successful, nearby Buffalo Lake may also be used as a reference location.

BERA - baseline ecological risk assessment

HHRA - human health risk assessment

USFWS - U.S. Fish and Wildlife Service

NA - not applicable

Table A2. Methods and Sample Mass Requirements and Prioritization

	Sample Preparation		Quantitative Analysis			Target Sample Mass[b] (g)		Analysis Priority	
Analyte	Protocol	Procedure	Protocol	Procedure	Holding Time[a]	BERA[c]	BERA and HHRA[d]	BERA[c]	BERA and HHRA[d]
TAL metals[e]	ALS SOP MET-TDIG (PSEP)	Acid digestion	EPA 6010C, EPA 6020A[f]	ICP-MS, ICP-AES	180 days at -20°C	2	2	1	1
Total mercury	ALS SOP MET 1631	Method (acid) digestion	EPA 1631E	CVAFS	1 year at -20°C	1.5	1.5	2	2
Methyl mercury	ALS SOP MET 1630T	Method (alcohol) digestion	EPA 1630M			NA	1.5	NA	3
Arsenic - inorganic	ALS SOP MET 1632	Method (acid) digestion	EPA 1632A	HG-QFAAS		NA	3	NA	4
PCB congeners	EPA 1668A	Method extraction	EPA 1668A	HRGC/HRMS		NA	10	NA	5
Dioxins/furans	EPA 1613B	Method extraction	EPA 1613B			NA	10	NA	6
Percent moisture	ALS SOP MET-TISP	Freeze-dry/ gravimetric	ALS SOP MET-TISP	Freeze-dry/ gravimetric		NA[g]	NA[g]	1	1
Percent lipids	EPA 1668A		EPA 1668A			NA	NA[h]	NA	5
Total Target Sample Mass[i] (g)						4.5[j]	30[k]		

Notes:

Crayfish and mussel samples will be wrapped in aluminum foil and double bagged in resealable plastic bags. All samples will be frozen and shipped overnight to the analytical laboratory. Upon receipt, the analytical laboratory will store the samples at -20°C until processing. Samples will be processed and extracted for analysis within the holding times listed.

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 typically uses a holding time of two years from the time of freeze-drying for total metals (per EPA comments dated March 11, 2016 on the February 2016 draft final version of this QAPP).

[a] Holding time based on applicable standard operating procedure (see Appendix C).

[b] Based on wet weight. The expected mass of a crayfish composite sample containing 5 organisms (each 3.5 to 4 in long) is 75 to 113 g ww (see Section 2.2.4).

[c] Crayfish and mussels collected at locations for baseline ecological risk assessment (BERA).

[d] Crayfish and mussels collected at locations for BERA and human health risk assessment (HHRA).

[e] Except mercury.

[f] Recommended method for analysis of calcium, iron, magnesium, potassium, and sodium is EPA 6010C. Recommended method for analysis of aluminum, antimony, arsenic, barium, beryllium, cadmium, chromium, cobalt, copper, lead, manganese, nickel, selenium, silver, thallium, vanadium, and zinc is EPA 6020A. Either EPA 6010C or 6020A may be used for TAL metals (except mercury) as long as the method reporting limits listed in Table A7-4 in the main text are achieved for non-detects.

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

[h] Percent lipids will be analyzed with PCB congeners; no additional sample mass required.

[i] The target sample size listed achieves the reporting limits in Table A7-4 in the main text and the lowest quantitation limits in Table A7-5 in the main text.

[j] Includes an additional 1 g to account for possible sample loss during sample processing.

[k] Includes an additional 2 g to account for possible sample loss during sample processing.

ALS - ALS Environmental
CVAFS - cold vapor atomic fluorescence spectrometry
HG-QFAAS - hydride generation - quartz furnace atomic adsorption spectrometry
HRGC/HRMS - high resolution gas chromatography/high resolution mass spectrometry
ICP-AES - inductively-coupled plasma - atomic emission spectrometry
ICP-MS - inductively-coupled plasma - mass spectrometry
NA - not applicable
PCB - polychlorinated biphenyl
PSEP - Puget Sound Estuary Program
SOP - standard operating procedure
TAL - target analyte list

Table A3. Target Number of Samples to be Collected

Sampling Area	Location Type	HHRA[a]	BERA[b]	Target Number of Composite Samples: Mussels: Soft Tissue	Crayfish: Whole Body Minus Stomach and Carapace	Crayfish: Stomach and Carapace Only	Crayfish: Whole Body (All Parts)	Total
Area 1	Site		X	6[c]			6[c]	12
Area 2	Site	X	X	6	6	6	calculated[d]	18
Area 3	Site		X	6[c]			6[c]	12
Area 4	Site		X	6[c]			6[c]	12
Area 5	Site	X	X	6	6	6	calculated[d]	18
Area 6	Site	X	X	6	6	6	calculated[d]	18
Rebecca Lake	Reference	X		6	6			12
Sanpoil River	Reference	X		6	6			12
Total				**48**	**30**	**18**	**18**	**114**
			EPA Splits [e]	***7***	***5***	***3***	***3***	***18***
			Field Splits [e]	***2***	***2***	***1***	***1***	***6***
			Field Replicates [e]	***10***	***6***	***4***	***4***	***24***

Notes:

For each composite, a target of five mussels and five crayfish was developed. Thus a target of 30 individuals each of mussels and crayfish will be collected from each sampling area.

[a] For crayfish, the HHRA will only inlcude samples for whole body minus stomach and carapace.

[b] For crayfish, the BERA will include data representing whole body samples, which, if not analyzed as whole body samples, will be calculated as described in footnote d of this table.

[c] Sample types that will only be used in the BERA (i.e., all mussel and crayfish samples from Areas 1, 3, and 4) will be analyzed for TAL metals, total mercury, and percent moisture. Analysis of methylmercury, inorganic arsenic, PCB congeners, dioxins/furans, and percent lipids will not be conducted for these samples because these analytes are only of concern for the HHRA.

[d] Whole body tissue concentrations will be calculated based on a weighted sum of whole body minus stomach and carapace, and stomach and carapace only samples. Crayfish samples for whole body minus stomach/carapace and stomach/carapace only samples will be comprised of the same individuals.

[e] EPA splits, field splits, and field replicates are targeted for analysis on a subset of 15%, 5%, and 20% of the total number of samples, respectively. Each sample analyzed as a split or replicate will need twice the targeted sample mass indicated in Table A2.

BERA - baseline ecological risk assessment

HHRA - human health risk assessment

ATTACHMENT A1

GENERAL SITE HEALTH AND SAFETY PLAN

ADDENDUM

MACROINVERTEBRATE TISSUE STUDY

CONTENTS

LIST OF TABLES A1-iii
ACRONYMS AND ABBREVIATIONS A1-iv
SITE HEALTH AND SAFETY PLAN ADDENDUM APPROVAL A1-v
SITE HEALTH AND SAFETY PLAN ADDENDUM ACKNOWLEDGEMENT A1-vi

1 INTRODUCTION A1-1-1

1.1 ORGANIZATION A1-1-2
1.2 SCOPE OF WORK A1-1-2
1.3 DEFINITIONS A1-1-2

2 SAFETY GUIDELINES FOR PHYSICAL HAZARDS A1-2-1

3 CHEMICAL HAZARD EVALUATION A1-3-1

4 PERSONAL PROTECTIVE EQUIPMENT AND SAFETY EQUIPMENT A1-4-1

4.1 PERSONAL PROTECTIVE EQUIPMENT A1-4-1
4.2 SAFETY EQUIPMENT A1-4-1

5 AIR MONITORING A1-5-1

6 EMERGENCY PLANNING A1-6-1

7 WORK ZONES A1-7-1

8 DECONTAMINATION A1-8-1

9 VEHICLE SAFETY, SPILL CONTAINMENT, AND SHIPPING INSTRUCTIONS A1-9-1

10 TASK-SPECIFIC SAFETY PROCEDURES A1-10-1

11 REFERENCE A1-11-1

Attachment A1-1. Site Map and Hospital Location Maps

Attachment A1-2. Cold-Stress Fact Sheet

Attachment A1-3. Heat-Related Illness Prevention Policy

LIST OF TABLES

Table 2-1. Summary of Activities and Potential Hazards

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

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

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

Table 5-1. Site-specific Air Monitoring Requirements

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

Table 6-1. Local Emergency Telephone Numbers

Table 6-2. Corporate Emergency Telephone Numbers

Table 6-3. Project Area Hospital Information

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
Windward	Windward Environmental, LLC
WISHA	Washington Industrial Safety and Health Act

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 (Windward Environmental, LLC [Windward]) for the Macroinvertebrate Tissue Study at the Upper Columbia River (UCR) site (Site) in support of the remedial investigation and feasibility study (RI/FS) for the Site.

____

Windward Task Manager Date

____

Windward Corporate 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 tissue 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 Windward 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
Employee signature	Company	Date
Employee signature	Company	Date
Employee signature	Company	Date
Employee signature	Company	Date
Employee signature	Company	Date
Employee signature	Company	Date

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 macroinvertebrate 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 Windward'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.

Windward 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 Windward'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 macroinvertebrate sampling are presented in this Addendum to the general SHSP. In addition, this Addendum provides detailed field site 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

Macroinvertebrate samples will be collected at locations in six sampling areas along the UCR and in two reference areas. It is anticipated that sampling will be carried out by one boat-based 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

All work will be done using the buddy system. Depending upon the time of year and the location of work, biting insects may be an issue when accessing any of the sampling locations during the sampling event. 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 sampling	Water hazards, slippery walking surfaces, cold/hypothermia (depending on sampling event), heat stress (depending on sampling event), material handling, adverse weather, work in remote areas

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

Potential Hazard	Yes	No	Proposed Safety Procedure
Slippery surfaces	X		Use caution; wear properly fitting shoes or boots with good gripping capacity; 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	Wear personal flotation devices (PFDs) at all times when working over water. Inspect the PFDs prior to use and do not use defective PFDs. Keep sampling equipment on boats organized at all times. Boats are required to be equipped with a throwable life ring, fire extinguisher, and warning horn, and all field members will be briefed on their storage locations.
Work in remote areas	X		Use the buddy system; carry radio and/or cellular telephone; bring sufficient equipment in case of accident or injury (first aid kit, shelter if appropriate)
Biting insects	X		Use repellents, as needed

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 tissue 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 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 Protection 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 addition of 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 onsite 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 (boat)
	Stop watch for monitoring heart rate		Windsock
	Thermoscan® thermometer (or equivalent) for heat stress monitoring	X	Cellular phone
			Radio sets
X	Survival kit	X	Global positioning system
X	Personal flotation device	X	Other Satellite telephone
	Cool vests		________

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 or facility 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] Facility emergency medical provider
- [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
Alternative Hospitals	Coulee Community Hospital, Grand Coulee, WA	(509) 633-1753	No
	Ferry County Memorial Hospital, Republic, WA	(509) 775-3333	No
	Lincoln Hospital, Davenport, WA	(509) 725-7101	No
	St Joseph's Hospital, Cheweleh, WA	(509) 935-8211	No
	Deer Park Hospital, Deer Park, WA	(509) 276-5061	No
	Deaconess Medical Center-Spokane, Spokane, WA	(509) 473-7178	No
	Holy Family Hospital, Spokane, WA	(509) 482-0111	No
	Sacred Heart Medical Center, Spokane, WA	(509) 474-3131	No
	Veterans Affairs Medical Center, Spokane, WA	(509) 434-7032	No

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

Local Resources	Name	Telephone	Notified Prior to Work (Yes/No)?
Site telephone	Field cellular 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.	TBD	Not applicable
Directions to Mount Carmel Hospital (from Highway 395)	Begin traveling SE 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)		

In case of serious injuries, death, or other emergency, the TAI and Windward task managers 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 Task Manager	Kris McCaig	Work: (509) 623-4501 Cellular: (509) 434-8542
Windward Task Manager	Berit Bergquist	Work: (206) 812-5403 Cellular: (206) 293-2632

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
Coulee Community Hospital	24 hours	509-633-1753	411 Fortuyn Road	Grand Coulee
Ferry County Memorial Hospital	24 hours	509-775-3333	36 Klondike Road	Republic
Lincoln Hospital	24 hours	509-725-7101	10 Nichols Street	Davenport
St Joseph's Hospital	24 hours	509-935-8211	500 East Webster Street	Chewelah

Table 6-3. Project Area Hospital Information (continued)

Facility Name	Open for Emergency Services	Telephone Number	Address	City
Mount Carmel Hospital	24 hours	509-684-2561	982 East Columbia Street	Colville
Deer Park Hospital	24 hours	509-276-5061	East 1015 'D' Street	Deer Park
Deaconess Medical Center-Spokane	24 hours	509-473-7178	West Fifth Avenue	Spokane
Holy Family Hospital	Dependent on case	509-482-0111	North 5633 Lidgerwood Avenue	Spokane
Sacred Heart Medical Center	24 hours	509-474-3131	West 101 Eighth Avenue	Spokane
Veterans Affairs Medical Center	7:30 am to 4:00 pm	509-434-7032	North 4815 Assembly Street	Spokane

If any health or safety issue arises, after the victim(s) receive appropriate medical treatment, the relevant field crew member(s) 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 A1) 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. Because the majority of sampling will be on the water, and in remote locations, no designation (e.g., traffic cones or caution tape) will be utilized.

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 tissue prior to the commencement of sampling at each location and upon completion of the study. This will include equipment such as measuring boards and scales. 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 tissue sampling event, as well as unexpected contact with the sampling equipment. Always move about the shore or upland area with caution. 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 with status updates at least every 4 hours while performing field collection activities.

When field personnel are working onboard a boat or near or over water, a PFD should be worn at all times. PFDs should be inspected daily prior to use and should not be used if defective. Information on boating safety is presented in the general SHSP (TCAI 2009, Section 9.2).

Some of the areas that will be sampled are accessible to the public. Field crew members should always be aware of their surroundings, and 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.

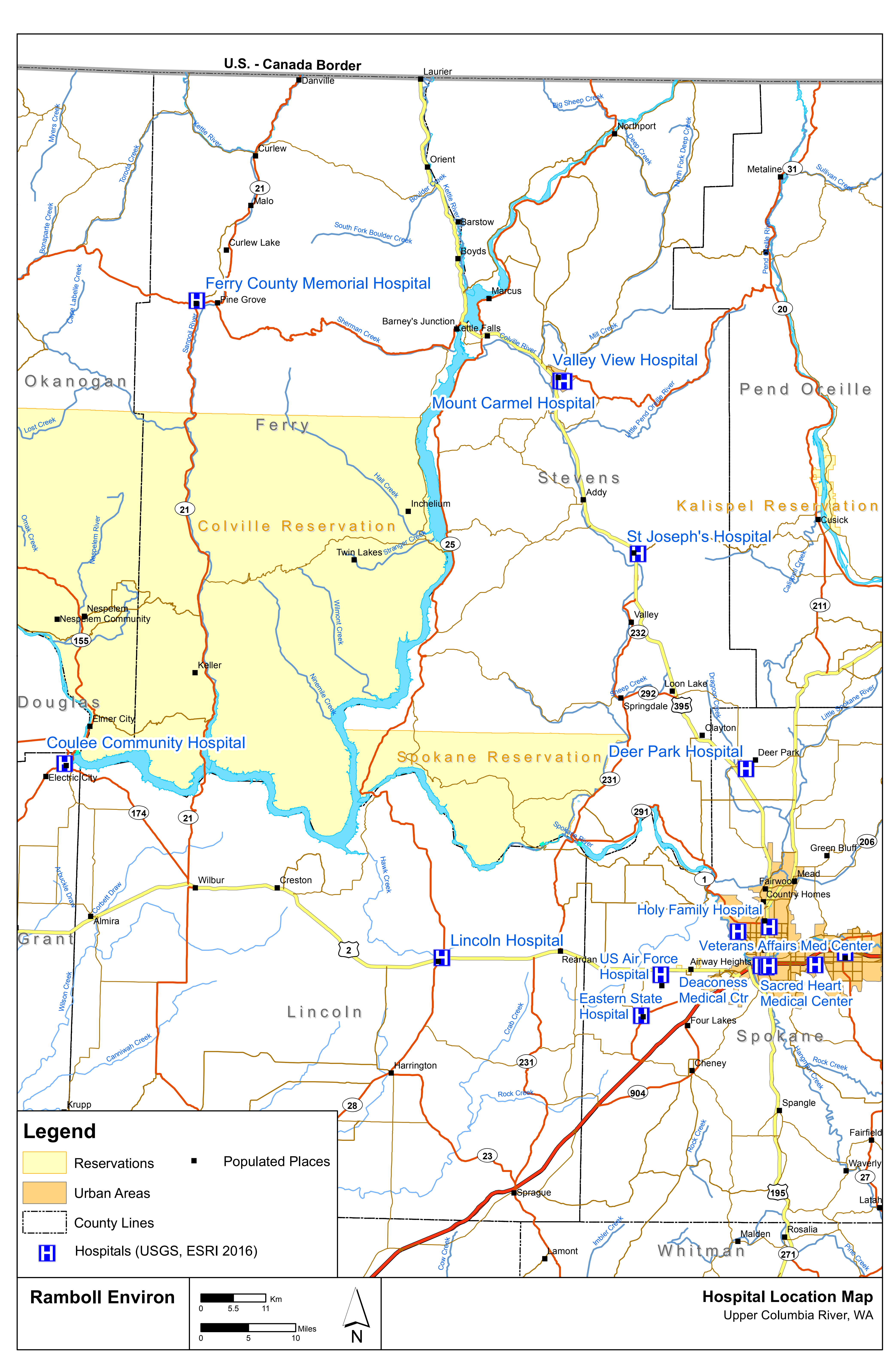
When handling sampling equipment or samples, nitrile gloves should always be worn, along with safety glasses or goggles when appropriate, or preservative chemicals (if required). Keep a 1-liter eye wash bottle accessible during all field work. Avoid getting preservatives on the skin or clothes. If any 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 eye wash 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, Bellevue, WA.

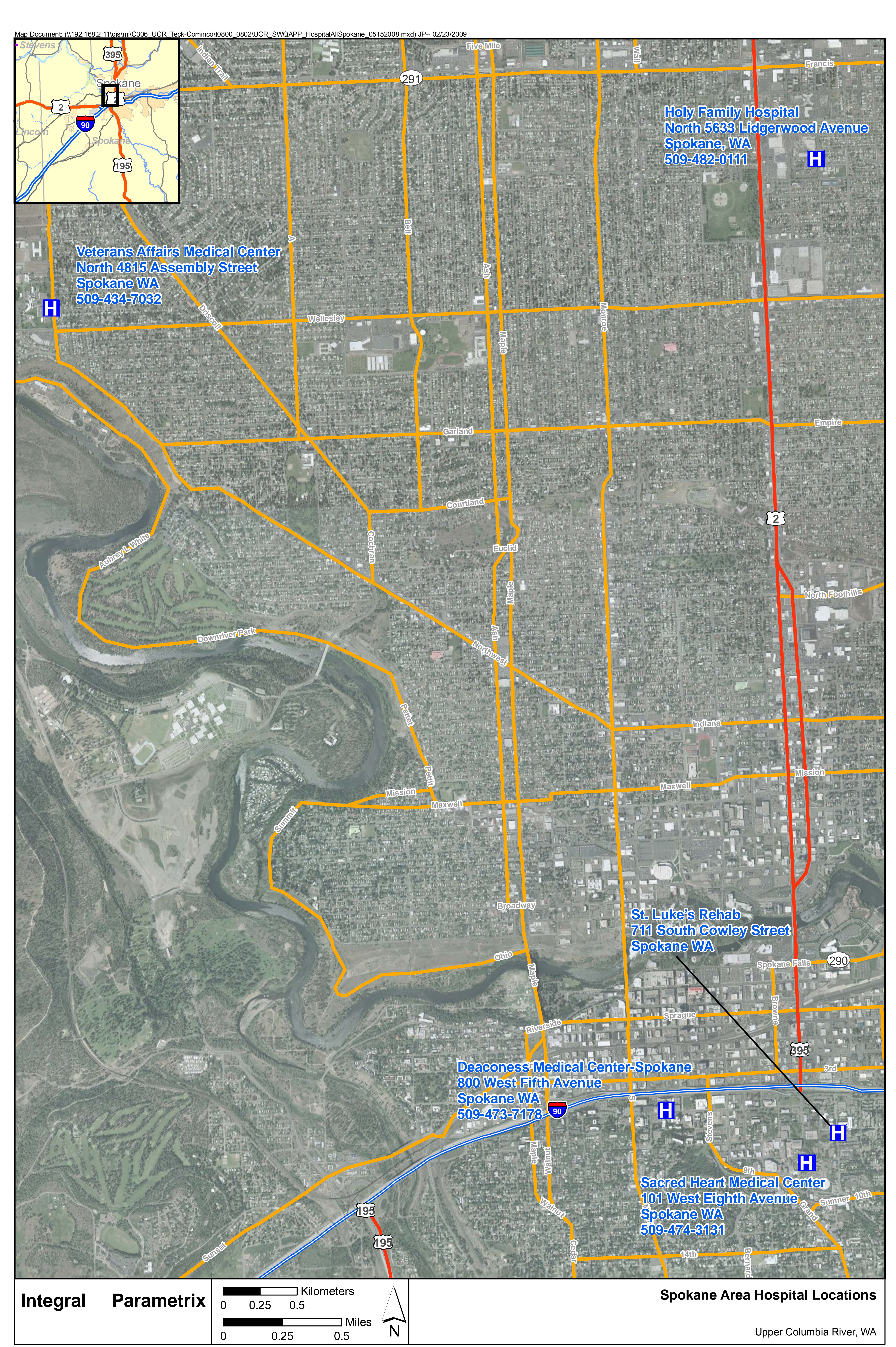
ATTACHMENT A1-1

SITE MAP AND HOSPITAL LOCATION MAPS



U.S. - Canada Border
Ferry County Memorial Hospital
Valley View Hospital
Mount Carmel Hospital
St Joseph's Hospital
Coulee Community Hospital
Deer Park Hospital
Holy Family Hospital
Lincoln Hospital
Veterans Affairs Med Center
US Air Force Hospital
Deaconess Medical Ctr
Sacred Heart Medical Center
Eastern State Hospital
Okanogan
Ferry
Stevens
Pend Oreille
Douglas
Grant
Lincoln
Spokane
Whitman
Colville Reservation
Spokane Reservation
Kalispel Reservation
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

Map Document: (\\192.168.2.11\gis\mi\C306_UCR_Teck-Cominco\t0800_0802\UCR_SWQAPP_HospitalAllSpokane_05152008.mxd) JP-- 02/23/2009



Integral Parametrix

Kilometers 0 0.25 0.5

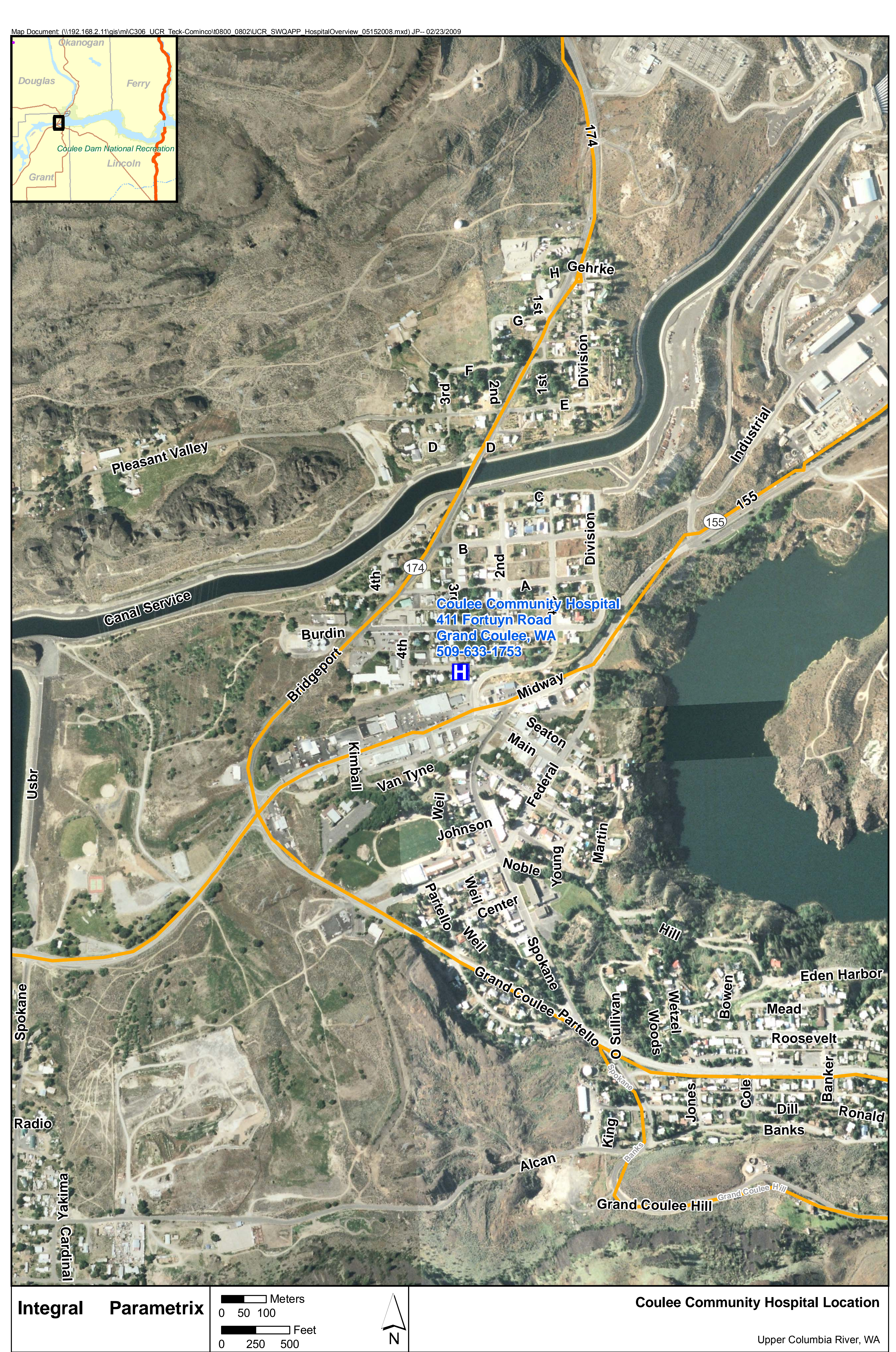
Miles 0 0.25 0.5

N

Spokane Area Hospital Locations

Upper Columbia River, WA

Map Document: (\\192.168.2.11\gis\ml\C306_UCR_Teck-Cominco\t0800_0802\UCR_SWQAPP_HospitalOverview_05152008.mxd) JP-- 02/23/2009



Integral Parametrix

Meters: 0 50 100

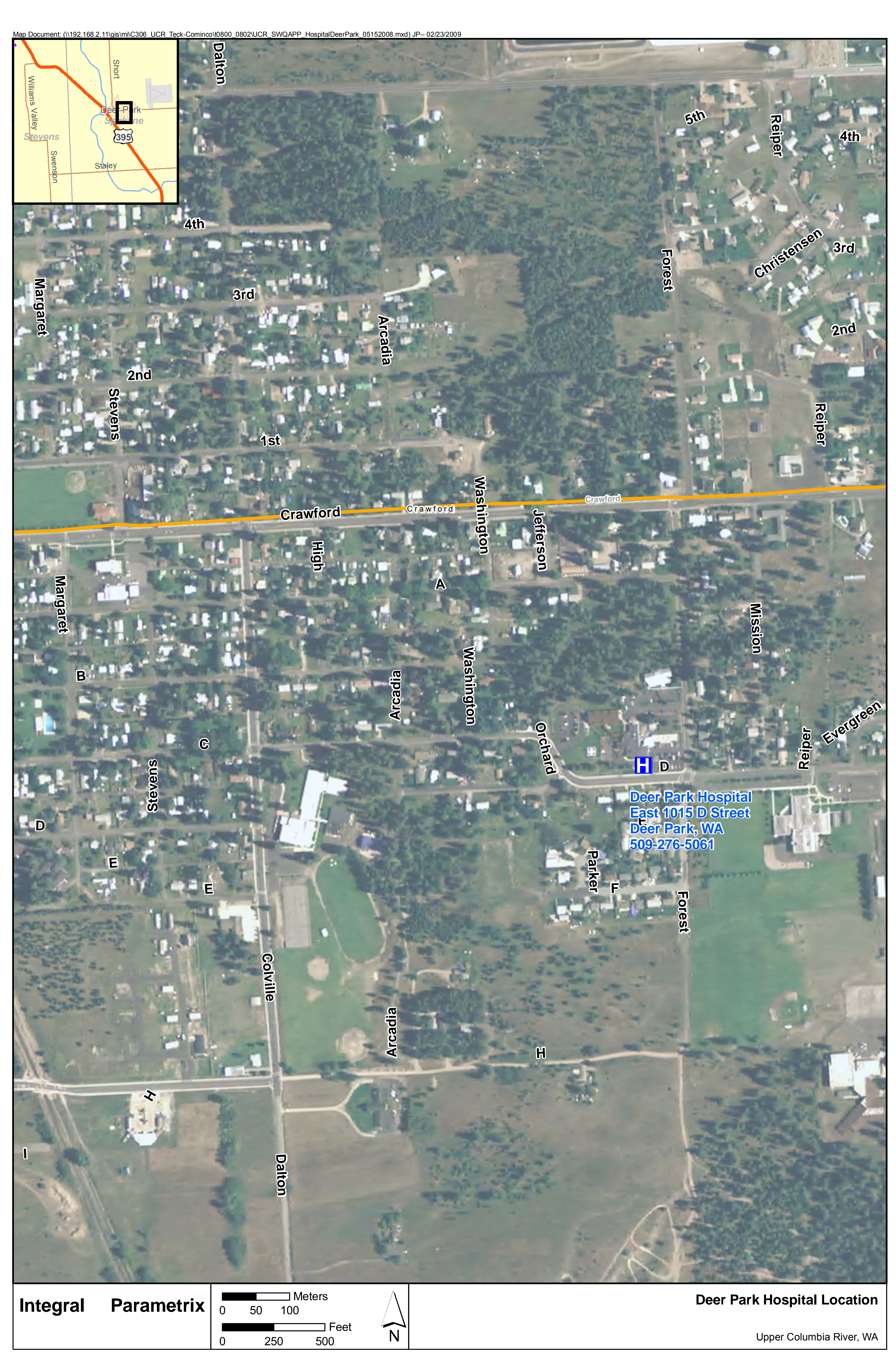
Feet: 0 250 500

N

Coulee Community Hospital Location

Upper Columbia River, WA

Map Document: (\\192.168.2.11\gis\mi\C306_UCR_Teck-Cominco\t0800_0802\UCR_SWQAPP_HospitalDeerPark_05152008.mxd) JP-- 02/23/2009



Integral Parametrix

Meters 0 50 100

Feet 0 250 500

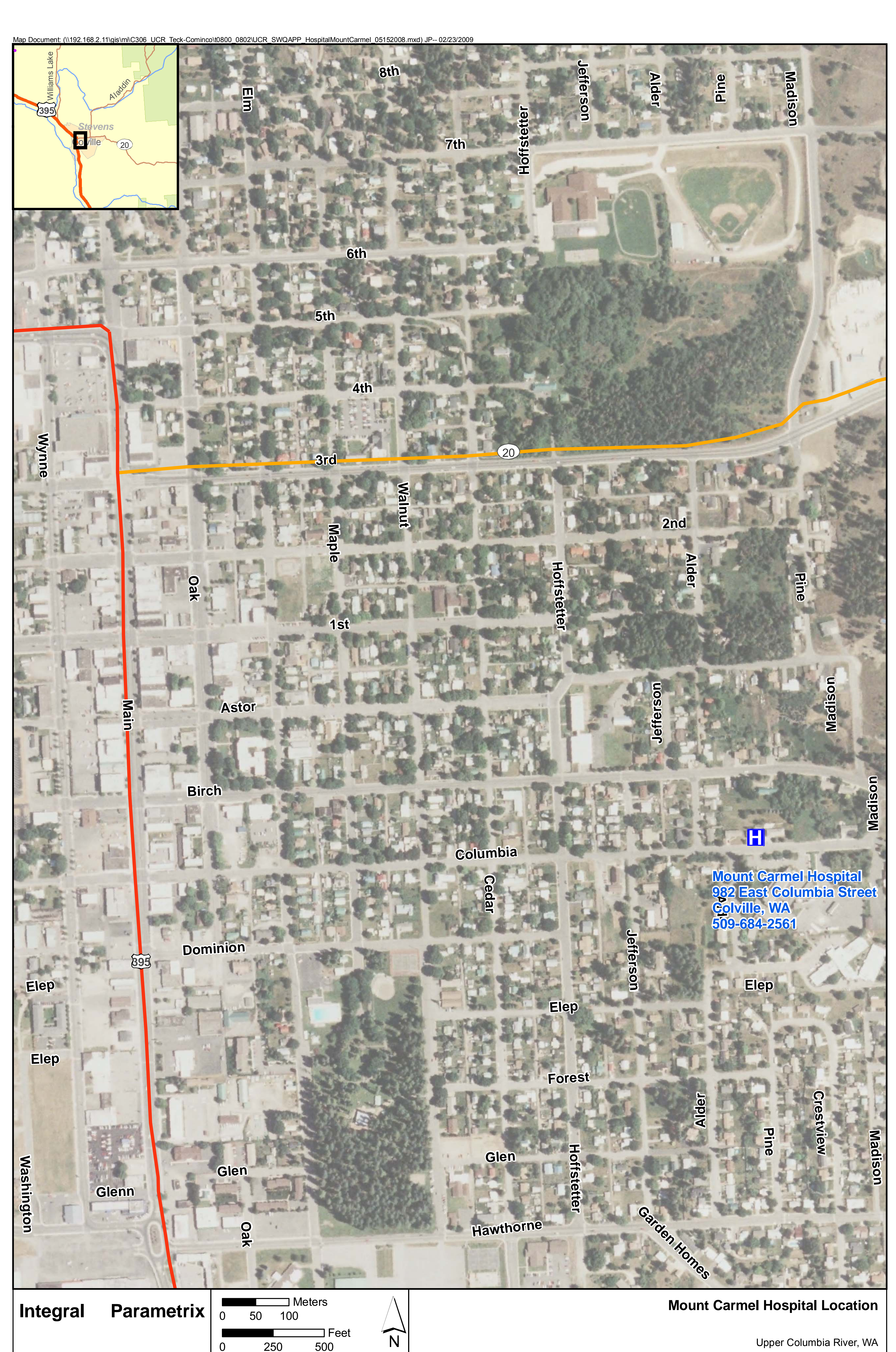
N

Deer Park Hospital Location

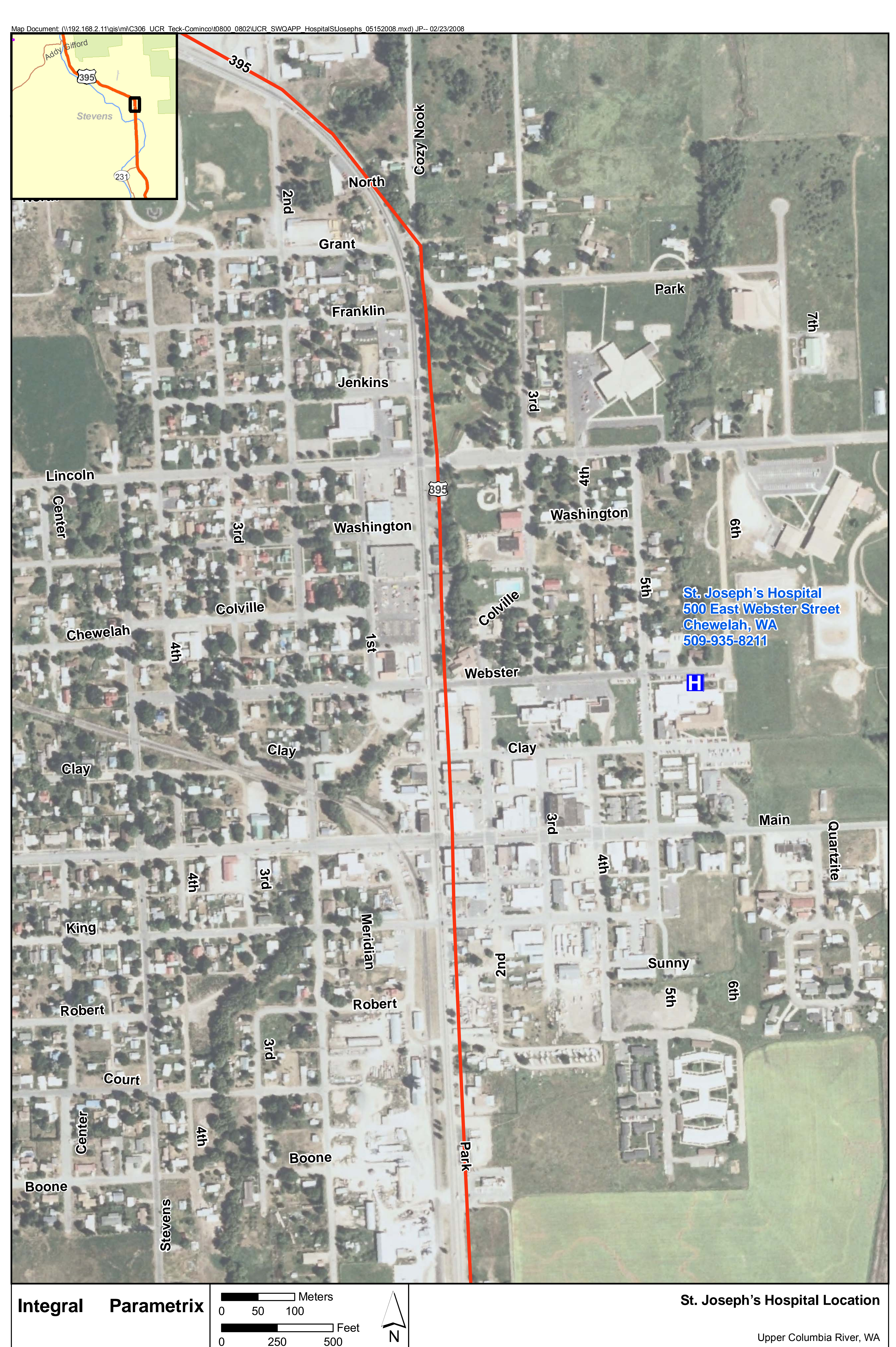
Upper Columbia River, WA

Map Document: (\\192.168.2.11\gis\mi\C306_UCR_Teck-Cominco\t0800_0802\UCR_SWQAPP_HospitalFerryCoMem_05152008.mxd) JP-- 02/23/2009
Ferry
21
20
Curlew Lake
Herron Creek
Ferry County Memorial Hospital
36 Klondike Road
Republic, WA
509-775-3333
H
Willow
21
Fish Hatchery
Torboy
21
Integral
Parametrix
Meters
0
50
100
Feet
0
250
500
N
Ferry County Memorial Hospital Location
Upper Columbia River, WA

Map Document: (\\192.168.2.11\gis\mi\C306_UCR_Teck-Cominco\t0800_0802\UCR_SWQAPP_HospitalLincoln_05152008.mxd) JP-- 02/23/2009
Lincoln Hospital
10 Nichols Street
Davenport, WA
509-725-7101
H
Mcinnis
Lincoln
Ross
Sinclair
Logan
Morgan
Park
Spring
Merriam
Marshall
Maxwell
Main
Washington
Adams
Sunset
8th
7th
6th
5th
4th
Nichols
Harker
3rd
2nd
1st
25
28
2
Integral
Parametrix
Meters
0 50 100
Feet
0 250 500
N
Lincoln Hospital Location
Upper Columbia River, WA



Map Document: (\\192.168.2.11\gis\mi\C306_UCR_Teck-Cominco\t0800_0802\UCR_SWQAPP_HospitalMountCarmel_05152008.mxd) JP-- 02/23/2009
Williams Lake
Aladdin
395
Stevens
Colville
20
8th
7th
6th
5th
4th
3rd
2nd
1st
Astor
Birch
Columbia
Dominion
Elep
Forest
Glen
Glenn
Hawthorne
Garden Homes
Elm
Jefferson
Alder
Pine
Madison
Hoffstetter
Wynne
Walnut
Maple
Oak
Main
Cedar
Crestview
Washington
Mount Carmel Hospital
982 East Columbia Street
Colville, WA
509-684-2561
Integral
Parametrix
Meters
0 50 100
Feet
0 250 500
N
Mount Carmel Hospital Location
Upper Columbia River, WA


Map Document: (\\192.168.2.11\gis\mi\C306_UCR_Teck-Cominco\t0800_0802\UCR_SWQAPP_HospitalStJosephs_05152008.mxd) JP-- 02/23/2008
Addy
Gifford
395
Stevens
231
395
North
Cozy Nook
2nd
Grant
Park
7th
Franklin
Jenkins
3rd
Lincoln
4th
Center
Washington
Washington
3rd
6th
5th
St. Joseph's Hospital
500 East Webster Street
Chewelah, WA
509-935-8211
Colville
Colville
Chewelah
1st
4th
Webster
H
Clay
Clay
Clay
Main
Quartzite
3rd
4th
3rd
4th
King
Meridian
2nd
Sunny
5th
6th
Robert
Robert
3rd
Court
Center
4th
Park
Boone
Boone
Stevens
Integral
Parametrix
Meters
0
50
100
Feet
0
250
500
N
St. Joseph's Hospital Location
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.

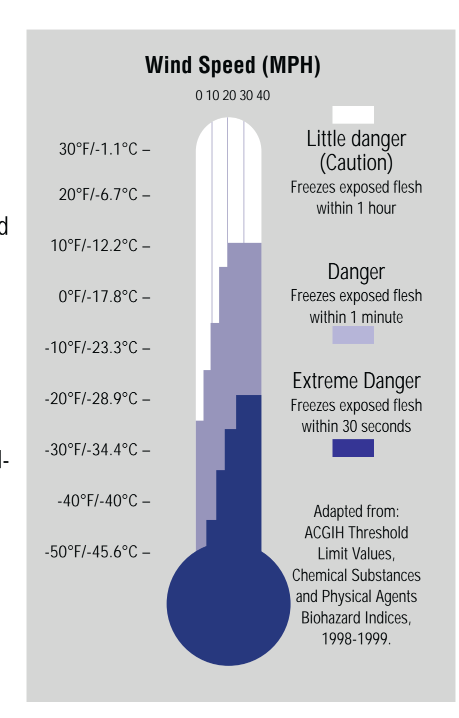
What to do: (water temperatures)

- 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**.

Relative Humidity	Temperature
70%	100°F / 37.8°C
60%	95°F / 35°C
50%	90°F / 32.2°C
40%	85°F / 29.4°C
30%	80°F / 26.7°C

= Danger
= Caution
= Less Hazardous

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

ATTACHMENT A2

MACROINVERTEBRATE TISSUE SAMPLING STANDARD OPERATING PROCEDURES

STANDARD OPERATING PROCEDURE SOP-1

RECORDING MACROINVERTEBRATE COLLECTION LOCATIONS

Scope and Applicability

This standard operating procedure (SOP) describes procedures used for recording mussel and crayfish tissue sampling stations across the Upper Columbia River (UCR) Site (hereafter the Site). 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)
- Boat-mounted GPS
- Spare batteries
- Charging unit.

A GPS hardware system, such as a Trimble GeoXH GPS (or equivalent device), should be used for recording sampling stations and re-visiting these locations as needed (e.g., to check crayfish traps). 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. If possible, GPS accuracy should be verified at the beginning or at the end of each sampling day.

Station Location Procedures

Sampling area boundaries and other applicable geographic information systems (GIS) data layers (e.g., aerial photographs, topography) will be uploaded into the hand-held GPS unit(s) prior to the sampling effort. A position will be recorded electronically at each location where mussels and crayfish are collected. Ancillary information will be recorded in the field logbook, and may include the personnel operating the GPS system, water depth of 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
- Wait for it to acquire the location of satellites
- Save the location into the GPS memory (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 in the field sampling report.

STANDARD OPERATING PROCEDURE SOP-2

SAMPLE LABELING

Scope and Applicability

This standard operating procedure (SOP) describes the general procedures for completing sample labels that will be used for macroinvertebrate tissue 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., field splits) 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), sample IDs for individual organisms, and sample IDs for composite samples.

Location ID

Mussel collection beaches or crayfish trap locations will be determined by the field crew during field sampling, and will each be assigned a unique identifier. These location IDs will consist of the following parts:

- Two-digit sampling area code—see Table A1 of the quality assurance project plan (QAPP) for sampling area codes (e.g., A1 = Sampling Area 1, RL = Rebecca Lake, and SR = Sanpoil River)
- Sample type code—MB for mussel beach and CT for crayfish trap
- Two-digit sequential number

Examples:

A4-CT03 = the third crayfish trap placed in Sampling Area 4

SR-MB01 = the first mussel beach in the Sanpoil River Reference Sampling Area.

These location IDs will be used to document sampling locations.

Sample IDs for Individual Organisms

Each individual organism will be assigned a unique identifier. The sample ID will include the location ID (as described above), the species code, and the individual number, as shown below.

- Two-digit sampling area code—see Table A1 for sampling area code (e.g., A1 = Sampling Area 1, RL = Rebecca Lake, and SR = Sanpoil River)
- Sample type code—MB for mussel beach and CT for crayfish trap
- Two-digit sequential number
- Species code—MU for mussels, CL for clam, and CR for crayfish
- Two-digit individual number—sequential number for each individual collected in a given sampling area (e.g., mussel #03 collected in Sampling Area 4). Note that this is important to be able to link analytical chemistry results.

Examples:

A4-MU-03 = Mussel individual #03 collected from Sampling Area 4

RL-CR-01 = Crayfish #01 collected from the Rebecca Lake Reference Sampling Area

Sample IDs for Composite Samples

Mussel and crayfish tissue composites will be prepared by the analytical laboratory upon receipt of samples; a compositing scheme (i.e., which individuals will be included in which composite) will be determined in consultation with EPA after sampling is completed. This compositing plan will document which individual organisms will be included in each composite sample. This information will also be documented by the analytical laboratory during processing. Unique sample identifiers will be assigned to these composites.

All mussel samples will be composited as soft tissue (i.e., the shell will be removed); samples will be designated as ST. Furthermore, as described in the FSP, crayfish analysis will be conducted based on 1) composites of whole-body samples for the baseline ecological risk assessment (BERA)-only samples (designated as WB), and 2) composites of both whole-body minus carapace/stomach (designated as PB) and carapace/stomach samples (designated as CS) for the BERA and human health risk assessment (HHRA) samples. It is essential that the same individuals are used in each composite so that whole body tissue concentrations can be mathematically calculated; therefore, there should be complementary tail meat and

remaining body results for each composite. The composite ID will be composed of the following elements:

- Two-digit sampling area code—see Table A1 for sampling area code (e.g., A1 = Sampling Area 1, RL = Rebecca Lake, and SR = Sanpoil River)
- Species code—MU for mussels, CL for clam, and CR for crayfish
- Composite number—a four-digit code starting with C, followed by a sequential three-digit number (e.g., C001)
- Tissue code—WB for whole body, PB for whole body minus carapace/stomach, CS for carapace/stomach, and ST for soft tissue.

Examples:

A6-MU-C001-ST = the first composite mussel soft tissue sample consisting of mussels from Sampling Area 6

A4-CR-C002-WB = the second composite whole-body tissue sample consisting of crayfish from Sampling Area 4

RL-CR-C006-CS = the sixth composite carapace and stomach tissue sample consisting of crayfish from the Rebecca Lake Reference Sampling Area.

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-3

MUSSEL TISSUE SAMPLE COLLECTION

Purpose

The purpose of this standard operating procedure (SOP) is to describe procedures for collecting mussels for the Upper Columbia River (UCR) benthic macroinvertebrate tissue study. The methods described here were in part derived from the quality assurance project plan (QAPP) for assessment of nearshore mussel tissue contamination for regional stormwater monitoring in Washington State, and the National Oceanic and Atmospheric Administration (NOAA) Mussel Watch Project (Lanksbury et al. 2010).

Scope and Applicability

Bivalves collected for this study will include the following species (when available):

- *Anodonta* clade 1 – winged/California floater
- *Anodonta* clade 2 – Oregon/western floater
- *Anodonta beringiana* – Yukon floater
- *Margaritifera falcata* – Western pearlshell
- *Gonidea angulata* – Western ridged mussel
- *Corbicular fluminea* – an invasive clam.

The mussels will be collected from the sample areas described in the field sampling plan (FSP). These sample areas include previously sampled Site locations, and were selected to cover a range of habitat types.

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 mussels
- Calipers (measuring to the nearest millimeter)
- Analytical balance (and calibrated taring weight)
- Resealable plastic bags and food-grade heavy duty aluminum foil
- Waterproof labels and tags
- Permanent marking pen
- Cooler and ice

- Nets for collecting mussels from wadeable waters
- Buckets (for holding mussels)
- Coolers for shipping mussels
- Chest or hip waders
- Personal protective equipment
- Protective gloves
- 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.

Sampling gear that has previously been used at other sites will be thoroughly inspected and cleaned before the field effort to prevent transport of aquatic invasive species (e.g., zebra mussels).

Procedures for Mussel Collection

Mussels (and/or clams) will be collected by handpicking on the beach or in wadeable waters using the process described below.

1. Select a sampling location (i.e., beach) in accordance with the FSP. Location selection will be based on best professional judgment and will use available information regarding previously sampled Site locations and areas where mussels are known to be abundant. Selection of sampling beaches in areas of high silt and fine sediments will increase the likelihood of locating mussel beds for floater species. In the upper portions the UCR, where rocky substrates (i.e., gravels, cobbles, and bounders) are more common, floater species have been found in this type of habitat. There rocky substrates are also the preferred habitat for the western pearlshell mussel.

2. Document sample location conditions, date, and time of arrival in the field logbook and take digital pictures of the area, as indicated in SOP-6 and SOP-9.

3. Ensure that a cultural resources monitor inspect and approve each sample location (i.e., beach) prior to beginning mussel collection. Any area(s) on the beach that do not pass the cultural resources review will be flagged and avoided during mussel

collection. Because mussels will be handpicked from the beach, significant sediment disturbance is not anticipated (i.e., the primary route of sediment disturbance would occur as a result of the crew walking the beach and looking for mussels).

4. Document the extent of the sampling location (i.e., start and end coordinates of the mussel sampling beach). Each beach area should cover a maximum of 150 m (approximately 500 ft) of shoreline, and should consist of the area from approximately 10 m (approximately 33 ft) above the waterline to the maximum wadeable water depth. Some areas may be smaller due to physical constraints (e.g., rocky outcrops bordering the beach area). Water level fluctuations in the riverine portion of the Site due to hydropower generation will be taken into account during sampling. During sampling, field crew members will walk the length of the beach, covering an approximately 2-meter (approximately 6.5 ft) wide transect. Five transects on the shore will be needed to cover the width of the sampling transect, along with an additional transect (or transects) in the wadeable waters.

5. When mussels are located visually, remove the mussel from the sediment surface (minimal sediment disturbance is anticipated) using gloved hands and place the mussel in a collection bucket. Any sediment remaining on the mussels will be rinsed off using site water.

6. Collect live mussels preferentially; however, dead mussels with tissue remaining in the shells will be collected if no live mussels can be located (or if insufficient live mussel tissue is available to fulfill analytical mass requirements). If dead mussels are observed but not collected, their general presence will be noted in the logbook or on the field forms. Only mussels greater than 2 cm in length will be collected (i.e., no maximum size is specified). If no mussels are located, document this information in the logbook and/or on the field forms (Attachment A3 of this FSP), and proceed to another sampling beach.

7. Collect sufficient mussels to achieve the targeted analytical mass of 4.5 g ww for the baseline ecological risk assessment (BERA)-only samples and 30 g ww for BERA and human health risk assessment (HHRA) samples (Table A2 of the FSP).[1] Because the mass of soft tissue that will be analyzed will not be known in the field (i.e., mussel shells will not be removed in the field), the field crew should collect additional individual mussels if they are concerned that the five mussels targeted for a composite

[1] The target analytical mass for BERA-only and BERA and HHRA samples takes into account sample loss due to sample processing (e.g., during homogenization and freeze drying).

will not provide sufficient analytical mass. Available literature (e.g., Bura et al. 2011) suggests that the soft tissue portion of the mussels likely to be encountered in the Site would be approximately 50 percent of the total weight of the mussel. This percent should be used in the field to estimate whether sufficient mussels have been collected to achieve the required analytical mass (i.e., the field crew will target at least 9 g ww for BERA-only samples and at least 60 g ww for BERA and HHRA samples, as described in Section 2.2.4 of the FSP).

8. Use best professional judgment while sampling; do not spend an unrealistic time in one location.
9. Identify the species of mussel (if possible). Nedeau et al. (2009) should be consulted to assist in taxonomic identification, and Teck American Incorporated (TAI) will report the location and consult with the U.S. Environmental Protection Agency (EPA) if western pearlshell mussels are found in the reservoir or riverine segments of the Site.
10. Record shell length, width, and breadth (see Figure A1 of the FSP); total weight; and general health condition on the specimen collection form (Attachment A3 of this FSP) as detailed in SOP-6. Photographs will be taken of all mussels (individually or as a group), as detailed in SOP-9, and photo IDs will be recorded on the specimen collection form.
11. Wrap mussels in food-grade heavy duty aluminum foil (dull side touching the mussel), and place the foil-wrapped mussel in a clean, resealable plastic bag.
12. Label mussel according to the labeling instructions in SOP-2.
13. Place the label and foil-wrapped/bagged mussel in a second clean, resealable plastic bag.
14. Bagged mussels will be held in coolers on wet ice for the remainder of the sampling day. Coolers will be filled by first placing a layer of bagged wet ice into the bottom of the cooler, followed by a layer of bagged mussels. Layers of bagged ice and mussels will be alternated, and voids will be filled with ice.
15. At the end of each sampling day, mussels will be transferred to a freezer or cooler with dry ice for holding until samples are shipped to the analytical laboratory.
16. Fulfill all necessary packaging, shipment, and sample custody procedures as detailed in SOP-7 and SOP-8, respectively.
17. Record any problems encountered during sample collection and corrective action taken in the field logbook as detailed in SOP-6.

Level of Effort

A total of six composite samples is targeted for each sampling area; each composite sample will be collected from a separate beach area. If no mussels are found after a reasonable effort to locate them, the field crew will document the area as searched and move on to another beach. In each sampling area, the field crew will attempt to collect mussels until sufficient tissue for six composite samples has been collected, or until a maximum of 12 beaches have been searched, whichever condition is met first. Any decisions to end sampling before this level of effort is met will be made in consultation with EPA.

Laboratory Processing

After samples are shipped to the analytical laboratory, soft tissue will be separated from the mussel shells for processing; any liquid inside the shell will be included with the sample during homogenization. A detailed description of the methods for these procedures and other analytical preparation steps are documented in Appendix D of the QAPP, specifically in the ALS Environmental (ALS) SOPs and project-specific SOP addendums.

STANDARD OPERATING PROCEDURE SOP-4

CRAYFISH TISSUE SAMPLE COLLECTION

Purpose

The purpose of this standard operating procedure (SOP) is to describe procedures for collecting crayfish from the Upper Columbia River (UCR) for the benthic macroinvertebrate tissue study. This SOP discusses collection of crayfish by use of baited crayfish traps and the procedures for processing captured crayfish.

Scope and Applicability

The crayfish species *Orconectes virilis* (northern/virile crayfish) and *Pacifastacus leniusculus* (signal crayfish) will be collected using crayfish traps. The use of bait attracts scavenging crayfish into the traps. Traps can be deployed in a variety of water depths (during the U.S. Fish and Wildlife Service [USFWS] surveys, traps were placed in water as deep as 60 ft), but will generally be placed in water less than 20 ft deep. Crayfish traps are identical to minnow traps used to catch small fish. Traps can be set individually with a line and a float, or in a series of up to three traps attached to a single line and float. The determination of trap locations is described in the field sampling plan (FSP).

Equipment and Materials

Required equipment and materials include:

- Minnow or crayfish traps (only traps with escape guards will be used)
- Bait (may include canned cat food, hot dog pieces, cut-up pieces of fish, and fish oils)
- Claw-proof bait bags (e.g., made out of nylon or cheesecloth) or bait canisters
- Chest waders or rubber boots (if traps are deployed in wading conditions; traps will primarily be deployed from a boat)
- Measuring board and calipers (measuring to the nearest millimeter)
- Analytical balance (and calibrated taring weight)
- Small floats or surveyor flagging
- Boat hook
- Twine
- Resealable plastic bags and food-grade heavy duty aluminum foil

- Disposable laboratory gloves (nitrile or latex); these gloves must always be worn when handling crayfish
- Waterproof labels and tags
- Permanent marking pen
- Cooler, ice scoops, and shovels
- Buckets (for holding crayfish)
- Plastic coolers for crayfish
- Personal protective equipment
- Protective gloves
- Radios
- Global positioning system (GPS) receivers
- Knife
- Depth finder
- Maps (Site)
- Digital camera
- Field forms and notebooks
- Pens and pencils
- First aid kits, and health and safety manuals (one each per crew).

Sampling gear that has previously been used at other sites will be thoroughly inspected and cleaned before the field effort to prevent transport of aquatic invasive species (e.g., zebra mussels).

Procedures

Crayfish will be collected and processed using the process described below. All non-native crayfish (i.e., northern/virile crayfish) will be retained regardless of size. For native (i.e., signal) crayfish at the HHRA and BERA locations, only individuals greater than 3.25 in. in length will be retained, as established by State of Washington fishing regulations. At the BERA-only locations, native crayfish of any size may be collected (using a Washington State Department of Fish and Wildlife research permit).

Deployment of Crayfish Traps

1. Select a sampling location as described in the FSP. Location selection will be based on best professional judgment in areas with potential crayfish habitat. Crayfish habitat includes areas with loose cobbles and boulders and tree/plant debris, as well as areas

near structures such as docks. In consultation with EPA, structures (e.g., refueling stations or docks) that could influence chemical concentrations will be avoided.

2. Document sample location conditions, date, and time of arrival in the field logbook, and take digital pictures of the area, as indicated in SOP-6 and SOP-9.
3. Document the sample location coordinates and number of traps placed at the location. Traps can be set individually with a line and a float, or in a series of up to three traps attached to a single line and float.
4. Bait and assemble each trap (e.g., use the hinges provided around the rim of each trap or use zip ties). Bait will be placed in a claw-proof bait bag (e.g., nylon or cheesecloth) or bait canister to ensure that crayfish do not consume the bait, which could affect sampling results. Cans of cat food will be the primary bait type; cans should be opened one day prior to sampling to improve sampling success. Other bait types may include hot dog pieces, cut-up pieces of fish, and fish oils to further improve catch success.
5. Attach a buoy (i.e., small visible float) to the trap to aid in retrieval of the trap. Buoys should be relatively inconspicuous to minimize vandalism.
6. Deploy the trap at the sampling station by lowering it into the water over the side of the boat (or from the shore when applicable), making sure that it does not get tangled in the buoy line. If a series of traps will be deployed, attach the trap to the next one in the sequence before deploying it. Floats do not need to be attached to any of the additional traps.
7. If the sample station is located in wadeable waters and is deployed from shore, tie a long piece of rope onto the trap and lower the trap at the edge of the habitat along the shoreline. Secure the end of the line to a structure on the shoreline, and use a long piece of surveyor flagging to mark where the line is tied.
8. Check traps daily (in the mornings and evenings) until the target number of crayfish is collected, or for a maximum of three overnight soaking periods.

Retrieving the Crayfish Trap

The trap will be retrieved and the crayfish collected as follows:

1. Arrive at the buoy attached to the trap, and snag the buoy line with a boat hook if using a boat to retrieve the traps. The previously collected GPS coordinates may be used to aid in locating the crayfish traps.
2. Pull the trap to the water surface using the buoy line, and bring the trap onboard the boat (or onto shore, if wading).
3. Open the trap and transfer the captured crayfish into the collection buckets; any sediment on the crayfish will be rinsed off using site water. If a string of traps is used,

proceed to the next trap in sequence and repeat steps 2 and 3.

4. Place crayfish from each trap into a bucket of water obtained locally on the site while they await processing.
5. Once the targeted number of crayfish have been collected from the sampling area (i.e., 30 crayfish) or after three overnight soaking periods, collect all traps and ropes and remove them from the sampling area.
6. In general, dead crayfish will not be retained for analysis. However, if few crayfish are found in a sampling area, dead crayfish may be retained in consultation with U.S. Environmental Protection Agency (EPA) oversight.
7. Record species other than crayfish in the traps on the non-target species form and release them immediately.
8. Process the crayfish for length (total and carapace; See Figure A2 of the FSP), weight, and enumeration record in the field forms (Attachment A3 of this FSP) according to SOP-6.

Crayfish Length and Weight Measurements

The following information should always be recorded on the field form (Attachment A3 of this FSP) for <u>each</u> crayfish that is collected from the Site or reference locations:

1. Place each crayfish on a measuring board and record its total length, and use calipers to measure the carapace length from the tip of its rostrum to the end of the telsun (central tail section or uropod). These measurements, which should be recorded to the nearest millimeter, are shown in detail in Figure A2 of the FSP.
2. Place a balance tray on an analytical scale, and press TARE. Wait for a reading of 0.0 g. Place the crayfish in the balance tray, and allow the weight reading to stabilize, and record the weight to the specified accuracy (e.g., 1.0 g). A Pesola scale may also be used to weigh crayfish.
3. Identify crayfish species (i.e., signal or northern/virile). Signal crayfish less than 3.25 in. in length will not be retained for the HHRA and BERA sampling areas. All crayfish (i.e., regardless of size) will be retained in other sampling areas (native signal crayfish will be collected using a Washington State Department of Fish and Wildlife research permit).As requested by EPA, Teck American Incorporated (TAI) will report the location and consult with EPA if signal crayfish are encountered in the Site.
4. Record length (total and carapace) and weight measurements on the specimen collection

form (Attachment A3 of this FSP) according to SOP-6, along with the crayfish identification number (ID), location ID, date, time, and any notes regarding the condition of the crayfish (e.g., missing claws, presence of eggs, molting). Take photographs of all crayfish (individually or as a group), as detailed in SOP-9.

5. Wrap crayfish in food-grade heavy duty aluminum foil (dull side touching crayfish), and place foil-wrapped crayfish in a clean, resealable plastic bag.
6. Label crayfish according to the label instructions in SOP-2.
7. Place label and foil-wrapped/bagged crayfish in a second clean, resealable plastic outer bag.
8. Bagged crayfish will be held in coolers on wet ice for the remainder of the sampling day. Coolers will be filled by first placing a layer of bagged wet ice into the bottom of the cooler, followed by a layer of bagged crayfish. Layers of bagged ice and crayfish will be alternated, and voids will be filled with ice.
9. At the end of each sampling day, crayfish will be transferred to a freezer or cooler with dry ice for holding until samples are shipped to the analytical laboratory.
10. Fulfill all necessary packaging, shipment, and sample custody procedures as detailed in SOP-7 and SOP-8, respectively.
11. Record any problems encountered during sample collection and corrective action taken in the field logbook as detailed in SOP-6.

Level of Effort

A total of six composite samples is targeted in each sampling area; each sample is to consist of five crayfish (i.e., a total of 30 crayfish in each sampling area). The target of five crayfish per composite was determined based on an analysis of the sizes of crayfish that are anticipated to be collected and the required analytical mass needed for each sample (i.e., 4.5 g ww for the baseline ecological risk assessment [BERA]-only samples and 30 g ww for BERA and human health risk assessment [HHRA] samples; see Table A2 of the FSP).[2] These calculations are discussed in more detail in Section 2.2.4 of the FSP. Traps will be checked twice daily (mornings and evenings) during sampling for a maximum of three nights in a given sampling area. Once the targeted number of crayfish is collected (n = 30) or the maximum period designated for trap deployment has been reached (whichever occurs first),

[2] The target analytical mass for BERA-only and BERA and HHRA samples takes into account sample loss due to sample processing (e.g., during homogenization and freeze drying).

all traps and ropes will be removed from the sampling area. Any decisions to end sampling before this level of effort is met will be made in consultation with EPA.

Laboratory Processing

After samples are shipped to the analytical laboratory, crayfish will be composited based on a compositing plan that will be developed after sampling is completed. The compositing plan must be finalized and approved by EPA so that chemical analyses can be completed within the sample holding times (six months for inorganic chemicals and one year for organic compounds, based on frozen samples that are stored at -20°F). Crayfish samples will be analyzed as the following tissue types to ensure that the resulting data best represent how human, fish, and wildlife would consume crayfish.

- For BERA-only samples (i.e., those collected from Sampling Areas 1, 3, and 4; Table A1 of the FSP), whole-body samples will be analyzed. Thus, no dissection is needed to prepare these samples for analysis.
- For BERA and/or HHRA samples (i.e., those collected from Sampling Areas 2, 5 and 6, and from reference areas), crayfish will be dissected and analyzed as 1) whole-body minus carapace and stomach samples (to represent the portion most commonly consumed by people), and 2) carapace- and stomach-only samples.

The methods for this tissue dissection are documented in Appendix D of the quality assurance project plan (QAPP), specifically in the ALS Environmental SOPs. It is essential that the same individuals are used in each composite so that whole-body tissue concentrations can be mathematically calculated; therefore, the resulting data will include both whole-body minus carapace/stomach and carapace/stomach samples consisting of the same individual crayfish.

STANDARD OPERATING PROCEDURE SOP-5

DECONTAMINATION OF SAMPLING EQUIPMENT

This standard operating procedure (SOP) describes procedures for decontaminating sampling and processing equipment contaminated by inorganic materials.

In general, mussel and crayfish sampling will not require equipment decontamination. Aluminum foil and/or plastic bags will be used as a barrier between weighing and measuring equipment. For example, a piece of aluminum foil will be used as a barrier between the specimen and the measuring board. Similarly, a resealable plastic bag may be used to hold organisms during weighing. Sampling equipment such as buckets and crayfish traps do not need to be decontaminated prior to use.

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 bucket(s) (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 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, between sampling stations, and at any other times specified by the field sampling plan (FSP). The specific procedures for decontaminating reusable sampling equipment are as follows:

1. Rinse the equipment thoroughly with tap or site water to remove any visible sediment or debris.
2. Pour a small amount of concentrated laboratory detergent (e.g., Alconox) into a bucket (e.g., about 1/2 tablespoon per 5-gallon bucket) and fill it halfway with tap or 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 site water. Equipment does not need to be dried before use.
5. If the decontaminated sampling equipment is not to be used immediately, wrap small items in aluminum foil (dull side facing the cleaned area).
6. If the sample collection or processing equipment is cleaned at the field laboratory and transported to the site, then the decontaminated equipment will be wrapped in aluminum foil (dull side facing the cleaned area) and stored and transported in a clean plastic bag (e.g., a trash bag) until ready for use, unless the FSP lists special handling procedures.

STANDARD OPERATING PROCEDURE SOP-6

FIELD DOCUMENTATION

Scope and Applicability

This standard operating procedure (SOP) presents the general information that will be documented for all macroinvertebrate tissue sampling activities conducted by Teck American Incorporated (TAI) field personnel. Proper record keeping will be implemented in the field to allow samples to be 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. Several types of field documents will be used for this purpose by field personnel.

Equipment and Materials

Equipment and materials used for this SOP are:

- Field logbook
- Field forms
- Black-ink pen
- Field forms
- 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 will be documented in the field logbook. Other types of information that may be included in the field logbook include the following:

- Project sampling name and type
- Name of person making entries and other field staff
- Onsite visitors, if any
- Observations made during sample collection, including collection complications, visible debris, and other details not entered onto the field form

- Any notable substrate characteristics, including presence of cobbles, boulders, woody debris, etc.
- Any vegetation that may influence mussel and/or crayfish abundance or distribution
- 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 mussel and/or crayfish abundances or distribution
- Specific measured characteristics of collected mussels and crayfish.

The field supervisor will maintain the field logbook and is responsible for ensuring that the field logbook and all field data forms are correct. Requirements for logbook entries will 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.
- 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.

Upon completion of the field sampling event, the field supervisor will be responsible for submitting all field logbooks to be copied. A discussion of copy distribution is provided 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 sampling and will include the following information:

- Project name and date
- Names of all members of the sampling team
- A brief description of the weather

- The time each sample was collected
- Sampling area identifier
- Sampling location details from the hand-held global positioning system (GPS) unit: latitude, longitude, positional accuracy, and elevation (unless recorded electronically)
- The sample identifier and analysis to be performed
- Descriptions of sampled habitat and collected mussels, clams, and/or crayfish
- A list of numbered photographs of the site
- 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. A discussion of copy distribution is provided below.

Photographs

Reference SOP-9 of the FSP for procedures regarding 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.

Set-up 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 work order number. The following documents may be included in this cabinet for each field event:

- Original field logbook(s)
- Original field data forms
- Photograph CDs (see SOP-9)
- Original signed chain-of-custody (COC) forms.

STANDARD OPERATING PROCEDURE SOP-7

SAMPLE PACKAGING AND SHIPPING

Scope and Applicability

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. This standard operating procedure (SOP) presents the method to be used when packaging samples that will either be hand-delivered or shipped by commercial carrier to the laboratory.

Equipment and Materials

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

- Field sampling plan (FSP) for the macroinvertebrate tissue study
- Project-specific field logbook
- Resealable airtight bags (assorted sizes)
- Food-grade heavy duty aluminum foil
- Dry ice
- Boxes and Styrofoam® coolers
- Drum liners or sturdy trash bags for securing samples within coolers
- Fiber-reinforced packing tape and duct tape
- Clear plastic packing tape
- Scissors or knife
- 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 would be needed as well
- COC seals
- Large plastic garbage bags (preferably 3 mil [0.003 in.] thick) for cooler lining
- Paper towels
- “Fragile,” “This End Up,” or “Handle With Care” labels and “Perishable Goods” labels
- Address labels for processing laboratory

- Airbills for overnight shipment.

Procedure

In some cases, samples may be transferred from the field to a local storage facility where they will be frozen. 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 potentially limiting factors to 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. In the field, samples will be maintained in coolers with wet ice at 4°C until they are packaged for shipping to the offsite analytical laboratory.

Sample Preparation

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 logbooks or 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-2.
2. Clean the outside of all dirty sample containers to remove any residual material that may lead to cross contamination.
3. Wrap individual organisms in food-grade heavy duty aluminum foil (dull side touching organism), and place in a resealable plastic bag. Label and bagged organism will be placed in a second resealable plastic bag such that the sample label can be read.
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, organisms will be frozen (either in a freezer or in a cooler on dry ice) until ready for shipping.

To Prepare Samples and Coolers for Shipping

1. Choose the appropriate size Styrofoam® cooler(s), make sure that the outside and inside of the cooler is clean of gross contamination, and contain it within a cardboard box.
2. Line the cooler with bubble wrap.
3. Concurrently with placing samples in the shipping cooler(s), 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. The COC form will note that the laboratory should freeze all samples and hold them until compositing instructions are received.
5. Check sample IDs for all organisms against the COC form to ensure all samples intended for shipment 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 (i.e., $<0°C$). The amount of dry ice that may be required should always be overestimated. 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. 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.
8. 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 (or multiple) COC form(s) for the samples contained in that particular cooler.
9. 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 around the opening between the lid and the bottom of the cooler and around the circumference of the cooler at both hinges.

10. 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 across the seal may be necessary if the outside of the cooler is wet.

11. 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 Shipping

Hand Delivery to the Testing Laboratory

1. The field supervisor will notify the laboratory contact and the team project QA/QC coordinator that samples will be delivered to the laboratory and the estimated arrival time.
2. All environmental samples that are hand-delivered to the testing 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” as well as “Perishable Goods” labels. If the shipment contains multiple coolers, indicate on the address label the number of coolers that the testing 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. Appropriate shipping labels will be used for dry ice.
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).
3. The field supervisor will notify the laboratory contact and the task analytical chemistry QA/QC coordinator that samples will be shipped and the estimated arrival date and time. All environmental samples will be shipped at <0°C overnight for next morning delivery. The field supervisor will provide copies of all COC forms to the task manager upon completion of the study.

STANDARD OPERATING PROCEDURE SOP-8

SAMPLE CUSTODY

Scope and Applicability

This standard operating procedure (SOP) describes procedures for maintaining custody of environmental samples collected during all macroinvertebrate tissue sampling activities conducted by Teck American Incorporated (TAI) field personnel. The procedure outlined herein will be used in conjunction with SOP-2, which covers sample labeling; SOP-6, 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.

At no time is it acceptable for samples to 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).

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 what analyses are to be performed on the shipped samples. Therefore, COCs must include information only on the samples within the shipping container, and samples shall not be shipped without an associated COC within 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. The COC form will also identify the sample collection date and time, the type of sample, the project, the sampling personnel, and the number of coolers shipped. The COC form will be sent to the laboratory along with the samples. The COC form(s) will be placed into a plastic resealable bag and secured to the inside lid of each cooler. A copy of the COC forms will be retained by the field supervisor for filing in the project files by the task manager 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(s), indicating the time and date that the transfer occurs.

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.
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 form(s) 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

As security against unauthorized handling of the samples during shipping, two custody seals will be affixed to 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 across the seal may be used.

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 the task manager. 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.

STANDARD OPERATING PROCEDURE SOP-9

DIGITAL CAMERA USE AND DOCUMENTATION PROCEDURES

Purpose

The purpose of this standard operating procedure (SOP) is to describe the use of digital cameras and procedures for digital camera data management.

Scope and Applicability

This SOP is applicable 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
- Spare batteries
- 12-volt charger
- Digital camera-carrying case and manual
- Field form
- Permanent marker
- Compass
- Personal computer.

Typical Camera Features

- Save photographs (in standard mode) directly to a memory stick or comparable device
- Auto focus; manual focus available if required
- Zoom
- 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 disk capacity
- 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 locations. Examples of field activities for which photo documentation will be useful include 1) collected mussels and crayfish; 2) station vicinity with associated river mile and compass directions noted; 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 will keep a daily hard copy log of all photographs. The following digital photograph data will be collected:

- Camera identifier (type, model, equipment number).
- Project and event identification (ID) number—this information is obtained from the field team leader.
- Team members—list each team member.
- Photograph number—record the number of the photograph and the photograph file name (as coded below).
- Date and time—as provided by the camera display.
- Description—the target of the photograph.
- Station—identify a station ID (such as sample location ID), if applicable.
- Northing and easting—list the coordinates of the subject of the photograph and the compass direction in which the picture is being taken. Coordinates can be acquired from a global positioning system (GPS) or from the geographic information system (GIS) if the location has been surveyed. Coordinates do not need to be recorded for a station if the location was surveyed prior to photograph collection.

Notes: Record any other pertinent information (including coordinates of location where the photograph was taken [see above]).

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 is the sampling area name (e.g., Rebecca Lake). 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 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 field team leader.

Sample Processing Coordinator Responsibilities

The field team leader will be responsible for 1) reviewing electronic photographs and the logs as they are made available to ensure consistency and completeness of annotations; 2) collecting and archiving the hard copies of the photograph logs; 3) reviewing electronic photographs and the logs as they are made available to ensure consistency and completeness of annotations; and 4) notifying the sampling team leader of apparent inconsistencies and making recommendations for corrective action.

Key Checks and Items

Important checks for digital camera management are:

- Make sure the camera's battery is fully charged on a daily basis
- Keep extra memory sticks available
- To save battery life, use flash only when necessary
- 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
- Leave enough time at the end of the field day to transfer the data.

STANDARD OPERATING PROCEDURE SOP-10

HANDLING AND REPORTING OF CULTURAL RESOURCES

Scope and Applicability

This standard operating procedure (SOP) describes the procedures to be followed by all Teck American Incorporated (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 macroinvertebrate tissue sampling effort. 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 and the applicable requirements, procedures, and standards of the National Park Service (NPS), 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 remedial investigation and feasibility study (RI/FS) are provided in the cultural resources coordination plan (CRCP).

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 slowing or halting ground-disturbing 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, 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 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 rainfall, stormwater, and other possible disturbances, or
- Assist in moving the 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 would jeopardize their integrity due to erosion or collection by unauthorized individuals.

The archaeological 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 1 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 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(s) 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 1 to the CRCP).

Discoveries When an Archeological Monitor is not Present

As previously stated, an archaeological monitor and/or tribal representative(s) 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 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 in recognizing these materials by a professional archaeologist prior to the initiation of any 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.

ATTACHMENT A3

EXAMPLES OF VARIOUS FIELD FORMS
MACROINVERTEBRATE TISSUE STUDY

This Addendum to the UCR RI/FS general SHSP provides examples of the various field forms that will be used during the macroinvertebrate tissue sampling effort. These forms include the following:

- Chain of Custody (COC) Form
- Custody Sea
- Sample Label
- Location Form
- Specimen Collection Form
- Non-Target Species Form
- Protocol Modification Form

_______ of _______

CHAIN-OF-CUSTODY/TEST REQUEST FORM

Project/Client Name: UCR Macroinvertebrate Sampling
Project Number:
Contact Name:
Sampled By:

Ship to:
Attn:
Shipper:
Form filled out by:

Shipping Date:
Airbill Number:
Turnaround requested:

Sample Collection Date (m/d/y)	Time	Sample Identification	Volume of Sample / # of Containers	Matrix	Test(s) Requested (check test(s) required)					Comments / Instructions [Jar tag number(s)]
Total Number of Containers				Purchase Order / Statement of Work #						

1) **Released by:**	2) **Released by:**	3) **Released by:**	4) **Released by:**	5) **Released by:**
Company:	Company:	Company:	Company:	Company:
Date/Time:	Date/Time:	Date/Time:	Date/Time:	Date/Time:
Rec'd by:	**Rec'd by:**	**Rec'd by:**	**Rec'd by:**	**Rec'd by:**
Company:	Company:	Company:	Company:	Company:
Date/Time:	Date/Time:	Date/Time:	Date/Time:	Date/Time:

200 West Mercer Street
Suite 401
Seattle, WA 98119
Tel: (206) 378-1364
Fax: (206) 217-9343

To be completed by Laboratory upon sample receipt:

Date of receipt::	Laboratory W.O. #:
Condition upon receipt:	Time of receipt:
Cooler temperature:	Received by:

CUSTODY SEAL

LAB SAMPLE
DO NOT TAMPER

DATE: ____________________

INITIALS: ________________

SAMPLE LABEL

Windward Environmental LLC 200 W. Mercer St., Suite 401, Seattle, WA 98119 Tel: (206) 378-1364 Fax: (206) 217-0089	
Project: UCR BMI	Sampler initials:
Collection date:	Collection time:
Sampling Area:	Location ID:
Species:	
Specimen ID:	
Length (mm):	Weight (g):

LOCATION FORM

Project Name: UCR Macroinvertebrate Sampling ***Field crew initials:***

Comments:

Date	Time	Location ID	Collection Method	Coordinates		Water Depth (ft)	Comments
				Latitude	Longitude		

Note: For mussel beaches, start and end coordinates will be recorded (two rows may be used if needed).

SPECIMAN COLLECTION FORM

Project Name: UCR Macroinvertebrate Sampling ***Field crew initials:***

Comments:

Collection Date	Collection Time	Location ID	Collection Method	Specimen ID #	Species	Length (mm)		Weight (g)	Photo ID	Comments
						Total	Carapace			

NON-TARGET SPECIES FORM

Project Name: UCR Macroinvertebrate Sampling *Field crew initials:*

Comments:

Collection Date	Collection Time	Location ID	Collection Method	Species	Count	Length Range (mm)	Total Weight (g ww)	Comments

PROTOCOL MODIFICATION FORM

Project Name: **UCR Macroinvertebrate Sampling**

Material to be Sampled: ______________________

Standard Procedure for Field Collection & 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:

Initiator's Name: ______________________ Date: __________

Project Manager: ______________________ Date: __________

QA Manager: ______________________ Date: __________

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

Parametrix

411 108th Avenue NE, Suite 1800
Bellevue, WA 98004

integral consulting inc.

7900 SE 28th Street, Suite 410
Mercer Island, WA 98040

April 2016

CONTENTS

INTRODUCTION A4-1

DISCOVERIES WHEN AN ARCHAEOLOGICAL MONITOR IS PRESENT A4-1

DISCOVERY OF HUMAN REMAINS A4-2

DISCOVERIES WHEN AN ARCHAEOLOGICAL MONITOR IS NOT PRESENT A4-3

CONFIDENTIALITY A4-3

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
NPS	National Park Service
RI/FS	remedial investigation and feasibility study
STI	Spokane Tribe of Indians
Teck	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 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 draft cultural resources coordination plan (CRCP).

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/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 Teck technical team will remain onsite 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 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 1 to the CRCP). Any discoveries within the boundaries of the Colville or the Spokane reservations must also be reported immediately to the respective Tribe.

The Teck 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(s) 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 1 to the CRCP).

DISCOVERIES WHEN AN ARCHAEOLOGICAL MONITOR IS NOT PRESENT

As previously stated, an archaeological monitor and/or Tribal representative(s) 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, Teck 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 Teck field personnel will be provided with training in recognizing these materials by a professional archaeologist prior to the initiation of any 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 Teck of any such inquiries. To the extent permitted by law prior to any release of information, Teck, 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

OVERVIEW OF THE U. S. FISH AND WILDLIFE SERVICE CRAYFISH AND MUSSEL SURVEYS

CONTENTS

LIST OF TABLES .... B-iii
ACRONYMS AND ABBREVIATIONS .... B-iv
UNITS OF MEASURE .... B-v
B1 INTRODUCTION B-1
B2 OVERVIEW OF CRAYFISH SURVEY B-1
B3 OVERVIEW OF MUSSEL SURVEY B-1
B4 REFERENCES B-2

LIST OF TABLES

Table B1. Summary of the USFWS Crayfish Survey Results

Table B2. Summary of the USFWS 2012 Mussel Survey Results

Table B3. Summary of the USFWS 2013 Mussel Survey Results

ACRONYMS AND ABBREVIATIONS

FSP	field sampling plan
UCR	Upper Columbia River
USFWS	U.S. Fish and Wildlife Service

UNITS OF MEASURE

m	meter(s)
m^2	square meter(s)

B1 INTRODUCTION

This document presents an overview of the crayfish and mussel surveys conducted by the U.S. Fish and Wildlife Service (USFWS). The objective of these surveys was to document where crayfish and mussels could be found in the Upper Columbia River (UCR) and several tributaries, and what species were present (USFWS unpublished). It is important to note, however, that because of the vast size of the UCR, this survey was not meant to be an exhaustive survey of all areas. Rather, select locations were identified for inclusion in the survey based on areas where mussels or crayfish had been reported, and to provide overall spatial coverage of the UCR.

B2 OVERVIEW OF CRAYFISH SURVEY

The USFWS crayfish survey was conducted from July to October of 2013. Baited crayfish traps were placed in more than 40 locations throughout the UCR, from the U.S-Canada border to the Grand Coulee Dam. In addition, several locations on the Kettle and Spokane Rivers (tributaries to the UCR) were included in the survey. Of the crayfish collected, the vast majority were non-native virile/northern crayfish; only five of the 348 crayfish collected were native signal crayfish. Crayfish were far more abundant in the downstream portion of the UCR, and abundance generally appeared to increase as stations progressed downstream. A summary of the locations, number of crayfish collected, and catch per unit effort (where a unit of effort is equal to one overnight trap soak) is provided in Table B1. A map showing the survey locations is provided in the field sampling plan (FSP) (Appendix A, Map A3).

B3 OVERVIEW OF MUSSEL SURVEY

The USFWS mussel surveys were conducted in both 2012 and 2013. In 2012, 24 beach areas were surveyed, each of which represented approximately 100 m of shoreline (from the shore to approximately 10 m above the waterline). In 2013, more than 40 locations were surveyed (revisiting many of the 2012 locations), including some locations on tributaries to the UCR (e.g., the Kettle and Sanpoil Rivers). Mussels or mussel shells were found at nearly all locations surveyed in 2012 when water levels were lower; mussel presence or absence was less consistent in 2013 when water levels were higher. A summary of the locations, number of mussels collected (dead and live), and mussel density at each location (in mussels per square meter) is provided in Tables B2 and B3, based on information provided

by USFWS (unpublished). A map showing the survey locations is provided in the FSP (Appendix A, Map A2).

B4 REFERENCES

USFWS. Unpublished. Data files containing results from 2012 and 2013 surveys of mussels and crayfish in the Upper Columbia River. Sent to John Toll, Windward Environmental, by Dustan Bott, EPA Region 10, on December 15, 2015.

TABLES

Table B1. Summary of the USFWS Crayfish Survey Results

Survey Locations	Approximate RM or Tributary	Sampling Month(s)	Water Depth Range (feet)	Crayfish Trap-Nights	Count of Crayfish			CPUE (crayfish/ trap-night)
					Total	Northern/ Virile (invasive)	Signal (native)	
Tom Bush Creek	742	Sept, Oct	2 to 18	23	0	0	0	0
Deadman's Eddy	737	Sept, Oct	6 to 228	24	0	0	0	0
Northport	735	Sept, Oct	2 to 25	31	0	0	0	0
Onion Creek	730	September	6 to 34	20	0	0	0	0
China Bar	724	July	4 to 54	32	0	0	0	0
North Gorge	719	July, October	5 to 64	34	2	0	2	0.059
Bossburg	714	July	10 to 25	10	0	0	0	0
Summer Island	710	July, October	8 to 29	22	0	0	0	0
Barstow Bridge	Kettle River	July	3 to 6	5	0	0	0	0
Napoleon	Kettle River	July	4 to 10	5	0	0	0	0
Kettle River Campground	Kettle River	July	3 to 9	5	0	0	0	0
Kamloop Island	Kettle River	July	12 to 25	7	0	0	0	0
Marcus	708	July	9 to 15	6	1	1	0	0.17
Marcus Nancy Creek	705	July	5.5 to 26	20	0	0	0	0
Marcus Pingston Creek	705	July	8 to 28	14	0	0	0	0
Marcus E. of St. Paul's Mission	705	July	9 to 23	12	0	0	0	0
Kettle Falls	700	July	2 to 24	42	7	7	0	0.17
Colville River (2 km upstream)	699	July	9 to 57	20	4	4	0	0.20
Hallam Creek	699	July	25 to 44	4	0	0	0	0
Haag Cove	698	July	6 to 18	9	0	0	0	0
French Rocks	692	July	3 to 27	46	15	15	0	0.33
Quilliscut Creek	686	July	9 to 19	10	0	0	0	0
Daisy	677	August	6 to 66	16	13	13	0	0.81
Cloverleaf	674	August	3 to 53	4	10	10	0	2.5
Gifford	672	August	5 to 40	20	17	17	0	0.85
Hunters	661	August	4 to 39	10	27	27	0	2.7
Enterprise	654	August	6 to 61	10	2	2	0	0.20
Camp Na-Bor-Lee	651	August	4 to 68	10	0	0	0	0
Spokane River Mouth	639	August	3 to 46	10	2	2	0	0.20
Porcupine Bay	Spokane River	August	5 to 50	10	0	0	0	0
Detillion	Spokane River	August	3 to 36	10	0	0	0	0
Crystal Cove	Spokane River	August	4 to 47	10	1	0	1	0.10
Fort Spokane Campground	Spokane River	August	3 to 63.7	4	1	0	1	0.25
Hawk Creek	635	July	9 to 23	10	1	0	1	0.10
Lincoln	632	July	4 to 23	10	1	1	0	0.10
Sterling Point	628	July	9 to 23	10	0	0	0	0
Jones Bay	620	July	6 to 17	8	9	9	0	1.1
Keller Ferry	615	July	3 to 19	9	56	56	0	6.2
Plum Point	604	July	4 to 22	10	35	35	0	3.5
Spring Canyon	600	July	1 to 22	10	31	31	0	3.1
Crescent Bay	597	July	6 to 20	10	73	73	0	7.3
Crescent Lake	Crescent Lake	July	2 to 13	10	40	40	0	4.0

Notes:
Source: USFWS (Unpublished)
Approximate river miles are calculated based on the centroid of crayfish trap locations.
CPUE - catch per unit effort
RM - river mile
USFWS - U. S. Fish and Wildlife Service

Table B2. Summary of the USFWS 2012 Mussel Survey Results

Survey Location	Approximate RM[a]	Date	Survey Type	Count of Mussels			Survey Area (m^2)	Density ($mussels/m^2$)	Bathymetry Contour	Mussel Species
				Total	Dead	Live				
Plum Point	603	4/30/2012	shore	49	49	0	7991	0.0061	1240	*Anodonta* spp.
Kwei Kwei	604	4/30/2012	shore	73	73	0	7834	0.0093	1240	*Anodonta* spp.
Swawilla Basin	613	4/30/2012	shore	9	9	0	6901	0.0013	1235	*Anodonta* spp.
Covington Cove	616	4/30/2012	shore	40	40	0	7148	0.0056	1245	*Anodonta* spp.
Whitestone	622	4/30/2012	shore	51	51	0	10555	0.0048	1235	*Anodonta* spp.
Sterling Point	628	4/30/2012	shore	6	6	0	6255	0.00096	1240	*Anodonta* spp.
Hawk Creek	633	4/30/2012	shore	14	14	0	5642	0.0025	1230	*Anodonta* spp.
Enterprise Bar	649	5/1/2012	shore	99	99	0	12869	0.0077	1235	*Anodonta* spp.
Wilmont Creek	654	5/1/2012	shore	60	60	0	14098	0.0043	1230	*Anodonta* spp.
Roger's Bar	657	5/1/2012	shore	104	104	0	4623	0.022	1240	*Anodonta* spp.
Hunter's	662	5/1/2012	shore	53	53	0	12228	0.0043	1240	*Anodonta* spp.
Bissell Island	674	5/2/2012	shore	52	52	0	6286	0.0083	1240	*Anodonta* spp.
Inchelium	677	5/2/2012	shore	45	45	0	9509	0.0047	1240	*Anodonta* spp.
Little Jim	680	5/2/2012	shore	124	124	0	3615	0.034	1240	*Anodonta* spp.; *Margaritifera falcata* shell remnants were also observed
Barnaby Island	686	5/3/2012	shore	21	21	0	8971	0.0023	1240	*Anodonta* spp.
Quillisicut Creek	686	5/3/2012	shore	125	125	0	8113	0.015	1240	*Anodonta* spp.
Colville River	700	5/2/2012	shore	212	212	0	7325	0.029	1245	*Anodonta* spp.
Hayes Island	704	5/3/2012	shore	1380	1380	0	6018	0.23	1245	*Anodonta* spp.
Across from Marcus Island	709	5/3/2012	shore	291	291	0	5796	0.050	1240	*Anodonta* spp.
Summer Island	711	4/30/2012	shore	390	390	0	3063	0.13	1245	*Anodonta* spp.
North Gorge	718	5/3/2012	shore	257	257	0	9659	0.027	1255	*Anodonta* spp.; *Margaritifera falcata* shell remnants were also observed
China Bar	724	5/4/2012	shore	24	24	0	14539	0.0017	1260	*Anodonta* spp.
North of Onion Creek	732	5/4/2012	shore	0	0	0	6658	0	1280	NA
Deadman's Eddy	738	5/4/2012	shore	14	14	0	7306	0.0019	1295	*Anodonta* spp.; *Margaritifera falcata* shell remnants were also observed

Notes:
Source: USFWS (Unpublished)
[a] No tributaries were surveyed in 2012.
NA - not applicable
RM - river mile
spp. - species
USFWS - U. S. Fish and Wildlife Service

Table B3. Summary of the USFWS 2013 Mussel Survey Results

Survey Location	Approximate RM or Tributary	Date	Survey Type	Count of Mussels Total	Dead	Live	Survey Area (m^2)	Density (mussels/m^2)	Bathymetry Contour	Mussel Species (number)
Plum Point	603	5/6/2013	shore	3	3	0	7386	0.00041	1255	*Anodonta* Clade 2
Kwei Kwei	604	5/6/2013	shore	0	0	0	7297	NA	1255	NA
Swawilla Basin	613	5/6/2013	shore	0	0	0	6848	NA	1255	NA
Ten Mile Creek Sanpoil	Sanpoil River	9/20/2013	in-water	166	0	0	513	0.32	NA	*Margaritifera falcata*
Thirty Mile Creek Sanpoil	Sanpoil River	9/20/2013	in-water	61	2	59	2132	0.029	NA	*Margaritifera falcata*
Silver Creek Sanpoil	Sanpoil River	9/20/2013	in-water	100	8	92	594	0.17	NA	*Margaritifera falcata*
Covington Cove	616	5/6/2013	shore	0	0	0	7030	NA	1255	NA
Whitestone	622	5/6/2013	shore	1	1	0	8027	0.00013	1255	*Anodonta* Clade 2
Sterling Point	628	5/6/2013	shore	0	0	0	5992	NA	1255	NA
Hawk Creek	633	5/6/2013	shore	0	0	0	6047	NA	1255	NA
Enterprise Bar	649	5/7/2013	shore	3	3	0	7859	0.00038	1260	*Anodonta* Clade 2
Wilmont Creek	654	5/7/2013	shore	14	14	0	9413	0.0015	1255	*Anodonta* Clade 2
Roger's Bar	657	5/7/2013	shore	43	43	0	7801	0.0055	1260	*Anodonta* Clade 2
Hunter's	662	5/7/2013	shore	7	7	0	9915	0.00071	1260	*Anodonta* Clade 2
Bissell Island	674	5/7/2013	shore	21	21	0	7302	0.0029	1255	*Anodonta* Clade 2
Inchelium	677	5/7/2013	shore	5	5	0	11027	0.00045	1260	*Anodonta* Clade 2
Little Jim Creek	680	5/3/2013	shore	2	2	0	4043	0.00050	1260	*Anodonta* Clade 2
Little Jim Creek	680	5/3/2013	in-water	0	0	0	937	NA	1255	NA
Barnaby Island	686	5/3/2013	shore	0	0	0	9720	NA	1260	NA
Quillisicut Creek	686	5/3/2013	shore	34	34	0	8983	0.0038	1260	*Anodonta* Clade 1
Bradbury Beach	699	5/1/2013	shore	11	11	0	5350	0.0021	1260	*Anodonta* Clade 2
Greenwood Loop, Colville River	Colville River	9/20/2013	in-water	>239	0	>239	199	1.2	NA	*Margaritifera falcata* (7); *Gonidia ungulata* (>232)
Hayes Island	704	4/30/2013	shore	461	461	0	4574	0.10	1260	*Anodonta* Clade 2
Barstow Kettle River	Kettle River	9/17/2013	in-water	8	0	8	6083	0.0013	NA	*Margaritifera falcata*
Downstream of Barstow	Kettle River	9/17/2013	in-water	>1600	0	>1600	2100	0.76	NA	*Margaritifera falcata*
Across from Marcus Island	709	4/30/2013	shore	22	22	0	5498	0.0040	1265	*Anodonta* Clade 2
Summer Island	710	5/3/2013	in-water	0	0	0	830	NA	1255	NA
Summer Island	710	4/30/2013	shore	32	32	0	4126	0.0078	1260	*Anodonta* Clade 2
North Gorge	719	4/30/2013	shore	9	9	0	9228	0.00098	1270	*Anodonta* Clade 2
China Bend boat launch	723	5/2/2013	shore	94	51	43	2100	0.045	1255	*Anodonta* Clade 1
Onion Creek	730	9/19/2013	in-water	0	0	0	4282	NA	NA	NA
North of Onion Creek	732	5/1/2013	shore	0	0	0	5016	NA	1275	NA
Northport Launch	735	9/16/2013	shore	130	130	0	1787	0.073	NA	*Anodonta* spp
Sheep Creek	737	5/1/2013	shore	0	0	0	3177	NA	1285	NA
Lower Sheep Creek	Sheep Creek	9/16/2013	shore	0	0	0	3090	NA	NA	NA
Deep Creek Mouth	737	5/1/2013	shore	0	0	0	480	NA	1285	NA
Deep Creek Mouth	737	9/19/2013	in-water	0	0	0	2207	NA	NA	NA
Deep Creek Tributary	Deep Creek	9/19/2013	shore	0	0	0	2738	NA	NA	NA
Deadman's Eddy	739	5/2/2013	in-water	7	7	0	1061	0.0066	1285	*Margaritifera falcata* (4); *Anodonta* Clade 2 (3)
Deadman's Eddy	739	5/1/2013	shore	149	149	0	2396	0.062	1290	*Margaritifera falcata* (5); *Anodonta* Clade 2 (144)
Deadman's Eddy, upstream end of gravel bar	739	9/18/2013	shore	20	16	4	6614	0.0030	NA	*Anodonta* Clade 2
Across from Tom Bush Creek	741	5/1/2013	shore	1	1	0	3968	0.00025	1295	*Margaritifera falcata*
Greenmile East Bank	741	9/18/2013	in-water	20	20	0	2022	0.0099	NA	*Anodonta* Clade 2
Black Sand Beach	743	9/18/2013	in-water	1	0	1	1085	0.00092	NA	*Anodonta* Clade 1
Near Canadian Border, west bank	744	5/1/2013	shore	3	3	0	2934	0.0010	1305	*Anodonta* Clade 2
Near Border west bank	745	9/18/2013	in-water	0	0	0	881	NA	NA	NA

Notes:
Source: USFWS (Unpublished)
NA - not applicable
RM - river mile
USFWS - U. S. Fish and Wildlife Service

APPENDIX C

QUALITY ASSURANCE MANUALS, SOPS, AND SAMPLE BENCHSHEETS FOR ALS ENVIRONMENTAL AND VISTA ANALYTICAL

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

UNCONTROLLED COPY

QUALITY ASSURANCE MANUAL

Doc ID:	ALSKL-QAM	Rev. Number:	24.1	Effective Date:	09/01/2015

Approved By: [signature] Date: 8/28/15
Laboratory Director – Jeff Grindstaff

Approved By: [signature] Date: 8/28/15
QA Manager – Carl Degner

Approved By: [signature] Date: 8/28/15
Technical Director, Metals – Jeff Coronado

Approved By: [signature] Date: 8/28/15
Technical Director, General Chemistry – Harvey Jacky

Approved By: [signature] Date: 8/28/15
Technical Director, Organics GC – Loren Portwood

Approved By: [signature] Date: 8/28/15
Technical Director, Organics GC/MS, HPLC – Jon James

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

UNCONTROLLED COPY

TABLE OF CONTENTS

1) Introduction and Scope ... 4
2) Organization ... 4
3) Management ... 5
4) Document Control ... 11
5) Review of Requests, Tenders and Contracts ... 11
6) Subcontracting of Tests ... 11
7) Purchasing Services and Supplies ... 12
8) Service to the Client ... 12
9) Complaints ... 13
10) Facilities and Equipment ... 13
11) Sample Management ... 14
12) Analytical Procedures ... 20
13) Measurement Traceability and Calibration ... 23
14) Assuring the Quality of Results ... 27
15) Control of Non-Conforming Environmental Testing Work ... 33
16) Corrective Action, Preventive Action, and Improvement ... 33
17) Control of Records ... 35
18) Audits ... 35
19) Management Review ... 37
20) Personnel ... 37
21) Reporting of Results ... 41
22) Summary of Changes and Document History ... 47
23) References for Quality System Standards, External Documents, Manuals, and Test Procedures ... 47
APPENDIX A – Glossary ... 49
APPENDIX B – Organization Charts, Key Personnel, and Report Signatories ... 54
APPENDIX C – ALS Environmental Confidentiality Agreement ... 69
APPENDIX D – Laboratory Floor Plan ... 74
APPENDIX E – Analytical Equipment ... 75
APPENDIX F – Containers, Preservation and Holding Times ... 82
APPENDIX G – Standard Operating Procedures ... 90
APPENDIX H – Data Qualifiers ... 100
APPENDIX I – Controlled and Normative Documents ... 102
APPENDIX J – Laboratory Accreditations ... 103

Current Data Quality Objectives (DQOs) may be requested from the laboratory for specified methods or projects.

UNCONTROLLED COPY

QA MANUAL CROSS REFERENCE TABLE

ALS QA Manual	ISO 17025:2005 Section	TNI Standard 2009 Volume 1, Module 2 Section
2	4.1	4.1
3	4.2	4.2
4	4.3	4.3
5	4.4	4.4
6	4.5	4.5
7	4.6	4.6
8	4.7	4.7
9	4.8	4.8
15	4.9	4.9
16	4.10	4.10
16	4.11	4.11
16	4.12	4.12
17	4.13	4.13
18	4.14	4.14
19	4.15	4.15
2, 12, 13, 14	5.1	5.1
20	5.2	5.2
10	5.3	5.3
12, 13, 14	5.4	5.4
10	5.5	5.5
13	5.6	5.6
11	5.7	5.7
11, 12, 13	5.8	5.8
14	5.9	5.9
21	5.10	5.10

UNCONTROLLED COPY

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 SOP CE-GEN001, *Laboratory Ethics and Data Integrity* 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

UNCONTROLLED COPY

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

UNCONTROLLED COPY

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

UNCONTROLLED COPY

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.

UNCONTROLLED COPY

- 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 SOP CE-QA001, Internal Audits. 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

UNCONTROLLED COPY

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.
- Faircall – 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 receive 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.

UNCONTROLLED COPY

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
Gregory Salata, Ph.D.	28	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	Semi-Volatile Organics Department Manager
Jon James, B.A.	24	HPLC, GC/MS Organics Department Manager
Les Kennedy, B.A.	24	Support Services Manager
Eileen Arnold, B.A.	33	Environmental Health and Safety Officer
Mike Sullivan, B.S.	15	Information Technology
Jeff Christian, B.S.	36	Director of Operations, Western USA

UNCONTROLLED COPY

4) Document Control

Procedures for control and maintenance of documents are described in SOP CE-GEN005, *Document Control*. 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 CE-QA007, *Making Entries onto Analytical Records*. 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 ADM-ARCH, *Data Archiving*.

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 CE-QA004, *Qualification of Subcontract Laboratories*. The Quality Assurance staff is responsible for maintaining a list of qualified subcontract laboratories.

UNCONTROLLED COPY

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. CE-QA012, Quality of Reagents and Standards and ADM-RLT, Reagent and Standards Login and Tracking 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 CE-GEN007, *Procurement and Control of Laboratory Services and Supplies*.

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 ADM-RLT, *Reagent and Standards Login and Tracking*.

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 CE-GEN010, *Handling Customer Feedback* is in place for these events.

UNCONTROLLED COPY

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 CE-GEN010, Handling Customer Feedback. 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

UNCONTROLLED COPY

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.

UNCONTROLLED COPY

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-GEN, *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

UNCONTROLLED COPY

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*.

UNCONTROLLED COPY

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.

UNCONTROLLED COPY

Figure 11-1

ALS Environmental Standard Chain of Custody Form

ALS Environmental

CHAIN OF CUSTODY

1317 South 13th Ave., Kelso, WA 98626 | 360.577.7222 | 800.695.7222 | 360.636.1068 (fax)

SR# ______ PAGE ______ OF ______ COC# ______

PROJECT NAME
PROJECT NUMBER
PROJECT MANAGER
COMPANY NAME
ADDRESS
CITY/STATE/ZIP
E-MAIL ADDRESS
PHONE # FAX #
SAMPLER'S SIGNATURE

SAMPLE I.D.	DATE	TIME	LAB I.D.	MATRIX	NUMBER OF CONTAINERS		Semivolatile Organics by GC/MS 625☐ 8270☐ 8270LL☐ SIM PAH☐	Volatile Organics 624☐ 8260☐ 8021☐ BTEX☐	Hydrocarbons (*see below) Gas☐ Diesel☐ Oil☐	Oil & Grease/TRPH 1664 HEM☐ 1664 SGT☐	PCBs Aroclors☐ Congeners☐	Pesticides/Herbicides 608☐ 8081☐ 8141☐ 8151☐	Chlorophenolics - 8151M Tri☐ Tetra☐ PCP☐	Metals, Total or Dissolved (See List below)	Cyanide☐ Hex-Chrom☐	(circle) pH, Cond., Cl, SO4, PO4, F, NO2, NO3, BOD, TSS, TDS, Turb.	(circle) NH3-N, COD, TKN, TOC, DOC, NO2+NO3, T-Phos	TOX 9020☐ AOX 1650☐ 506☐	Alkalinity ☐ CO3 ☐ HCO3 ☐	Dioxins/Furans 1613☐ 8290☐	Dissolved Gases RSK 175 ☐Methane ☐Ethane☐ CO2☐ Ethene☐				REMARKS

REPORT REQUIREMENTS

___ I. Routine Report: Method Blank, Surrogate, as required

___ II. Report Dup., MS, MSD as required

___ III. CLP Like Summary (no raw data)

___ IV. Data Validation Report

___ V. EDD

INVOICE INFORMATION

P.O. # ______

Bill To: ______

TURNAROUND REQUIREMENTS

____ 24 hr. ____ 48 hr.

____ 5 day

____ Standard (15 working days)

____ Provide FAX Results

______ Requested Report Date

Circle which metals are to be analyzed:

Total Metals: Al As Sb Ba Be B Ca Cd Co Cr Cu Fe Pb Mg Mn Mo Ni K Ag Na Se Sr Tl Sn V Zn Hg

Dissolved Metals: Al As Sb Ba Be B Ca Cd Co Cr Cu Fe Pb Mg Mn Mo Ni K Ag Na Se Sr Tl Sn V Zn Hg

***INDICATE STATE HYDROCARBON PROCEDURE: AK CA WI NORTHWEST OTHER: ______ (CIRCLE ONE)**

SPECIAL INSTRUCTIONS/COMMENTS:

☐ Sample Shipment contains USDA regulated soil samples (check box if applicable)

RELINQUISHED BY:		RECEIVED BY:		RELINQUISHED BY:		RECEIVED BY:	
Signature	Date/Time	Signature	Date/Time	Signature	Date/Time	Signature	Date/Time
Printed Name	Firm	Printed Name	Firm	Printed Name	Firm	Printed Name	Firm

Copyright 2012 by ALS Group

UNCONTROLLED COPY

Figure 11-2

ALS Environmental Cooler Receipt and Preservation Form

PC________

Cooler Receipt and Preservation Form

Client ______________________________ Service Request ***K15***______________________

Received:____________ Opened:____________ By:__________ Unloaded:____________ By:__________

1. Samples were received via? *Mail* *Fed Ex* *UPS* *DHL* *PDX* *Courier* *Hand Delivered*
2. Samples were received in: (circle) *Cooler* *Box* *Envelope* *Other*________________ *NA*
3. Were custody seals on coolers? NA Y N If yes, how many and where?____________________

   If present, were custody seals intact? Y N If present, were they signed and dated? Y N

Raw Cooler Temp	Corrected. Cooler Temp	Raw Temp Blank	Corrected Temp Blank	Corr. Factor	Thermometer ID	Cooler/COC ID NA	Tracking Number NA	Filed

4. Packing material: *Inserts* *Baggies* *Bubble Wrap* *Gel Packs* *Wet Ice* *Dry Ice* *Sleeves* ________________
5. Were custody papers properly filled out (ink, signed, etc.)? NA Y N
6. Did all bottles arrive in good condition (unbroken)? *Indicate in the table below.* NA Y N
7. Were all sample labels complete (i.e analysis, preservation, etc.)? NA Y N
8. Did all sample labels and tags agree with custody papers? *Indicate major discrepancies in the table on page 2.* NA Y N
9. Were appropriate bottles/containers and volumes received for the tests indicated? NA Y N
10. Were the pH-preserved bottles (*see SMO GEN SOP*) received at the appropriate pH? *Indicate in the table below* NA Y N
11. Were VOA vials received without headspace? *Indicate in the table below.* NA Y N
12. Was C12/Res negative? NA Y N

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:____

____

____

____

***Page*____*of*_____**

UNCONTROLLED COPY

Cooler Receipt and Preservation Form

Client ____Service Request *K15*______________________

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:

____

____

____

____

____

____

____

____

____

Page_____ of_____

UNCONTROLLED COPY

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 CE-GEN009, *Preparation of Standard Operating Procedures*. 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 annual 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 CE-GEN005, *Document Control*. 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 CE-QA007, *Making Entries onto Analytical Records*. 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

UNCONTROLLED COPY

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

UNCONTROLLED COPY

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.

UNCONTROLLED COPY

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 ADM-SEMC, *Support Equipment Monitoring and Calibration*.

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 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

UNCONTROLLED COPY

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* (SOP 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 SOC-CAL, *Calibration of Instruments for Organics Chromatographic Analyses*.

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 SOC-CAL, *Calibration of Instruments for Organics Chromatographic Analyses*.

UNCONTROLLED COPY

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 SOC-CAL, *Calibration of Instruments for Organics Chromatographic Analyses*.

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.

UNCONTROLLED COPY

13.16 Pipets

The calibration of pipets and autopipettors used to make critical-volume measurements is verified following SOP ADM-VOLWARE, *Checking Volumetric Labware*. 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

UNCONTROLLED COPY

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 CE-QA009, *Control Limits*.

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.

UNCONTROLLED COPY

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 CE-QA011, *Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation*, 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 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

UNCONTROLLED COPY

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.

UNCONTROLLED COPY

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

UNCONTROLLED COPY

(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:

Relative Percent Difference (RPD) = (S1 - S2) x 100 ÷ 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 CE-QA009, *Control Limits*.

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

UNCONTROLLED COPY

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 CE-QA010, *Estimate of Uncertainty of Analytical Measurements*. 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 CE-QA008, *Nonconformance and Corrective Action*. 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

UNCONTROLLED COPY

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 CE-QA008, *Nonconformance and Corrective Action*.

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 CE-GEN004, *Preventive Action*, 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;

UNCONTROLLED COPY

- 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

UNCONTROLLED COPY

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 CE-QA001, *Internal Audits*. 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 CE-QA006, *Proficiency Sample Testing Analysis*. 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.

UNCONTROLLED COPY

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 SOP CE-QA005, *Laboratory Management Review*. 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 and 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-

UNCONTROLLED COPY

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 ADM-TRAIN, *ALS-Kelso Training Procedure*. 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.

UNCONTROLLED COPY

- 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.

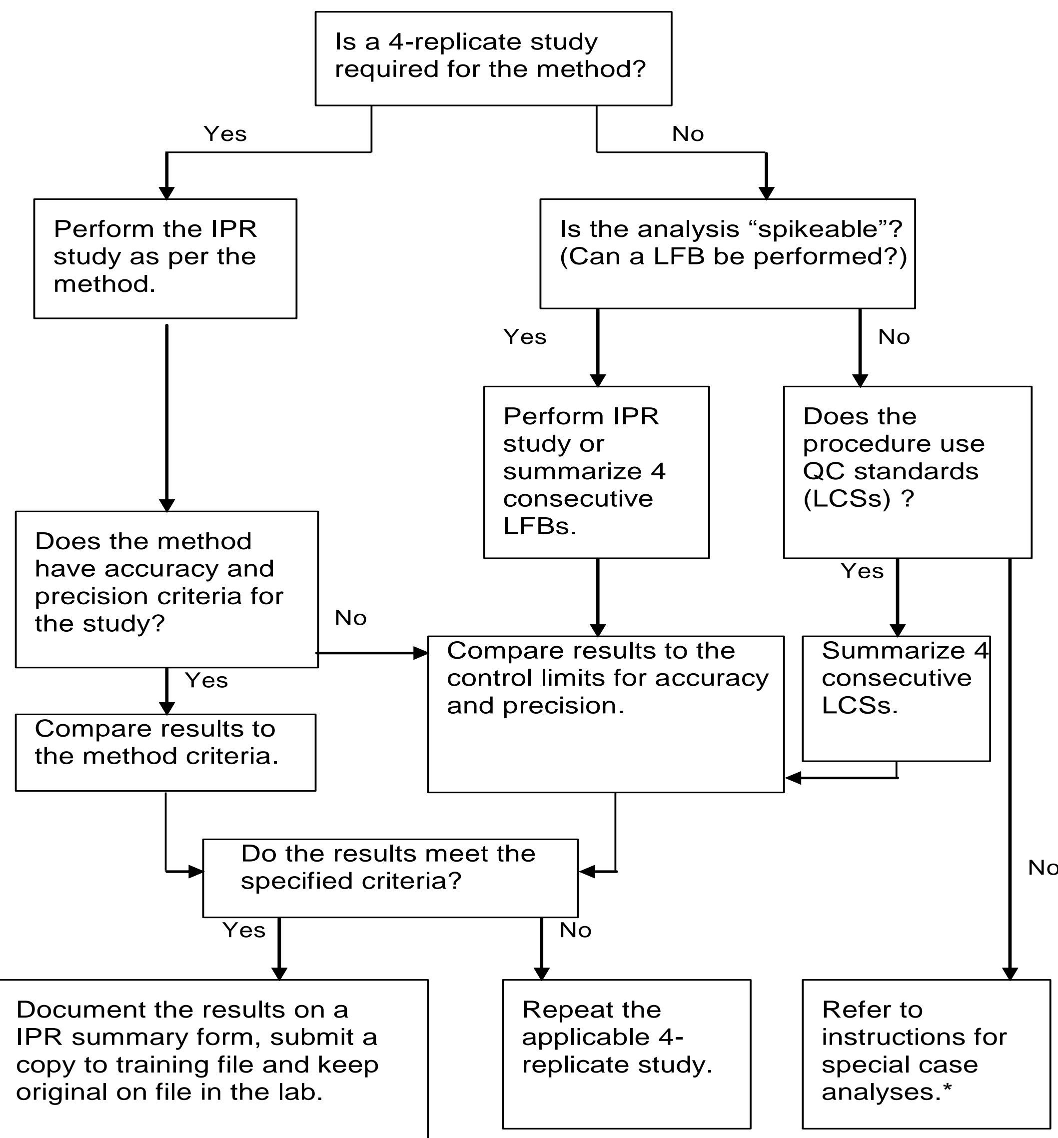
20.4 Documentation of Training

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 ADM-TRAIN, *ALS-Kelso Training Procedure*.

UNCONTROLLED COPY

Figure 20-1

Demonstration of Proficiency Flowchart



UNCONTROLLED COPY

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 CE-QA007, *Making Entries onto Analytical Records*.

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 CE-QA002, *Manual Integration Policy* and SOP ADM-MI, *Manual Integration of Chromatographic Peaks*.

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.

UNCONTROLLED COPY

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.

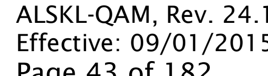
- Initial Calibration – Following the analysis of calibration standards according to the applicable SOP the data is fit to an applicable and allowed calibration model (correlation coefficient, linear, average response factor, quadratic, etc.) and the resulting calibration is compared to specified criteria. 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.

UNCONTROLLED COPY



- 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

UNCONTROLLED COPY

procedure described in the 2003 NELAC standards, Appendix D.1.1.2.1 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 SOC-CONF, *Confirmation Procedures for GC and HPLC Analyses*. Confirmation Data 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.

UNCONTROLLED COPY

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 CE-QA010, *Estimation of Uncertainty of Analytical Measurements*.

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.

UNCONTROLLED COPY

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.

UNCONTROLLED COPY

22) Summary of Changes and Document History

Revision Number	Effective Date	Document Editor	Description of Changes
24.1	9/1/2015	L. Wolf	Update QA Manager to Carl Degner, and related revision of key personnel and organization charts. Updated SOP list. Minor error corrections to existing content.

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), 2003 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.

UNCONTROLLED COPY

- 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.

UNCONTROLLED COPY

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.

UNCONTROLLED COPY

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.

UNCONTROLLED COPY

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.

UNCONTROLLED COPY

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.

UNCONTROLLED COPY

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

UNCONTROLLED COPY

APPENDIX B – Organization Charts, Key Personnel, and Report Signatories

UNCONTROLLED COPY

ALS Environmental

USA

August 28, 2015

Raj Naran
Vice President
ALS Group USA Corp
North America

- Tish Davis — Group Controller USA
- Phllip Zagotti — Controller USA Life Sciences & Tribology
- Chris Pugliano — National HSE Manager
- Liana Campbell — Compliance Manager North America
- Albert Valle — LIMS Manager USA
- Hiren Prajapati — IT Manager USA
- Ann Yeager — HR Director Life Sciences & Industrial Divisions USA
- Jarrod Evans — Marketing Manager Life Sciences Division

Greg Grandits — Director of Operations Eastern USA
- Jeff Ogle — Laboratory Director Cincinnati, OH
- Jeff Glaser — Laboratory Director Holland, MI
- Hoai Van — Laboratory Director Houston, TX
- Mike Kimmel — Laboratory Director Jacksonville, FL
- Peter Frick — Laboratory Director Middletown, PA
- Jason Badman — Operations Manager Rochester, NY

Hoai Van — Technical Director USA

Jeff Christian — Director of Operations Western USA
- Rick Bagan — Laboratory Director Everett, WA
- Roy French — Laboratory Director Fort Collins, CO
- Jeff Grindstaff — Laboratory Director Kelso, WA
- Brent Stephens — Laboratory Director Salt Lake City, UT
- Kelly Horiuchi — Laboratory Director Simi Valley, CA
- Ralph Poulsen — Laboratory Director Tucson, AZ
- Lee Wolf — Corporate QA Manager USA

UNCONTROLLED COPY

ALS Environmental

Kelso, Washington Laboratory

August 28, 2015

- Laboratory Director — Jeff Grindstaff
  - HR Rep — Dena Jurhs (dashed line to Corporate Human Resources)
  - EH&S Coordinator — Eileen Arnold (dashed line to Corporate EH&S)
  - IT Support (dashed line to Corporate Information Technology Development/Operations)
  - Quality Assurance Program Manager — Carl Degner (dashed line to Corporate Quality Assurance)
    - QA Associate — T. Caron
  - Technical Sales — Todd Poyfair (dashed line to Corporate Business Development/ Marketing)
  - Facilities — Steve Solinger
    - D. Iverson, J. Wygant
  - Support Services Manager — Les Kennedy — See 2nd Page
  - Metals — Jeff Coronado — See 2nd Page
  - Client Services — Greg Salata — See 2nd Page
  - General Chemistry — Harvey Jacky — See 2nd Page
  - SVG Organics — Jeff Grindstaff (Acting) — See 2nd Page
  - SVM Organics — Jon James — See 2nd Page

UNCONTROLLED COPY

ALS Environmental

Kelso, Washington Laboratory

August 28, 2015
Operations

Laboratory Director
Jeff Grindstaff

Support Services Manager – Les Kennedy

- **Sample Receiving**: K. Reed, A. Rynevich, K. Smith, S. Wolf, B. Wygant
- **Sample Custodian**: S. Davis, P. Dowell
- **Bottle Preparation**: R. Benz, K. Lane, S. Maximiliano, B. Totten

Metals – Jeff Coronado

- **Metals**: E. Arnold (S), J. Bailey (S), L. Jording (S), R. Moore (S), E. Cabrera, J. Chan, A. Cheatley, D. Madden, E. Mcallister, S. Patterson, T. Reinhardt, M. Ritola, M. Smith

Client Services – Greg Salata

- **Project Management**: H. Holmes, A. Juell, C. Leaf, J. Malloch, S. Samy
- **Report Production**: A. Sheldon (S), L. Bohannon, R. Hall, M. Jacobson

General Chemistry – Harvey Jacky

- **General Chemistry**: C. Mihai-Lazar (S), T. Hanganu (S), N. Bakotich (S), A. Bradbury (TL), B. Hetland (TL), E. Schloss (TL), G. Beatley, A. Cheatley, C. Christian (T), B. Devine, I. Franks, A. Hamilton, E. Lazar, C. Sethe, N. Slater, M. Specht

SVG Organics – Jeff Grindstaff (Acting)

- **GC**: L. Portwood (S), K. Clark (S), C. Harvey, R. Hayes, C. Jones (PT), J. Johnson, L. Lee, K. Miller, S. Murray, M. Ottoson, A. Petersen, B. Sheldon, S. Smith, J. Tennant, C. Vecchitto, B. Willard, C. Williams

SVM Organics – Jon James

- **LC**: C. Schroeder (S), D. Haderly, M. Hall, R. Holden, S. Jones, J. Smith
- **Volatiles**: K. Reasoner-Weiderman (S), S. Chappelle, M. Kanaly, J. McCoy, G. Roettger, Y. Xu
- **SVM**: C. Hart (S), K. Bailey, M. Brandner, E. Bruno, N. Cisney, S. Eldridge, D. Flatz, J. Laycock (T), C. Pollard, L. Murrsan, C. Smith, L. Weiskopf

UNCONTROLLED COPY

Jeffrey A. Grindstaff

1317 S. 13th Avenue • Kelso, WA 98626 • +1 360 577 7222

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
Interpretation of Mass Spectra I, 1992

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

Affiliations

American Chemical Society, 1989

Publications

Mr. Grindstaff has a number of publications and presentations. For a complete list, contact ALS.

Laboratory Director

2011 - Present

Responsible for all phases of laboratory operations at the Kelso, (WA) facility, including project planning, budgeting and quality assurance. Primary duties include the direct management of the Kelso laboratory

Previous Experience

Columbia Analytical Services, Inc.
Kelso, WA
Laboratory Director, '10-'11

Responsibilities the same as above.

Columbia Analytical Services, Inc.
Kelso, WA
Technical Manager III, Pharmaceutical GC/MS, VOC and SVOC Laboratories, '97-'10

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.

Columbia Analytical Services, Inc.
Kelso, WA
Manager, GC/MS VOA Laboratory, '94-'97

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.

Columbia Analytical Services, Inc.
Kelso, WA
Scientist III, GC/MS VOA Laboratory, '91-'94

Responsible included scheduling workload, data review, instrument maintenance and troubleshooting and personnel training and evaluation. Also responsible for supervision of extraction personnel and instrument analysts. Additional supervisory duties included report generation and data review for GC analyses. Responsibilities also included project management and client service.

Enseco-CRL
Ventura, CA
Chemist, '90-'91

Established GC/MS department including inventory maintenance, preparation of state certification data packages, method development, SOPs, and extended data programs. Performed daily maintenance and troubleshooting of GC and GC/MS instrumentation. Scheduled and performed routine and non-routine VOA analyses.

Coast to Coast Analytical Services.
San Luis Obispo, CA
GC/MS Chemist VOA Laboratory, '05-'07

Responsible for Standard Preparation for VOA analyses, instrument calibration, tuning and maintenance. Also implemented and further developed EPA methods for quantitative analysis of pesticides and priority pollutants

UNCONTROLLED COPY

Gregory G. Salata, Ph.D.

1317 S. 13th Avenue | Kelso, WA 98626 | +1 360 577 7222

Client Services Manager

2013 – Present

Management of the Client Services Departments: Project Management, Electronic Data Deliverables and Report Generation, and Sample Management. Oversee the client services for approximately $15 million in revenue annually. Personally responsible for approximately $4 million of direct technical project management annually providing technical and regulatory interpretation assistance, as well as project organization of work received by the laboratory.

Education

University of California-San Diego, Revelle College, La Jolla, CA
BA, Chemistry, 1987

Texas A&M University, College Station, TX
MS, Oceanography, 1993

Texas A&M University, College Station, TX
Ph.D., Oceanography, 1999

Affiliations

Society of Environmental Toxicology and Chemistry (SETAC)

Publication

Dr. Salata has a number of publications and published abstracts. For a complete list, contact ALS|Kelso.

Previous Experience

ALS Group USA Corp dba ALS Environmental — **Project Manager V '11 -'13**
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 annually providing technical and regulatory interpretation assistance, as well as project organization of work received and reported by the laboratory. Specializes in complex or highly sensitive projects which may involve difficult matrices and analytes..

Columbia Analytical Services, Inc. — **Project Manager V, '03 – '11**
Kelso, WA

Responsibilities include Project Management, including quotation preparation and data reporting, as well as providing technical support to the laboratory as needed. Responsibilities also include oversight of the organic extractions lab, managing resources and providing technical support for all organic preparation work flows

B&B Laboratories — **Project Manager**, '99-'03
College Station, TX

Supervisor/responsible for analysis of TPH (waters, tissues, sediments), organotins (waters, tissues, sediments), Atterberg Limits (sediments), and total organic/inorganic carbon (sediments, waters). Also responsible for report generation on specific projects. Instrumentation operated included GCs with FID and FPD detectors, Combustion TOC, Water TOC, and Dionex Accelerated Solvent Extractor.

Texas A&M University — **Graduate Student**,'91-'99
College Station, TX

While working toward MS in Oceanography, performed organic extractions for pesticides, PCBs, PAHs, and butyltins. While working toward Ph.D. in Oceanography determined stable carbon isotope ratios in sediments, waters, and bacterial phospholipid fatty acids. Other responsibilities included field sample collection, and operation/maintenance of FinniganMAT 252 isotope ratio MS.

Science Applications International — **Analytical Chemist**, '89-'90
San Diego, CA

Performed organic extraction and GC/FID analysis on sediment/rock samples for the Exxon Valdez oil spill.

Analytical Technologies — **GC Chemist**, '87-'89
San Diego, CA

Responsible for analysis of volatile organics using purge and trap and GC/PID/ELCD.

UNCONTROLLED COPY

Carl S. Degner

1317 S. 13th Avenue | Kelso, WA 98626 | +1 360 577 7222

Education

University of Houston, Houston, TX
MS in Environmental Management 1998

University of Houston, Houston, TX
BS in Biochemistry/ Biophysical Science 1984

Kelso QA Manager

2015 – Present

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. Provide QA input and policy as needed for operations, development initiatives, special projects, planning, and information technology implementation.

Previous Experience

ALS Group USA Corp.
Kelso, WA
Technical Manager, SVM Laboratory '11–'14
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 PCs on client specific project requirements.

Columbia Analytical Services, Inc.
Kelso, WA
Technical Manager, SVM Laboratory '01–'11
Essentially the same as current duties above.

Columbia Analytical Services, Inc.
Kelso, WA
Scientist IV, SVM Laboratory '98 – '01
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.

Environ Express Laboratory
LaPorte, TX
Project Chemist/Principal Organic Scientist '93 – '98
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.

BETZ Analytical Services
The Woodlands, TX
GCMS Supervisor, '91– '93
GCMS Operator: '90–'91
Supervised GC/MS Volatiles laboratory and oversaw staff's operation of four GC/MS systems. Served as system manager for HP 1000 utilizing RTE-A software. As operator performed routine sample analysis in Volatiles laboratory.

Harris County Pollution Control
Pasadena, TX
Instrument Technician, '88 – '90
Operated and maintained various equipment in instrumentation laboratory, including GC/MS, GC, HPLC, UV-Vis and Fluorescence spectrophotometers. Selected to meet with EPA and Texas Air Control Board at industrial sampling sites.

Harris County Pollution Control
Pasadena, TX
Chemist, '84 – '88
Performed wide variety of inorganic analyses utilizing gravimetric, titrimetric, and colorimetric techniques. Brought analytical methods online e.g. TKN, ortho-Phosphate. Performed metals digestions and analyzed samples using Flame AAS and GFAA.

UNCONTROLLED COPY

Eileen M. Arnold

1317 S. 13th Avenue • Kelso, WA 98626 • +1 360 577 7222

Scientist, Metals Laboratory/Kelso Health and Safety Officer

2011 - Present

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.

Environmental, Health and Safety Officer responsibilities include development and implementation of the Kelso Health and Safety program, including accident investigation and incident review, maintenance of all safety related equipment, review of monthly safety audits, and completion of all Federal and State mandated EH&S reports.

Education

Immaculata College,
Immaculata, PA
BA, Chemistry, 1977

Affiliations

American Chemical Society, Member since 1987.

Previous Experience

Columbia Analytical Services, Inc.
Kelso, WA
Scientist IV Metals Laboratory/Kelso Health and Safety Officer, *'94-'11*

Duties as described above.

Columbia Analytical Services, Inc.
Kelso, WA
Project Chemist, '92-'94

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.

Columbia Analytical Services, Inc.
Kelso, WA
Scientist IV Metals Laboratory, '87-'92

Duties include the operation and maintenance of the Inductively Coupled Argon Plasma (ICAP) Emission Spectrometer. This involves digestion, instrumental analysis, and report generation for environmental samples using approved EPA techniques.

Dow Corning Corporation.
Springfield, OR
Chemist, '86-'87

Responsibilities included ICP and atomic absorption work in silicon manufacturing. Methods development for ICP analysis of minor impurities found in silicon.

Ametek, Inc.
Harleysville, PA
Chemist, '86-'87

Responsibilities included product research and development chemist involved in production of thin-film semiconductors for use as solar cells. Work involved AA and SEM techniques

Janbridge, Inc..
Philadelphia, PA
Chemist, '78-'82

Responsibilities included maintaining electroplating process lines through wet chemical analysis techniques, and performed Quality Assurance testing on printed circuit boards.

UNCONTROLLED COPY

Jeffrey A. Coronado

1317 S. 13th Avenue | Kelso, WA 98626 | +1 360 577 7222

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 Immunaossay Training Course - EnSys Inc., 1995

Winter Conference on Plasma Spectrochemistry - San Diego, CA, 1994

ICP-MS Training Course - VG-Elemental, 1992

Technical Manager IV, Metals Department Manager

1992 - Present

Management of the Kelso Metals Department with a staff of 22 chemists and technicians, and annual revenues approaching $4 million. Responsible for data quality and timeliness, annual budgeting, revenues, expenses, workload coordination, method development efforts, and resource allocation. 2001 to Present—Project Manager: 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.2008 to Present— Participation in the corporate Information Technology governance team ensuring software development activities are in line with the companies operational objectives.2010 to Present— Participation in multiple LIMS development teams responsible for defining the CAS product. Team leader for defining specifications of the Sample Preparation Module to capture preparation information across all laboratory departments.

Previous Experience

Columbia Analytical Services, Inc.
Kelso, WA
Metals Department Manager, '92 - present

Responsibilities included management of all aspects of the metal laboratory operation, including personnel training and evaluation, review of all metals data, and report generation. Also responsible for client service on a number of ongoing CAS accounts. Technical duties include primary analytical responsibility for trace level metals analysis by ICP/MS. Analyses range from routine water and soil analysis, to marine tissues, as well as industrial applications such as ultra-trace QA/QC work for various semiconductor clients. Also responsible for a number of specialized sample preparation techniques including trace metals in seawater by reductive precipitation, and arsenic and selenium speciation by ion-exchange chromatography. Developed methodology for performing mercury analysis at low part per trillion levels by cold vapor atomic fluorescence.

Columbia Analytical Services, Inc.
Kelso, WA
Supervisor, GFAA Laboratory, '89 - '92

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.

UNCONTROLLED COPY

Harvey Jacky

1317 S. 13th Avenue | Kelso, WA 98626 | +1 360 577 7222

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

Presentations

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

General Chemistry Department Manager

2008 – Present

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

Columbia Analytical Services, Inc. — **Project Manager III, '99 – '08**
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.

Coffey Laboratories — **Director of Project Management, '97 – '99**
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.

Coffey Laboratories — **Project Manager/Chemist, '97 - '99**
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. Clients included the Oregon Department of Transportation, Hazmat Unit, Portland, Oregon; Raythion Demilitarization Co., Umatilla, Oregon; Hydroblast - Wastewater Evaporator Systems, Vancouver, Washington; and Union Pacific Railroad, Northwest Region, Klamath Falls, Oregon.

Coffey Laboratories — **Technical Sales Representative, '95 – '97**
Portland, OR

Responsible for marketing and sales, including actively prospecting for new potential clients. Additional responsibilities included procurement and preparation of all major project bids; ensuring that client expectations were met; and maintaining customer satisfaction. Served as consultant regarding industrial compliance issues, environmental remediation projects, and hazardous waste management.

Coffey Laboratories — **Senior Chemist/Laboratory Chemical Hygiene Officer, '88 - '95**
Portland, OR

Responsibilities: Performed analytical tests including Anions by Ion Chromatography (EPA 300.0), PAHs by HPLC (EPA 8310), Cyanides (EPA 335), and other inorganic, wet chemistry, and organic analytical tests on a wide variety of sample matrices. Responsible for the initial quality assurance review of work performed, supervised and managed personnel. Developed and implemented Laboratory Chemical Hygiene Plan. Directed personnel in regards to safety issues and hazardous waste management. Served as consultant and teacher regarding analytical methodology, environmental compliance, and industrial hygiene.

UNCONTROLLED COPY

Jonathan (Jon) James

1317 S. 13th Avenue | Kelso, WA 98626 | +1 360 577 7222

VOA/MS, GC/MS and HPLC Department Manager

2009 – Present

Oversee the operation of the Volatiles GC/MS, Semivolatile GC/MS and HPLC laboratories. Responsibilities include organizing and prioritizing workload, training and development of staff, working with PCs on client specific project requirements, departmental budgets, workload coordination, method development efforts and resource allocation. Responsible for the quality and timeliness of analytical reports. Other responsibilities include ensuring compliance with CAS QA protocols and assisting staff with troubleshooting equipment and procedural problems.

Education

Evergreen State College
Olympia, WA
BA, Chemistry/Biology
1991

Introduction to LC Methods Development & Troubleshooting, *Hewlett-Packard, Tacoma, WA,* 1995.
HPLC Maintenance Seminar, *Waters, Portland, OR,* 1994.
GC/HPLC Maintenance Seminar, *Hewlett-Packard, Olympia, WA,* 1993.
Gas Chromatography Seminar, *Curtis Matheson Scientific, Kelso, WA,* 1992.
HPLC Seminar, *Hewlett-Packard, Kelso, WA,* 1991.

Previous Experience

Columbia Analytical Services, Inc.
Kelso, WA
Manager VOA and PHC/HPLC Laboratories, '04– '09

Oversee daily operation of the Volatiles GC/MS and PHC/HPLC laboratories. Responsibilities include organizing and prioritizing workload, initiating process improvements, training and development of staff and working wit PCs on client specific project requirements. Responsible for analytical duties as listed below for Scientist IV. Other responsibilities include ensuring compliance with CAS QA protocols and assisting staff with troubleshooting equipment and procedural problems.

Columbia Analytical Services, Inc.
Kelso, WA
Scientist IV, VOA Laboratory, '99 - '04

Perform sample analysis and data review for EPA methods 524.2, 624 and 8260. Duties also include Project Management.

Columbia Analytical Services, Inc.
Kelso, WA
Project Chemist, Supervisor Pesticides GC Laboratory, '98 - '99

Primary responsibilities included workload scheduling, data review, instrument maintenance and troubleshooting, and personnel training and evaluation. Also responsible for supervision of extraction personnel and instrument analysts.

Columbia Analytical Services, Inc.
Kelso, WA
Analyst, SVOC GC Lab '92 - '98

Primary responsibilities included analysis of samples using GC and HPLC techniques, report generation, data review, preparation of analytical standards, maintenance of instrumentation, Client Services and some Project Management. Routine duties included analysis of soil and water samples for pesticides, PCBs, CLP Pesticides, Explosives and PAHs using EPA methods.

Columbia Analytical Services, Inc.
Kelso, WA
Analyst, Organic Extractions Lab, '91 – '92

. Responsibilities included extraction of soil and water samples for various SVOCs, and TCLP extraction of SVOC and VOC compounds using TCLP equipment. Other duties included performing cleanup procedures, validation studies, MDL studies, and the training of employees in advanced extraction procedures and techniques..

UNCONTROLLED COPY

Loren E. Portwood

1317 S. 13th Avenue | Kelso, WA 98626 | +1 360 577 7222

Technical Manager I, Semivolatile Organics Laboratory

2011 – Present

Responsible for the overall operation and supervision of the Semivolatile Organics Gas chromatograph department. Perform method development.

Previous Experience

ALS Group USA Corp.
Kelso, WA
Columbia Analytical Services, Inc.
Kelso, WA

Technical Manager I, DW Laboratory, '08 – '13

Responsible for management of the Semivolatile 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.

Columbia Analytical Services, Inc.
Kelso, WA

Technical Manager I, DW Laboratory, '08 – '11

Plan, conduct, and, as lead analyst, supervise analyses using advanced instrumentation such as HPLC with post column derivatization, GC/MS, and GC/ECD. 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

Columbia Analytical Services, Inc.
Kelso, WA

Lead Analyst DW Laboratory, , '02 – '08

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..

Columbia Analytical Services, Inc.
Kelso, WA

Petroleum Hydrocarbons Supervisor, , '98 – '02

Analysis, reporting, and archiving of water, soil, and product samples for semi-volatile petroleum hydrocarbons and miscellaneous FID tests. Methods include EPA methods 8100, 8310, 8315, 8330, 8040, 8015 and various state modifications of 8015 (OR, WA, CA, AK). Additional analyses include solvent scans, alcohols, glycols, and EPA methods 413.2 and 418.1. Other responsibilities include sample preparation and instrument maintenance

Columbia Analytical Services, Inc.
Kelso, WA

Petroleum Hydrocarbons, '93 – '98

SVOC sample preparations for water, soil, and oil to be analyzed in the GC, GC/MS, and PH Departments. These extraction methods included hazardous waste, wastewater, and drinking water procedures. Other responsibilities included extract cleanup via Florisil®, GPC, and Hg.

Treclen Laboratories
Spokane, WA

Bench Chemist, Extractions, '92 – '93

Inorganic water and soil testing and sample preparation by EPA methods.

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

UNCONTROLLED COPY

Lester "Les" Kennedy

1317 S. 13th Avenue • Kelso, WA 98626 • +1 360 577 7222

Education

Lower Columbia College, Longview, WA
Coursework, general Studies, 1988 - 1990

Portland Bible College
Portland, OR
Batchelor ofTheology, 2009

Support Services Manager/Sample Management Manager

2010 - Present

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

Columbia Analytical Services, Inc.
Kelso, WA
Project Manager '99 -'11
SMO Supervisor, '06 -'11

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.

Columbia Analytical Services, Inc.
Kelso, WA
Supervisor Organic Extractions Laboratory, '97-'99

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.

Columbia Analytical Services, Inc.
Kelso, WA
Senior Analyst, GC/MS Laboratory, '96-'97

Primary duties were performing analyses by EPA Method 8270, SIM TCL. SIM PAH, including all steps in the data review and reporting process.

Columbia Analytical Services, Inc.
Kelso, WA
Senior Analyst, Organic Extractions Laboratory, '93-'96

Primary responsibilities include managing workload; 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.

Columbia Analytical Services, Inc.
Kelso, WA
Analyst, Organic Extractions Laboratory, '91-'93

Duties primarily as listed above

UNCONTROLLED COPY

Jeffery D. Christian

1317 S. 13th Avenue | Kelso, WA 98626 | +1 360 577 7222

Director of Operation, Western USA

2011 – Present

Responsible for oversight of operating units In the territory designated Western reporting to the COO. Primary responsibilities include establishment of consistent quality, technical, and client service enhancements across the group, as well as the financial performance of the individual operating units. In addition, a significant role is to represent operations as a member of the management team consisting of the Directors of Operations of other territories, Laboratory Directors for all locations, and senior management of the North America Environmental Division of ALS USA.

Previous Experience

Columbia Analytical Services, Inc.
Kelso, WA
Chief Operating Officer/Vice President – '10 to '11

Responsible for oversight of operating units of Columbia Analytical Services, Inc. with all Laboratory Directors reporting to the COO. Primary responsibilities include establishment of consistent quality, technical, and client service enhancements across the company, as well as the financial performance of the individual operating units. In addition, a significant role is to represent operations as a member of the Senior Management Team (SMT) consisting of the Chief Executive Officer, Chief Financial Officer, Chief Quality Officer, and the Director of Information Technology.

Columbia Analytical Services, Inc.
Kelso, WA
Vice President/Kelso Laboratory Director '93–'10

Responsible for all phases of laboratory operations, including project planning, budgeting, and quality assurance.

Columbia Analytical Services, Inc.
Kelso, WA
Operations Manager, Kelso Laboratory '92–'93

. Responsibilities included directing the daily operation of the Kelso laboratory. Other responsibilities and duties included functioning as a technical consultant to clients, providing assistance in developing and planning analytical schemes to match client objectives, and writing and developing analytical procedures/methods. Also, served as Project Manager for State of Alaska Department of Environmental Conservation contract and Coordinator for EPA Special Analytical Services (SAS) contracts.. Always leave an extra space after this paragraph to separate from the next job.

Columbia Analytical Services, Inc.
Kelso, WA
Project Chemist & Manager, Metals Analysis Lab, '89–'92

Responsible for directing the daily operation of the Metals Laboratory, including the sample preparation, AAS, ICP-OES, and ICP-MS Laboratories

Weyerhaeuser Technology Center,
Federal Way, WA
Scientist '86–'89

Responsibilities included supervising atomic spectroscopy laboratory which included flame and furnace AAS, ICP-OES, and sample preparation capabilities to handle a wide variety of sample types. Interfaced with internal and external clients to provide technical support. Wrote and developed analytical procedures/methods.

Weyerhaeuser Technology Center,
Federal Way, WA
Lead Technician, Metals Lab '81–'86

Responsibilities included primary ICP and AAS analyst for EPA-CLP contract work. Extensive experience in wide variety of environmental and product-related testing.

ITT Rayonier, Olympic Research Division,
Shelton, WA
Research Assistant, '78–'81

Responsibilities included performing water quality tests, product-related analytical tests, corrosion tests operated pilot equipment specific to the pulp and paper ind

Education

Evergreen State College – Olympia, WA
BS in Chemistry 1993

Coursework, *Pacific Lutheran University, Tacoma, WA*. 1988-1989.
Coursework, *Tacoma Community College, Tacoma, WA* 1970-1971, 1988-1989.
CERTIFICATION, Chemistry, *L.H. Bates Technical, Tacoma, WA*, 1976-1978.
Coursework, *Central Washington University, Ellensburg, WA*. 1969-1970.
Numerous Training/Educational Activities via Conferences, Professional Seminars, and Factory Training, 1989-2010

Publications

Mr. Christian has a number of publications and presentations. For a list of these publications and presentations, please contact ALS

UNCONTROLLED COPY

APPROVED SIGNATORIES FOR FINAL ANALYTICAL REPORTS

ALS Environmental, Kelso, WA

CHRISTIAN, JEFF
CORONADO, JEFFREY
DEGNER, CARL
GRINDSTAFF, JEFF
HOLMES, HOWARD
JACKY, HARVEY
JAMES, JON
JUELL, AMANDA
KENNEDY, LES
LEAF, CHRIS
MALLOCH, JANET
MIHAI-LAZAR, CARMEN
MOORE, RACHEL
SALATA, GREGORY
SAMY, SHAR
SCHROEDER, COLLEEN

Update: May, 2015 Approved by: Gregory Salata, Client Services Manager

UNCONTROLLED COPY

APPENDIX C

ALS Environmental Confidentiality Agreement

UNCONTROLLED COPY

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

UNCONTROLLED COPY

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

UNCONTROLLED COPY

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.

UNCONTROLLED COPY

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: ______________________________________

UNCONTROLLED COPY

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

UNCONTROLLED COPY

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,OHOUS, 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 Parr 6300 Isoparabolic	 1987 2005	 LM LM	 4 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 (3): COD (2) Kjeldahl, Lachat 46-place (1)	 1989 1999	 LM LM	 4 3
Dissolved Oxygen Meter - YSI Model 58 (2)	1988, 1991	LM	4
Distillation apparatus (Midi) - Easy Still (2)	1996, 2000	LM	5
Drying Ovens (12): Shel-Lab and VWR models	1990-2010	LM	13
Air Drying Cabinets	2011	LM	-
Flash Point Tester (1): Petroleum Systems Services	2005	LM	3
Flow-Injection Analyzers (2): Bran-Leubbe Lachat 8500	 2002 2007	 LM LM	 2 2
Ion Chromatographs (4) Dionex DX-120 with Peaknet Data System Dionex ICS-2500 with Chromchem Data System Dionex ICS-2000 with Chromchem Data System Dionex ICS-1600 with Chromchem Data System	 1998 2002 2006 2009	 LM LM LM LM	 3 3 3 3
Meters (ISE and pH) (4) Fisher Scientific Accument Model 50 Fisher Scientific Accument Model 25 Fisher Scientific Accument Model 20 Fisher Scientific Accument Model AR25	 1997 1993 2000 1992	 LM LM LM LM	 4 4 4 4

UNCONTROLLED COPY

Microscope - Olympus	1988	LM	1
Muffle Furnace- Sybron Thermolyne Model F-A1730	1991	LM	13
Shatter Box (2): GP 1000 SPEX 8530	1989 2011	LM	5
Sieve Shakers (2): CE Tyler - Portable RX 24 WS Tyler - RX 86	 1990 1991	 LM LM	 5 5
Thomas-Wiley Laboratory Mill, Model 4	1989	LM	5
Total Organic Carbon (TOC) Analyzers (4) Coulemetrics Model 5012 OI 1010 Teledyne Tekmar Fusion 1 Analytik Jena 2500	 1997 2000 2009 2013	 LM LM LM LM	 3 3 3 3
Total Organic Halogen (TOX) Analyzers (2): Mitsubishi TOX-100	2001	LM	2
Turbidimeter - Hach Model 2100N	1996	LM	5
UV-Visible Spectrophotometers (4): SpectraMax 384 Plus Beckman-Coulter DU520 Perkin Elmer Lambda 25 Abrazix	 2009 2005 2008 2011	 LM LM LM LM	 4 4 4 2
Discrete Autoanlayzer –Westco SmartChem AD20-1	2011	LM	2
Vacuum Pumps (3): Welch Duo-Seal Model 1376 Busch R-5 Series Single Stage Chem Star 1402N-01	 1990 1991 2011	LM	13
Water Baths/Incubators (5): Various Fisher Scientific and VWR Models	1986 - 2009	LM	13
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 Various Mettler, Sartorius, and Ohaus models	1988-2010	MM	12
Atomic Absorption Spectrophotometers (4): Varian SpectrAA Zeeman/220 AA Perkin Elmer AAnalyst 200 Flame AA CETAC Mercury Analyzer M-6100	 2000 2005 2010	 LM MM MM	 2 2 2
Buck AA Spectrophotometer Model 205	2008	LM	2

UNCONTROLLED COPY

Atomic Fluorescence Spectrophotometer Brooks-Rand Model III (1)	2005	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 Thermo Scientific Model iCAP 6500	 2007 2012	 MM MM	 3 3
Inductively Coupled Plasma Mass Spectrometers (ICP-MS) (3): Agilent 7700 Thermo X-Series Nexion Model 300D	 2014 2006 2011	 MM MM MM	 2 2 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 OHaus EP613	 1999 - 2011	 MM	 15
Centrifuge – Sorvall GLC-1 (2)	2014	LM	15
Drying Ovens (2) Fisher Model 655G VWR Model 1305U	 1991 1999	 LM LM	 15 15
Evaporators/concentrators Organomation N-Evap (6) Organomation S-Evap (8) Biotage Turbovap (2)	 1990-2010 1990-2010 2013	 LM LM LM	 15 15 15
Extractor Heaters: Lab-Line Multi-Unit for Soxhlet and Continuous Liquid-Liquid Extractions (90)	1987-2007	LM	9
Solids Extractors: Sonic Bath VWR Sonic Horn (4) Soxhtherm Gerhardt (2) OI Analytical (5)	 1994 1994 2000 2008	 LM LM LM LM LM	 6 6 6 6

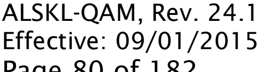
UNCONTROLLED COPY

Extractors, TCLP (8):			
Millipore TCLP Zero Headspace Extractors (20)	1992-2011	LM	2
TCLP 12 position Extractor/Tumbler (2)	1989-2011	LM	2
Gel Permeation Chromatography (GPC) (3)			
J2 Scientific AccuPrep (2)	2005, 2010	LM	4
Gilson (1)	2013	LM	4
Muffle Furnace (2)	2006, 2009	LM	4
Solid Phase Extractors (18) – Horizon SPE-Dex 4790	2003, 2006,2008	LM	4
Microwave Extractor – Mars 6	2014	LM	2
GC SEMIVOLATILE ORGANICS INSTRUMENT LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Gas Chromatographs (18):			
Hewlett-Packard 5890 GC with HP 7673 Autosampler and Dual ECD Detectors	1995	LM	6
Agilent 6890 GC with Agilent 7683 Autosampler and Dual ECD Detectors (6)	2001, 2005, 2007,2011	LM	6
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	6
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	6
Agilent 7890A Dual NPD Detectors and Agilent 7683B autosampler	2012	LM	3
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
Thremo 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	6

UNCONTROLLED COPY

Semivolatile GC/MS Systems (11): Agilent 6890/5973 with ATAS Optic2 LVI and HP 7673 Autosampler (2) Agilent 5890/5970 and HP 7673 Autosampler Agilent 5890/5972 with ATAS Optic2 LVI and HP 7673 Autosampler (2) Agilent 6890/5973 with ATAS Optic3 LVI and HP 7683 Autosampler Agilent 6890/5973 with Agilent PTV Injector and 7683 Autosampler Agilent7890A/5975C with Agilent 7693 Autosampler (4)	1997, 2001 1990 1993, 1994 2005 2007 2010 - 2011	LM LM LM LM LM LM	6 6 6 6 6 6
Semivolatile GC/MS/MS – Waters Quattro Micro GC Micromass with Agilent 6890, Agilent PTV Injector, 7683B Autosampler	2008	MM	2
HPLC LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balance - Mettler BB240	1994	MM	6
Drying Oven - Fisher Model 630F	1991	LM	5
Evaporator – Turbo Vap	2009	LM	6
Centrifuge (2) Beckman Coulter Eppendorf	 2002 2012	 LM LM	 6 6
High-Performance Liquid Chromatographs (3): Agilent 1260 Infinity with Diode Array UV Detector	2011	LM	4
High-Performance LC/MS (3) Spectrometer - Thermo Electron TSQ Vantage LC/MS/MS and autosampler API 5000 LC/MS/MS and SIL-20AC autosampler AB Sciex 5500 and Schimadzu DGU 20A5	 2005 2008 2011	 MM MM MM	 2 4 4
Agilent 1100 HPLC -UV/Fluorescence detector	2003	LM	3
VOLATILE ORGANICS LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balance - Mettler PE 160	1989	MM	5
Fisher Vortex Mixer	1989	LM	5

UNCONTROLLED COPY



Drying Ovens (1): Boekel 107801	 1989	 LM	 5
Sonic Water Bath - Branson Model 2200	1989	LM	5
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 6890/5973 Tekmar 3000 Purge and Trap Concentrator Varian Archon 5100 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	 1989 1995 1996 2001 2001 2001 2005 2005 2005 2007 2007 2007 2010, 2011 2010, 2011 2013 2013 2013 2014 2014 2014	 LM LM LM LM LM LM LM LM LM LM LM LM LM LM LM LM LM LM LM LM	 5 5 5 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4 4
Agilent 7890 GC with FID Encon Evolution Purge and Trap Concentrator Encon Centurion Autosampler	2013	LM	3
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 M5155 printers.	2010 - 2015	LM	NA
Approximately 220+ Dell/HP PC workstations running Windows XP/Windows 7 on LAN connected via 100BT/1GigE network	2010 - 2015	LM	NA

UNCONTROLLED COPY

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
Thruput, MARRS, Stealth, Harold, Blackbird, EDDGE, CASLIMS, & LabCoat reporting software systems.	1998 - 2014	LM	NA
Data processing terminals (79) - Enviroquant, Target, Saturn, MassHunter, Chromeleon	1996 - 2014	LM	NA

UNCONTROLLED COPY

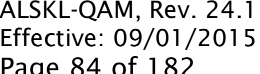
APPENDIX F – Containers, Preservation and Holding Times

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

UNCONTROLLED COPY

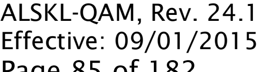
DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	HOLDING TIME
Ferrous Iron (ALS SOP)	W, D	G Amber	Cool, 4˚C	24 hours
Fluoride (EPA 300.0, 9056, SM 4500 F-C)	W, S	P,G	Cool, 4˚C	28 days
Formaldehyde (ASTM D6303)	W	G Amber	Cool, 4˚C	48 hours
Hardness (SM 2340C)	W, DW	P,G	HNO_3 to pH<2	6 months
Hydrogen 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_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

UNCONTROLLED COPY



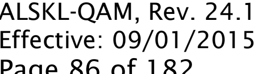
DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	HOLDING TIME
Phenolics, Total (EPA 420.1, 9056)	W, S	G Amber	Cool, 4˚C, H_2SO_4 to pH<4	28 days
Phosphorus, Total (EPA 365.3)	W	P,G	Cool, 4˚C, H_2SO_4 to pH<2	28 days
Residue, Filterable (TDS) (SM 2540C)	W	P,G	Cool, 4˚C	7 days
Residue, Nonfilterable (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 < 4ºC	28 days
Chromium VI (EPA 7195/7191)	W	P,G	Cool, 4˚C	24 hours

UNCONTROLLED COPY



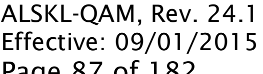
DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	HOLDING TIME
Mercury (1631E)	W	F	Cool, 4˚C, HCl or H_2SO_4 to pH<2	90 days
Mercury (1631E)	S	F	Freeze < -15ºC	1 Yr
Mercury (7471)	S	P,G	Cool, 4˚C	28 days
Mercury (EPA 245.1, 7470, 7471)	W, DW	P,G	HNO_3 to pH<2	28 days
Metals (200.7, 200.8, 200.9, 6010, 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, 4ºC, 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

UNCONTROLLED COPY



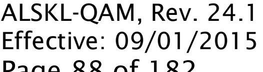
DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	HOLDING TIME
Purgeable Aromatic Hydrocarbons (including BTEX and MTBE 624, 8260)	W	G, Teflon-Lined,Septum Cap, No Headspace	Residual Chlorine Present: 10% $Na_2S_2O_3$,, HCl to pH<2, Cool 4°C	14 days
Purgeable Aromatic Hydrocarbons (including BTEX 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				
Nonyl Phenols	W	G, Teflon-Lined Cap	H_2SO_4 to pH<2, Cool, 4°C	28 days
Organotins (CAS 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

UNCONTROLLED COPY



DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	HOLDING TIME
PBDE/PBB – ROHS GC/MS	W, S, T	G	Cool, 4˚C	40 days after extraction
Pharma Personal Care Products 1694	W, S	Amber G, Teflon-Lined Cap	Cool, < 6˚C	7[f] days until extraction; 30 days after extraction
Nitroaromatics and Nitramines 8330B	W, S	G, Teflon-Lined Cap	Cool, 4˚C	S 14, W 7 days until extraction; 40 days after extraction
Nitroaromatics/Nitroamines HPLC/MS/MS	W, S, T	G	Cool, 4˚C Tissues < -10 C	S 14, W 7 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˚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[f] days until extraction; 40 days after extraction
Base/Neutral Extractable Semivolatile Organics (EPA 8270)	S	G, Teflon-Lined Cap	Cool, 4˚C[g]	14[f] days 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˚C[g]	30 days until extraction; 30 days after extraction

UNCONTROLLED COPY



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 , <6ºC	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 4ºC	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 4ºC	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

UNCONTROLLED COPY

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.

UNCONTROLLED COPY

APPENDIX G – Standard Operating Procedures

Corporate General and Quality Assurance SOPs

SOP TITLE	SOP ID	Revision
Laboratory Ethics and Data Integrity	CE-GEN001	2.00
(proprietary– client specific)	CE-GEN002	1.00
Records Management Policy	CE-GEN003	1.00
Preventive Action	CE-GEN004	1.00
Document Control	CE-GEN005	1.00
Data Recall	CE-GEN006	0.00
Procurement and Control of Laboratory Services and Supplies	CE-GEN007	0.00
Method Development	CE-GEN008	0.00
Establishing Standard Operating Procedures	CE-GEN009	0.00
Handling Customer Feedback	CE-GEN010	0.00
Assigning and TSR to a Project	CE-GEN011	0.00
Policy for the Use of Accreditation Organization Names, Symbols, and Logos	CE-GEN012	0.00
(proprietary – client specific)	CE-GEN013	0.00
(proprietary– client specific)	CE-GEN014	0.00
Internal Audits	CE-QA001	1.00
Manual Integration Policy	CE-QA002	1.00
Training Policy	CE-QA003	1.00
Qualification of Subcontract Laboratories	CE-QA004	2.00
Laboratory Management Review	CE-QA005	1.00
Proficiency Testing Sample Analysis	CE-QA006	1.00
Making Entries onto Analytical Records	CE-QA007	1.00
Nonconformance and Corrective Action	CE-QA008	1.00
Control Limits	CE-QA009	1.00
Estimation of Uncertainty of Analytical Measurements	CE-QA010	0.00
Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation	CE-QA011	0.00
Quality of Reagents and Standards	CE-QA012	0.00

UNCONTROLLED COPY

LABORATORY SOPs

SOP TITLE	SOP ID	Revision
DATA ARCHIVING	ADM-ARCH	6
DOCUMENTING LABORATORY BALANCE AND TEMPERATURE CHECKS	ADM-BAL	6
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
SOFTWARE QUALITY ASSURANCE AND DATA SECURITY	ADM-SWQADATA	0
ALS KELSO TRAINING PROCEDURE	ADM-TRAIN	2
CHECKING VOLUMETRIC LABWARE	ADM-VOLWARE	4
SOP FOR WISCONSIN PROJECTS LABORATORY PRACTICES AND PROJECT MANAGEMENT, WI ADMINISTRATIVE CODE, CHAPTER NR 149	ADM-WISC	1
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

UNCONTROLLED COPY

SEPARATORY FUNNEL LIQUID-LIQUID EXTRACTION	EXT-3510	11
CONTINUOUS LIQUID - LIQUID EXTRACTION	EXT-3520	16
SOLID PHASE EXTRACTION	EXT-3535	6
SOXHLET EXTRACTION	EXT-3540	11
AUTOMATED SOXHLET EXTRACTION	EXT-3541	10
ULTRASONIC EXTRACTION	EXT-3550	10
WASTE DILUTION EXTRACTION	EXT-3580	6
SILICA GEL CLEANUP	EXT-3630	5
GEL PERMEATION CHROMATOGRAPHY	EXT-3640A	8
REMOVAL OF SULFUR USING COPPER	EXT-3660	7
REMOVAL OF SULFUR USING MERCURY	EXT-3660M	3
SULFURIC ACID CLEANUP	EXT-3665	6
CARBON CLEANUP	EXT-CARCU	4
DIAZOMETHANE PREPARATION	EXT-DIAZ	6
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	3
FACILITY AND LABORATORY CLEANING	FAC-CLEAN	2
OPERATION AND MAINTENANCE OF LABORATORY REAGENT WATER SYSTEMS	FAC-WATER	2
FLASHPOINT DETERMINATION - SETAFLASH	GEN-1020	7
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	7
SETTEABLE SOLIDS	GEN-160.5	5
HALIDES, ADSORBABLE ORGANIC (AOX)	GEN-1650	4

UNCONTROLLED COPY

GRAVIMETRIC DETERMINATION OF HEXANE EXTRACTABLE MATERIAL (1664)	GEN-1664	9
ALKALINITY TOTAL	GEN-2320	9
HARDNESS, TOTAL	GEN-2340	8
DETERMINATION OF INORGANIC ANIONS IN DRINKING WATER BY ION CHROMATOGRAPHY	GEN-300.1	8
ACIDITY	GEN-305.2	4
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	10
NITRATE/NITRITE, NITRITE BY FLOW INJECTION ANALYSIS	GEN-353.2	9
PHOSPHORUS DETERMINATION USING COLORMETRIC PROCEDURE	GEN-365.3	12
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
HALIDES, EXTRACTABLE ORGANIC (EOX)	GEN-9020M	4
TOTAL SULFIDES BY METHYLENE BLUE DETERMINATION	GEN-9030	10

UNCONTROLLED COPY

TOTAL HALIDES BY OXIDATIVE COMBUSTION AND MICROCOULOMETRY	GEN-9076	2
TOTAL CARBON IN SOIL	GEN-ASTM	9
AUTOFLUFF	GEN-AUTOFLU	2
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	12
STANDARD TEST METHODS FOR DETERMINING SEDIMENT CONCENTRATION IN WATER SAMPLES	GEN-D3977	0
CARBONATE (CO3) BY EVOLUTION AND COLUMETRIC TITRATION	GEN-D513-82M	1
SULFIDE, SOLUBLE DETERMINATION OF SOLUBLE SULFIDE IN SEDIMENT	GEN-DIS.S2	3
BULK DENSITY OF SOLID WASTE FRACTIONS	GEN-E1109	1
FDA EXTRACTABLES	GEN-FDAEX	2
FERROUS IRON IN WATER	GEN-FeII	4
FLUORIDE BY ION SELECTIVE ELECTRODE	GEN-FISE	9
FORMALDEHYDE COLORIMETRIC DETERMINATION	GEN-FORM	3
HYDROGEN PEROXIDE BY PERMANGANATE TITRATION	GEN-H2O2	2
HYDROGEN HALIDES BY ION CHROMATOGTRAPHY (METHOD 26)	GEN-HA26	3
HYDAZINE IN WATER USING COLORIMETRIC PROCEDURE	GEN-HYD	2
TOTAL SULFUR FOR ION CHROMATOGRAPHY	GEN-ICS	2
ION CHROMATOGRAPHY	GEN-IONC	17
COLOR, NCASI	GEN-NCAS	3
NITROCELLULOSE IN SOIL	GEN-NCEL	1
OXYGEN CONSUMPTION RATE	GEN-O2RATE	1
CARBON, TOTAL ORGANIC DETERMINATION (WALKELY BLACK METHOD)	GEN-OSU	3

UNCONTROLLED COPY

Ph IN SOIL AND SOLIDS	GEN-Phs	13
Ph IN WATER	GEN-Phw	13
PARTICLE SIZE DETERMINATION - ASTM PROCEDURE	GEN-PSASTM	2
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	1
SUBSAMPLING AND COMPOSITING OF SAMPLES	GEN-SUBS	6
SOLIDS, TOTAL DISSOLVED (TDS)	GEN-TDS	11
THIOCYANATE	GEN-THIOCN	2
NITROGEN, TOTAL AND SOLUBLE KJELDAHL	GEN-TKN	14
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	11
TURBIDITY MEASUREMENT	GEN-TURB	6
GLASSWASHING FOR INORGANIC ANALYSES	GEN-WASH	4
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	2
DETERMINATION OF HORMONES IN DRINKING WATER BY SOLID PHASE EXTRACTION AND LIQUID CHROMATOGRAPHY ELECTROSPRAY IONIZATION	LCP-539	2
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/Tandam 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	4
Acrylamide by High Performance Liquid Chromatography/tandem mass spectrometry (HPLC/ms/ms)..	LCP-ACRYL	2
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-	LCP-LCMS4	2
NITROGUANIDINE BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY	LCP-NITG	7

UNCONTROLLED COPY

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	0
PERFLUORINATED COMPOUNDS BY HPLC/MS/MS	LCP-PFC	4
DETERMINATION OF PHTHALATES IN FOOD BY LIQUID CHROMATOGRAPHY TANDEM MASS SPECTROMETRY (LC/MSMS)	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	13
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	12
METALS DIGESTION	MET-3020A	15
METALS DIGESTION	MET-3050B	14
CLOSED VESSEL OIL DIGESTION	MET-3051M	3
CLOSED VESSEL DIGESTION OF SILICEOUS AND ORGANICALLY BASED MATRICIES	MET-3052M	1
DETERMINATION OF METALS & TRACE ELEMENTS BY INDUCTIVELY COUPLED PLASMA-MS (METHOD 6020)	MET-6020	16
ARSENIC BY BOROHYDRIDE REDUCTION ATOMIC ABSORPTION	MET-7062	4
METALS DIGESTION FOR HEXAVALENT CHROMIUM	MET-7195	9
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
BIOACCESSIBILITY OF METALS IN SOIL AND SOLID WASTE	MET-BIOACC	1
METALS DIGESTION OF AQUEOUS SAMPLES	MET-DIG	15
SAMPLE FILTRATION FOR METALS ANALYSIS	MET-FILT	4
METALS LABORATORY GLASSWARE CLEANING	MET-GC	5
DETERMINATION OF TRACE METALS BY GRAPHITE FURNACE ATOMIC ABSORPTION SPECTROMETRY (GFAA)	MET-GFAA	21

UNCONTROLLED COPY

DETERMINATION OF METALS AND TRACE ELEMENTS BY ICP/AES	MET-ICP	25
DETERMINATION OF METALS & TRACE ELEMENTS BY INDUCTIVELY COUPLED PLASMA-MS (METHOD 200.8)	MET-ICPMS	16
TRACE METALS IN WATER BY PRECONCENTRATION USING REDUCTIVE PRECIPITATION FOLLOWED BY ICP-MS	MET-RPMS	7
METALS AND SEMIVOLATILES SPLP EXTRACTION (EPA METHOD 1312)	MET-SPLP	1
WASTE EXTRACTION TEST (WET) PROCEDURE (STLC) for NONVOLATILE and SEMIVOLATILE PARAMETERS	MET-STLC	2
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	4
TISSUE SAMPLE PREPARATION	MET-TISP	9
ANALYSIS OF WATER AND SOLID SAMPLES FOR ALIPHATIC HYDROCARBONS	PET-ALIPHAT	2
GASOLINE RANGE ORGANICS BY GAS CHROMATOGRAPHY	PET-GRO	10
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
BOTTLE ORDER PREPARATION AND SHIPPING	SMO-BORD	16
SAMPLE DISPOSAL	SMO-DISP	12
FOREIGN SOILS HANDLING TREATMENT	SMO-FSHT	11
SAMPLE RECEIVING	SMO-GEN	31
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	0
1,2-DIBROMOETHANE (EDB) AND 1,2-DIBROMO-3-CHLORO-PROPANE (DBCP) IN SOLIDS BY MICROEXTRACTION AND GAS CHROMATOGRAPHY	SOC-8011S	0
GLYCOLS	SOC-8015	11
ORGANOCHLORINE PESTICIDES BY GAS CHROMATOGRAPHY: CAPILLARY COLUMN TECHNIQUE	SOC-8081	18
PCBS AS AROCLORS	SOC-8082Ar	16
CONGENER-SPECIFIC DETERMINATION OF PCBS BY GC/ECD	SOC-8082Co	13
DETERMINATION OF NITROGEN OR PHOSPHORUS CONTAINING PESTICIDES	SOC-8141	13
CHLORINATED HERBICIDES	SOC-8151	16
CHLORINATED PHENOLS METHOD 8151 MODIFIED	SOC-8151M	11
METHANOL IN PROCESS LIQUIDS AND STATIONARY SOURCE EMISSIONS	SOC-9403	8

UNCONTROLLED COPY

HAZARDOUS AIR POLLUTANTS (HAPS) IN PULP AND PAPER INDUSTRY CONDENSATES	SOC-9901	5
HAPS AND OTHER COMPOUNDS IN IMPINGER/CANISTER SAMPLES FROM WOOD PRODUCTS FACILITIES	SOC-9902	4
ALCOHOLS	SOC-ALC	2
BUTYLTINS	SOC-BUTYL	13
CALIBRATION OF INSTRUMENTS FOR ORGANICS CHROMATOGRAPHIC ANALYSES	SOC-CAL	9
CONFIRMATION PROCEDURE FOR GC AND HPLC ANALYSES	SOC-CONF	6
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	10
ORGANOCHLORINE PESTICIDES AND PCBS IN DRINKING WATER	SVD-508_1	8
CHLORINATED HEBICIDES IN DRINKING WATER	SVD-515.4	10
N-NITROSAMINES BY GC/MS/MS	SVD-521	6
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS (METHOD 525.2)	SVD-525	9
ENDOTHALL IN DRINKING WATER BY GC/MS	SVD-548	10
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
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS - METHOD 8270D	SVM-8270D	4
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS - LOW LEVEL PROCEDURE	SVM-8270L	9
POLYNUCLEAR AROMATIC HYDROCARBONS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY SIM	SVM-8270P	9
Quantifying and Reporting Alkylated Homologs of Polycyclic Aromatic Hydrocarbons for Gulf Oil Spill Analyses	SVM-8270PQAH	0
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS SELECTED ION MONITORING	SVM-8270S	7
QUANTITATIVE GEOCHEMICAL BIOMARKERS BY GC/MS SELECTIVE ION MONITORING	SVM-BIO	1
OCTAMETHYLCYCLOTETRASILOXANE (D4) IN AQUEOUS SAMPLES BY GC/MS	SVM-D4AQ	0
OCTAMETHYLCYCLOTETRASILOXANE (D4) IN SEDIMENTS AND BIOSOLIDS BY GC/MS	SVM-D4SO	0
OCTAMETHYLCYCLOTETRASILOXANE (D4) IN BIOLOGICAL MATRICES BY GC/MS	SVM-D4TI	0
NONYLPHENOLS ISOMERS AND NONYLPHENOL ETHOXYLATES	SVM-NONYL	5

UNCONTROLLED COPY

ORGANOPHOSPHOROUS PESTICIDES BY GC/MS/MS	SVM-OPPMS2	2
CHLORINATED PESTICIDES BY GC/MS/MS	SVM-PESTMS2	4
POLYBROMINATED DIPHENYL ETHERS (PBDEs) AND POLYBROMINATED BIPHENYLS (PBBs) BY GC/MS	SVM-ROHS	2
DIMP	SVM-SIM	0
1,2,3-TRICHLOROPROPANE BY ISOTOPE DILUTION-GC/MS SIM	SVM-TCP	0
PURGE AND TRAP FOR AQUEOUS SAMPLES	VOC-5030	9
PURGE AND TRAP/EXTRACTION FOR VOC IN SOIL AND WASTE SAMPLES , CLOSED SYSTEM	VOC-5035	10
VOLATILE ORGANIC COMPOUNDS BY GC/MS	VOC-524.2	16
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	18
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
ZERO HEADSPACE EXTRACTION (EPA METHOD 1311)	VOC-ZHE	8

UNCONTROLLED COPY

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.

UNCONTROLLED COPY

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.

UNCONTROLLED COPY

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 20XX.rX.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

UNCONTROLLED COPY

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
Michigan DEQ	9949
Minnesota DOH	053-999-457
Montana DPHHS	CERT0047
Nevada DEP	WA012762015-3
New Jersey DEP	WA005
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
Wisconsin DNR	998386840
Wyoming (EPA Region8)	-
Miscellaneous	
Foreign Soil Permit	USDA
Plant Import Permit	USDA
Controlled Substances Permit	US DEA
Controlled Substances Permit	WA DOH

UNCONTROLLED COPY

Oregon

Environmental Laboratory Accreditation Program

Department of Agriculture, Laboratory Division
Department of Environmental Quality, Laboratory Division
Oregon Health Authority, Public Health Division

NELAP Recognized

ORELAP Fields of Accreditation

ORELAP ID: WA100010
EPA CODE: WA01276
Certificate: WA100010 - 010

ALS Environmental, Kelso
1317 South 13th Ave.
Kelso WA 98626

Issue Date: 02/11/2015 ***Expiration Date:*** 02/10/2016

As of 02/11/2015 ***this list supercedes all previous lists for this certificate number.***
Customers. Please verify the current accreditation standing with ORELAP.

MATRIX : Biological Tissue

Reference	Code	Description
ALS Kelso LCP-PFC 4	**60001505**	**ALS Kelso - Perfluorinated Compounds by HPLC-MS-MS**

Analyte Code	Analyte
6911	Perfluorobutane Sulfonate (PFBS)
9562	Perfluorodecane Sulfonate (PFDS)
6905	Perfluorodecanoic acid (PFDA)
6903	Perfluorododecanoic (PFDDA)
6908	Perfluoroheptanoic acid (PFHA)
6910	Perfluorohexane Sulfonate (PFHS)
6913	Perfluorohexanoic acid (PFHXA)
6906	Perfluorononanoic acid (PFNA)
6912	Perfluorooctanoic acid
6909	Perfluorooctanoic Sulfonate (PFOS)
6914	Perfluoropentanoic acid (PFPEA)
6904	Perfluoroundecanoic acid (PFUDA)

Reference	Code	Description
CAS SOC-Butyl	**60035009**	**Butyltin by GC/Flame Photometric Detector**

Analyte Code	Analyte
1201	Butyltin trichloride
1202	Dibutyltin dichloride
1209	Tetrabutyltin
1203	Tributyltin chloride

Reference	Code	Description
EPA 1631E	**10237204**	**Mercury in Water by Oxidation, Purge & Trap, and Cold Vapor Atomic Fluorescence**

Analyte Code	Analyte
1095	Mercury

Reference	Code	Description
EPA 1632A	**10123407**	**Arsenic in Water by Gaseous Hydride Atomic Absorption**

Analyte Code	Analyte
1010	Arsenic
1012	Arsenite (As+3)
6138	Dimethylarsinic acid (DMA)
1207	Monomethylarsonic acid (MMA)

Reference	Code	Description
EPA 3540C	**10140202**	**Soxhlet Extraction**

Analyte Code	Analyte
8031	Extraction/Preparation

UNCONTROLLED COPY

ORELAP Fields of Accreditation

ORELAP ID: WA100010
EPA CODE: WA01276
Certificate: WA100010 - 010

ALS Environmental, Kelso

1317 South 13th Ave.
Kelso WA 98626

Issue Date: 02/11/2015 ***Expiration Date:*** 02/10/2016

As of 02/11/2015 ***this list supercedes all previous lists for this certificate number.***
Customers. Please verify the current accreditation standing with ORELAP.

EPA 3541 **10140406** **Automated Soxhlet Extraction**

Analyte Code	Analyte
8031	Extraction/Preparation

EPA 3630C **10146802** **Silica gel cleanup**

Analyte Code	Analyte
8031	Extraction/Preparation

EPA 3640A **10147203** **Gel Preparation Cleanup**

Analyte Code	Analyte
8031	Extraction/Preparation

EPA 365.3 **10070607** **Phosphorous - Colorimetric, two reagent.**

Analyte Code	Analyte
1908	Total Phosphate

EPA 3660B **10148400** **Sulfur cleanup**

Analyte Code	Analyte
8031	Extraction/Preparation

EPA 3665A **10148808** **Sulfuric Acid / permanganate Cleanup**

Analyte Code	Analyte
8031	Extraction/Preparation

EPA 5035A **10284807** **Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples**

Analyte Code	Analyte
8031	Extraction/Preparation

EPA 6010C **10155803** **ICP - AES**

Analyte Code	Analyte
1000	Aluminum
1005	Antimony
1010	Arsenic
1015	Barium
1020	Beryllium
1025	Boron
1030	Cadmium
1040	Chromium
1050	Cobalt
1055	Copper
1070	Iron
1075	Lead
1090	Manganese
1100	Molybdenum
1105	Nickel
1140	Selenium
1150	Silver
1175	Tin
1185	Vanadium
1190	Zinc

UNCONTROLLED COPY

ORELAP Fields of Accreditation

ORELAP ID: WA100010
EPA CODE: WA01276
Certificate: WA100010 - 010

ALS Environmental, Kelso
1317 South 13th Ave.
Kelso WA 98626

Issue Date: 02/11/2015 **Expiration Date:** 02/10/2016

As of 02/11/2015 ***this list supercedes all previous lists for this certificate number.***
Customers. Please verify the current accreditation standing with ORELAP.

EPA 6020A **10156408** **Inductively Coupled Plasma-Mass Spectrometry**

Analyte Code	Analyte
1000	Aluminum
1005	Antimony
1010	Arsenic
1015	Barium
1020	Beryllium
1030	Cadmium
1040	Chromium
1050	Cobalt
1055	Copper
1070	Iron
1075	Lead
1090	Manganese
1100	Molybdenum
1105	Nickel
1140	Selenium
1150	Silver
1165	Thallium
1185	Vanadium
1190	Zinc

EPA 7196A **10162400** **Chromium Hexavalent colorimetric**

Analyte Code	Analyte
1045	Chromium VI

EPA 7471B **10166402** **Mercury by Cold Vapor Atomic Absorption**

Analyte Code	Analyte
1095	Mercury

EPA 7742 **10169207** **Selenium by Borohydride Reduction and Atomic Absorption**

Analyte Code	Analyte
1140	Selenium

EPA 8081B **10178800** **Organochlorine Pesticides by GC/ECD**

Analyte Code	Analyte
8580	2,4'-DDD
8585	2,4'-DDE
8590	2,4'-DDT
7355	4,4'-DDD
7360	4,4'-DDE
7365	4,4'-DDT
7005	Alachlor
7025	Aldrin
7110	alpha-BHC (alpha-Hexachlorocyclohexane)
7240	alpha-Chlordane
7115	beta-BHC (beta-Hexachlorocyclohexane)
7250	Chlordane (tech.)
7300	Chlorpyrifos
7925	cis-Nonachlor
7105	delta-BHC
7470	Dieldrin
7510	Endosulfan I
7515	Endosulfan II
7520	Endosulfan sulfate
7540	Endrin

UNCONTROLLED COPY

Analyte Code	Analyte
7530	Endrin aldehyde
7535	Endrin ketone
7120	gamma-BHC (Lindane, gamma-HexachlorocyclohexanE)
7245	gamma-Chlordane
7685	Heptachlor
7690	Heptachlor epoxide
6275	Hexachlorobenzene
4835	Hexachlorobutadiene
7725	Isodrin
7810	Methoxychlor
7870	Mirex
3890	Oxychlordane
8250	Toxaphene (Chlorinated camphene)

EPA 8082A **10179201** **Polychlorinated Biphenyls (PCBs) by GC/ECD**

Analyte Code	Analyte
9095	2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (BZ-206)
9090	2,2',3,3',4,4',5,5'-Octachlorobiphenyl (BZ-194)
9103	2,2',3,3',4,4',5,6-Octachlorobiphenyl (BZ-195)
9065	2,2',3,3',4,4',5-Heptachlorobiphenyl (BZ-170)
9020	2,2',3,3',4,4'-Hexachlorobiphenyl (BZ-128)
9112	2,2',3,3',4,5',6,6'-Octachlorobiphenyl (BZ-201)
9116	2,2',3,3',4,5,6'-Heptachlorobiphenyl (BZ-174)
9114	2,2',3,3',4,5',6'-Heptachlorobiphenyl (BZ-177)
9120	2,2',3,3',4,6'-Hexachlorobiphenyl (BZ-132)
9133	2,2',3,4,4',5,5',6-Octachlorobiphenyl (BZ-203)
9134	2,2',3,4,4',5,5'-Heptachlorobiphenyl (BZ-180)
9075	2,2',3,4,4',5',6-Heptachlorobiphenyl (BZ-183)
9025	2,2',3,4,4',5'-Hexachlorobiphenyl (BZ-138)
9139	2,2',3,4,4',6,6'-Heptachlorobiphenyl (BZ-184)
9080	2,2',3,4',5,5',6-Heptachlorobiphenyl (BZ-187)
9030	2,2',3,4,5,5'-Hexachlorobiphenyl (BZ-141)
9151	2,2',3,4',5',6-Hexachlorobiphenyl (BZ-149)
8975	2,2',3,4,5'-Pentachlorobiphenyl (BZ-87)
9155	2,2',3,4',5-Pentachlorobiphenyl (BZ-90)
9154	2,2',3,4',5'-Pentachlorobiphenyl (BZ-97)
9035	2,2',3,5,5',6-Hexachlorobiphenyl (BZ-151)
9166	2,2',3,5',6-Pentachlorobiphenyl (BZ-95)
8945	2,2',3,5'-Tetrachlorobiphenyl (BZ-44)
9040	2,2',4,4',5,5'-Hexachlorobiphenyl (BZ-153)
9174	2,2',4,4',5,6'-Hexachlorobiphenyl (BZ-154)
9175	2,2',4,4',5-Pentachlorobiphenyl (BZ-99)
8980	2,2',4,5,5'-Pentachlorobiphenyl (BZ-101)
8950	2,2',4,5'-Tetrachlorobiphenyl (BZ-49)
8955	2,2',5,5'-Tetrachlorobiphenyl (BZ-52)
8930	2,2',5-Trichlorobiphenyl (BZ-18)
9085	2,3,3',4,4',5,5'-Heptachlorobiphenyl (BZ-189)
9050	2,3,3',4,4',5-Hexachlorobiphenyl (BZ-156)
9045	2,3,3',4,4',5'-Hexachlorobiphenyl (BZ-157)
9193	2,3,3',4,4',6-Hexachlorobiphenyl (BZ-158)
8985	2,3,3',4,4'-Pentachlorobiphenyl (BZ-105)
8990	2,3,3',4',6-Pentachlorobiphenyl (BZ-110)
9207	2,3,3',4'-Tetrachlorobiphenyl (BZ-56)
9055	2,3',4,4',5,5'-Hexachlorobiphenyl (BZ-167)
9218	2,3',4,4',5',6-Hexachlorobiphenyl (BZ-168)
9005	2,3,4,4',5-Pentachlorobiphenyl (BZ-114)
8995	2,3',4,4',5-Pentachlorobiphenyl (BZ-118)
9000	2,3',4,4',5'-Pentachlorobiphenyl (BZ-123)
9220	2,3',4,4',6-Pentachlorobiphenyl (BZ-119)
9221	2,3,4,4'-Tetrachlorobiphenyl (BZ-60)

UNCONTROLLED COPY

Analyte Code	Analyte
8960	2,3',4,4'-Tetrachlorobiphenyl (BZ-66)
9230	2,3',4',5-Tetrachlorobiphenyl (BZ-70)
9239	2,3',4'-Trichlorobiphenyl (BZ-33)
8920	2,3-Dichlorobiphenyl (BZ-5)
9250	2,4,4',5-Tetrachlorobiphenyl (BZ-74)
9252	2,4,4'-Trichlorobiphenyl (BZ-28)
8940	2,4',5-Trichlorobiphenyl (BZ-31)
8915	2-Chlorobiphenyl (BZ-1)
9060	3,3',4,4',5,5'-Hexachlorobiphenyl (BZ-169)
9015	3,3',4,4',5-Pentachlorobiphenyl (BZ-126)
8965	3,3',4,4'-Tetrachlorobiphenyl (BZ-77)
8970	3,4,4',5-Tetrachlorobiphenyl (BZ-81)
9266	3,4,4'-Trichlorobiphenyl (BZ-37)
8880	Aroclor-1016 (PCB-1016)
8885	Aroclor-1221 (PCB-1221)
8890	Aroclor-1232 (PCB-1232)
8895	Aroclor-1242 (PCB-1242)
8900	Aroclor-1248 (PCB-1248)
8905	Aroclor-1254 (PCB-1254)
8910	Aroclor-1260 (PCB-1260)
8912	Aroclor-1262 (PCB-1262)
8913	Aroclor-1268 (PCB-1268)
9105	Decachlorobiphenyl (BZ-209)

EPA 8270D **10186002** **Semivolatile Organic compounds by GC/MS**

Analyte Code	Analyte
5660	4-Bromophenyl phenyl ether (BDE-3)
5562	Azobenzene
5570	Benzaldehyde
5640	Biphenyl
6545	n-Nitrosodi-n-propylamine

EPA 8270D SIM **10242509** **Semivolatile Organic compounds by GC/MS Selective Ion Monitoring**

Analyte Code	Analyte
5155	1,2,4-Trichlorobenzene
4610	1,2-Dichlorobenzene
6221	1,2-Diphenylhydrazine
4615	1,3-Dichlorobenzene
4620	1,4-Dichlorobenzene
6380	1-Methylnaphthalene
9501	1-Methylphenanthrene
6852	2,3,5-Trimethylnaphthalene
6835	2,4,5-Trichlorophenol
6840	2,4,6-Trichlorophenol
6000	2,4-Dichlorophenol
6130	2,4-Dimethylphenol
6175	2,4-Dinitrophenol
6185	2,4-Dinitrotoluene (2,4-DNT)
6188	2,6-Dimethylnaphthalene
6190	2,6-Dinitrotoluene (2,6-DNT)
5795	2-Chloronaphthalene
5800	2-Chlorophenol
6360	2-Methyl-4,6-dinitrophenol (4,6-Dinitro-2-methylphenol)
6385	2-Methylnaphthalene
6400	2-Methylphenol (o-Cresol)
6460	2-Nitroaniline
6490	2-Nitrophenol
6412	3 & 4 Methylphenol
5660	4-Bromophenyl phenyl ether (BDE-3)

UNCONTROLLED COPY

Analyte Code	Analyte
5700	4-Chloro-3-methylphenol
5825	4-Chlorophenyl phenylether
6410	4-Methylphenol (p-Cresol)
6470	4-Nitroaniline
6500	4-Nitrophenol
5500	Acenaphthene
5505	Acenaphthylene
5555	Anthracene
5575	Benzo(a)anthracene
5580	Benzo(a)pyrene
5605	Benzo(e)pyrene
5590	Benzo(g,h,i)perylene
9309	Benzo(j)fluoranthene
5600	Benzo(k)fluoranthene
5585	Benzo[b]fluoranthene
5630	Benzyl alcohol
5640	Biphenyl
5760	bis(2-Chloroethoxy)methane
5765	bis(2-Chloroethyl) ether
5780	bis(2-Chloroisopropyl) ether
5670	Butyl benzyl phthalate
5680	Carbazole
5855	Chrysene
6065	Di(2-ethylhexyl) phthalate (bis(2-Ethylhexyl)phthalate, DEHP)
5895	Dibenz(a,h) anthracene
5905	Dibenzofuran
6070	Diethyl phthalate
6135	Dimethyl phthalate
5925	Di-n-butyl phthalate
6200	Di-n-octyl phthalate
6265	Fluoranthene
6270	Fluorene
6275	Hexachlorobenzene
4835	Hexachlorobutadiene
4840	Hexachloroethane
6315	Indeno(1,2,3-cd) pyrene
6320	Isophorone
5005	Naphthalene
5015	Nitrobenzene
6525	n-Nitrosodiethylamine
6545	n-Nitrosodi-n-propylamine
6535	n-Nitrosodiphenylamine
6605	Pentachlorophenol
6608	Perylene
6615	Phenanthrene
6625	Phenol
6665	Pyrene

EPA 8330B **10308006** **Nitroaromatics, Nitramines and Nitrate Esters by High Performance Liquid Chromatography (HPLC)**

Analyte Code	Analyte
6885	1,3,5-Trinitrobenzene (1,3,5-TNB)
6160	1,3-Dinitrobenzene (1,3-DNB)
9651	2,4,6-Trinitrotoluene (2,4,6-TNT)
6185	2,4-Dinitrotoluene (2,4-DNT)
6190	2,6-Dinitrotoluene (2,6-DNT)
9303	2-Amino-4,6-dinitrotoluene (2-am-dnt)
9507	2-Nitrotoluene
6150	3,5-Dinitroaniline
9510	3-Nitrotoluene
9306	4-Amino-2,6-dinitrotoluene (4-am-dnt)

UNCONTROLLED COPY

ORELAP Fields of Accreditation

ORELAP ID: WA100010
EPA CODE: WA01276
Certificate: WA100010 - 010

ALS Environmental, Kelso
1317 South 13th Ave.
Kelso WA 98626

Issue Date: 02/11/2015 ***Expiration Date:*** 02/10/2016

As of 02/11/2015 ***this list supercedes all previous lists for this certificate number.***
Customers. Please verify the current accreditation standing with ORELAP.

Analyte Code	Analyte
9513	4-Nitrotoluene
6415	Methyl-2,4,6-trinitrophenylnitramine (tetryl)
5015	Nitrobenzene
6485	Nitroglycerin
9522	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)
9558	Pentaerythritoltetranitrate
9432	RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine)

NELAP RECOGNIZED TNI ACCREDITATION BODY

UNCONTROLLED COPY

ORELAP Fields of Accreditation

ORELAP ID: WA100010
EPA CODE: WA01276
Certificate: WA100010 - 010

ALS Environmental, Kelso

1317 South 13th Ave.
Kelso WA 98626

Issue Date: 02/11/2015 **Expiration Date:** 02/10/2016

As of 02/11/2015 ***this list supercedes all previous lists for this certificate number.***
Customers. Please verify the current accreditation standing with ORELAP.

MATRIX : Drinking Water

Reference	Code	Description
EPA 180.1	**10011402**	**Turbidity - Nephelometric**

Analyte Code	Analyte
2055	Turbidity

Reference	Code	Description
EPA 200.7 4.4	**10013806**	**ICP - metals**

Analyte Code	Analyte
1000	Aluminum
1005	Antimony
1015	Barium
1020	Beryllium
1025	Boron
1030	Cadmium
1035	Calcium
1040	Chromium
1055	Copper
1760	Hardness (calc.)
1070	Iron
1085	Magnesium
1090	Manganese
1100	Molybdenum
1105	Nickel
1125	Potassium
1990	Silica as SiO_2
1150	Silver
1155	Sodium
1185	Vanadium
1190	Zinc

Reference	Code	Description
EPA 200.8 5.4	**10014605**	**Metals by ICP-MS**

Analyte Code	Analyte
1000	Aluminum
1005	Antimony
1010	Arsenic
1015	Barium
1020	Beryllium
1030	Cadmium
1040	Chromium
1055	Copper
1075	Lead
1090	Manganese
1105	Nickel
1140	Selenium
1150	Silver
1165	Thallium

Reference	Code	Description
EPA 245.1 3	**10036609**	**Mercury by Cold Vapor Atomic Absorption**

Analyte Code	Analyte
1095	Mercury

Reference	Code	Description
EPA 300.0 2.1	**10053200**	**Methods for the Determination of Inorganic Substances in Environmental Samples**

Analyte Code	Analyte
1575	Chloride
1730	Fluoride

UNCONTROLLED COPY

Analyte Code	Analyte
1810	Nitrate as N
1820	Nitrate-nitrite
1840	Nitrite as N
2000	Sulfate

EPA 300.1 **10053608** **Ion chromatography - anions.**

Analyte Code	Analyte
1535	Bromate
1540	Bromide
1570	Chlorate
1595	Chlorite

EPA 314.0 **10055400** **Perchlorate in Drinking Water by Ion Chromatography**

Analyte Code	Analyte
1895	Perchlorate

EPA 335.4 **10061208** **Methods for the Determination of Inorganic Substances in Environmental Samples**

Analyte Code	Analyte
1645	Total cyanide

EPA 353.2 2 **10067604** **Nitrate/Nitrite Nitrogen - Automated, Cadmium**

Analyte Code	Analyte
1810	Nitrate as N
1840	Nitrite as N
1825	Total nitrate+nitrite

EPA 504.1 **10082607** **EDB/DBCP/TCP micro-extraction, GC/ECD**

Analyte Code	Analyte
5180	1,2,3-Trichloropropane
4570	1,2-Dibromo-3-chloropropane (DBCP)
4585	1,2-Dibromoethane (EDB, Ethylene dibromide)

EPA 508.1 2 **10086405** **Chlorinated Pesticides, Herbicides, and Organohalides, liquid/solid extraction by GC/ECD**

Analyte Code	Analyte
7355	4,4'-DDD
7360	4,4'-DDE
7365	4,4'-DDT
7025	Aldrin
7110	alpha-BHC (alpha-Hexachlorocyclohexane)
7240	alpha-Chlordane
7115	beta-BHC (beta-Hexachlorocyclohexane)
7250	Chlordane (tech.)
7105	delta-BHC
7470	Dieldrin
7510	Endosulfan I
7515	Endosulfan II
7520	Endosulfan sulfate
7540	Endrin
7530	Endrin aldehyde
7535	Endrin ketone
7120	gamma-BHC (Lindane, gamma-HexachlorocyclohexanE)
7245	gamma-Chlordane
7685	Heptachlor
7690	Heptachlor epoxide

UNCONTROLLED COPY

Analyte Code	Analyte
7810	Methoxychlor
8870	PCBs
8250	Toxaphene (Chlorinated camphene)

EPA 515.4 1 **10088503** **Chlorinated acids Liquid/Solid and GC/ECD**

Analyte Code	Analyte
8655	2,4,5-T
8545	2,4-D
8560	2,4-DB
8600	3,5-Dichlorobenzoic acid
6500	4-Nitrophenol
8505	Acifluorfen
8530	Bentazon
8540	Chloramben
8555	Dalapon
8570	DCPA di acid degradate
8595	Dicamba
8605	Dichloroprop (Dichlorprop)
8620	Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)
6605	Pentachlorophenol
8645	Picloram
8650	Silvex (2,4,5-TP)

EPA 524.2 4.1 **10088809** **Volatile Organic Compounds GC/MS Capillary Column**

Analyte Code	Analyte
5105	1,1,1,2-Tetrachloroethane
5160	1,1,1-Trichloroethane
5110	1,1,2,2-Tetrachloroethane
5165	1,1,2-Trichloroethane
4630	1,1-Dichloroethane
4640	1,1-Dichloroethylene
4670	1,1-Dichloropropene
5150	1,2,3-Trichlorobenzene
5180	1,2,3-Trichloropropane
5155	1,2,4-Trichlorobenzene
5210	1,2,4-Trimethylbenzene
4570	1,2-Dibromo-3-chloropropane (DBCP)
4610	1,2-Dichlorobenzene
4635	1,2-Dichloroethane (Ethylene dichloride)
4655	1,2-Dichloropropane
5215	1,3,5-Trimethylbenzene
4615	1,3-Dichlorobenzene
4660	1,3-Dichloropropane
4620	1,4-Dichlorobenzene
4665	2,2-Dichloropropane
4535	2-Chlorotoluene
4540	4-Chlorotoluene
4910	4-Isopropyltoluene (p-Cymene)
4375	Benzene
4385	Bromobenzene
4390	Bromochloromethane
4395	Bromodichloromethane
4397	Bromoethane (Ethyl Bromide)
4400	Bromoform
4455	Carbon tetrachloride
4475	Chlorobenzene
4575	Chlorodibromomethane
4485	Chloroethane (Ethyl chloride)
4505	Chloroform

UNCONTROLLED COPY

ALS Environmental, Kelso

1317 South 13th Ave.
Kelso WA 98626

Issue Date: 02/11/2015 **Expiration Date:** 02/10/2016

As of 02/11/2015 ***this list supercedes all previous lists for this certificate number.***
Customers. Please verify the current accreditation standing with ORELAP.

Analyte Code	Analyte
4645	cis-1,2-Dichloroethylene
4680	cis-1,3-Dichloropropene
4595	Dibromomethane (Methylene bromide)
4625	Dichlorodifluoromethane (Freon-12)
4765	Ethylbenzene
4835	Hexachlorobutadiene
4900	Isopropylbenzene
5240	m+p-xylene
4950	Methyl bromide (Bromomethane)
4960	Methyl chloride (Chloromethane)
5000	Methyl tert-butyl ether (MTBE)
4975	Methylene chloride (Dichloromethane)
5005	Naphthalene
4435	n-Butylbenzene
5090	n-Propylbenzene
5250	o-Xylene
4440	sec-Butylbenzene
5100	Styrene
4445	tert-Butylbenzene
5115	Tetrachloroethylene (Perchloroethylene)
5140	Toluene
5205	Total trihalomethanes
4700	trans-1,2-Dichloroethylene
4685	trans-1,3-Dichloropropylene
5170	Trichloroethene (Trichloroethylene)
5175	Trichlorofluoromethane (Fluorotrichloromethane, Freon 11)
5235	Vinyl chloride
5260	Xylene (total)

EPA 525.2 2 **10090003** **Semi-Volatile by SPE extraction and GC/MS**

Analyte Code	Analyte
6185	2,4-Dinitrotoluene (2,4-DNT)
6190	2,6-Dinitrotoluene (2,6-DNT)
4310	Acetochlor
7005	Alachlor
7065	Atrazine
5580	Benzo(a)pyrene
6062	bis(2-Ethylhexyl)adipate
7160	Butachlor
5670	Butyl benzyl phthalate
8550	Dacthal (DCPA)
6065	Di(2-ethylhexyl) phthalate (bis(2-Ethylhexyl)phthalate, DEHP)
6070	Diethyl phthalate
6135	Dimethyl phthalate
5925	Di-n-butyl phthalate
6200	Di-n-octyl phthalate
7555	EPTC (Eptam, s-ethyl-dipropyl thio carbamate)
6275	Hexachlorobenzene
6285	Hexachlorocyclopentadiene
6320	Isophorone
7835	Metolachlor
7845	Metribuzin
7875	Molinate
8045	Propachlor (Ramrod)
8125	Simazine
8180	Terbacil

UNCONTROLLED COPY

EPA 548.1 1 **10092805** **Endothall by Ion Exchange, Methylation and GC/MS**

Analyte Code	*Analyte*
7525	Endothall

EPA 549.2 **10093206** **Diquat/Paraquat, Liquid/Solid Extraction and HPLC/UV**

Analyte Code	*Analyte*
9390	Diquat
9528	Paraquat

EPA 552.2 1 **10095804** **Haloacetic Acid/Dalapon, Liquid/Liquid Extraction, Derivitization and GC/ECD**

Analyte Code	*Analyte*
9312	Bromoacetic acid
9315	Bromochloroacetic acid
9336	Chloroacetic acid
9357	Dibromoacetic acid
9360	Dichloroacetic acid
9414	Total haloacetic acids
9642	Trichloroacetic acid

SM 2120 B 20th ED **20224004** **Color by Visual Comparison**

Analyte Code	*Analyte*
1605	Color

SM 2320 B 20th ED **20045209** **Alkalinity by Titration**

Analyte Code	*Analyte*
1505	Alkalinity as CaCO3

SM 2340 B 20th ED **20046202** **Hardness by calculation**

Analyte Code	*Analyte*
1750	Hardness

SM 2510 B 20th ED **20048208** **Conductivity by Probe**

Analyte Code	*Analyte*
1610	Conductivity

SM 2540 C 20th ED **20050004** **Total Dissolved Solids**

Analyte Code	*Analyte*
1955	Residue-filterable (TDS)

SM 4500-Cl F 20th ED **20080506** **Residual Chlorine by DPD Ferrous Titration**

Analyte Code	*Analyte*
1945	Residual free chlorine

SM 4500-F⁻ C 20th ED **20102005** **Fluoride by Ion Selective Electrode**

Analyte Code	*Analyte*
1730	Fluoride

SM 4500-H+ B 20th ED **20104807** **pH by Probe**

Analyte Code	*Analyte*

UNCONTROLLED COPY

Analyte Code	Analyte
1900	pH

SM 4500-P E 20th ED **20123802** **Phosphorus by Ascorbic Acid Reduction**

Analyte Code	Analyte
1870	Orthophosphate as P

SM 5310 C 20th ED **20138403** **Total Organic Carbon by Persulfate-Ultraviolet Oxidation Method**

Analyte Code	Analyte
2040	Total organic carbon

SM 9215 B (PCA) 20th ED **20181208** **Heterotrophic Plate Count Pour Plate (plate count agar): Heterotrophic Bacteria**

Analyte Code	Analyte
2555	Heterotrophic plate count

SM 9223 B (Colilert-18® Multiple-tube) 20th ED **20229407** **Chromogenic/Fluorogenic Quantitative: Total Coliform and E. coli**

Analyte Code	Analyte
2530	Fecal coliforms

SM 9223 B (Colilert®) 20th ED **20212208** **Chromogenic/Fluorogenic Qualitative (Colilert®): Total Coliform and E. coli**

Analyte Code	Analyte
2525	Escherichia coli
2500	Total coliforms

SM 9223 B (Colilert®-18) 20th ED **20214204** **Chromogenic/Fluorogenic Qualitative (Colilert®-18): Total Coliform and E. coli**

Analyte Code	Analyte
2525	Escherichia coli
2500	Total coliforms

SM 9223 B (Colilert®-18) 21st ED **20214408** **Chromogenic/Fluorogenic Qualitative (Colilert®-18): Total Coliform and E. coli**

Analyte Code	Analyte
2525	Escherichia coli
2500	Total coliforms

UNCONTROLLED COPY

ORELAP Fields of Accreditation

ORELAP ID: WA100010
EPA CODE: WA01276
Certificate: WA100010 - 010

ALS Environmental, Kelso

1317 South 13th Ave.
Kelso WA 98626

Issue Date: 02/11/2015 ***Expiration Date:*** 02/10/2016

As of 02/11/2015 ***this list supercedes all previous lists for this certificate number. Customers. Please verify the current accreditation standing with ORELAP.***

MATRIX : Non-Potable Water

Reference	Code	Description
ALS Kelso LCP-Acryl 1	**60001712**	**ALS Kelso - Acrylamide by HPLC/MS/MS**

Analyte Code	*Analyte*
4330	Acrylamide

Reference	Code	Description
ALS Kelso LCP-PFC 4	**60001505**	**ALS Kelso - Perfluorinated Compounds by HPLC-MS-MS**

Analyte Code	*Analyte*
6911	Perfluorobutane Sulfonate (PFBS)
9562	Perfluorodecane Sulfonate (PFDS)
6905	Perfluorodecanoic acid (PFDA)
6903	Perfluorododecanoic (PFDDA)
6908	Perfluoroheptanoic acid (PFHA)
6910	Perfluorohexane Sulfonate (PFHS)
6913	Perfluorohexanoic acid (PFHXA)
6906	Perfluorononanoic acid (PFNA)
6912	Perfluorooctanoic acid
6909	Perfluorooctanoic Sulfonate (PFOS)
6914	Perfluoropentanoic acid (PFPEA)
6904	Perfluoroundecanoic acid (PFUDA)

Reference	Code	Description
ASTM D1426-08B	**30007397**	**Ammonia by Titration**

Analyte Code	*Analyte*
1515	Ammonia as N

Reference	Code	Description
ASTM D1426-98B	**30023406**	**Ammonia by Titration**

Analyte Code	*Analyte*
1515	Ammonia as N

Reference	Code	Description
ASTM D3590-02(06)A	**30016819**	**Total Kjeldahl Nitrogen in Water**

Analyte Code	*Analyte*
1795	Kjeldahl nitrogen - total

Reference	Code	Description
ASTM D3590-89B	**30016809**	**Total Kjeldahl Nitrogen in Water**

Analyte Code	*Analyte*
1795	Kjeldahl nitrogen - total

Reference	Code	Description
ASTM D4129 05	**30018907**	**Total and Organic Carbon in Water by High Temperature Oxidation and by Coulometric Detection**

Analyte Code	*Analyte*
2040	Total organic carbon

Reference	Code	Description
CAS PestMS2 (1699 modified) 2	**60035101**	**Chlorinated Pesticides by GC/MS/MS**

Analyte Code	*Analyte*
8580	2,4'-DDD
8585	2,4'-DDE
8590	2,4'-DDT
7355	4,4'-DDD
7360	4,4'-DDE
7365	4,4'-DDT
7025	Aldrin
7110	alpha-BHC (alpha-Hexachlorocyclohexane)

UNCONTROLLED COPY

Analyte Code	Analyte
7240	alpha-Chlordane
7115	beta-BHC (beta-Hexachlorocyclohexane)
7300	Chlorpyrifos
7925	cis-Nonachlor
7105	delta-BHC
7470	Dieldrin
7510	Endosulfan I
7515	Endosulfan II
7520	Endosulfan sulfate
7540	Endrin
7530	Endrin aldehyde
7535	Endrin ketone
7120	gamma-BHC (Lindane, gamma-HexachlorocyclohexanE)
7245	gamma-Chlordane
7685	Heptachlor
7690	Heptachlor epoxide
6275	Hexachlorobenzene
7725	Isodrin
7810	Methoxychlor
7870	Mirex
5553	Octachlorostyrene
3890	Oxychlordane
7910	trans-Nanochlor

CAS SOC-Butyl **60035009** **Butyltin by GC/Flame Photometric Detector**

Analyte Code	Analyte
1201	Butyltin trichloride
1202	Dibutyltin dichloride
1209	Tetrabutyltin
1203	Tributyltin chloride

Enterolert® **60030208** **Chromogenic/Fluorogenic Quantitative (Enterolert®): Enterococci**

Analyte Code	Analyte
2520	Enterococci

EPA 1020A **10117007** **Ignitability Setaflash Closed-cup Method**

Analyte Code	Analyte
1780	Ignitability

EPA 160.4 **10256801** **Total Volatile Solids, ignition @ 550 C.**

Analyte Code	Analyte
4075	Vol. residue, density, water & solids content of coatings

EPA 1630 **10122608** **Methyl Mercury by Purge & Trap Cold Vapor Atomic Fluorescence Spectrometry**

Analyte Code	Analyte
1205	Methyl Mercury

EPA 1631E **10237204** **Mercury in Water by Oxidation, Purge & Trap, and Cold Vapor Atomic Fluorescence**

Analyte Code	Analyte
1095	Mercury

EPA 1632A **10123407** **Arsenic in Water by Gaseous Hydride Atomic Absorption**

Analyte Code	Analyte

UNCONTROLLED COPY

ORELAP Fields of Accreditation

ORELAP ID: WA100010
EPA CODE: WA01276
Certificate: WA100010 - 010

ALS Environmental, Kelso
1317 South 13th Ave.
Kelso WA 98626

Issue Date: 02/11/2015 ***Expiration Date:*** 02/10/2016

As of 02/11/2015 ***this list supercedes all previous lists for this certificate number.***
Customers. Please verify the current accreditation standing with ORELAP.

Analyte Code	Analyte
1010	Arsenic
1012	Arsenite (As+3)
6138	Dimethylarsinic acid (DMA)
1207	Monomethylarsonic acid (MMA)

EPA 1650C **10125005** **Adsorbable Organic Halides by Adsorption and Coulometric Titration**

Analyte Code	Analyte
4345	Adsorbable organic halogens (AOX)

EPA 1653A **10125403** **Chlorinated Phenolics by "In Situ" Acetylation and GC/MS**

Analyte Code	Analyte
6735	2,3,4,6-Tetrachlorophenol
6835	2,4,5-Trichlorophenol
6840	2,4,6-Trichlorophenol
6805	3,4,5-Trichlorocatechol
6815	3,4,5-Trichloroguaiacol
6810	3,4,6-Trichlorocatechol
6820	3,4,6-Trichloroguaiacol
6825	4,5,6-Trichloroguaiacol
6605	Pentachlorophenol
6720	Tetrachlorocatechol
6725	Tetrachloroguaiacol
6875	Trichlorosyringol

EPA 1664A (HEM) **10127807** **N-Hexane Extractable Material (Oil and Grease) by Extraction and Gravimetry**

Analyte Code	Analyte
1803	n-Hexane Extractable Material (O&G)
1860	Oil & Grease

EPA 1694 1.0 **10132908** **Pharmaceuticals and Personal Care Products by HPLC/MS/MS**

Analyte Code	Analyte
6769	17a-estradiol
6771	17a-ethynylestradiol
6773	17ß-estradiol
4307	Acetominophen
7052	Androstenedione
7065	Atrazine
9301	Bisphenol A
5675	Caffeine
7194	Carbamazepine
7375	DEET
7086	Diazepam
7087	Diclofenac
6075	Diethylstilbestrol
7253	Estriol
7254	Estrone
7257	Fluoxetine
7258	Gemfibrozil
7219	Hydrocodone
7259	Ibuprofen
7719	Iopromide
7313	Meprobamate
7316	Methadone
7269	Naproxen
7317	Oxybenzone
7318	Pentoxifylline

NELAP RECOGNIZED TNI ACCREDITATION BODY
UNCONTROLLED COPY

Analyte Code	Analyte
7284	Progesterone
9585	Salicylic acid
7297	Sulfamethoxazole
7301	Testosterone
7304	Triclosan
7307	Trimethoprim

EPA 180.1 **10011402** **Turbidity - Nephelometric**

Analyte Code	Analyte
2055	Turbidity

EPA 200.7 4.4 **10013806** **ICP - metals**

Analyte Code	Analyte
1000	Aluminum
1005	Antimony
1010	Arsenic
1015	Barium
1020	Beryllium
1025	Boron
1030	Cadmium
1035	Calcium
1040	Chromium
1055	Copper
1760	Hardness (calc.)
1070	Iron
1075	Lead
1085	Magnesium
1090	Manganese
1100	Molybdenum
1105	Nickel
1125	Potassium
1140	Selenium
1990	Silica as SiO2
1150	Silver
1155	Sodium
1160	Strontium
1175	Tin
1180	Titanium
1185	Vanadium
1190	Zinc

EPA 200.8 5.4 **10014605** **Metals by ICP-MS**

Analyte Code	Analyte
1000	Aluminum
1005	Antimony
1010	Arsenic
1015	Barium
1020	Beryllium
1030	Cadmium
1040	Chromium
1050	Cobalt
1055	Copper
1070	Iron
1075	Lead
1090	Manganese
1100	Molybdenum
1105	Nickel

UNCONTROLLED COPY

Analyte Code	Analyte
1140	Selenium
1150	Silver
1165	Thallium
3035	Uranium
1185	Vanadium
1190	Zinc

EPA 200.9 2.2 **10015404** **Metals by Graphite Atomic Absorption**

Analyte Code	Analyte
1010	Arsenic
1040	Chromium
1075	Lead
1140	Selenium
1165	Thallium

EPA 245.1 3 **10036609** **Mercury by Cold Vapor Atomic Absorption**

Analyte Code	Analyte
1095	Mercury

EPA 300.0 2.1 **10053200** **Methods for the Determination of Inorganic Substances in Environmental Samples**

Analyte Code	Analyte
1540	Bromide
1575	Chloride
1730	Fluoride
1810	Nitrate as N
1820	Nitrate-nitrite
1840	Nitrite as N
2000	Sulfate

EPA 3005A **10133207** **Acid Digestion of waters for Total Recoverable or Dissolved Metals**

Analyte Code	Analyte
8031	Extraction/Preparation

EPA 3010A **10133605** **Acid Digestion of Aqueous samples and Extracts for Total Metals**

Analyte Code	Analyte
8031	Extraction/Preparation

EPA 3020A **10134404** **Acid Digestion of Aqueous samples and Extracts for Total Metals for Analysis by GFAA**

Analyte Code	Analyte
8031	Extraction/Preparation

EPA 314.0 **10055400** **Perchlorate in Drinking Water by Ion Chromatography**

Analyte Code	Analyte
1895	Perchlorate

EPA 330.4 **10059004** **Residual Chlorine - DPD-FAS Titration**

Analyte Code	Analyte
1940	Total residual chlorine

EPA 335.4 **10061208** **Methods for the Determination of Inorganic Substances in Environmental Samples**

Analyte Code	Analyte

UNCONTROLLED COPY

Analyte Code	Analyte
1645	Total cyanide

EPA 3510C **10138202** **Separatory Funnel Liquid-liquid extraction**

Analyte Code	Analyte
8031	Extraction/Preparation

EPA 3520C **10139001** **Continuous Liquid-liquid extraction**

Analyte Code	Analyte
8031	Extraction/Preparation

EPA 353.2 2 **10067604** **Nitrate/Nitrite Nitrogen - Automated, Cadmium**

Analyte Code	Analyte
1810	Nitrate as N
1820	Nitrate-nitrite
1840	Nitrite as N
1825	Total nitrate+nitrite

EPA 3535A **10139409** **Solid-Phase Extraction (SPE)**

Analyte Code	Analyte
8031	Extraction/Preparation

EPA 3610B **10144602** **Alumina Cleanup**

Analyte Code	Analyte
8031	Extraction/Preparation

EPA 3620C **10146006** **Florisil Cleanup**

Analyte Code	Analyte
8031	Extraction/Preparation

EPA 3630C **10146802** **Silica gel cleanup**

Analyte Code	Analyte
8031	Extraction/Preparation

EPA 3640A **10147203** **Gel Preparation Cleanup**

Analyte Code	Analyte
8031	Extraction/Preparation

EPA 365.3 **10070607** **Phosphorous - Colorimetric, two reagent.**

Analyte Code	Analyte
1870	Orthophosphate as P
1908	Total Phosphate

EPA 3660B **10148400** **Sulfur cleanup**

Analyte Code	Analyte
8031	Extraction/Preparation

EPA 3665A **10148808** **Sulfuric Acid / permanganate Cleanup**

Analyte Code	Analyte
8031	Extraction/Preparation

UNCONTROLLED COPY

ORELAP Fields of Accreditation

ORELAP ID: WA100010
EPA CODE: WA01276
Certificate: WA100010 - 010

ALS Environmental, Kelso

1317 South 13th Ave.
Kelso WA 98626

Issue Date: 02/11/2015 **Expiration Date:** 02/10/2016

As of 02/11/2015 ***this list supercedes all previous lists for this certificate number. Customers. Please verify the current accreditation standing with ORELAP.***

EPA 420.1 **10079206** **Phenolics - Spectrophotometric, manual.**

Analyte Code	Analyte
1905	Total phenolics

EPA 5030B **10153409** **Purge and trap for aqueous samples**

Analyte Code	Analyte
8031	Extraction/Preparation

EPA 6010C **10155803** **ICP - AES**

Analyte Code	Analyte
1000	Aluminum
1005	Antimony
1010	Arsenic
1015	Barium
1020	Beryllium
1025	Boron
1030	Cadmium
1035	Calcium
1040	Chromium
1050	Cobalt
1055	Copper
1070	Iron
1075	Lead
1085	Magnesium
1090	Manganese
1100	Molybdenum
1105	Nickel
1125	Potassium
1140	Selenium
1150	Silver
1155	Sodium
1160	Strontium
1165	Thallium
1175	Tin
1180	Titanium
1185	Vanadium
1190	Zinc

EPA 6020A **10156408** **Inductively Coupled Plasma-Mass Spectrometry**

Analyte Code	Analyte
1000	Aluminum
1005	Antimony
1010	Arsenic
1015	Barium
1020	Beryllium
1030	Cadmium
1040	Chromium
1050	Cobalt
1055	Copper
1070	Iron
1075	Lead
1090	Manganese
1100	Molybdenum
1105	Nickel
1140	Selenium
1150	Silver
1160	Strontium

NELAP RECOGNIZED TNI ACCREDITATION BODY

UNCONTROLLED COPY

Analyte Code	Analyte
1165	Thallium
3035	Uranium
1185	Vanadium
1190	Zinc

EPA 608 **10103603** **Organochlorine Pesticides & PCBs by GC/ECD**

Analyte Code	Analyte
7355	4,4'-DDD
7360	4,4'-DDE
7365	4,4'-DDT
7025	Aldrin
7110	alpha-BHC (alpha-Hexachlorocyclohexane)
8880	Aroclor-1016 (PCB-1016)
8885	Aroclor-1221 (PCB-1221)
8890	Aroclor-1232 (PCB-1232)
8895	Aroclor-1242 (PCB-1242)
8900	Aroclor-1248 (PCB-1248)
8905	Aroclor-1254 (PCB-1254)
8910	Aroclor-1260 (PCB-1260)
7115	beta-BHC (beta-Hexachlorocyclohexane)
7250	Chlordane (tech.)
7105	delta-BHC
7470	Dieldrin
7510	Endosulfan I
7515	Endosulfan II
7520	Endosulfan sulfate
7540	Endrin
7530	Endrin aldehyde
7120	gamma-BHC (Lindane, gamma-HexachlorocyclohexanE)
7685	Heptachlor
7690	Heptachlor epoxide
7810	Methoxychlor
8250	Toxaphene (Chlorinated camphene)

EPA 624 **10107207** **Volatile Organic Compounds by purge and trap GC/MS**

Analyte Code	Analyte
5160	1,1,1-Trichloroethane
5110	1,1,2,2-Tetrachloroethane
5165	1,1,2-Trichloroethane
4630	1,1-Dichloroethane
4640	1,1-Dichloroethylene
5155	1,2,4-Trichlorobenzene
4610	1,2-Dichlorobenzene
4635	1,2-Dichloroethane (Ethylene dichloride)
4655	1,2-Dichloropropane
4615	1,3-Dichlorobenzene
4620	1,4-Dichlorobenzene
4500	2-Chloroethyl vinyl ether
4995	4-Methyl-2-pentanone (MIBK)
4325	Acrolein (Propenal)
4340	Acrylonitrile
4375	Benzene
4395	Bromodichloromethane
4400	Bromoform
4455	Carbon tetrachloride
4475	Chlorobenzene
4575	Chlorodibromomethane
4485	Chloroethane (Ethyl chloride)
4505	Chloroform

UNCONTROLLED COPY

Analyte Code	Analyte
4680	cis-1,3-Dichloropropene
4625	Dichlorodifluoromethane (Freon-12)
4765	Ethylbenzene
4950	Methyl bromide (Bromomethane)
4960	Methyl chloride (Chloromethane)
4975	Methylene chloride (Dichloromethane)
5100	Styrene
5115	Tetrachloroethylene (Perchloroethylene)
5140	Toluene
4700	trans-1,2-Dichloroethylene
4685	trans-1,3-Dichloropropylene
5170	Trichloroethene (Trichloroethylene)
5175	Trichlorofluoromethane (Fluorotrichloromethane, Freon 11)
5235	Vinyl chloride
5260	Xylene (total)

EPA 625 **10300002** **Base/Neutrals and Acids by GC/MS**

Analyte Code	Analyte
5155	1,2,4-Trichlorobenzene
4610	1,2-Dichlorobenzene
6221	1,2-Diphenylhydrazine
4615	1,3-Dichlorobenzene
4620	1,4-Dichlorobenzene
6840	2,4,6-Trichlorophenol
6000	2,4-Dichlorophenol
6130	2,4-Dimethylphenol
6175	2,4-Dinitrophenol
6185	2,4-Dinitrotoluene (2,4-DNT)
6190	2,6-Dinitrotoluene (2,6-DNT)
5795	2-Chloronaphthalene
5800	2-Chlorophenol
6360	2-Methyl-4,6-dinitrophenol (4,6-Dinitro-2-methylphenol)
6490	2-Nitrophenol
5945	3,3'-Dichlorobenzidine
5660	4-Bromophenyl phenyl ether
5700	4-Chloro-3-methylphenol
5825	4-Chlorophenyl phenylether
6500	4-Nitrophenol
5500	Acenaphthene
5505	Acenaphthylene
5555	Anthracene
5595	Benzidine
5575	Benzo(a)anthracene
5580	Benzo(a)pyrene
5590	Benzo(g,h,i)perylene
5600	Benzo(k)fluoranthene
5585	Benzo[b]fluoranthene
5760	bis(2-Chloroethoxy)methane
5765	bis(2-Chloroethyl) ether
5780	bis(2-Chloroisopropyl) ether
5670	Butyl benzyl phthalate
5855	Chrysene
6065	Di(2-ethylhexyl) phthalate (bis(2-Ethylhexyl)phthalate, DEHP)
5895	Dibenz(a,h) anthracene
6070	Diethyl phthalate
6135	Dimethyl phthalate
5925	Di-n-butyl phthalate
6200	Di-n-octyl phthalate
6265	Fluoranthene
6270	Fluorene

UNCONTROLLED COPY

Analyte Code	Analyte
6275	Hexachlorobenzene
4835	Hexachlorobutadiene
6285	Hexachlorocyclopentadiene
4840	Hexachloroethane
6315	Indeno(1,2,3-cd) pyrene
6320	Isophorone
5005	Naphthalene
5015	Nitrobenzene
6530	n-Nitrosodimethylamine
6545	n-Nitrosodi-n-propylamine
6535	n-Nitrosodiphenylamine
6605	Pentachlorophenol
6615	Phenanthrene
6625	Phenol
6665	Pyrene

EPA 6850 **10304606** **Perchlorate in Water, Soils and Solid Wastes Using High Performance Liquid Chromatography/Electrospray Ionization/Mass Spectrometry**

Analyte Code	Analyte
1895	Perchlorate

EPA 7010 **10157809** **Metals by Graphite Furnace Atomic Absorption**

Analyte Code	Analyte
1010	Arsenic
1040	Chromium
1075	Lead
1140	Selenium
1165	Thallium

EPA 7062 **10159407** **Antimony and Arsenic by Borohydride Reduction and Atomic Absorption**

Analyte Code	Analyte
1010	Arsenic

EPA 7195 **10162002** **Chromium, Hexavalent (Coprecipitation) by Graphite Furnace Atomic Absorption**

Analyte Code	Analyte
1045	Chromium VI

EPA 7196A **10162400** **Chromium Hexavalent colorimetric**

Analyte Code	Analyte
1045	Chromium VI

EPA 7470A **10165807** **Mercury in Liquid Waste by Cold Vapor Atomic Absorption**

Analyte Code	Analyte
1095	Mercury

EPA 7742 **10169207** **Selenium by Borohydride Reduction and Atomic Absorption**

Analyte Code	Analyte
1140	Selenium

EPA 8015C **10173805** **Non-halogenated organics using GC/FID**

Analyte Code	Analyte
9369	Diesel range organics (DRO)
4785	Ethylene glycol

UNCONTROLLED COPY

Analyte Code	Analyte
9408	Gasoline range organics (GRO)

EPA 8081B **10178800** **Organochlorine Pesticides by GC/ECD**

Analyte Code	Analyte
8580	2,4'-DDD
8585	2,4'-DDE
8590	2,4'-DDT
7355	4,4'-DDD
7360	4,4'-DDE
7365	4,4'-DDT
7005	Alachlor
7025	Aldrin
7110	alpha-BHC (alpha-Hexachlorocyclohexane)
7240	alpha-Chlordane
7115	beta-BHC (beta-Hexachlorocyclohexane)
7250	Chlordane (tech.)
7300	Chlorpyrifos
7925	cis-Nonachlor
7105	delta-BHC
7470	Dieldrin
7510	Endosulfan I
7515	Endosulfan II
7520	Endosulfan sulfate
7540	Endrin
7530	Endrin aldehyde
7535	Endrin ketone
7120	gamma-BHC (Lindane, gamma-HexachlorocyclohexanE)
7245	gamma-Chlordane
7685	Heptachlor
7690	Heptachlor epoxide
6275	Hexachlorobenzene
4835	Hexachlorobutadiene
4840	Hexachloroethane
7725	Isodrin
7810	Methoxychlor
7870	Mirex
3890	Oxychlordane
8250	Toxaphene (Chlorinated camphene)
7910	trans-Nanochlor

EPA 8082A **10179201** **Polychlorinated Biphenyls (PCBs) by GC/ECD**

Analyte Code	Analyte
9095	2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (BZ-206)
9090	2,2',3,3',4,4',5,5'-Octachlorobiphenyl (BZ-194)
9103	2,2',3,3',4,4',5,6-Octachlorobiphenyl (BZ-195)
9065	2,2',3,3',4,4',5-Heptachlorobiphenyl (BZ-170)
9020	2,2',3,3',4,4'-Hexachlorobiphenyl (BZ-128)
9112	2,2',3,3',4,5',6,6'-Octachlorobiphenyl (BZ-201)
9116	2,2',3,3',4,5,6'-Heptachlorobiphenyl (BZ-174)
9114	2,2',3,3',4,5',6'-Heptachlorobiphenyl (BZ-177)
9120	2,2',3,3',4,6'-Hexachlorobiphenyl (BZ-132)
9133	2,2',3,4,4',5,5',6-Octachlorobiphenyl (BZ-203)
9134	2,2',3,4,4',5,5'-Heptachlorobiphenyl (BZ-180)
9075	2,2',3,4,4',5',6-Heptachlorobiphenyl (BZ-183)
9025	2,2',3,4,4',5'-Hexachlorobiphenyl (BZ-138)
9139	2,2',3,4,4',6,6'-Heptachlorobiphenyl (BZ-184)
9080	2,2',3,4',5,5',6-Heptachlorobiphenyl (BZ-187)
9030	2,2',3,4,5,5'-Hexachlorobiphenyl (BZ-141)
9151	2,2',3,4',5',6-Hexachlorobiphenyl (BZ-149)

UNCONTROLLED COPY

Analyte Code	Analyte
8975	2,2',3,4,5'-Pentachlorobiphenyl (BZ-87)
9155	2,2',3,4',5-Pentachlorobiphenyl (BZ-90)
9154	2,2',3,4',5'-Pentachlorobiphenyl (BZ-97)
9035	2,2',3,5,5',6-Hexachlorobiphenyl (BZ-151)
9166	2,2',3,5',6-Pentachlorobiphenyl (BZ-95)
8945	2,2',3,5'-Tetrachlorobiphenyl (BZ-44)
9040	2,2',4,4',5,5'-Hexachlorobiphenyl (BZ-153)
9174	2,2',4,4',5,6'-Hexachlorobiphenyl (BZ-154)
9175	2,2',4,4',5-Pentachlorobiphenyl (BZ-99)
8980	2,2',4,5,5'-Pentachlorobiphenyl (BZ-101)
8950	2,2',4,5'-Tetrachlorobiphenyl (BZ-49)
8955	2,2',5,5'-Tetrachlorobiphenyl (BZ-52)
8930	2,2',5-Trichlorobiphenyl (BZ-18)
9085	2,3,3',4,4',5,5'-Heptachlorobiphenyl (BZ-189)
9050	2,3,3',4,4',5-Hexachlorobiphenyl (BZ-156)
9045	2,3,3',4,4',5'-Hexachlorobiphenyl (BZ-157)
9193	2,3,3',4,4',6-Hexachlorobiphenyl (BZ-158)
8985	2,3,3',4,4'-Pentachlorobiphenyl (BZ-105)
8990	2,3,3',4',6-Pentachlorobiphenyl (BZ-110)
9207	2,3,3',4'-Tetrachlorobiphenyl (BZ-56)
9055	2,3',4,4',5,5'-Hexachlorobiphenyl (BZ-167)
9217	2,3,4,4',5,6-Hexachlorobiphenyl (BZ-166)
9218	2,3',4,4',5',6-Hexachlorobiphenyl (BZ-168)
9005	2,3,4,4',5-Pentachlorobiphenyl (BZ-114)
8995	2,3',4,4',5-Pentachlorobiphenyl (BZ-118)
9000	2,3',4,4',5'-Pentachlorobiphenyl (BZ-123)
9220	2,3',4,4',6-Pentachlorobiphenyl (BZ-119)
9221	2,3,4,4'-Tetrachlorobiphenyl (BZ-60)
8960	2,3',4,4'-Tetrachlorobiphenyl (BZ-66)
9230	2,3',4',5-Tetrachlorobiphenyl (BZ-70)
9239	2,3',4'-Trichlorobiphenyl (BZ-33)
8920	2,3-Dichlorobiphenyl (BZ-5)
9250	2,4,4',5-Tetrachlorobiphenyl (BZ-74)
9252	2,4,4'-Trichlorobiphenyl (BZ-28)
8940	2,4',5-Trichlorobiphenyl (BZ-31)
9256	2,4'-Dichlorobiphenyl (BZ-8)
8915	2-Chlorobiphenyl (BZ-1)
9060	3,3',4,4',5,5'-Hexachlorobiphenyl (BZ-169)
9015	3,3',4,4',5-Pentachlorobiphenyl (BZ-126)
8965	3,3',4,4'-Tetrachlorobiphenyl (BZ-77)
8970	3,4,4',5-Tetrachlorobiphenyl (BZ-81)
9266	3,4,4'-Trichlorobiphenyl (BZ-37)
8880	Aroclor-1016 (PCB-1016)
8885	Aroclor-1221 (PCB-1221)
8890	Aroclor-1232 (PCB-1232)
8895	Aroclor-1242 (PCB-1242)
8900	Aroclor-1248 (PCB-1248)
8905	Aroclor-1254 (PCB-1254)
8910	Aroclor-1260 (PCB-1260)
8912	Aroclor-1262 (PCB-1262)
8913	Aroclor-1268 (PCB-1268)
9105	Decachlorobiphenyl (BZ-209)

EPA 8141B **10182204** **Organophosphorous Pesticides by GC/NPD**

Analyte Code	Analyte
7075	Azinphos-methyl (Guthion)
7125	Bolstar (Sulprofos)
7300	Chlorpyrifos
7315	Coumaphos
7395	Demeton-o

UNCONTROLLED COPY

ORELAP Fields of Accreditation

ORELAP ID: WA100010
EPA CODE: WA01276
Certificate: WA100010 - 010

ALS Environmental, Kelso
1317 South 13th Ave.
Kelso WA 98626

Issue Date: 02/11/2015 ***Expiration Date:*** 02/10/2016

As of 02/11/2015 ***this list supercedes all previous lists for this certificate number.***
Customers. Please verify the current accreditation standing with ORELAP.

Analyte Code	Analyte
7385	Demeton-s
7410	Diazinon
8610	Dichlorovos (DDVP, Dichlorvos)
7475	Dimethoate
8625	Disulfoton
7550	EPN
7570	Ethoprop
7600	Fensulfothion
7605	Fenthion
7770	Malathion
7785	Merphos
7825	Methyl parathion (Parathion, methyl)
7850	Mevinphos
7955	Parathion, ethyl
7985	Phorate
8110	Ronnel
8155	Sulfotepp
8200	Tetrachlorvinphos (Stirophos, Gardona) Z-isomer
8245	Tokuthion (Prothiophos)
8275	Trichloronate

EPA 8151A **10183207** **Chlorinated Herbicides by GC/ECD**

Analyte Code	Analyte
8655	2,4,5-T
8545	2,4-D
8560	2,4-DB
8555	Dalapon
8595	Dicamba
8605	Dichloroprop (Dichlorprop)
8620	Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)
7775	MCPA
7780	MCPP
8650	Silvex (2,4,5-TP)

EPA 8260C **10307003** **Volatile Organics: GC/MS (capillary column)**

Analyte Code	Analyte
5105	1,1,1,2-Tetrachloroethane
5185	1,1,1-Trichloro-2,2,2-trifluoroethane
5160	1,1,1-Trichloroethane
5110	1,1,2,2-Tetrachloroethane
5195	1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)
5165	1,1,2-Trichloroethane
5167	1,1,2-Trichlorofluoroethane
4630	1,1-Dichloroethane
4640	1,1-Dichloroethylene
4670	1,1-Dichloropropene
5150	1,2,3-Trichlorobenzene
5180	1,2,3-Trichloropropane
5155	1,2,4-Trichlorobenzene
5210	1,2,4-Trimethylbenzene
4570	1,2-Dibromo-3-chloropropane (DBCP)
4585	1,2-Dibromoethane (EDB, Ethylene dibromide)
4610	1,2-Dichlorobenzene
4635	1,2-Dichloroethane (Ethylene dichloride)
4655	1,2-Dichloropropane
6800	1,3,5-Trichlorobenzene
5215	1,3,5-Trimethylbenzene
4615	1,3-Dichlorobenzene
4660	1,3-Dichloropropane

UNCONTROLLED COPY

ORELAP Fields of Accreditation

ORELAP ID: WA100010
EPA CODE: WA01276
Certificate: WA100010 - 010

ALS Environmental, Kelso

1317 South 13th Ave.
Kelso WA 98626

Issue Date: 02/11/2015 ***Expiration Date:*** 02/10/2016

As of 02/11/2015 ***this list supercedes all previous lists for this certificate number.***
Customers. Please verify the current accreditation standing with ORELAP.

Analyte Code	Analyte
4620	1,4-Dichlorobenzene
4735	1,4-Dioxane (1,4- Diethyleneoxide)
4510	1-Chlorohexane
4665	2,2-Dichloropropane
4410	2-Butanone (Methyl ethyl ketone, MEK)
4500	2-Chloroethyl vinyl ether
4535	2-Chlorotoluene
4860	2-Hexanone
5020	2-Nitropropane
4536	4-Bromofluorobenzene
4540	4-Chlorotoluene
4910	4-Isopropyltoluene (p-Cymene)
4995	4-Methyl-2-pentanone (MIBK)
4305	Acetamide
4315	Acetone
4320	Acetonitrile
4325	Acrolein (Propenal)
4330	Acrylamide
4340	Acrylonitrile
4355	Allyl chloride (3-Chloropropene)
4375	Benzene
4385	Bromobenzene
4390	Bromochloromethane
4395	Bromodichloromethane
4400	Bromoform
4450	Carbon disulfide
4455	Carbon tetrachloride
4475	Chlorobenzene
4575	Chlorodibromomethane
4485	Chloroethane (Ethyl chloride)
4505	Chloroform
4525	Chloroprene (2-Chloro-1,3-butadiene)
4705	cis & trans-1,2-Dichloroethene
4645	cis-1,2-Dichloroethylene
4680	cis-1,3-Dichloropropene
4595	Dibromomethane (Methylene bromide)
4625	Dichlorodifluoromethane (Freon-12)
4725	Diethyl ether
4755	Ethyl acetate
4810	Ethyl methacrylate
4765	Ethylbenzene
4835	Hexachlorobutadiene
4870	Iodomethane (Methyl iodide)
4875	Isobutyl alcohol (2-Methyl-1-propanol)
4900	Isopropylbenzene
5240	m+p-xylene
4925	Methacrylonitrile
4950	Methyl bromide (Bromomethane)
4960	Methyl chloride (Chloromethane)
5000	Methyl tert-butyl ether (MTBE)
4975	Methylene chloride (Dichloromethane)
5245	m-Xylene
5005	Naphthalene
4435	n-Butylbenzene
5090	n-Propylbenzene
5250	o-Xylene
4440	sec-Butylbenzene
5100	Styrene
4370	T-amylmethylether (TAME)
4445	tert-Butylbenzene
5115	Tetrachloroethylene (Perchloroethylene)

UNCONTROLLED COPY

Analyte Code	Analyte
5140	Toluene
4700	trans-1,2-Dichloroethylene
4685	trans-1,3-Dichloropropylene
4605	trans-1,4-Dichloro-2-butene
5170	Trichloroethene (Trichloroethylene)
5175	Trichlorofluoromethane (Fluorotrichloromethane, Freon 11)
5225	Vinyl acetate
5235	Vinyl chloride
5260	Xylene (total)

EPA 8270D **10186002** **Semivolatile Organic compounds by GC/MS**

Analyte Code	Analyte
6715	1,2,4,5-Tetrachlorobenzene
5155	1,2,4-Trichlorobenzene
4610	1,2-Dichlorobenzene
6221	1,2-Diphenylhydrazine
6885	1,3,5-Trinitrobenzene (1,3,5-TNB)
4615	1,3-Dichlorobenzene
4620	1,4-Dichlorobenzene
6420	1,4-Naphthoquinone
6630	1,4-Phenylenediamine
5790	1-Chloronaphthalene
6380	1-Methylnaphthalene
6425	1-Naphthylamine
6735	2,3,4,6-Tetrachlorophenol
6835	2,4,5-Trichlorophenol
6795	2,4,6-Trichloroaniline
6840	2,4,6-Trichlorophenol
6000	2,4-Dichlorophenol
6130	2,4-Dimethylphenol
6175	2,4-Dinitrophenol
6185	2,4-Dinitrotoluene (2,4-DNT)
5992	2,5-Dichlorophenol
6005	2,6-Dichlorophenol
6190	2,6-Dinitrotoluene (2,6-DNT)
5735	2-Chloroaniline
5795	2-Chloronaphthalene
5800	2-Chlorophenol
6360	2-Methyl-4,6-dinitrophenol (4,6-Dinitro-2-methylphenol)
5145	2-Methylaniline (o-Toluidine)
6385	2-Methylnaphthalene
6400	2-Methylphenol (o-Cresol)
6430	2-Naphthylamine
6460	2-Nitroaniline
6490	2-Nitrophenol
5050	2-Picoline (2-Methylpyridine)
6412	3 & 4 Methylphenol
5945	3,3'-Dichlorobenzidine
6120	3,3'-Dimethylbenzidine
6355	3-Methylcholanthrene
6405	3-Methylphenol (m-Cresol)
6465	3-Nitroaniline
5540	4-Aminobiphenyl
5660	4-Bromophenyl phenyl ether (BDE-3)
5700	4-Chloro-3-methylphenol
5745	4-Chloroaniline
5825	4-Chlorophenyl phenylether
6410	4-Methylphenol (p-Cresol)
6470	4-Nitroaniline
6500	4-Nitrophenol

UNCONTROLLED COPY

ORELAP Fields of Accreditation

ORELAP ID: WA100010
EPA CODE: WA01276
Certificate: WA100010 - 010

ALS Environmental, Kelso

1317 South 13th Ave.
Kelso WA 98626

Issue Date: 02/11/2015 ***Expiration Date:*** 02/10/2016

As of 02/11/2015 ***this list supercedes all previous lists for this certificate number.***
Customers. Please verify the current accreditation standing with ORELAP.

Analyte Code	Analyte
6125	a-a-Dimethylphenethylamine
5500	Acenaphthene
5505	Acenaphthylene
5510	Acetophenone
5545	Aniline
5555	Anthracene
5560	Aramite
7065	Atrazine
5562	Azobenzene
5570	Benzaldehyde
5575	Benzo(a)anthracene
5580	Benzo(a)pyrene
5605	Benzo(e)pyrene
5590	Benzo(g,h,i)perylene
9309	Benzo(j)fluoranthene
5600	Benzo(k)fluoranthene
5585	Benzo[b]fluoranthene
5610	Benzoic acid
5630	Benzyl alcohol
5640	Biphenyl
5760	bis(2-Chloroethoxy)methane
5765	bis(2-Chloroethyl) ether
5780	bis(2-Chloroisopropyl) ether
5670	Butyl benzyl phthalate
7180	Caprolactam
5680	Carbazole
7260	Chlorobenzilate
5855	Chrysene
6065	Di(2-ethylhexyl) phthalate (bis(2-Ethylhexyl)phthalate, DEHP)
7405	Diallate
7410	Diazinon
5895	Dibenz(a,h) anthracene
5905	Dibenzofuran
6070	Diethyl phthalate
7475	Dimethoate
6135	Dimethyl phthalate
5925	Di-n-butyl phthalate
6200	Di-n-octyl phthalate
7580	Famphur
6265	Fluoranthene
6270	Fluorene
6275	Hexachlorobenzene
4835	Hexachlorobutadiene
6285	Hexachlorocyclopentadiene
4840	Hexachloroethane
6290	Hexachlorophene
6315	Indeno(1,2,3-cd) pyrene
7725	Isodrin
6320	Isophorone
7740	Kepone
6345	Methapyrilene
7825	Methyl parathion (Parathion, methyl)
5005	Naphthalene
5015	Nitrobenzene
6525	n-Nitrosodiethylamine
6530	n-Nitrosodimethylamine
6545	n-Nitrosodi-n-propylamine
6535	n-Nitrosodiphenylamine
6555	n-Nitrosomorpholine
6560	n-Nitrosopiperidine
6565	n-Nitrosopyrrolidine

UNCONTROLLED COPY

Analyte Code	Analyte
7955	Parathion, ethyl
6590	Pentachlorobenzene
6600	Pentachloronitrobenzene
6605	Pentachlorophenol
6608	Perylene
6615	Phenanthrene
6625	Phenol
6650	Pronamide (Kerb)
6665	Pyrene
5095	Pyridine
6685	Safrole
8235	Thionazin (Zinophos)

EPA 8270D SIM **10242509** **Semivolatile Organic compounds by GC/MS Selective Ion Monitoring**

Analyte Code	Analyte
4735	1,4-Dioxane (1,4- Diethyleneoxide)
6380	1-Methylnaphthalene
9501	1-Methylphenanthrene
6852	2,3,5-Trimethylnaphthalene
6188	2,6-Dimethylnaphthalene
6385	2-Methylnaphthalene
5500	Acenaphthene
5505	Acenaphthylene
5555	Anthracene
5575	Benzo(a)anthracene
5580	Benzo(a)pyrene
5590	Benzo(g,h,i)perylene
9309	Benzo(j)fluoranthene
5600	Benzo(k)fluoranthene
5585	Benzo[b]fluoranthene
5640	Biphenyl
5670	Butyl benzyl phthalate
5680	Carbazole
5855	Chrysene
6065	Di(2-ethylhexyl) phthalate (bis(2-Ethylhexyl)phthalate, DEHP)
5895	Dibenz(a,h) anthracene
5905	Dibenzofuran
5910	Dibenzothiophene
6070	Diethyl phthalate
6135	Dimethyl phthalate
5925	Di-n-butyl phthalate
6200	Di-n-octyl phthalate
6265	Fluoranthene
6270	Fluorene
6315	Indeno(1,2,3-cd) pyrene
5005	Naphthalene
6605	Pentachlorophenol
6608	Perylene
6615	Phenanthrene
6665	Pyrene

EPA 8315A **10188008** **Determination of Carbonyl Compounds by HPLC/UV-VIS**

Analyte Code	Analyte
4815	Formaldehyde

EPA 8321B **10189205** **Solvent Extractable non-volatile compounds by HPLC/TS/MS**

Analyte Code	Analyte

UNCONTROLLED COPY

Analyte Code	Analyte
7010	Aldicarb (Temik)
7040	Aminocarb
7080	Barban
7130	Bromacil
7195	Carbaryl (Sevin)
7205	Carbofuran (Furaden)
7275	Chloropropham
7505	Diuron
7610	Fenuron
7630	Fluometuron
7765	Linuron (Lorox)
7800	Methiocarb (Mesurol)
7805	Methomyl (Lannate)
7855	Mexacarbate
7885	Monuron
7915	Neburon
7940	Oxamyl
8075	Propham
8080	Propoxur (Baygon)
8120	Siduron

EPA 8330B **10308006** **Nitroaromatics, Nitramines and Nitrate Esters by High Performance Liquid Chromatography (HPLC)**

Analyte Code	Analyte
6885	1,3,5-Trinitrobenzene (1,3,5-TNB)
6160	1,3-Dinitrobenzene (1,3-DNB)
9651	2,4,6-Trinitrotoluene (2,4,6-TNT)
6185	2,4-Dinitrotoluene (2,4-DNT)
6190	2,6-Dinitrotoluene (2,6-DNT)
9303	2-Amino-4,6-dinitrotoluene (2-am-dnt)
9507	2-Nitrotoluene
6150	3,5-Dinitroaniline
9510	3-Nitrotoluene
9306	4-Amino-2,6-dinitrotoluene (4-am-dnt)
9513	4-Nitrotoluene
6415	Methyl-2,4,6-trinitrophenylnitramine (tetryl)
5015	Nitrobenzene
6485	Nitroglycerin
9522	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)
9558	Pentaerythritoltetranitrate
9432	RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine)

EPA 9012B **10243206** **Total and Amenable Cyanide (automated colorimetric with off-line distillation)**

Analyte Code	Analyte
1510	Amenable cyanide
1645	Total cyanide

EPA 9020B **10194408** **Total Organic Halides**

Analyte Code	Analyte
2045	Total organic halides (TOX)

EPA 9040C **10244403** **pH Electrometric Measurement**

Analyte Code	Analyte
1900	pH

UNCONTROLLED COPY

ORELAP Fields of Accreditation

ORELAP ID: WA100010
EPA CODE: WA01276
Certificate: WA100010 - 010

ALS Environmental, Kelso

1317 South 13th Ave.
Kelso WA 98626

Issue Date: 02/11/2015 ***Expiration Date:*** 02/10/2016

As of 02/11/2015 ***this list supercedes all previous lists for this certificate number. Customers. Please verify the current accreditation standing with ORELAP.***

EPA 9060A **10244801** **Total Organic Carbon**

Analyte Code	Analyte
2040	Total organic carbon

NCASI 94.03 0 **60031507** **Methanol in Process Liquids and Wastewaters**

Analyte Code	Analyte
4930	Methanol

NCASI 99.01 **60002804** **Selected HAPS in Condensates by GC/FID**

Analyte Code	Analyte
4930	Methanol

NWTPH-Dx **90018409** **Oregon DEQ TPH Diesel Range**

Analyte Code	Analyte
9369	Diesel range organics (DRO)
9506	Residual Range Organics (RRO)

NWTPH-Gx **90018603** **Oregon DEQ TPH Gasoline Range Organics by GC/FID-PID Purge & Trap**

Analyte Code	Analyte
9408	Gasoline range organics (GRO)

NWTPH-HCID **90013200** **Oregon DEQ Total Petroleum Hydrocarbon ID**

Analyte Code	Analyte
2050	Total Petroleum Hydrocarbons (TPH)

SM 2120 B 20th ED **20224004** **Color by Visual Comparison**

Analyte Code	Analyte
1605	Color

SM 2310 B 20th ED **20044206** **Acidity by Titration**

Analyte Code	Analyte
1500	Acidity, as $CaCO_3$

SM 2320 B 20th ED **20045209** **Alkalinity by Titration**

Analyte Code	Analyte
1505	Alkalinity as $CaCO_3$

SM 2340 B 20th ED **20046202** **Hardness by calculation**

Analyte Code	Analyte
1750	Hardness

SM 2340 C 20th ED **20047205** **Hardness by EDTA Titration**

Analyte Code	Analyte
1750	Hardness

SM 2510 B-97 online **20048606** **Conductivity by Probe**

Analyte Code	Analyte
1610	Conductivity

UNCONTROLLED COPY

SM 2540 B 20th ED **20049007** **Total Solids**

Analyte Code	Analyte
1950	Residue-total

SM 2540 C 20th ED **20050004** **Total Dissolved Solids**

Analyte Code	Analyte
1955	Residue-filterable (TDS)

SM 2540 D 20th ED **20050800** **Total Suspended Solids**

Analyte Code	Analyte
1960	Residue-nonfilterable (TSS)

SM 2540 D-2011 **20051212** **Total Suspended Solids Dried at 103 - 105 C**

Analyte Code	Analyte
1960	Residue-nonfilterable (TSS)

SM 2540 D-97 online **20051201** **Total Suspended Solids Dried at 103 - 105C**

Analyte Code	Analyte
1960	Residue-nonfilterable (TSS)

SM 2540 F 20th ED **20051803** **Settleable Solids**

Analyte Code	Analyte
1965	Residue-settleable

SM 4500-Cl C 20th ED **20078802** **Chlorine by Iodometric Method II**

Analyte Code	Analyte
1575	Chloride

SM 4500-Cl F 20th ED **20080506** **Residual Chlorine by DPD Ferrous Titration**

Analyte Code	Analyte
1945	Residual free chlorine

SM 4500-CN E 20th ED **20092404** **Cyanide by Colorimetric Determination**

Analyte Code	Analyte
1635	Cyanide
1645	Total cyanide

SM 4500-CN G 20th ED **20093203** **Cyanide Amenable to Chlorination after Distillation**

Analyte Code	Analyte
1510	Amenable cyanide

SM 4500-CN$^-$ E-97 online **20096406** **Cyanide by Colorimetric Method**

Analyte Code	Analyte
1635	Cyanide

SM 4500-F$^-$ C 20th ED **20102005** **Fluoride by Ion Selective Electrode**

Analyte Code	Analyte
1730	Fluoride

ORELAP Fields of Accreditation

ORELAP ID: WA100010
EPA CODE: WA01276
Certificate: WA100010 - 010

ALS Environmental, Kelso

1317 South 13th Ave.
Kelso WA 98626

Issue Date: 02/11/2015 **Expiration Date:** 02/10/2016

As of** 02/11/2015 **this list supercedes all previous lists for this certificate number.
Customers. Please verify the current accreditation standing with ORELAP.

SM 4500-H+ B 20th ED **20104807** **pH by Probe**

Analyte Code	Analyte
1900	pH

SM 4500-NH3 E 20th ED **20109802** **Ammonia by Selective Ion Probe**

Analyte Code	Analyte
1515	Ammonia as N

SM 4500-NH3 G 20th ED **20111006** **Ammonia by Automated Phenate**

Analyte Code	Analyte
1515	Ammonia as N

SM 4500-O G 20th ED **20121204** **Dissolved Oxygen by Membrane Electrode Method**

Analyte Code	Analyte
1880	Oxygen, dissolved

SM 4500-S2 F-2011 **20126663** **Sulfide by Iodometric Method**

Analyte Code	Analyte
2005	Sulfide

SM 4500-S2⁻ D 20th ED **20125400** **Sulfide by Methylene Blue Method**

Analyte Code	Analyte
2005	Sulfide

SM 4500-S2⁻ D-97 online **20125808** **Sulfide by Methylene Blue Method**

Analyte Code	Analyte
2005	Sulfide

SM 4500-S2⁻ F 20th ED **20126209** **Sulfide by Iodometric Titration**

Analyte Code	Analyte
2005	Sulfide

SM 4500-SO3⁻ B 20th ED **20130205** **Sulfite by Iodometric Method**

Analyte Code	Analyte
2015	Sulfite-SO3

SM 5210 B 20th ED **20134809** **Biochemical Oxygen Demand, 5-Day (BOD5)**

Analyte Code	Analyte
1530	Biochemical oxygen demand

SM 5220 C 20th ED **20135608** **Chemical Oxygen Demand by Closed Reflux and Titration**

Analyte Code	Analyte
1565	Chemical oxygen demand

SM 5310 C 20th ED **20138403** **Total Organic Carbon by Persulfate-Ultraviolet Oxidation Method**

Analyte Code	Analyte
2040	Total organic carbon

UNCONTROLLED COPY

SM 5540 C 20th ED **20144609** **Surfactants as MBAS**

Analyte Code	Analyte
2025	Surfactants - MBAS

SM 5550 B 20th ED **20145306** **Tannin and Lignin**

Analyte Code	Analyte
9597	Tannin & Lignin

SM 9215 B (PCA) 20th ED **20181208** **Heterotrophic Plate Count Pour Plate (plate count agar): Heterotrophic Bacteria**

Analyte Code	Analyte
2555	Heterotrophic plate count

SM 9221 B (LTB) + C MPN 20th ED **20186805** **Multiple Tube Fermentation Quantitative (LTB): Total Coliform**

Analyte Code	Analyte
2500	Total coliforms

SM 9221 E (EC) 20th ED **20226806** **Multiple Tube Fermentation Quantitative (EC): Fecal Coliform**

Analyte Code	Analyte
2530	Fecal coliforms

SM 9222 D (m-FC) 20th ED **20209603** **Membrane Filtration Quantitative (m-FC): Fecal Coliform**

Analyte Code	Analyte
2530	Fecal coliforms

SM 9223 B (Colilert®) 20th ED **20212208** **Chromogenic/Fluorogenic Qualitative (Colilert®): Total Coliform and E. coli**

Analyte Code	Analyte
2525	Escherichia coli
2500	Total coliforms

SM 9223 B (Colilert®-18 Quanti-Tray®) 20th ED **20213201** **Chromogenic/Fluorogenic Quantitative (Colilert®-18): Total Coliform and E. coli**

Analyte Code	Analyte
2525	Escherichia coli
2500	Total coliforms

SM 9223 B (Colilert®-18) 20th ED **20214204** **Chromogenic/Fluorogenic Qualitative (Colilert®-18): Total Coliform and E. coli**

Analyte Code	Analyte
2525	Escherichia coli
2500	Total coliforms

SM 9223 B (Colilert®-18) 21st ED **20214408** **Chromogenic/Fluorogenic Qualitative (Colilert®-18): Total Coliform and E. coli**

Analyte Code	Analyte
2525	Escherichia coli
2500	Total coliforms

WI(95) DRO **90019457** **Wisconsin DNR - Modified Method for Determination of Diesel Range Organics by GC-FID**

Analyte Code	Analyte
9369	Diesel range organics (DRO)

UNCONTROLLED COPY

ORELAP Fields of Accreditation

ORELAP ID: WA100010
EPA CODE: WA01276
Certificate: WA100010 - 010

ALS Environmental, Kelso

1317 South 13th Ave.
Kelso WA 98626

Issue Date: 02/11/2015 ***Expiration Date:*** 02/10/2016

As of 02/11/2015 ***this list supercedes all previous lists for this certificate number.***
Customers. Please verify the current accreditation standing with ORELAP.

WI(95) GRO **90019468** **Wisconsin DNR - Modified Method for Determining Gasoline Range Organics GC-FID**

Analyte Code	Analyte
9408	Gasoline range organics (GRO)

NELAP RECOGNIZED TNI ACCREDITATION BODY

UNCONTROLLED COPY

ORELAP Fields of Accreditation

ORELAP ID: WA100010
EPA CODE: WA01276
Certificate: WA100010 - 010

ALS Environmental, Kelso

1317 South 13th Ave.
Kelso WA 98626

Issue Date: 02/11/2015 **Expiration Date:** 02/10/2016

As of 02/11/2015 ***this list supercedes all previous lists for this certificate number. Customers. Please verify the current accreditation standing with ORELAP.***

MATRIX : Solids

Reference	Code	Description
ALS Kelso LCP-Acryl 1	**60001712**	**ALS Kelso - Acrylamide by HPLC/MS/MS**

Analyte Code	*Analyte*
4330	Acrylamide

Reference	Code	Description
ALS Kelso LCP-PFC 4	**60001505**	**ALS Kelso - Perfluorinated Compounds by HPLC-MS-MS**

Analyte Code	*Analyte*
6911	Perfluorobutane Sulfonate (PFBS)
9562	Perfluorodecane Sulfonate (PFDS)
6905	Perfluorodecanoic acid (PFDA)
6903	Perfluorododecanoic (PFDDA)
6908	Perfluoroheptanoic acid (PFHA)
6910	Perfluorohexane Sulfonate (PFHS)
6913	Perfluorohexanoic acid (PFHXA)
6906	Perfluorononanoic acid (PFNA)
6912	Perfluorooctanoic acid
6909	Perfluorooctanoic Sulfonate (PFOS)
6914	Perfluoropentanoic acid (PFPEA)
6904	Perfluoroundecanoic acid (PFUDA)

Reference	Code	Description
ASTM D1426-08B	**30007397**	**Ammonia by Titration**

Analyte Code	*Analyte*
1515	Ammonia as N

Reference	Code	Description
ASTM D3590-02(06)A	**30016819**	**Total Kjeldahl Nitrogen in Water**

Analyte Code	*Analyte*
1795	Kjeldahl nitrogen - total

Reference	Code	Description
ASTM D4129 05	**30018907**	**Total and Organic Carbon in Water by High Temperature Oxidation and by Coulometric Detection**

Analyte Code	*Analyte*
2040	Total organic carbon

Reference	Code	Description
ASTM D422-63	**30030854**	**Partical Size Distribution (Grain sizing)**

Analyte Code	*Analyte*
6118	Distribution of particle sizes

Reference	Code	Description
CAS PestMS2 (1699 modified) 2	**60035101**	**Chlorinated Pesticides by GC/MS/MS**

Analyte Code	*Analyte*
8580	2,4'-DDD
8585	2,4'-DDE
8590	2,4'-DDT
7355	4,4'-DDD
7360	4,4'-DDE
7365	4,4'-DDT
7025	Aldrin
7110	alpha-BHC (alpha-Hexachlorocyclohexane)
7240	alpha-Chlordane
7115	beta-BHC (beta-Hexachlorocyclohexane)
7300	Chlorpyrifos
7925	cis-Nonachlor
7105	delta-BHC

UNCONTROLLED COPY

ORELAP Fields of Accreditation

ORELAP ID: WA100010
EPA CODE: WA01276
Certificate: WA100010 - 010

ALS Environmental, Kelso

1317 South 13th Ave.
Kelso WA 98626

Issue Date: 02/11/2015 **Expiration Date:** 02/10/2016

As of 02/11/2015 ***this list supercedes all previous lists for this certificate number.***
Customers. Please verify the current accreditation standing with ORELAP.

Analyte Code	Analyte
7470	Dieldrin
7510	Endosulfan I
7515	Endosulfan II
7520	Endosulfan sulfate
7540	Endrin
7530	Endrin aldehyde
7535	Endrin ketone
7120	gamma-BHC (Lindane, gamma-HexachlorocyclohexanE)
7245	gamma-Chlordane
7685	Heptachlor
7690	Heptachlor epoxide
6275	Hexachlorobenzene
7725	Isodrin
7810	Methoxychlor
7870	Mirex
5553	Octachlorostyrene
3890	Oxychlordane
7910	trans-Nanochlor

CAS SOC-Butyl **60035009** **Butyltin by GC/Flame Photometric Detector**

Analyte Code	Analyte
1201	Butyltin trichloride
1202	Dibutyltin dichloride
1209	Tetrabutyltin
1203	Tributyltin chloride

EPA 1020A **10117007** **Ignitability Setaflash Closed-cup Method**

Analyte Code	Analyte
1780	Ignitability

EPA 1110A **10235208** **Corrosivity Toward Steel**

Analyte Code	Analyte
1615	Corrosivity

EPA 1311 **10118806** **Toxicity Characteristic Leaching Procedure**

Analyte Code	Analyte
8031	Extraction/Preparation

EPA 1312 **10119003** **Synthetic Precipitation Leaching Procedure**

Analyte Code	Analyte
8031	Extraction/Preparation

EPA 160.3 **10009800** **Total Solids, dried @ 103-105 C.**

Analyte Code	Analyte
1950	Residue-total

EPA 1630 **10122608** **Methyl Mercury by Purge & Trap Cold Vapor Atomic Fluorescence Spectrometry**

Analyte Code	Analyte
1205	Methyl Mercury

EPA 1631E **10237204** **Mercury in Water by Oxidation, Purge & Trap, and Cold Vapor Atomic Fluorescence**

Analyte Code	Analyte

UNCONTROLLED COPY

Analyte Code	Analyte
1095	Mercury

EPA 1664A (HEM) **10127807** **N-Hexane Extractable Material (Oil and Grease) by Extraction and Gravimetry**

Analyte Code	Analyte
1803	n-Hexane Extractable Material (O&G)
1860	Oil & Grease

EPA 300.0 2.1 **10053200** **Methods for the Determination of Inorganic Substances in Environmental Samples**

Analyte Code	Analyte
1575	Chloride
1730	Fluoride
2000	Sulfate

EPA 3050B **10135601** **Acid Digestion of Sediments, Sludges, and soils**

Analyte Code	Analyte
8031	Extraction/Preparation

EPA 314.0 **10055400** **Perchlorate in Drinking Water by Ion Chromatography**

Analyte Code	Analyte
1895	Perchlorate

EPA 350.1 2 **10063602** **Ammonia Nitrogen - Colorimetric, Auto Phenate**

Analyte Code	Analyte
1515	Ammonia as N

EPA 353.2 2 **10067604** **Nitrate/Nitrite Nitrogen - Automated, Cadmium**

Analyte Code	Analyte
1810	Nitrate as N
1840	Nitrite as N
1825	Total nitrate+nitrite

EPA 3540C **10140202** **Soxhlet Extraction**

Analyte Code	Analyte
8031	Extraction/Preparation

EPA 3541 **10140406** **Automated Soxhlet Extraction**

Analyte Code	Analyte
8031	Extraction/Preparation

EPA 3550C **10142004** **Ultrasonic Extraction**

Analyte Code	Analyte
8031	Extraction/Preparation

EPA 3580A **10143007** **Waste Dilution**

Analyte Code	Analyte
8031	Extraction/Preparation

UNCONTROLLED COPY

EPA 3620C **10146006** **Florisil Cleanup**

Analyte Code	*Analyte*
8031	Extraction/Preparation

EPA 3630C **10146802** **Silica gel cleanup**

Analyte Code	*Analyte*
8031	Extraction/Preparation

EPA 3640A **10147203** **Gel Preparation Cleanup**

Analyte Code	*Analyte*
8031	Extraction/Preparation

EPA 365.3 **10070607** **Phosphorous - Colorimetric, two reagent.**

Analyte Code	*Analyte*
1870	Orthophosphate as P
1908	Total Phosphate

EPA 3660B **10148400** **Sulfur cleanup**

Analyte Code	*Analyte*
8031	Extraction/Preparation

EPA 3665A **10148808** **Sulfuric Acid / permanganate Cleanup**

Analyte Code	*Analyte*
8031	Extraction/Preparation

EPA 5030B **10153409** **Purge and trap for aqueous samples**

Analyte Code	*Analyte*
8031	Extraction/Preparation

EPA 5035A **10284807** **Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples**

Analyte Code	*Analyte*
8031	Extraction/Preparation

EPA 6010C **10155803** **ICP - AES**

Analyte Code	*Analyte*
1000	Aluminum
1005	Antimony
1010	Arsenic
1015	Barium
1020	Beryllium
1025	Boron
1030	Cadmium
1035	Calcium
1040	Chromium
1050	Cobalt
1055	Copper
1070	Iron
1075	Lead
1085	Magnesium
1090	Manganese
1100	Molybdenum
1105	Nickel

UNCONTROLLED COPY

Analyte Code	Analyte
1125	Potassium
1140	Selenium
1150	Silver
1155	Sodium
1160	Strontium
1165	Thallium
1175	Tin
1180	Titanium
1185	Vanadium
1190	Zinc

EPA 6020A **10156408** **Inductively Coupled Plasma-Mass Spectrometry**

Analyte Code	Analyte
1000	Aluminum
1005	Antimony
1010	Arsenic
1015	Barium
1020	Beryllium
1030	Cadmium
1040	Chromium
1050	Cobalt
1055	Copper
1070	Iron
1075	Lead
1090	Manganese
1100	Molybdenum
1105	Nickel
1140	Selenium
1150	Silver
1160	Strontium
1165	Thallium
1185	Vanadium
1190	Zinc

EPA 6850 **10304606** **Perchlorate in Water, Soils and Solid Wastes Using High Performance Liquid Chromatography/Electrospray Ionization/Mass Spectrometry**

Analyte Code	Analyte
1895	Perchlorate

EPA 7062 **10159407** **Antimony and Arsenic by Borohydride Reduction and Atomic Absorption**

Analyte Code	Analyte
1010	Arsenic

EPA 7196A **10162400** **Chromium Hexavalent colorimetric**

Analyte Code	Analyte
1045	Chromium VI

EPA 7471B **10166402** **Mercury by Cold Vapor Atomic Absorption**

Analyte Code	Analyte
1095	Mercury

EPA 7742 **10169207** **Selenium by Borohydride Reduction and Atomic Absorption**

Analyte Code	Analyte
1140	Selenium

NELAP RECOGNIZED TNI ACCREDITATION BODY

UNCONTROLLED COPY

ORELAP Fields of Accreditation

ORELAP ID: WA100010
EPA CODE: WA01276
Certificate: WA100010 - 010

ALS Environmental, Kelso

1317 South 13th Ave.
Kelso WA 98626

Issue Date: 02/11/2015 **Expiration Date:** 02/10/2016

As of 02/11/2015 ***this list supercedes all previous lists for this certificate number.***
Customers. Please verify the current accreditation standing with ORELAP.

EPA 8015C **10173805** **Non-halogenated organics using GC/FID**

Analyte Code	Analyte
9369	Diesel range organics (DRO)
4785	Ethylene glycol
9408	Gasoline range organics (GRO)

EPA 8081B **10178800** **Organochlorine Pesticides by GC/ECD**

Analyte Code	Analyte
8580	2,4'-DDD
8585	2,4'-DDE
8590	2,4'-DDT
7355	4,4'-DDD
7360	4,4'-DDE
7365	4,4'-DDT
7005	Alachlor
7025	Aldrin
7110	alpha-BHC (alpha-Hexachlorocyclohexane)
7240	alpha-Chlordane
7115	beta-BHC (beta-Hexachlorocyclohexane)
7250	Chlordane (tech.)
7300	Chlorpyrifos
7925	cis-Nonachlor
7105	delta-BHC
7470	Dieldrin
7510	Endosulfan I
7515	Endosulfan II
7520	Endosulfan sulfate
7540	Endrin
7530	Endrin aldehyde
7535	Endrin ketone
7120	gamma-BHC (Lindane, gamma-HexachlorocyclohexanE)
7245	gamma-Chlordane
7685	Heptachlor
7690	Heptachlor epoxide
6275	Hexachlorobenzene
4835	Hexachlorobutadiene
4840	Hexachloroethane
7725	Isodrin
7810	Methoxychlor
7870	Mirex
3890	Oxychlordane
8250	Toxaphene (Chlorinated camphene)
7910	trans-Nanochlor

EPA 8082A **10179201** **Polychlorinated Biphenyls (PCBs) by GC/ECD**

Analyte Code	Analyte
9095	2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (BZ-206)
9090	2,2',3,3',4,4',5,5'-Octachlorobiphenyl (BZ-194)
9103	2,2',3,3',4,4',5,6-Octachlorobiphenyl (BZ-195)
9065	2,2',3,3',4,4',5-Heptachlorobiphenyl (BZ-170)
9020	2,2',3,3',4,4'-Hexachlorobiphenyl (BZ-128)
9112	2,2',3,3',4,5',6,6'-Octachlorobiphenyl (BZ-201)
9116	2,2',3,3',4,5,6'-Heptachlorobiphenyl (BZ-174)
9114	2,2',3,3',4,5',6'-Heptachlorobiphenyl (BZ-177)
9120	2,2',3,3',4,6'-Hexachlorobiphenyl (BZ-132)
9133	2,2',3,4,4',5,5',6-Octachlorobiphenyl (BZ-203)
9134	2,2',3,4,4',5,5'-Heptachlorobiphenyl (BZ-180)
9075	2,2',3,4,4',5',6-Heptachlorobiphenyl (BZ-183)

UNCONTROLLED COPY

Analyte Code	Analyte
9025	2,2',3,4,4',5'-Hexachlorobiphenyl (BZ-138)
9139	2,2',3,4,4',6,6'-Heptachlorobiphenyl (BZ-184)
9080	2,2',3,4',5,5',6-Heptachlorobiphenyl (BZ-187)
9030	2,2',3,4,5,5'-Hexachlorobiphenyl (BZ-141)
9151	2,2',3,4',5',6-Hexachlorobiphenyl (BZ-149)
8975	2,2',3,4,5'-Pentachlorobiphenyl (BZ-87)
9155	2,2',3,4',5-Pentachlorobiphenyl (BZ-90)
9154	2,2',3,4',5'-Pentachlorobiphenyl (BZ-97)
9035	2,2',3,5,5',6-Hexachlorobiphenyl (BZ-151)
9166	2,2',3,5',6-Pentachlorobiphenyl (BZ-95)
8945	2,2',3,5'-Tetrachlorobiphenyl (BZ-44)
9040	2,2',4,4',5,5'-Hexachlorobiphenyl (BZ-153)
9174	2,2',4,4',5,6'-Hexachlorobiphenyl (BZ-154)
9175	2,2',4,4',5-Pentachlorobiphenyl (BZ-99)
8980	2,2',4,5,5'-Pentachlorobiphenyl (BZ-101)
8950	2,2',4,5'-Tetrachlorobiphenyl (BZ-49)
8955	2,2',5,5'-Tetrachlorobiphenyl (BZ-52)
8930	2,2',5-Trichlorobiphenyl (BZ-18)
9085	2,3,3',4,4',5,5'-Heptachlorobiphenyl (BZ-189)
9050	2,3,3',4,4',5-Hexachlorobiphenyl (BZ-156)
9193	2,3,3',4,4',6-Hexachlorobiphenyl (BZ-158)
8985	2,3,3',4,4'-Pentachlorobiphenyl (BZ-105)
8990	2,3,3',4',6-Pentachlorobiphenyl (BZ-110)
9207	2,3,3',4'-Tetrachlorobiphenyl (BZ-56)
9055	2,3',4,4',5,5'-Hexachlorobiphenyl (BZ-167)
9218	2,3',4,4',5',6-Hexachlorobiphenyl (BZ-168)
9005	2,3,4,4',5-Pentachlorobiphenyl (BZ-114)
8995	2,3',4,4',5-Pentachlorobiphenyl (BZ-118)
9000	2,3',4,4',5'-Pentachlorobiphenyl (BZ-123)
9220	2,3',4,4',6-Pentachlorobiphenyl (BZ-119)
9221	2,3,4,4'-Tetrachlorobiphenyl (BZ-60)
8960	2,3',4,4'-Tetrachlorobiphenyl (BZ-66)
9230	2,3',4',5-Tetrachlorobiphenyl (BZ-70)
9239	2,3',4'-Trichlorobiphenyl (BZ-33)
8920	2,3-Dichlorobiphenyl (BZ-5)
9250	2,4,4',5-Tetrachlorobiphenyl (BZ-74)
9252	2,4,4'-Trichlorobiphenyl (BZ-28)
8940	2,4',5-Trichlorobiphenyl (BZ-31)
9256	2,4'-Dichlorobiphenyl (BZ-8)
8915	2-Chlorobiphenyl (BZ-1)
9060	3,3',4,4',5,5'-Hexachlorobiphenyl (BZ-169)
9015	3,3',4,4',5-Pentachlorobiphenyl (BZ-126)
8965	3,3',4,4'-Tetrachlorobiphenyl (BZ-77)
8970	3,4,4',5-Tetrachlorobiphenyl (BZ-81)
9266	3,4,4'-Trichlorobiphenyl (BZ-37)
8880	Aroclor-1016 (PCB-1016)
8885	Aroclor-1221 (PCB-1221)
8890	Aroclor-1232 (PCB-1232)
8895	Aroclor-1242 (PCB-1242)
8900	Aroclor-1248 (PCB-1248)
8905	Aroclor-1254 (PCB-1254)
8910	Aroclor-1260 (PCB-1260)
8912	Aroclor-1262 (PCB-1262)
8913	Aroclor-1268 (PCB-1268)
9105	Decachlorobiphenyl (BZ-209)

EPA 8141B **10182204** **Organophosphorous Pesticides by GC/NPD**

Analyte Code	Analyte
7075	Azinphos-methyl (Guthion)
7125	Bolstar (Sulprofos)

Analyte Code	Analyte
7300	Chlorpyrifos
7315	Coumaphos
7395	Demeton-o
7385	Demeton-s
7410	Diazinon
8610	Dichlorovos (DDVP, Dichlorvos)
7475	Dimethoate
8625	Disulfoton
7550	EPN
7570	Ethoprop
7600	Fensulfothion
7605	Fenthion
7770	Malathion
7785	Merphos
7825	Methyl parathion (Parathion, methyl)
7850	Mevinphos
7955	Parathion, ethyl
7985	Phorate
8110	Ronnel
8155	Sulfotepp
8200	Tetrachlorvinphos (Stirophos, Gardona) Z-isomer
8245	Tokuthion (Prothiophos)
8275	Trichloronate

EPA 8151A **10183207** **Chlorinated Herbicides by GC/ECD**

Analyte Code	Analyte
8655	2,4,5-T
8545	2,4-D
8560	2,4-DB
8555	Dalapon
8595	Dicamba
8605	Dichloroprop (Dichlorprop)
8620	Dinoseb (2-sec-butyl-4,6-dinitrophenol, DNBP)
7775	MCPA
7780	MCPP
8650	Silvex (2,4,5-TP)

EPA 8260C **10307003** **Volatile Organics: GC/MS (capillary column)**

Analyte Code	Analyte
5105	1,1,1,2-Tetrachloroethane
5185	1,1,1-Trichloro-2,2,2-trifluoroethane
5160	1,1,1-Trichloroethane
5110	1,1,2,2-Tetrachloroethane
5195	1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)
5165	1,1,2-Trichloroethane
5167	1,1,2-Trichlorofluoroethane
4630	1,1-Dichloroethane
4640	1,1-Dichloroethylene
4670	1,1-Dichloropropene
5150	1,2,3-Trichlorobenzene
5180	1,2,3-Trichloropropane
5155	1,2,4-Trichlorobenzene
5210	1,2,4-Trimethylbenzene
4570	1,2-Dibromo-3-chloropropane (DBCP)
4585	1,2-Dibromoethane (EDB, Ethylene dibromide)
4610	1,2-Dichlorobenzene
4635	1,2-Dichloroethane (Ethylene dichloride)
4655	1,2-Dichloropropane
6800	1,3,5-Trichlorobenzene

UNCONTROLLED COPY

ORELAP Fields of Accreditation

ORELAP ID: WA100010
EPA CODE: WA01276
Certificate: WA100010 - 010

ALS Environmental, Kelso

1317 South 13th Ave.
Kelso WA 98626

Issue Date: 02/11/2015 **Expiration Date:** 02/10/2016

As of 02/11/2015 ***this list supercedes all previous lists for this certificate number.***
Customers. Please verify the current accreditation standing with ORELAP.

Analyte Code	Analyte
5215	1,3,5-Trimethylbenzene
4615	1,3-Dichlorobenzene
4660	1,3-Dichloropropane
4620	1,4-Dichlorobenzene
4735	1,4-Dioxane (1,4- Diethyleneoxide)
4510	1-Chlorohexane
4665	2,2-Dichloropropane
4410	2-Butanone (Methyl ethyl ketone, MEK)
4500	2-Chloroethyl vinyl ether
4535	2-Chlorotoluene
4860	2-Hexanone
5020	2-Nitropropane
4536	4-Bromofluorobenzene
4540	4-Chlorotoluene
4910	4-Isopropyltoluene (p-Cymene)
4995	4-Methyl-2-pentanone (MIBK)
4305	Acetamide
4315	Acetone
4320	Acetonitrile
4325	Acrolein (Propenal)
4330	Acrylamide
4340	Acrylonitrile
4355	Allyl chloride (3-Chloropropene)
4375	Benzene
4385	Bromobenzene
4390	Bromochloromethane
4395	Bromodichloromethane
4400	Bromoform
4450	Carbon disulfide
4455	Carbon tetrachloride
4475	Chlorobenzene
4575	Chlorodibromomethane
4485	Chloroethane (Ethyl chloride)
4505	Chloroform
4525	Chloroprene (2-Chloro-1,3-butadiene)
4705	cis & trans-1,2-Dichloroethene
4645	cis-1,2-Dichloroethylene
4680	cis-1,3-Dichloropropene
4595	Dibromomethane (Methylene bromide)
4625	Dichlorodifluoromethane (Freon-12)
4725	Diethyl ether
4755	Ethyl acetate
4810	Ethyl methacrylate
4765	Ethylbenzene
4835	Hexachlorobutadiene
4870	Iodomethane (Methyl iodide)
4875	Isobutyl alcohol (2-Methyl-1-propanol)
4900	Isopropylbenzene
5240	m+p-xylene
4925	Methacrylonitrile
4950	Methyl bromide (Bromomethane)
4960	Methyl chloride (Chloromethane)
5000	Methyl tert-butyl ether (MTBE)
4975	Methylene chloride (Dichloromethane)
5245	m-Xylene
5005	Naphthalene
4435	n-Butylbenzene
5090	n-Propylbenzene
5250	o-Xylene
4440	sec-Butylbenzene
5100	Styrene

UNCONTROLLED COPY

Analyte Code	Analyte
4370	T-amylmethylether (TAME)
4445	tert-Butylbenzene
5115	Tetrachloroethylene (Perchloroethylene)
5140	Toluene
4700	trans-1,2-Dichloroethylene
4685	trans-1,3-Dichloropropylene
4605	trans-1,4-Dichloro-2-butene
5170	Trichloroethene (Trichloroethylene)
5175	Trichlorofluoromethane (Fluorotrichloromethane, Freon 11)
5225	Vinyl acetate
5235	Vinyl chloride
5260	Xylene (total)

EPA 8270D **10186002** **Semivolatile Organic compounds by GC/MS**

Analyte Code	Analyte
6715	1,2,4,5-Tetrachlorobenzene
5155	1,2,4-Trichlorobenzene
4610	1,2-Dichlorobenzene
6221	1,2-Diphenylhydrazine
6885	1,3,5-Trinitrobenzene (1,3,5-TNB)
4615	1,3-Dichlorobenzene
4620	1,4-Dichlorobenzene
6420	1,4-Naphthoquinone
6630	1,4-Phenylenediamine
5790	1-Chloronaphthalene
6380	1-Methylnaphthalene
6425	1-Naphthylamine
6735	2,3,4,6-Tetrachlorophenol
6835	2,4,5-Trichlorophenol
6795	2,4,6-Trichloroaniline
6840	2,4,6-Trichlorophenol
6000	2,4-Dichlorophenol
6130	2,4-Dimethylphenol
6175	2,4-Dinitrophenol
6185	2,4-Dinitrotoluene (2,4-DNT)
5992	2,5-Dichlorophenol
6005	2,6-Dichlorophenol
6190	2,6-Dinitrotoluene (2,6-DNT)
5735	2-Chloroaniline
5795	2-Chloronaphthalene
5800	2-Chlorophenol
6360	2-Methyl-4,6-dinitrophenol (4,6-Dinitro-2-methylphenol)
5145	2-Methylaniline (o-Toluidine)
6385	2-Methylnaphthalene
6400	2-Methylphenol (o-Cresol)
6430	2-Naphthylamine
6460	2-Nitroaniline
6490	2-Nitrophenol
5050	2-Picoline (2-Methylpyridine)
6412	3 & 4 Methylphenol
5945	3,3'-Dichlorobenzidine
6120	3,3'-Dimethylbenzidine
6355	3-Methylcholanthrene
6405	3-Methylphenol (m-Cresol)
6465	3-Nitroaniline
5540	4-Aminobiphenyl
5660	4-Bromophenyl phenyl ether (BDE-3)
5700	4-Chloro-3-methylphenol
5745	4-Chloroaniline
5825	4-Chlorophenyl phenylether

NELAP RECOGNIZED TNI ACCREDITATION BODY

ORELAP Fields of Accreditation

ORELAP ID: WA100010
EPA CODE: WA01276
Certificate: WA100010 - 010

ALS Environmental, Kelso

1317 South 13th Ave.
Kelso WA 98626

Issue Date: 02/11/2015 ***Expiration Date:*** 02/10/2016

As of 02/11/2015 ***this list supercedes all previous lists for this certificate number. Customers. Please verify the current accreditation standing with ORELAP.***

Analyte Code	Analyte
6410	4-Methylphenol (p-Cresol)
6470	4-Nitroaniline
6500	4-Nitrophenol
6125	a-a-Dimethylphenethylamine
5500	Acenaphthene
5505	Acenaphthylene
5510	Acetophenone
5545	Aniline
5555	Anthracene
5560	Aramite
7065	Atrazine
5562	Azobenzene
5570	Benzaldehyde
5575	Benzo(a)anthracene
5580	Benzo(a)pyrene
5605	Benzo(e)pyrene
5590	Benzo(g,h,i)perylene
9309	Benzo(j)fluoranthene
5600	Benzo(k)fluoranthene
5585	Benzo[b]fluoranthene
5610	Benzoic acid
5630	Benzyl alcohol
5640	Biphenyl
5760	bis(2-Chloroethoxy)methane
5765	bis(2-Chloroethyl) ether
5780	bis(2-Chloroisopropyl) ether
5670	Butyl benzyl phthalate
7180	Caprolactam
5680	Carbazole
7260	Chlorobenzilate
5855	Chrysene
6065	Di(2-ethylhexyl) phthalate (bis(2-Ethylhexyl)phthalate, DEHP)
7405	Diallate
7410	Diazinon
5895	Dibenz(a,h) anthracene
5905	Dibenzofuran
6070	Diethyl phthalate
7475	Dimethoate
6135	Dimethyl phthalate
5925	Di-n-butyl phthalate
6200	Di-n-octyl phthalate
7580	Famphur
6265	Fluoranthene
6270	Fluorene
6275	Hexachlorobenzene
4835	Hexachlorobutadiene
6285	Hexachlorocyclopentadiene
4840	Hexachloroethane
6290	Hexachlorophene
6315	Indeno(1,2,3-cd) pyrene
7725	Isodrin
6320	Isophorone
7740	Kepone
6345	Methapyrilene
7825	Methyl parathion (Parathion, methyl)
5005	Naphthalene
5015	Nitrobenzene
6525	n-Nitrosodiethylamine
6530	n-Nitrosodimethylamine
6545	n-Nitrosodi-n-propylamine
6535	n-Nitrosodiphenylamine

UNCONTROLLED COPY

Analyte Code	Analyte
6555	n-Nitrosomorpholine
6560	n-Nitrosopiperidine
6565	n-Nitrosopyrrolidine
7955	Parathion, ethyl
6590	Pentachlorobenzene
6600	Pentachloronitrobenzene
6605	Pentachlorophenol
6608	Perylene
6615	Phenanthrene
6625	Phenol
6650	Pronamide (Kerb)
6665	Pyrene
5095	Pyridine
6685	Safrole
8235	Thionazin (Zinophos)

EPA 8270D SIM **10242509** **Semivolatile Organic compounds by GC/MS Selective Ion Monitoring**

Analyte Code	Analyte
4735	1,4-Dioxane (1,4- Diethyleneoxide)
6380	1-Methylnaphthalene
6852	2,3,5-Trimethylnaphthalene
6188	2,6-Dimethylnaphthalene
6385	2-Methylnaphthalene
5500	Acenaphthene
5505	Acenaphthylene
5555	Anthracene
5575	Benzo(a)anthracene
5580	Benzo(a)pyrene
5605	Benzo(e)pyrene
5590	Benzo(g,h,i)perylene
9309	Benzo(j)fluoranthene
5600	Benzo(k)fluoranthene
5585	Benzo[b]fluoranthene
5640	Biphenyl
5670	Butyl benzyl phthalate
5680	Carbazole
5855	Chrysene
6065	Di(2-ethylhexyl) phthalate (bis(2-Ethylhexyl)phthalate, DEHP)
5895	Dibenz(a,h) anthracene
5905	Dibenzofuran
5910	Dibenzothiophene
6070	Diethyl phthalate
6135	Dimethyl phthalate
5925	Di-n-butyl phthalate
6200	Di-n-octyl phthalate
6265	Fluoranthene
6270	Fluorene
6315	Indeno(1,2,3-cd) pyrene
5005	Naphthalene
6545	n-Nitrosodi-n-propylamine
6605	Pentachlorophenol
6608	Perylene
6615	Phenanthrene
6665	Pyrene

EPA 8321B **10189205** **Solvent Extractable non-volatile compounds by HPLC/TS/MS**

Analyte Code	Analyte
7710	3-Hydroxycarbofuran
7010	Aldicarb (Temik)

UNCONTROLLED COPY

Analyte Code	Analyte
7015	Aldicarb sulfone
7020	Aldicarb sulfoxide
7195	Carbaryl (Sevin)
7205	Carbofuran (Furaden)
7800	Methiocarb (Mesurol)
7805	Methomyl (Lannate)
7940	Oxamyl
8080	Propoxur (Baygon)

EPA 8330B **10308006** **Nitroaromatics, Nitramines and Nitrate Esters by High Performance Liquid Chromatography (HPLC)**

Analyte Code	Analyte
6885	1,3,5-Trinitrobenzene (1,3,5-TNB)
6160	1,3-Dinitrobenzene (1,3-DNB)
9651	2,4,6-Trinitrotoluene (2,4,6-TNT)
6185	2,4-Dinitrotoluene (2,4-DNT)
6190	2,6-Dinitrotoluene (2,6-DNT)
9303	2-Amino-4,6-dinitrotoluene (2-am-dnt)
9507	2-Nitrotoluene
6150	3,5-Dinitroaniline
9510	3-Nitrotoluene
9306	4-Amino-2,6-dinitrotoluene (4-am-dnt)
9513	4-Nitrotoluene
6415	Methyl-2,4,6-trinitrophenylnitramine (tetryl)
5015	Nitrobenzene
6485	Nitroglycerin
9522	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)
9558	Pentaerythritoltetranitrate
9432	RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine)

EPA 9012B **10243206** **Total and Amenable Cyanide (automated colorimetric with off-line distillation)**

Analyte Code	Analyte
1510	Amenable cyanide
1645	Total cyanide

EPA 9013A **10308802** **Cyanide Extraction Procedure for Solids and Oils**

Analyte Code	Analyte
8031	Extraction/Preparation

EPA 9020B **10194408** **Total Organic Halides**

Analyte Code	Analyte
2045	Total organic halides (TOX)

EPA 9030B **10195605** **Acid-Soluble and Acid-Insoluble sulfides: Distillation**

Analyte Code	Analyte
2005	Sulfide

EPA 9034 **10196006** **Titrimetric Procedure for Acid-Soluble and Acid-Insoluble Sulfides**

Analyte Code	Analyte
2005	Sulfide

EPA 9045D **10244607** **Soil and Waste pH**

Analyte Code	Analyte
1900	pH

UNCONTROLLED COPY

ORELAP Fields of Accreditation

ORELAP ID: WA100010
EPA CODE: WA01276
Certificate: WA100010 - 010

ALS Environmental, Kelso

1317 South 13th Ave.
Kelso WA 98626

Issue Date: 02/11/2015 **Expiration Date:** 02/10/2016

As of 02/11/2015 ***this list supercedes all previous lists for this certificate number. Customers. Please verify the current accreditation standing with ORELAP.***

EPA 9056A **10199607** **Determination of Inorganic Anions by Ion Chromatography**

Analyte Code	Analyte
1575	Chloride
1730	Fluoride
1805	Nitrate
1835	Nitrite
2000	Sulfate

EPA 9071A **10201408** **Oil and Grease Extraction Method for sludge and sediment samples**

Analyte Code	Analyte
1860	Oil & Grease

NWTPH-Dx **90018409** **Oregon DEQ TPH Diesel Range**

Analyte Code	Analyte
9369	Diesel range organics (DRO)
9506	Residual Range Organics (RRO)

NWTPH-Gx **90018603** **Oregon DEQ TPH Gasoline Range Organics by GC/FID-PID Purge & Trap**

Analyte Code	Analyte
9408	Gasoline range organics (GRO)

NWTPH-HCID **90013200** **Oregon DEQ Total Petroleum Hydrocarbon ID**

Analyte Code	Analyte
2050	Total Petroleum Hydrocarbons (TPH)

PLUMB 1981 **60006259** **Extraction/Preparation**

Analyte Code	Analyte
6118	Distribution of particle sizes
8031	Extraction/Preparation

WI(95) DRO **90019457** **Wisconsin DNR - Modified Method for Determination of Diesel Range Organics by GC-FID**

Analyte Code	Analyte
9369	Diesel range organics (DRO)

UNCONTROLLED COPY

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:

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:

Initial Accreditation Date:	*Issue Date:*	*Revision Date:*
July 19, 2011	March 13, 2014	February 25, 2015
Expiration Date:	*Accreditation No.:*	*Certificate No.:*
March 13, 2016	65188	L14-51-R2

Tracy Szerszen
President/Operations Manager

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

UNCONTROLLED COPY

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental-Kelso

1317 South 13th Avenue, Kelso, WA 98626
Lee Wolf 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	Nephelometer	Turbidity
Aqueous	EPA 2340B	Calculation by 6010	Hardness as $CaCO_{3)}$
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 + Nitrite as N
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	Automated Colorimetry	Nitrate + Nitrite as N
Aqueous	EPA 1632	HG-CT-GC-AAS	Arsenic (III)
Aqueous	EPA 1632	HG-CT-GC-AAS	Arsenic (V)
Aqueous	EPA 1632	HG-CT-GC-AAS	Total Inorganic Arsenic
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
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

UNCONTROLLED COPY

PJLA

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental-Kelso

1317 South 13th Avenue, Kelso, WA 98626

Lee Wolf Phone: 360-577-7222

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard/Method	Technology	Analyte
Aqueous	EPA 8260C SIM	GC-MS	Trichloroethene
Aqueous	EPA 8260C SIM	GC-MS	Vinyl chloride
Aqueous	EPA 9020B	Microcoulometric-titration detector	Total Organic Halides (TOX)
Aqueous	EPA 9040C	pH Meter	pH
Aqueous	EPA 9060A	TOC Meter	Total Organic Carbons (TOC)
Aqueous	SM 10200 H	Colorimetry	Chlorophyll-A
Aqueous	SM 2130B	Nephelometer	Turbidity
Aqueous	SM 2320B	Titrimetry	Total Alkalinity (as $CaCO_3$)
Aqueous	SM 2510B	Conductivity Meter	Specific Conductance
Aqueous	SM 2540B	Balance	Solids, Total
Aqueous	SM 2540C	Balance	Solids, Total Dissolved
Aqueous	SM 2540D	Balance	Solids, Total Suspended
Aqueous	SM 4500-CN- G	Colorimetry	Cyanide, Amenable
Aqueous	SM 4500-P-E	Colorimetry	ortho-phosphorous
Aqueous	SM 4500-S2 D	Distillation Unit	Sulfide
Aqueous	SM 4500-CN E	Colorimetry	Total Cyanide
Aqueous	SM4500-NH3 G	Colorimetry	Ammonia
Aqueous	SM5220C	Titrimetry	Chemical Oxygen Demand (COD)
Aqueous	SM5310C	TOC Meter	Total Organic Carbons (TOC)
Aqueous	SOP-LCP-PFC	HPLC/MS/MS	Perfluor-n butanoic acid (PFBA)
Aqueous	SOP-LCP-PFC	HPLC/MS/MS	Perfluor-n octanesulfonate (PFOS)
Aqueous	SOP-LCP-PFC	HPLC/MS/MS	Perfluor-n octanoic acid (PFOA)
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

UNCONTROLLED COPY

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental-Kelso

1317 South 13th Avenue, Kelso, WA 98626
Lee Wolf Phone: 360-577-7222

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard/Method	Technology	Analyte
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
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

UNCONTROLLED COPY

PJLA

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental-Kelso

1317 South 13th Avenue, Kelso, WA 98626
Lee Wolf Phone: 360-577-7222

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard/Method	Technology	Analyte
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	TOC Meter	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	pH Meter	pH
Solid	EPA 9056A	IC	Nitrate as N
Solid	EPA 9056A	IC	Nitrite as N
Solid	EPA 9071B	Gravimetry	Hexane Extractable Material (HEM)
Solid	GEN-AVS	Colorimetry	Acid Volatile Sulfides
Solid	GEN-NCEL	Colorimetry	Nitrocellulose
Solid	LCP-LCMS4	HPLC/MS/MS	1,3,5-Trinitrobenzene
Solid	LCP-LCMS4	HPLC/MS/MS	1,3-Dinitrobenzene
Solid	LCP-LCMS4	HPLC/MS/MS	2,4,6-Trinitrotoluene
Solid	LCP-LCMS4	HPLC/MS/MS	2,4-Dinitrotoluene
Solid	LCP-LCMS4	HPLC/MS/MS	2,6-Dinitrotoluene
Solid	LCP-LCMS4	HPLC/MS/MS	2-Amino-4,6-dinitrotoluene
Solid	LCP-LCMS4	HPLC/MS/MS	3,5-Dinitroaniline
Solid	LCP-LCMS4	HPLC/MS/MS	4-Amino-2,6-dinitrotoluene
Solid	LCP-LCMS4	HPLC/MS/MS	HMX (Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine)
Solid	LCP-LCMS4	HPLC/MS/MS	Pentaerythritoltetranitrate
Solid	LCP-LCMS4	HPLC/MS/MS	RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine)
Solid	LCP-LCMS4	HPLC/MS/MS	Tetryl (methyl-2,4,6-trinitrophenylnitramine)

UNCONTROLLED COPY

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental-Kelso
1317 South 13th Avenue, Kelso, WA 98626
Lee Wolf Phone: 360-577-7222

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard/Method	Technology	Analyte
Solid	LCP-Nitro	HPLC/MS/MS	2,4-Dinitrophenol
Solid	LCP-Nitro	HPLC/MS/MS	Picramic Acid
Solid	LCP-Nitro	HPLC/MS/MS	Picric Acid
Solid	PSEP	Gravimetry	Particle Size
Solid	SOP-GEN-AVS	Colorimetry	Acid Volatile Sulfides
Tissue	EPA 1631E	CVAFS	Mercury (low level)
Tissue	EPA 1632	HG-CT-GC-AAS	Arsenic (III)
Tissue	EPA 1632	HG-CT-GC-AAS	Arsenic (V)
Tissue	EPA 1632	HG-CT-GC-AAS	Total Inorganic Arsenic
Tissue	EPA 6010B, C/200.7	ICP	Aluminum
Tissue	EPA 6010B, C/200.7	ICP	Antimony
Tissue	EPA 6010B, C/200.7	ICP	Arsenic
Tissue	EPA 6010B, C/200.7	ICP	Barium
Tissue	EPA 6010B, C/200.7	ICP	Beryllium
Tissue	EPA 6010B, C/200.7	ICP	Boron
Tissue	EPA 6010B, C/200.7	ICP	Cadmium
Tissue	EPA 6010B, C/200.7	ICP	Calcium
Tissue	EPA 6010B, C/200.7	ICP	Chromium, total
Tissue	EPA 6010B, C/200.7	ICP	Cobalt
Tissue	EPA 6010B, C/200.7	ICP	Copper
Tissue	EPA 6010B, C/200.7	ICP	Iron
Tissue	EPA 6010B, C/200.7	ICP	Lead
Tissue	EPA 6010B, C/200.7	ICP	Magnesium
Tissue	EPA 6010B, C/200.7	ICP	Manganese
Tissue	EPA 6010B, C/200.7	ICP	Molybdenum
Tissue	EPA 6010B, C/200.7	ICP	Nickel
Tissue	EPA 6010B, C/200.7	ICP	Potassium
Tissue	EPA 6010B, C/200.7	ICP	Selenium
Tissue	EPA 6010B, C/200.7	ICP	Silver
Tissue	EPA 6010B, C/200.7	ICP	Sodium
Tissue	EPA 6010B, C/200.7	ICP	Strontium
Tissue	EPA 6010B, C/200.7	ICP	Thallium
Tissue	EPA 6010B, C/200.7	ICP	Tin
Tissue	EPA 6010B, C/200.7	ICP	Titanium
Tissue	EPA 6010B, C/200.7	ICP	Vanadium

UNCONTROLLED COPY

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental-Kelso
1317 South 13th Avenue, Kelso, WA 98626
Lee Wolf Phone: 360-577-7222

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard/Method	Technology	Analyte
Tissue	EPA 6010B, C/200.7	ICP	Zinc
Tissue	EPA 6020A/200.8	ICP-MS	Aluminum
Tissue	EPA 6020A/200.8	ICP-MS	Antimony
Tissue	EPA 6020A/200.8	ICP-MS	Arsenic
Tissue	EPA 6020A/200.8	ICP-MS	Barium
Tissue	EPA 6020A/200.8	ICP-MS	Beryllium
Tissue	EPA 6020A/200.8	ICP-MS	Boron
Tissue	EPA 6020A/200.8	ICP-MS	Cadmium
Tissue	EPA 6020A/200.8	ICP-MS	Chromium, total
Tissue	EPA 6020A/200.8	ICP-MS	Cobalt
Tissue	EPA 6020A/200.8	ICP-MS	Copper
Tissue	EPA 6020A/200.8	ICP-MS	Iron
Tissue	EPA 6020A/200.8	ICP-MS	Lead
Tissue	EPA 6020A/200.8	ICP-MS	Manganese
Tissue	EPA 6020A/200.8	ICP-MS	Molybdenum
Tissue	EPA 6020A/200.8	ICP-MS	Nickel
Tissue	EPA 6020A/200.8	ICP-MS	Selenium
Tissue	EPA 6020A/200.8	ICP-MS	Silver
Tissue	EPA 6020A/200.8	ICP-MS	Strontium
Tissue	EPA 6020A/200.8	ICP-MS	Thallium
Tissue	EPA 6020A/200.8	ICP-MS	Tin
Tissue	EPA 6020A/200.8	ICP-MS	Titanium
Tissue	EPA 6020A/200.8	ICP-MS	Vanadium
Tissue	EPA 6020A/200.8	ICP-MS	Zinc
Tissue	EPA 7471A, B	CVAA	Mercury
Tissue	EPA 7742	AA, Borohydride Reduction; GFAA	Selenium
Tissue	EPA 8081A, B	GC-ECD	Aldrin
Tissue	EPA 8081A, B	GC-ECD	Alpha-BHC
Tissue	EPA 8081A, B	GC-ECD	DDD (4,4)
Tissue	EPA 8081A, B	GC-ECD	DDE (4,4)
Tissue	EPA 8081A, B	GC-ECD	DDT (4,4)
Tissue	EPA 8081A, B	GC-ECD	delta-BHC
Tissue	EPA 8081A, B	GC-ECD	Dieldrin
Tissue	EPA 8081A, B	GC-ECD	Endosulfan I
Tissue	EPA 8081A, B	GC-ECD	Endosulfan II

UNCONTROLLED COPY

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental-Kelso

1317 South 13th Avenue, Kelso, WA 98626

Lee Wolf Phone: 360-577-7222

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard/Method	Technology	Analyte
Tissue	EPA 8081A, B	GC-ECD	Endosulfan sulfate
Tissue	EPA 8081A, B	GC-ECD	Endrin
Tissue	EPA 8081A, B	GC-ECD	Endrin aldehyde
Tissue	EPA 8081A, B	GC-ECD	Endrin ketone
Tissue	EPA 8081A, B	GC-ECD	gamma-BHC
Tissue	EPA 8081A, B	GC-ECD	gamma-Chlordane
Tissue	EPA 8081A, B	GC-ECD	Heptachlor
Tissue	EPA 8081A, B	GC-ECD	Heptachlor Epoxide (beta)
Tissue	EPA 8081A, B	GC-ECD	Methoxychlor
Tissue	EPA 8081A, B	GC-ECD	Toxaphene (total)
Tissue	EPA 8081B	GC-ECD	2,4-DDD
Tissue	EPA 8081B	GC-ECD	2,4-DDE
Tissue	EPA 8081B	GC-ECD	2,4-DDT
Tissue	EPA 8081B	GC-ECD	Chlorpyrifos
Tissue	EPA 8081B	GC-ECD	cis-Nonachlor
Tissue	EPA 8081B	GC-ECD	Hexachlorobenzene
Tissue	EPA 8081B	GC-ECD	Hexachloroethane
Tissue	EPA 8081B	GC-ECD	Hexchlorobutadiene
Tissue	EPA 8081B	GC-ECD	Isodrin
Tissue	EPA 8081B	GC-ECD	Mirex
Tissue	EPA 8081B	GC-ECD	Oxychlordane
Tissue	EPA 8081B	GC-ECD	trans-Nonachlor
Tissue	EPA 8082A	GC-ECD	2,2',3,3',4,4',5,5',6,6' Decachlorobiphenyl (PCB 209)
Tissue	EPA 8082A	GC-ECD	2,2’,3,3’,4,4’,5,5’,6-Nonachlorobiphenyl (PCB 206)
Tissue	EPA 8082A	GC-ECD	2,2’,3,3’,4,4’,5,6-Octachlorobiphenyl (PCB 195)
Tissue	EPA 8082A	GC-ECD	2,2’,3,3’,4,4’,5-Heptachlorobiphenyl (PCB 170)
Tissue	EPA 8082A	GC-ECD	2,2’,3,3’,4,4’-Hexachlorobiphenyl (PCB 128)
Tissue	EPA 8082A	GC-ECD	2,2’,3,4,4’,5,5’-Heptachlorobiphenyl (PCB 180)
Tissue	EPA 8082A	GC-ECD	2,2’,3,4,4’,5’,6-Heptachlorobiphenyl (PCB 183)
Tissue	EPA 8082A	GC-ECD	2,2’,3,4,4’,5’-Hexachlorobiphenyl (PCB 138)
Tissue	EPA 8082A	GC-ECD	2,2’,3,4,4’,6,6’-Heptachlorobiphenyl (PCB 184)
Tissue	EPA 8082A	GC-ECD	2,2’,3,4,5’-Pentachlorobiphenyl (PCB 87)
Tissue	EPA 8082A	GC-ECD	2,2’,3,4’,5,5’,6-Heptachlorobiphenyl (PCB 187)
Tissue	EPA 8082A	GC-ECD	2,2’,3,4’,5-Pentachlorobiphenyl (PCB 90)
Tissue	EPA 8082A	GC-ECD	2,2’,3,5’-Tetrachlorobiphenyl (PCB 44)
Tissue	EPA 8082A	GC-ECD	2,2’,4,4’,5,5’-Hexachlorobiphenyl (PCB 153)

UNCONTROLLED COPY

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental-Kelso

1317 South 13th Avenue, Kelso, WA 98626
Lee Wolf Phone: 360-577-7222

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard/Method	Technology	Analyte
Tissue	EPA 8082A	GC-ECD	2,2',4,5,5'-Pentachlorobiphenyl (PCB 101)
Tissue	EPA 8082A	GC-ECD	2,2',5,6'-Tetrachlorbiphenyl (PCB 53)
Tissue	EPA 8082A	GC-ECD	2,2',5-Trichlorobiphenyl (PCB 18)
Tissue	EPA 8082A	GC-ECD	2,3,3',4,4',5,5'-Heptachlorobiphenyl (PCB 189)
Tissue	EPA 8082A	GC-ECD	2,3,3',4,4',5'-Hexachlorobiphenyl (PCB 157)
Tissue	EPA 8082A	GC-ECD	2,3,3',4,4',5-Hexachlorobiphenyl (PCB 156)
Tissue	EPA 8082A	GC-ECD	2,3,3',4,4',6-Hexachlorobiphenyl (PCB 158)
Tissue	EPA 8082A	GC-ECD	2,3,3',4,4'-Pentachlorobiphenyl (PCB 105)
Tissue	EPA 8082A	GC-ECD	2,3,4,4',5-Pentachlorobiphenyl (PCB 114)
Tissue	EPA 8082A	GC-ECD	2,3,4,4'-Tetrachlorobiphenyl (PCB 60)
Tissue	EPA 8082A	GC-ECD	2,3',4,4',5,5' Hexachlorobiphenyl (PCB 167)
Tissue	EPA 8082A	GC-ECD	2,3',4,4',5',6-Hexachlorobiphenyl (PCB 168)
Tissue	EPA 8082A	GC-ECD	2,3',4,4',5-Pentachlorobiphenyl (PCB 118)
Tissue	EPA 8082A	GC-ECD	2,3',4,4',5-Pentachlorobiphenyl (PCB 123)
Tissue	EPA 8082A	GC-ECD	2,3',4,4'-Tetrachlorobiphenyl (PCB 66)
Tissue	EPA 8082A	GC-ECD	2,4,4'-Trichlorobiphenyl (PCB 28)
Tissue	EPA 8082A	GC-ECD	2,4'-Dichlorobiphenyl (PCB 8)
Tissue	EPA 8082A	GC-ECD	3,3',4,4',5,5'-Hexachlorobiphenyl (PCB 169)
Tissue	EPA 8082A	GC-ECD	3,3',4,4',5-Pentachlorobiphenyl (PCB 126)
Tissue	EPA 8082A	GC-ECD	3,3',4,4'-Tetrachlorobiphenyl (PCB 77)
Tissue	EPA 8082A	GC-ECD	3,4,4',5-Tetrachlorobiphenyl (PCB 81)
Tissue	EPA 8082A	GC-ECD	Aroclor 1016
Tissue	EPA 8082A	GC-ECD	Aroclor 1221
Tissue	EPA 8082A	GC-ECD	Aroclor 1232
Tissue	EPA 8082A	GC-ECD	Aroclor 1242
Tissue	EPA 8082A	GC-ECD	Aroclor 1248
Tissue	EPA 8082A	GC-ECD	Aroclor 1254
Tissue	EPA 8082A	GC-ECD	Aroclor 1260
Tissue	EPA 8082A	GC-ECD	Aroclor 1262
Tissue	EPA 8082A	GC-ECD	Aroclor 1268
Tissue	EPA 8270 SIM	GC-MS	PBDE 100
Tissue	EPA 8270 SIM	GC-MS	PBDE 128
Tissue	EPA 8270 SIM	GC-MS	PBDE 138
Tissue	EPA 8270 SIM	GC-MS	PBDE 153
Tissue	EPA 8270 SIM	GC-MS	PBDE 154
Tissue	EPA 8270 SIM	GC-MS	PBDE 17

UNCONTROLLED COPY

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental-Kelso

1317 South 13th Avenue, Kelso, WA 98626
Lee Wolf Phone: 360-577-7222

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard/Method	Technology	Analyte
Tissue	EPA 8270 SIM	GC-MS	PBDE 183
Tissue	EPA 8270 SIM	GC-MS	PBDE 190
Tissue	EPA 8270 SIM	GC-MS	PBDE 203
Tissue	EPA 8270 SIM	GC-MS	PBDE 206
Tissue	EPA 8270 SIM	GC-MS	PBDE 209
Tissue	EPA 8270 SIM	GC-MS	PBDE 28
Tissue	EPA 8270 SIM	GC-MS	PBDE 47
Tissue	EPA 8270 SIM	GC-MS	PBDE 66
Tissue	EPA 8270 SIM	GC-MS	PBDE 71
Tissue	EPA 8270 SIM	GC-MS	PBDE 85
Tissue	EPA 8270 SIM	GC-MS	PBDE 99
Tissue	EPA 8270 SIM PAH	GC-MS	2-Methylnaphthalene
Tissue	EPA 8270 SIM PAH	GC-MS	Acenaphthene
Tissue	EPA 8270 SIM PAH	GC-MS	Acenaphthylene
Tissue	EPA 8270 SIM PAH	GC-MS	Anthracene
Tissue	EPA 8270 SIM PAH	GC-MS	Benzo(a)anthracene
Tissue	EPA 8270 SIM PAH	GC-MS	Benzo(a)pyrene
Tissue	EPA 8270 SIM PAH	GC-MS	Benzo(b)fluoranthene
Tissue	EPA 8270 SIM PAH	GC-MS	Benzo(g,h,i)perylene
Tissue	EPA 8270 SIM PAH	GC-MS	Benzo(k)fluoranthene
Tissue	EPA 8270 SIM PAH	GC-MS	Chrysene
Tissue	EPA 8270 SIM PAH	GC-MS	Dibenzo(a,h)anthracene
Tissue	EPA 8270 SIM PAH	GC-MS	Fluoranthene
Tissue	EPA 8270 SIM PAH	GC-MS	Fluorene
Tissue	EPA 8270 SIM PAH	GC-MS	Indeno(1,2,3, cd)pyrene
Tissue	EPA 8270 SIM PAH	GC-MS	Naphthalene
Tissue	EPA 8270 SIM PAH	GC-MS	Phenanthrene
Tissue	EPA 8270 SIM PAH	GC-MS	Pyrene
Tissue	EPA 8270D SIM	GC-MS	1,2,4,5-Tetrachlorobenzene
Tissue	EPA 8270D SIM	GC-MS	1,2,4-Trichlorobenzene
Tissue	EPA 8270D SIM	GC-MS	1,2-Dichlorobenzene
Tissue	EPA 8270D SIM	GC-MS	1,3-Dichlorobenzene
Tissue	EPA 8270D SIM	GC-MS	1,4-Dichlorobenzene
Tissue	EPA 8270D SIM	GC-MS	2,3,4,6-Tetrachlorophenol
Tissue	EPA 8270D SIM	GC-MS	2,4,5-Trichlorophenol
Tissue	EPA 8270D SIM	GC-MS	2,4,6-Trichlorophenol

UNCONTROLLED COPY

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental-Kelso

1317 South 13th Avenue, Kelso, WA 98626

Lee Wolf Phone: 360-577-7222

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard/Method	Technology	Analyte
Tissue	EPA 8270D SIM	GC-MS	2,4-Dichlorophenol
Tissue	EPA 8270D SIM	GC-MS	2,4-Dimethylphenol
Tissue	EPA 8270D SIM	GC-MS	2,4-Dinitrophenol
Tissue	EPA 8270D SIM	GC-MS	2,4-Dinitrotoluene
Tissue	EPA 8270D SIM	GC-MS	2,6-Dichlorophenol
Tissue	EPA 8270D SIM	GC-MS	2,6-Dinitrotoluene
Tissue	EPA 8270D SIM	GC-MS	2-Chloronaphthalene
Tissue	EPA 8270D SIM	GC-MS	2-Chlorophenol
Tissue	EPA 8270D SIM	GC-MS	2-Methyl-4,6-Dinitrophenol
Tissue	EPA 8270D SIM	GC-MS	2-Methylnaphthalene
Tissue	EPA 8270D SIM	GC-MS	2-Methylphenol
Tissue	EPA 8270D SIM	GC-MS	2-Nitroaniline
Tissue	EPA 8270D SIM	GC-MS	2-Nitrophenol
Tissue	EPA 8270D SIM	GC-MS	3,3-Dichlorobenzidine
Tissue	EPA 8270D SIM	GC-MS	3-Nitroaniline
Tissue	EPA 8270D SIM	GC-MS	4-Bromophenyl-phenylether
Tissue	EPA 8270D SIM	GC-MS	4-Chloro-3-methylphenol
Tissue	EPA 8270D SIM	GC-MS	4-Chloroaniline
Tissue	EPA 8270D SIM	GC-MS	4-Chlorophenyl-phenylether
Tissue	EPA 8270D SIM	GC-MS	4-Methylphenol (and/or 3-Methylphenol)
Tissue	EPA 8270D SIM	GC-MS	4-Nitroaniline
Tissue	EPA 8270D SIM	GC-MS	4-Nitrophenol
Tissue	EPA 8270D SIM	GC-MS	Acenaphthene
Tissue	EPA 8270D SIM	GC-MS	Acenaphthylene
Tissue	EPA 8270D SIM	GC-MS	Anthracene
Tissue	EPA 8270D SIM	GC-MS	Benzo(a)anthracene
Tissue	EPA 8270D SIM	GC-MS	Benzo(a)pyrene
Tissue	EPA 8270D SIM	GC-MS	Benzo(b)fluoranthene
Tissue	EPA 8270D SIM	GC-MS	Benzo(g,h,i)perylene
Tissue	EPA 8270D SIM	GC-MS	Benzo(k)fluoranthene
Tissue	EPA 8270D SIM	GC-MS	Benzoic acid
Tissue	EPA 8270D SIM	GC-MS	Benzyl alcohol
Tissue	EPA 8270D SIM	GC-MS	bis(2-Chloroethoxy)methane
Tissue	EPA 8270D SIM	GC-MS	bis(2-Chloroethyl)ether
Tissue	EPA 8270D SIM	GC-MS	bis(2-Chloroisopropyl)ether
Tissue	EPA 8270D SIM	GC-MS	bis(2-ethylhexy)phthalate

UNCONTROLLED COPY

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental-Kelso

1317 South 13th Avenue, Kelso, WA 98626

Lee Wolf Phone: 360-577-7222

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard/Method	Technology	Analyte
Tissue	EPA 8270D SIM	GC-MS	Butyl benzyl phthalate
Tissue	EPA 8270D SIM	GC-MS	Carbazole
Tissue	EPA 8270D SIM	GC-MS	Chrysene
Tissue	EPA 8270D SIM	GC-MS	Dibenzo(a,h)anthracene
Tissue	EPA 8270D SIM	GC-MS	Dibenzofuran
Tissue	EPA 8270D SIM	GC-MS	Diethyl phthalate
Tissue	EPA 8270D SIM	GC-MS	Dimethylphthalate
Tissue	EPA 8270D SIM	GC-MS	di-n-butylphthalate
Tissue	EPA 8270D SIM	GC-MS	Di-n-octylphthalate
Tissue	EPA 8270D SIM	GC-MS	Fluoranthene
Tissue	EPA 8270D SIM	GC-MS	Fluorene
Tissue	EPA 8270D SIM	GC-MS	Hexachlorobenzene
Tissue	EPA 8270D SIM	GC-MS	Hexachlorobutadiene
Tissue	EPA 8270D SIM	GC-MS	Hexachlorocyclopentadiene
Tissue	EPA 8270D SIM	GC-MS	Hexachloroethane
Tissue	EPA 8270D SIM	GC-MS	Indeno(1,2,3, cd)pyrene
Tissue	EPA 8270D SIM	GC-MS	Isophorone
Tissue	EPA 8270D SIM	GC-MS	Naphthalene
Tissue	EPA 8270D SIM	GC-MS	Nitrobenzene
Tissue	EPA 8270D SIM	GC-MS	N-Nitrosodiethylamine
Tissue	EPA 8270D SIM	GC-MS	N-Nitrosodimethylamine
Tissue	EPA 8270D SIM	GC-MS	N-Nitroso-di-n-propylamine
Tissue	EPA 8270D SIM	GC-MS	N-Nitrosodiphenylamine
Tissue	EPA 8270D SIM	GC-MS	Pentachlorophenol
Tissue	EPA 8270D SIM	GC-MS	Phenanthrene
Tissue	EPA 8270D SIM	GC-MS	Phenol
Tissue	EPA 8270D SIM	GC-MS	Pyrene
Tissue	EPA 8330B	HPLC	1,3,5-Trinitrobenzene
Tissue	EPA 8330B	HPLC	1,3-Dinitrobenzene
Tissue	EPA 8330B	HPLC	2,4,6-Trinitrotoluene
Tissue	EPA 8330B	HPLC	2,4-Dinitrotoluene
Tissue	EPA 8330B	HPLC	2,6-Dinitrotoluene
Tissue	EPA 8330B	HPLC	2-Amino-4,6-dinitrotoluene
Tissue	EPA 8330B	HPLC	2-Nitrotoluene
Tissue	EPA 8330B	HPLC	3,5-Dinitroaniline
Tissue	EPA 8330B	HPLC	3-Nitrotoluene

UNCONTROLLED COPY

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental-Kelso
1317 South 13th Avenue, Kelso, WA 98626
Lee Wolf Phone: 360-577-7222

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard/Method	Technology	Analyte
Tissue	EPA 8330B	HPLC	4-Amino-2,6-dinitrotoluene
Tissue	EPA 8330B	HPLC	4-Nitrotoluene
Tissue	EPA 8330B	HPLC	HMX (octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine)
Tissue	EPA 8330B	HPLC	Nitrobenzene
Tissue	EPA 8330B	HPLC	Nitroglycerin
Tissue	EPA 8330B	HPLC	Pentachloronitrobenzene
Tissue	EPA 8330B	HPLC	Pentaerythritoltetranitrate
Tissue	EPA 8330B	HPLC	RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine)
Tissue	EPA 8330B	HPLC	Tetryl (methyl-2,4,6-trinitrophenylnitramine)
Tissue	OPPMS2	GC/MS/MS	Azinphos-methyl (Guthion)
Tissue	OPPMS2	GC/MS/MS	Chlorpyrifos
Tissue	OPPMS2	GC/MS/MS	Demeton O & S
Tissue	OPPMS2	GC/MS/MS	Diazinon
Tissue	OPPMS2	GC/MS/MS	Dichlorvos
Tissue	OPPMS2	GC/MS/MS	dimethoate
Tissue	OPPMS2	GC/MS/MS	Disulfoton
Tissue	OPPMS2	GC/MS/MS	Ethoprop
Tissue	OPPMS2	GC/MS/MS	Parathion, ethyl
Tissue	OPPMS2	GC/MS/MS	Parathion, methyl
Tissue	OPPMS2	GC/MS/MS	Phorate
Tissue	OPPMS2	GC/MS/MS	Ronnel
Tissue	OPPMS2	GC/MS/MS	Stirophos
Tissue	OPPMS2	GC/MS/MS	Sulfotepp
Tissue	SOC-Butyl	GC-FPD	Di-n-butyltin
Tissue	SOC-Butyl	GC-FPD	n-Butyltin
Tissue	SOC-Butyl	GC-FPD	Tetra-n-butyltin
Tissue	SOC-Butyl	GC-FPD	Tri-n-butyltin
Tissue	SOC-PESTMS2	GC/MS/MS	Aldrin
Tissue	SOC-PESTMS2	GC/MS/MS	Alpha-BHC
Tissue	SOC-PESTMS2	GC/MS/MS	beta-BHC
Tissue	SOC-PESTMS2	GC/MS/MS	DDD (4,4)
Tissue	SOC-PESTMS2	GC/MS/MS	DDE (4,4)
Tissue	SOC-PESTMS2	GC/MS/MS	DDT (4,4)
Tissue	SOC-PESTMS2	GC/MS/MS	delta-BHC
Tissue	SOC-PESTMS2	GC/MS/MS	Dieldrin
Tissue	SOC-PESTMS2	GC/MS/MS	Endosulfan I

UNCONTROLLED COPY

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental-Kelso

1317 South 13th Avenue, Kelso, WA 98626

Lee Wolf Phone: 360-577-7222

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard/Method	Technology	Analyte
Tissue	SOC-PESTMS2	GC/MS/MS	Endosulfan II
Tissue	SOC-PESTMS2	GC/MS/MS	Endosulfan sulfate
Tissue	SOC-PESTMS2	GC/MS/MS	Endrin
Tissue	SOC-PESTMS2	GC/MS/MS	Endrin aldehyde
Tissue	SOC-PESTMS2	GC/MS/MS	Endrin ketone
Tissue	SOC-PESTMS2	GC/MS/MS	gamma-BHC
Tissue	SOC-PESTMS2	GC/MS/MS	Heptachlor
Tissue	SOC-PESTMS2	GC/MS/MS	Heptachlor Epoxide (beta)
Tissue	SOC-PESTMS2	GC/MS/MS	Methoxychlor
Tissue	SOP LCP-LCMS4	HPLC/MS/MS	1,3,5-Trinitrobenzene
Tissue	SOP LCP-LCMS4	HPLC/MS/MS	1,3-Dinitrobenzene
Tissue	SOP LCP-LCMS4	HPLC/MS/MS	2,4,6-Trinitrotoluene
Tissue	SOP LCP-LCMS4	HPLC/MS/MS	2,4-Dinitrotoluene
Tissue	SOP LCP-LCMS4	HPLC/MS/MS	2,6-Dinitrotoluene
Tissue	SOP LCP-LCMS4	HPLC/MS/MS	2-Amino-4,6-dinitrtoluene
Tissue	SOP LCP-LCMS4	HPLC/MS/MS	3,5-Dinitroaniline
Tissue	SOP LCP-LCMS4	HPLC/MS/MS	4-Amino-2,6-dinitrotoluene
Tissue	SOP LCP-LCMS4	HPLC/MS/MS	HMX (octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine)
Tissue	SOP LCP-LCMS4	HPLC/MS/MS	Pentaerythritoltetranitrate
Tissue	SOP LCP-LCMS4	HPLC/MS/MS	RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine)
Tissue	SOP LCP-LCMS4	HPLC/MS/MS	Tetryl (methyl-2,4,6-trinitrophenylnitramine)
Tissue	SOP LCP-Nitro	HPLC/MS/MS	2,4-Dinitrophenol
Tissue	SOP LCP-Nitro	HPLC/MS/MS	Picramic Acid
Tissue	SOP LCP-Nitro	HPLC/MS/MS	Picric Acid
Tissue	SOP-LCP-PFC	HPLC/MS/MS	Perfluorobutane Sulfonate
Tissue	SOP-LCP-PFC	HPLC/MS/MS	Perfluorobutanesulfonic Acid
Tissue	SOP-LCP-PFC	HPLC/MS/MS	Perfluorobutanoic Acid
Tissue	SOP-LCP-PFC	HPLC/MS/MS	Perfluorodecane Sulfonate
Tissue	SOP-LCP-PFC	HPLC/MS/MS	Perfluorodecanoic Acid
Tissue	SOP-LCP-PFC	HPLC/MS/MS	Perfluorododecanoic Acid
Tissue	SOP-LCP-PFC	HPLC/MS/MS	Perfluoroheptanoic Acid
Tissue	SOP-LCP-PFC	HPLC/MS/MS	Perfluorohexane Sulfonate
Tissue	SOP-LCP-PFC	HPLC/MS/MS	Perfluorohexanoic Acid
Tissue	SOP-LCP-PFC	HPLC/MS/MS	Perfluorohexylsulfonic Acid
Tissue	SOP-LCP-PFC	HPLC/MS/MS	Perfluorononanoic Acid
Tissue	SOP-LCP-PFC	HPLC/MS/MS	Perfluorooctane Sulfonate

UNCONTROLLED COPY

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental-Kelso

1317 South 13th Avenue, Kelso, WA 98626
Lee Wolf Phone: 360-577-7222

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard/Method	Technology	Analyte
Tissue	SOP-LCP-PFC	HPLC/MS/MS	Perfluorooctanoic Acid
Tissue	SOP-LCP-PFC	HPLC/MS/MS	Perfluorooctylsulfonic Acid
Tissue	SOP-LCP-PFC	HPLC/MS/MS	Perfluoropentanoic Acid
Tissue	SOP-LCP-PFC	HPLC/MS/MS	Perfluoroundecanoic Acid
Aqueous/Drinking Water	EPA 200.9	GFAA	Antimony
Aqueous/Drinking Water	EPA 200.9	GFAA	Selenium
Aqueous/Drinking Water	EPA 200.9	GFAA	Thallium
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/Drinking Water	EPA 200.9	GFAA	Arsenic
Aqueous/Drinking Water	EPA 200.9	GFAA	Lead
Aqueous/Solid	ASTM D 1426-93B	ISE	Nitrogen, Total Kjeldahl (TKN)
Aqueous/Solid	EPA 1020A	Closed Cup Flashpoint	Ignitability
Aqueous/Solid	EPA 1630	CVAFS	Methyl Mercury
Aqueous/Solid	EPA 314.0	IC	Perchlorate
Aqueous/Solid	EPA 350.1	Colorimetry	Ammonia
Aqueous/Solid	EPA 365.3	Colorimetry	Total Phosphorus
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
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

UNCONTROLLED COPY

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental-Kelso

1317 South 13th Avenue, Kelso, WA 98626

Lee Wolf Phone: 360-577-7222

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard/Method	Technology	Analyte
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
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

UNCONTROLLED COPY

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental-Kelso

1317 South 13th Avenue, Kelso, WA 98626

Lee Wolf 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	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 7010	GFAA	Antimony
Aqueous/Solid	EPA 7010	GFAA	Arsenic
Aqueous/Solid	EPA 7010	GFAA	Chromium, total
Aqueous/Solid	EPA 7010	GFAA	Lead
Aqueous/Solid	EPA 7010	GFAA	Selenium
Aqueous/Solid	EPA 7010	GFAA	Thallium
Aqueous/Solid	EPA 7742	AA, Borohydride Reduction; GFAA	Selenium
Aqueous/Solid	EPA 8011	GC-ECD	Ethylene Dibromide
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	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
Aqueous/Solid	EPA 8081A, B	GC-ECD	gamma-BHC
Aqueous/Solid	EPA 8081A, B	GC-ECD	gamma-Chlordane

UNCONTROLLED COPY

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental-Kelso
1317 South 13th Avenue, Kelso, WA 98626
Lee Wolf 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	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)
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)

UNCONTROLLED COPY

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental-Kelso

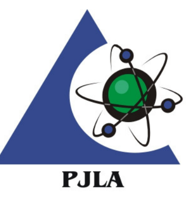
1317 South 13th Avenue, Kelso, WA 98626

Lee Wolf Phone: 360-577-7222

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard/Method	Technology	Analyte
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 8151A	GC-ECD	2,4,5-T
Aqueous/Solid	EPA 8151A	GC-ECD	2,4,5-TP (Silvex)
Aqueous/Solid	EPA 8151A	GC-ECD	2,4-D
Aqueous/Solid	EPA 8151A	GC-ECD	2,4-DB
Aqueous/Solid	EPA 8151A	GC-ECD	Dalapon
Aqueous/Solid	EPA 8151A	GC-ECD	Dicamba
Aqueous/Solid	EPA 8151A	GC-ECD	Dichloroprop

UNCONTROLLED COPY



Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental-Kelso
1317 South 13th Avenue, Kelso, WA 98626
Lee Wolf Phone: 360-577-7222

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard/Method	Technology	Analyte
Aqueous/Solid	EPA 8151A	GC-ECD	Dinoseb
Aqueous/Solid	EPA 8151A	GC-ECD	MCPA
Aqueous/Solid	EPA 8151A	GC-ECD	MCPP
Aqueous/Solid	EPA 8260B, C	GC-MS	1,1,1,2-Tetrachloroethane
Aqueous/Solid	EPA 8260B, C	GC-MS	1,1,1-Trichloroethane
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

UNCONTROLLED COPY

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental-Kelso

1317 South 13th Avenue, Kelso, WA 98626
Lee Wolf 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	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
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

UNCONTROLLED COPY

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental-Kelso

1317 South 13th Avenue, Kelso, WA 98626

Lee Wolf 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	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
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-Dichlorophenol
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

UNCONTROLLED COPY

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental-Kelso

1317 South 13th Avenue, Kelso, WA 98626

Lee Wolf Phone: 360-577-7222

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard/Method	Technology	Analyte
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
Aqueous/Solid	EPA 8270C, D	GC-MS	Anthracene
Aqueous/Solid	EPA 8270C, D	GC-MS	Azinphos-methyl (Guthion)
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	Chlorpyrifos
Aqueous/Solid	EPA 8270C, D	GC-MS	Chrysene
Aqueous/Solid	EPA 8270C, D	GC-MS	Demeton O & S
Aqueous/Solid	EPA 8270C, D	GC-MS	Diazinon
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	Dichlorvos
Aqueous/Solid	EPA 8270C, D	GC-MS	Diethyl phthalate
Aqueous/Solid	EPA 8270C, D	GC-MS	dimethoate
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

UNCONTROLLED COPY

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental-Kelso

1317 South 13th Avenue, Kelso, WA 98626

Lee Wolf Phone: 360-577-7222

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard/Method	Technology	Analyte
Aqueous/Solid	EPA 8270C, D	GC-MS	Disulfoton
Aqueous/Solid	EPA 8270C, D	GC-MS	Ethoprop
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-Nitrosodiethylamine
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
Aqueous/Solid	EPA 8270C, D	GC-MS	o-Toluidine
Aqueous/Solid	EPA 8270C, D	GC-MS	Parathion, ethyl
Aqueous/Solid	EPA 8270C, D	GC-MS	Parathion, methyl
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	Phorate
Aqueous/Solid	EPA 8270C, D	GC-MS	Pyrene
Aqueous/Solid	EPA 8270C, D	GC-MS	Pyridine
Aqueous/Solid	EPA 8270C, D	GC-MS	Ronnel
Aqueous/Solid	EPA 8270C, D	GC-MS	Stirophos
Aqueous/Solid	EPA 8270C, D	GC-MS	Sulfotepp
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 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

UNCONTROLLED COPY

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental-Kelso

1317 South 13th Avenue, Kelso, WA 98626

Lee Wolf Phone: 360-577-7222

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard/Method	Technology	Analyte
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	PBDE 100
Aqueous/Solid	EPA 8270 SIM	GC-MS	PBDE 128
Aqueous/Solid	EPA 8270 SIM	GC-MS	PBDE 138
Aqueous/Solid	EPA 8270 SIM	GC-MS	PBDE 153
Aqueous/Solid	EPA 8270 SIM	GC-MS	PBDE 154
Aqueous/Solid	EPA 8270 SIM	GC-MS	PBDE 17
Aqueous/Solid	EPA 8270 SIM	GC-MS	PBDE 183
Aqueous/Solid	EPA 8270 SIM	GC-MS	PBDE 190
Aqueous/Solid	EPA 8270 SIM	GC-MS	PBDE 203
Aqueous/Solid	EPA 8270 SIM	GC-MS	PBDE 206
Aqueous/Solid	EPA 8270 SIM	GC-MS	PBDE 209
Aqueous/Solid	EPA 8270 SIM	GC-MS	PBDE 28
Aqueous/Solid	EPA 8270 SIM	GC-MS	PBDE 47
Aqueous/Solid	EPA 8270 SIM	GC-MS	PBDE 66
Aqueous/Solid	EPA 8270 SIM	GC-MS	PBDE 71
Aqueous/Solid	EPA 8270 SIM	GC-MS	PBDE 85
Aqueous/Solid	EPA 8270 SIM	GC-MS	PBDE 99
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 8330B	HPLC	1,3,5-Trinitrobenzene
Aqueous/Solid	EPA 8330B	HPLC	1,3-Dinitrobenzene
Aqueous/Solid	EPA 8330B	HPLC	2,4,6-Trinitrotoluene
Aqueous/Solid	EPA 8330B	HPLC	2,4-Dinitrotoluene

UNCONTROLLED COPY

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental-Kelso
1317 South 13th Avenue, Kelso, WA 98626
Lee Wolf Phone: 360-577-7222

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard/Method	Technology	Analyte
Aqueous/Solid	EPA 8330B	HPLC	2,6-Dinitrotoluene
Aqueous/Solid	EPA 8330B	HPLC	2-Amino-4,6-dinitrtoluene
Aqueous/Solid	EPA 8330B	HPLC	2-Nitrotoluene
Aqueous/Solid	EPA 8330B	HPLC	3,5-Dinitroaniline
Aqueous/Solid	EPA 8330B	HPLC	3-Nitrotoluene
Aqueous/Solid	EPA 8330B	HPLC	4-Amino-2,6-dinitrotoluene
Aqueous/Solid	EPA 8330B	HPLC	4-Nitrotoluene
Aqueous/Solid	EPA 8330B	HPLC	HMX (octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine)
Aqueous/Solid	EPA 8330B	HPLC	Nitrobenzene
Aqueous/Solid	EPA 8330B	HPLC	Nitroglycerin
Aqueous/Solid	EPA 8330B	HPLC	Pentachloronitrobenzene
Aqueous/Solid	EPA 8330B	HPLC	Pentaerythritoltetranitrate
Aqueous/Solid	EPA 8330B	HPLC	RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine)
Aqueous/Solid	EPA 8330B	HPLC	Tetryl (methyl-2,4,6-trinitrophenylnitramine)
Aqueous/Solid	EPA 9012B	Colorimetry	Total Cyanide
Aqueous/Solid	EPA 9030B	Distillation Unit	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	EPA 9065	Spectrophotometer	Total Phenolics
Aqueous/Solid	LCP-NITG	HPLC/UV	Nitroguanidine
Aqueous/Solid	NWTPH-Dx	GC-FID	Residual Range Organics
Aqueous/Solid	SM4500 NH3 G	Colorimetry	Ammonia
Aqueous/Solid	SOC-Butyl	GC-FPD	Di-n-butyltin
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	SOC-OTTO	GC-ECD	Otto Fuel
Aqueous/Solid	SOC-PESTMS2	GC/MS/MS/MS	Aldrin
Aqueous/Solid	SOC-PESTMS2	GC/MS/MS/MS	Alpha-BHC
Aqueous/Solid	SOC-PESTMS2	GC/MS/MS/MS	beta-BHC
Aqueous/Solid	SOC-PESTMS2	GC/MS/MS/MS	DDD (4,4)
Aqueous/Solid	SOC-PESTMS2	GC/MS/MS/MS	DDE (4,4)
Aqueous/Solid	SOC-PESTMS2	GC/MS/MS/MS	DDT (4,4)

UNCONTROLLED COPY

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental-Kelso

1317 South 13th Avenue, Kelso, WA 98626

Lee Wolf Phone: 360-577-7222

Accreditation is granted to the facility to perform the following testing:

Matrix	Standard/Method	Technology	Analyte
Aqueous/Solid	SOC-PESTMS2	GC/MS/MS/MS	delta-BHC
Aqueous/Solid	SOC-PESTMS2	GC/MS/MS/MS	Dieldrin
Aqueous/Solid	SOC-PESTMS2	GC/MS/MS/MS	Endosulfan I
Aqueous/Solid	SOC-PESTMS2	GC/MS/MS/MS	Endosulfan II
Aqueous/Solid	SOC-PESTMS2	GC/MS/MS/MS	Endosulfan sulfate
Aqueous/Solid	SOC-PESTMS2	GC/MS/MS/MS	Endrin
Aqueous/Solid	SOC-PESTMS2	GC/MS/MS/MS	Endrin aldehyde
Aqueous/Solid	SOC-PESTMS2	GC/MS/MS/MS	Endrin ketone
Aqueous/Solid	SOC-PESTMS2	GC/MS/MS/MS	gamma-BHC
Aqueous/Solid	SOC-PESTMS2	GC/MS/MS/MS	Heptachlor
Aqueous/Solid	SOC-PESTMS2	GC/MS/MS/MS	Heptachlor Epoxide (beta)
Aqueous/Solid	SOC-PESTMS2	GC/MS/MS/MS	Methoxychlor
Aqueous/Solid	SOP-LCP-PFC	HPLC/MS/MS	Perfluorobutane sulfonate
Aqueous/Solid	SOP-LCP-PFC	HPLC/MS/MS	Perfluorobutanesulfonic Acid
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	Perfluorohexylsulfonic 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	Perfluorooctylsulfonic Acid
Aqueous/Solid	SOP-LCP-PFC	HPLC/MS/MS	Perfluoropentanoic acid
Aqueous/Solid	SOP-LCP-PFC	HPLC/MS/MS	Perfluoroundecanoic acid

UNCONTROLLED COPY

Certificate of Accreditation: Supplement

ISO/IEC 17025:2005 and DoD-ELAP

ALS Environmental-Kelso

1317 South 13th Avenue, Kelso, WA 98626
Lee Wolf Phone: 360-577-7222

Accreditation is granted to the facility to perform the following testing:

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 3520C	Continuous Liquid-Liquid Extraction	Extractable Prep
Aqueous	EPA 3535A	Solid Phase Extraction	Prep Method
Aqueous	EPA 5030B	Purge and Trap for Volatiles	Volatile Prep
Aqueous	SOP-MET-DIG	Acid Digestion	Metals Digestion
Solid	EPA 3050B	Acid Digestion	Metals Digestion
Solid	EPA 3060	Alkaline Digestion for Cr(VI)	Alkaline Digestion for Cr(VI) only
Solid	EPA 3541	Automated Soxhlet Extraction	Extractable Prep
Solid	EPA 3550B	Ultrasonic Extraction	Extractable Prep
Solid	EPA 5035A	Purge and Trap for Volatiles	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

UNCONTROLLED COPY

END

OF

DOCUMENT

UNCONTROLLED COPY

Confidential Proprietary

DOCUMENT TITLE:	**SAMPLE PREPARATION OF BIOLOGICAL TISSUE FOR METALS ANALYSIS BY GFAA, ICP-OES, AND ICP-MS**
REFERENCED METHOD:	
SOP ID:	**MET-TDIG**
REV. NUMBER:	**4**
EFFECTIVE DATE:	**10/31/2013**

IF THIS SOP IS ACCESSED ELECTRONICALLY, IT IS AN UNCONTROLLED COPY AND WILL NOT BE UPDATED.

ALS-Kelso SOP Annual Review Statement

SOP Code: MET-TDIG
Revision: 4

An annual review of the SOP listed was completed on (date): **2/9/15**

☒ The SOP reflects current practices and requires no procedural changes.

Supervisor: L.J   Date: 2/9/15

☐ 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
8.2.3.4	Add Hg to the spike list. Add 6.0mL of 1000ppm Hg standard.	2/9/15	L.J.

METALS SPIKING SOLUTIONS CONCENTRATIONS FORM

Solution Name	Element	mLs of 1000ppm Solution	Final Volume	Solution Conc. mg/L	Enter mls Added
	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	
K-MET SS1	Cr	100*	1000ml	20	
	Cu	100*	1000ml	25	
	Fe	100*	1000ml	100	
	Pb	100*	1000ml	50	
*** Add after HNO3	Mn	100*	1000ml	50	
and before cas cal	Ni	100*	1000ml	50	
-14	Sb***	50	1000ml	50	
when making the	V	100*	1000ml	50	
solution	Zn	100*	1000ml	50	
	HNO3	25.0	500ml	-	
K-MET SS2	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	500	12	
	HNO3	25	500ml	-	
K-MET SS4	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.

Standard	mls of standard	ppm	Logbook #	Exp. Date

SAMPLE PREPARATION OF BIOLOGICAL TISSUE FOR METALS ANALYSIS BY GFAA, ICP-OES, AND ICP-MS

ALS-KELSO

SOP ID:	MET-TDIG	Rev. Number:	4	Effective Date:	10/31/2013

Approved By:	Department Supervisor – Jeff Coronado	Date:	9/20/13 9/23/13
Approved By:	QA Manager – Suzanne LeMay	Date:	9/23/13
Approved By:	Laboratory Director – Jeff Grindstaff	Date:	9/23/13

Issue Date:	___________	Doc Control ID#:	___________	Issued To:	___________

Confidential Proprietary

Standard Operating Procedure

For

SAMPLE PREPARATION OF BIOLOGICAL TISSUE BY GFAA, ICP-OES, AND ICP-MS

Confidential

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 graphite furnace atomic absorption (GFAA), Inductively Coupled Plasma-Optical Emission Spectroscopy (ICP-OES), or Inductively Coupled Plasma-Mass Spectrometry (ICP-MS). 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 GFAA, 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.

3.1.1. Preparation Batch - A preparation batch is composed of one to twenty field samples, all of the same matrix, with a maximum time between the start of processing of the first and last samples in the batch to be 24 hours.

Proprietary

3.2. **Method Blank (MB)** - The method blank or Preparation Blank (PB) is an artificial sample is designed to monitor the introduction of artifacts into the analytical process. The method blank is carried through the entire analytical procedure.

3.3. **Laboratory Control Samples (LCS)** - The LCS is laboratory created sample 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.

3.4. **Matrix Spike/Duplicate Matrix Spike (MS/DMS)** - In the matrix spike analysis, predetermined quantities of target analytes is 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 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. **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

Refer to the determinative method for a discussion of interferences.

5. SAFETY

5.1. Follow all ALS safety practices as described in the ALS Safety Manual.

5.2. Each chemical compound or reagent should be treated as a potential health hazard. Exposure to these chemicals should be reduced to the lowest possible level. A reference file of material safety data sheets is available to all personnel involved in these analyses. ALS also maintains a file of OSHA regulations regarding the safe handling of the chemicals specified in this method.

5.3. Nitric Acid is extremely corrosive. Care should be taken while working with this chemical. Personal protective equipment shall include safety glasses (with side shields), gloves and a lab coat. Follow normal precautions as per the ALS Safety Manual.

6. SAMPLE, COLLECTION, PRESERVATION AND STORAGE

Samples are typically collected in plastic containers and iced or refrigerated at 4 ± 2°C from time of collection until preparation or analysis. Sample may be frozen at ≤-10°C or as specified by project requirements.

7. APPARATUS AND EQUIPMENT

7.1. Lyophilizing apparatus, LABCONCO Model 7948040 freeze dryer.

7.2. Conventional laboratory oven capable of precise temperature control at 105°C.

7.3. Analytical balance capable of weighing to 0.1 mg.

7.4. 50 mL graduated poly tubes.

8. STANDARDS AND REAGENTS

8.1. 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.

8.1.1. Reagent water - ASTM Type I or Type II water

8.1.2. Concentrated nitric acid.

8.2. Standards

8.2.1. 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.

8.2.2. Metals spiking solutions: Five solutions are needed to prepare the matrix spiking standards: SS1, SS2, SS3, SS4, and SS5.

8.2.3. 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.

8.2.3.1. 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.

8.2.3.2. 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.

8.2.3.3. SS3 (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.

8.2.3.4. 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.

8.2.3.5. 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.

9. PREVENTIVE MAINTENANCE

9.1. 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.

9.2. 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.

9.3. Routine cleaning of the sample handling and digestion apparatus is necessary. Refer to the SOP for Metals Laboratory Glassware Cleaning.

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 ADM-TRAIN, *ALS-Kelso Training Procedure* is also the responsibility of the department supervisor/manager.

10.3. 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.

11. PROCEDURE

11.1. 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.

11.2. Slice the sample into thin pieces prior to filling the drying vessel. Homogenize the sample before freeze drying.

11.3. 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.

11.4. When the sample is frozen, remove from freezer and follow the manufacturer's instructions for operation of the freezer dryer.

11.5. Blend the dry solids to obtain a homogeneous sample. The sample may be stored dry until digestion.

11.6. Sample Digestion

11.6.1. Transfer 300 mg of dried sample, weighed to the nearest 0.1 mg, to a 50 mL decomposition vessel.

11.6.2. Add 4.5mL concentrated nitric acid to the vessel.

11.6.3. 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.

11.6.4. 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.

11.6.5. 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.

12. QA/QC REQUIREMENTS

12.1. 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 QC Samples are:

12.1.1. Method Blank

12.1.1.1. A method blank is prepared in an empty digestion vessel and digested with every batch of 20 (or fewer) samples to demonstrate that there are no

IF THIS SOP IS ACCESSED ELECTRONICALLY, IT IS AN UNCONTROLLED COPY AND WILL NOT BE UPDATED.

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.

12.1.2. Lab Control Sample (LCS)

12.1.2.1. 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.

12.1.2.2. 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.

12.1.2.3. 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.

12.1.3. Sample duplicates

12.1.3.1. 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.

12.1.4. Matrix Spike

12.1.4.1. 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.

12.1.4.2. 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.

13. REPORTING

13.1. Refer to the SOP for *Data Reporting and Report Generation* for reporting guidelines.

14. DATA REVIEW AND ASSESSMENT

14.1. 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. (See appendix A)

14.2. Spiking sheets are completed including all spike data and volumes of spiking solutions used.

14.3. 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.

15. CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

15.1. 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.

15.2. Handling out-of-control or unacceptable data

15.2.1. 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.

15.2.2. 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

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.

17. TRAINING

17.1. Training outline (See ADM-TRAIN, *ALS-Kelso Training Procedure*)

17.1.1. Read and understand the SOP/Method. 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 trainee is expected to transition from a role of assisting, to performing the procedure with minimal oversight from an experienced analyst. This incremental step in training shall be documented at each step by use of the Training Plan or a modified version of the Training Plan outlined in ADM-TRAIN, *ALS-Kelso Training Procedure.*

17.1.3. Perform an initial precision and recovery (IPR) study for tissue samples. Summaries of the IPR are reviewed and signed by the Technical Director. Along with the completed Training Plan, copies are 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 ADM-TRAIN, *ALS-Kelso Training Procedure*.

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. This section is not applicable because this procedure is a laboratory developed method.

19. REFERENCES

19.1. Recommended Guidelines for Measuring Metals in Puget Sound Marine Water, Sediment, and Tissue Samples; April 1997.

20. CHANGES SINCE THE LAST REVISION

20.1. Reformatted SOP to ALS branding.
20.2. Replaced "CAS" references with "ALS".
20.3. Updated SOP references.
20.4. Sec. 8.2.3.5: Corrected instruction to match current procedure.

Table 1

Selected Elements and Analysis Procedures for Tissue Samples

Element	GFAA	ICP-OES	ICP-MS
Aluminum		√	√
Antimony		√	√
Arsenic	√		√
Barium		√	√
Beryllium		√	√
Boron		√	
Cadmium	√	√	√
Calcium		√	
Chromium		√	√
Cobalt		√	√
Copper		√	√
Iron		√	
Lead	√	√	√
Lithium		√	
Magnesium		√	
Manganese		√	√
Molybdenum		√	√
Nickel		√	√
Potassium		√	
Phosphorus		√	
Selenium	√		
Silver		√	√
Sodium		√	
Strontium		√	
Thallium	√	√	√
Tin		√	√
Vanadium		√	
Zinc		√	√

Confidential Proprietary

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 effect the operation of the Stoppering Tray Dryer. If an event occurs, the alarm indicator will flash and the beeper

IF THIS SOP IS ACCESSED ELECTRONICALLY, IT IS AN UNCONTROLLED COPY AND WILL NOT BE UPDATED.

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 that 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.
7. 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).

Confidential

Proprietary

ALS Standard Operating Procedure

DOCUMENT TITLE:	TISSUE SAMPLE PREPARATION
REFERENCED METHOD:	N/A
SOP ID:	MET-TISP
REVISION NUMBER:	9
EFFECTIVE DATE:	07/01/2014

UNCONTROLLED COPY

TISSUE SAMPLE PREPARATION

ALS-KELSO

SOP ID:	MET-TISP	Rev. Number:	9	Effective Date:	07/01/2014

Approved By: ______________________ Date: 6/12/14
Department Manager/Technical Director – Jeff Coronado

Approved By: ______________________ Date: 6/12/14
QA Manager – Lee Wolf

Approved By: ______________________ Date: 6/12/14
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

UNCONTROLLED COPY

TABLE OF CONTENTS

1.SCOPE AND APPLICATION 3
2.METHOD SUMMARY 3
3.DEFINITIONS 3
4.INTERFERENCES 4
5.SAFETY 4
6.SAMPLE COLLECTION, CONTAINERS, PRESERVATION AND STORAGE 4
7.STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS 4
8.APPARATUS AND EQUIPMENT 4
9.PREVENTIVE MAINTENANCE 5
10.RESPONSIBILITIES 5
11.PROCEDURE 5
12.QA/QC REQUIREMENTS 13
13.DATA REDUCTION AND REPORTING 13
14.CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA 13
15.METHOD PERFORMANCE 14
16.POLLUTION PREVENTION AND WASTE MANAGEMENT 14
17.TRAINING 14
18.METHOD MODIFICATIONS 15
19.REFERENCES 15
20.CHANGES SINCE THE LAST REVISION 15

UNCONTROLLED COPY

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.

UNCONTROLLED COPY

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.

5.2. 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.

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

Note: 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.

8.1. Hobart Food Chopper, or comparable device.

8.2. Tissumizer.

UNCONTROLLED COPY

8.3. Waring blender, or similar device.

8.4. Freeze-drier, Labonco or equivalent.

8.5. Glass or PTFE cutting boards.

8.6. Knives and cutting implements – refer to Procedure section.

8.7. Standard laboratory glassware (beakers, scintillation vials, etc.)

8.8. VOA vial – pre-cleaned, 40ml with Teflon-lined cap.

8.9. Pre-cleaned glass jars with PTFE lined lids, various sizes.

8.10. Gloves: Should be talc free and of non-contaminating materials.

8.11. 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 chemists 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

UNCONTROLLED COPY

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 in Section 11.1.1.

11.1.1. Tissue Samples with Limited Quantity

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.

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.

The project chemist 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.

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 as described in Section 11.1.1.

11.2. Sample Homogenization

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. Samples for Organics Analyses:

Equipment used for the processing of tissue samples for organics analyses should be of 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 should be talc free and of non-contaminating materials. 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 precleaned, 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 40ml voa vial and libeled "FOR VOA ANALYSIS ONLY".

UNCONTROLLED COPY

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:

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.

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:

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".

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. Precleaned, 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.

11.2.4. Sample Preparation

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

UNCONTROLLED COPY

container is labeled with the sample I.D., the word "homogenized", initialed, and dated.

11.2.4.1.Whole Fish Tissue

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.

11.2.4.2.Edible Fish Tissue

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 Chemist 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.

11.2.4.3.Shellfish Tissue

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

UNCONTROLLED COPY

organisms constitute a single sample, the edible tissues are collected, composited and homogenized.

11.2.4.3.1.Bivalve mollusks (oysters, clams, mussels, and scallops).

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.

11.2.4.3.2.Crabs

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.

11.2.4.3.3.Shrimp and Crayfish - Edible tissue includes the tail meat.

11.2.4.3.4.Lobster - Edible tissue includes the tail and claw meat.

11.2.4.4.Worms

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 chemist to seek guidance from the client.

11.2.4.5.Eggs

Avian eggs are typically removed from the shell and blended. Aquatic eggs are blended including the soft shell.

11.2.4.6.Internal Organs Extraction

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.

11.2.4.7.Plant Tissue

Plant tissue should be handled using the size reduction, homogenization and implement cleaning steps outlined in Sections 11.2.1, 11.2.2, 11.2.3, and 11.2.4. Where these procedures are inappropriate, specific written procedures or guidance from the client is recommended.

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.

11.2.4.8.Small Mammals and Rodents

UNCONTROLLED COPY

11.2.4.8.1. There are two primary concerns in working with small mammals and rodents: safety and sample homogenization.

11.2.4.8.2. 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.

11.2.4.8.3. 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.

11.2.4.8.4. Homogenization should be done while the carcass is still partially frozen.

11.2.4.8.5. If the hide is to be included in the homogenization, snip the feet from the animal using stainless steel scissors.

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.

11.2.4.8.6. 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.

11.2.4.8.7. 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

UNCONTROLLED COPY

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. General Provision for Handling Large Sample Mass

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 under Section 10.2.3. For very large specimens (i.e. >20 pounds), high grade stainless steel containers are used (large bowls or small drums).

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

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.

11.3.1. 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.

11.3.2. Tissue Samples with Limited Quantity

Samples and sample composites that are quantity limited will be handled by the same procedure as described in Section 10.1.1

11.3.3. Compositing Procedure

11.3.3.1. Each tissue sample is first homogenized as per instructions in Section 11.2.

UNCONTROLLED COPY

11.3.3.2. 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.

11.3.3.3. 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.

11.3.4. Tissue Samples Requiring VOC Analyses

A separate aliquot of the composite homogenate should be placed in 40ml 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 as per Section 11.2. 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.

UNCONTROLLED COPY

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 bench 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

UNCONTROLLED COPY

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 Environmental Health and Safety Manual.

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

UNCONTROLLED COPY

of assisting, to performing the procedure with minimal oversight from an experienced analyst.

17.2. Training is documented following ADM-TRAIN, ALS-Kelso Training Procedure.

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. Reformatted SOP to current ALS format and style.
20.2. Replaced "CAS" references with "ALS".
20.3. Few minor changes (correct typos and errors, etc.).
20.4. Added section 4.
20.5. Section 10.2 – Copied to section 4.2.
20.6. Section 12 – Revised to clarify various blank requirements.
20.7. Section 14 replaced with current standard language.
20.8. Updated SOP references.
20.9. Inserted appendices.

UNCONTROLLED COPY

APPENDIX A
Fish Filleting Procedure

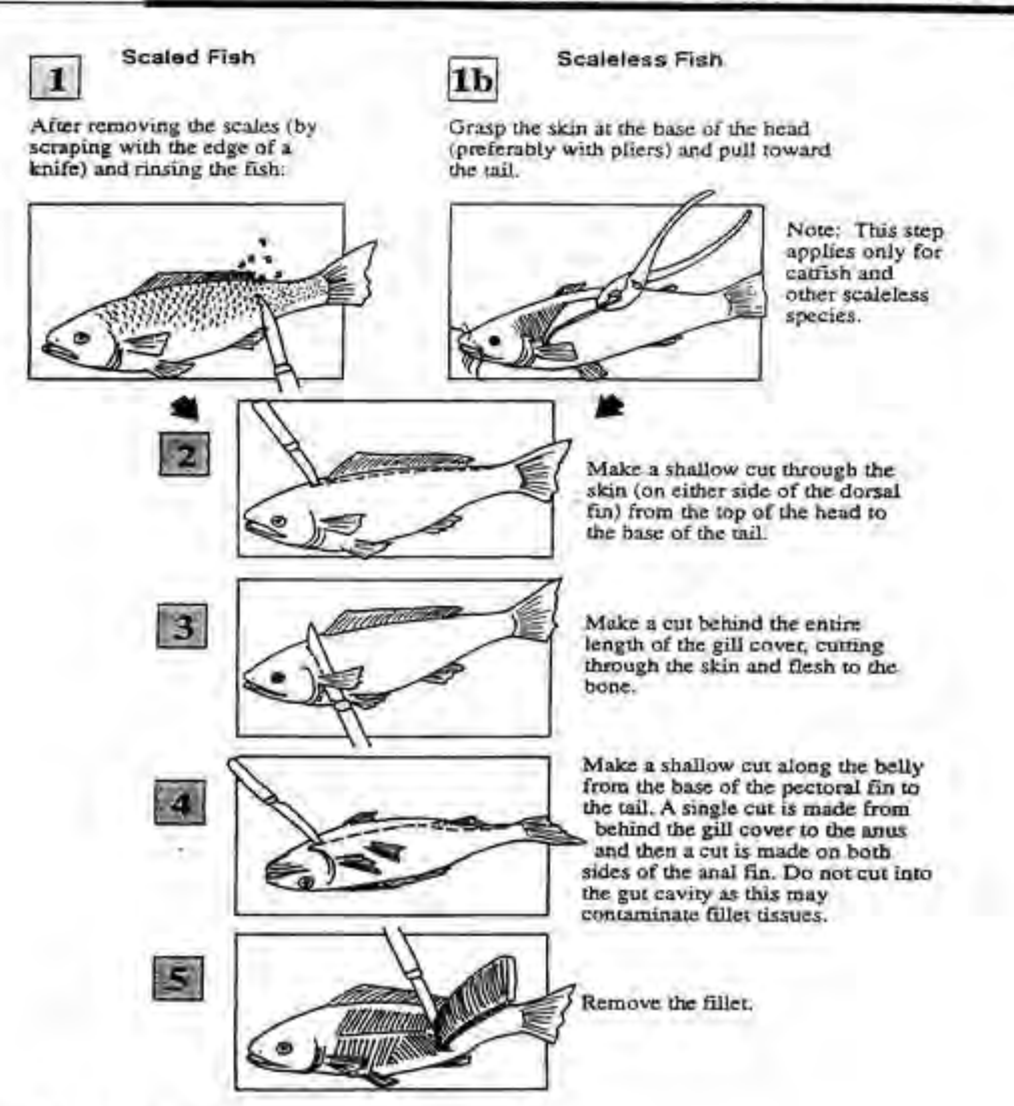
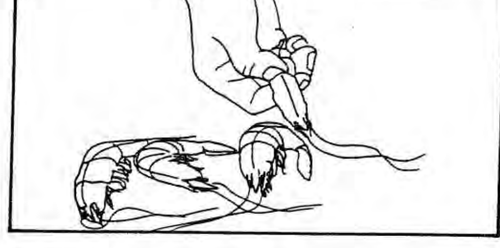


Figure 7-3. Illustration of basic fish filleting procedure.

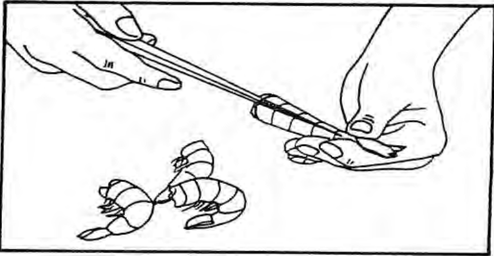
APPENDIX B
General Procedure for Removing Edible Tissues from Shellfish

Heading, peeling and deveining shrimp

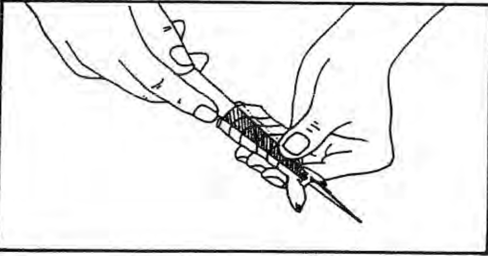
To head a shrimp, hold it in one hand. With your thumb behind shrimp head, push head off. Be sure to push just the head off so that you do not lose any meat.



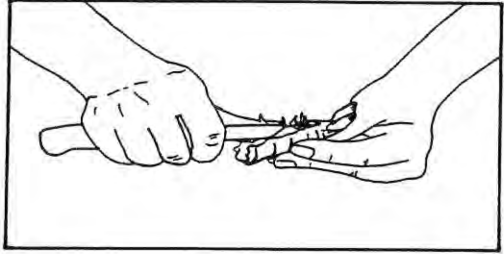
If using a deveiner, insert it at head end, just above the vein.



Push through shrimp to the tail and split and remove shell. This removes vein at the same time.



If you prefer to use a paring knife, shell shrimp with your fingers or knife. Then use knife to gently remove vein.

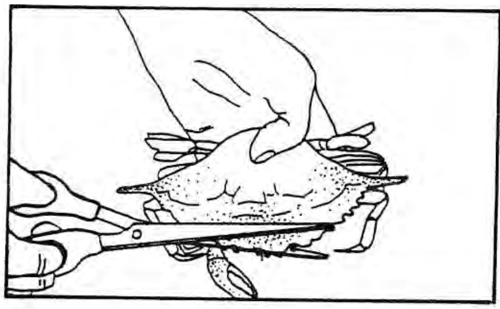


Source: UNC Sea Grant Publication UNC-SG-88-02

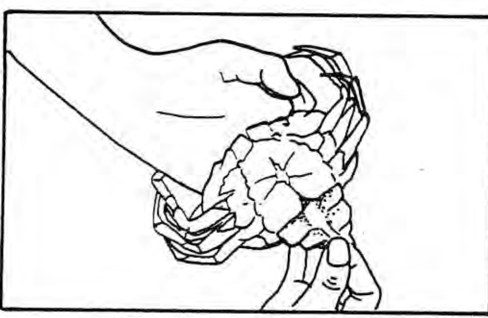
G-3

Cleaning soft-shell crabs

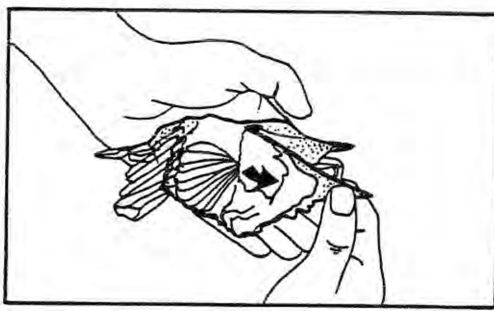
Hold crab in one hand and cut across body just behind eyes to remove eyes and mouth.



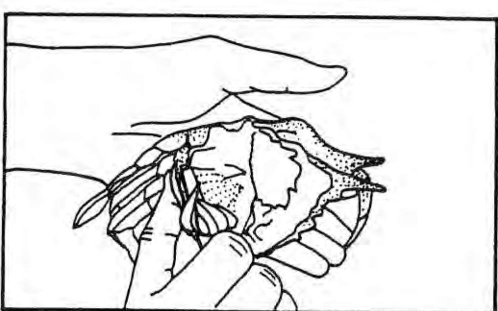
Turn crab on its back. Lift and remove apron and vein attached to it.



Turn crab over and lift one side of top shell.



With a small knife, scrape off grayish-feathery gills. Repeat procedure on other side.

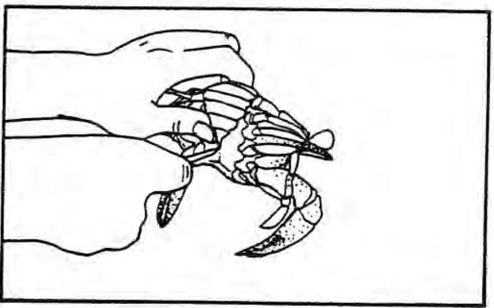


Source: UNC Sea Grant Publication UNC-SG-88-02

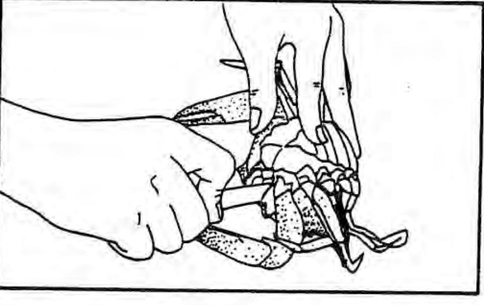
UNCONTROLLED COPY

Cleaning hard-shell crabs

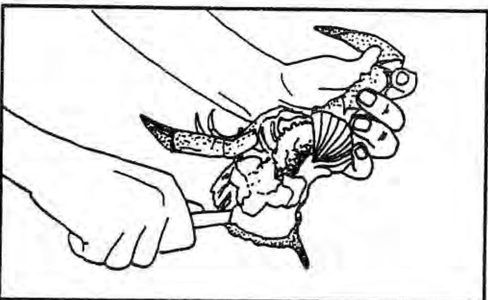
Hold crab in one hand. Turn crab over and stab straight down at point of apron with a knife.



Make two cuts from this point to form a V-pattern that will remove mouth.

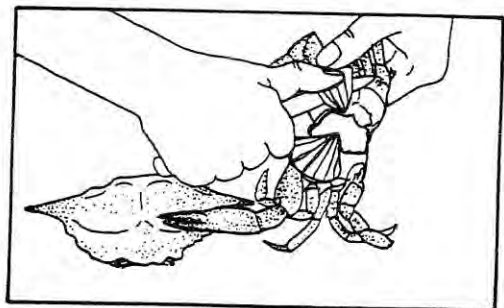


Do not remove knife after making second cut. Firmly press crab shell to cutting surface without breaking back shell. With other hand, grasp crab by legs and claws on the side where you are holding knife, and pull up. This should pull crab body free from back shell.

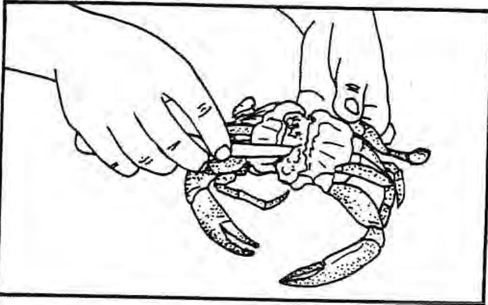


UNCONTROLLED COPY

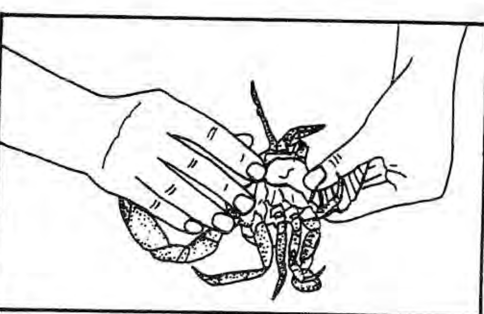
Remove gray, feathery gills, which are attached just above legs. Cut and scrape upward to remove gills.



Remove all loose material—viscera and eggs—from body cavity.



If apron did not come loose with shell, remove it.



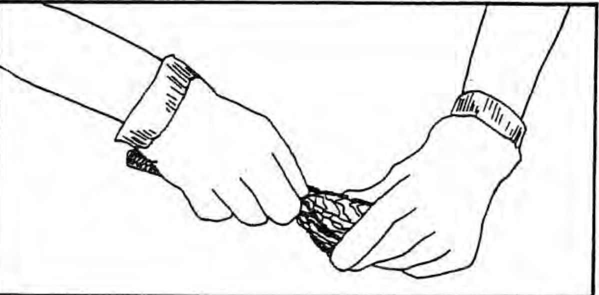
Source: UNC Sea Grant Publication UNC-SG-88-02

UNCONTROLLED COPY

Shucking oysters

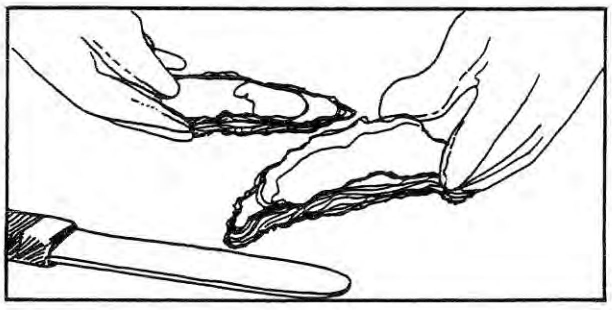
Oyster shells are especially sharp; be sure to wear gloves to protect your hands. Chip off a small piece of shell from the thin lip of the oyster until there is a small opening.

Insert knife blade into the opening and cut muscle free from top and bottom shells.



3

Remove oyster meat from the shell.

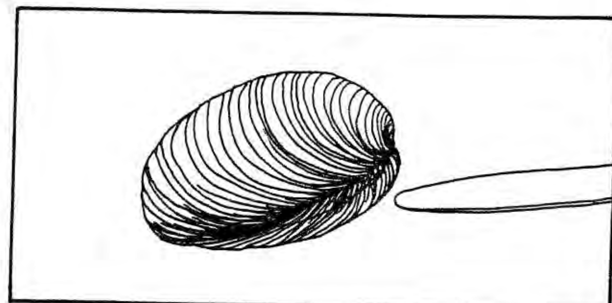


Source: UNC Sea Grant Publication UNC-SG-88-02

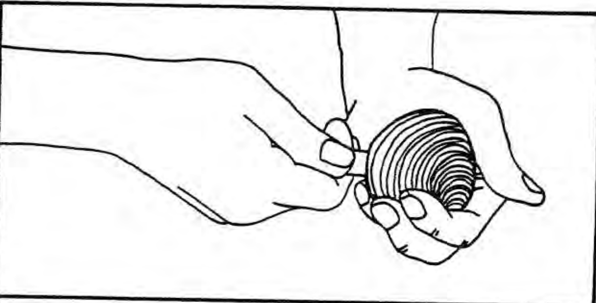
UNCONTROLLED COPY

Shucking clams

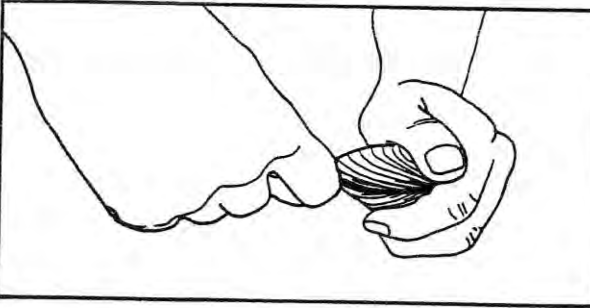
In the back of clam near the hinge is a black ligament. Toward the front where ligament ends is a weak spot. Insert your knife at this spot.



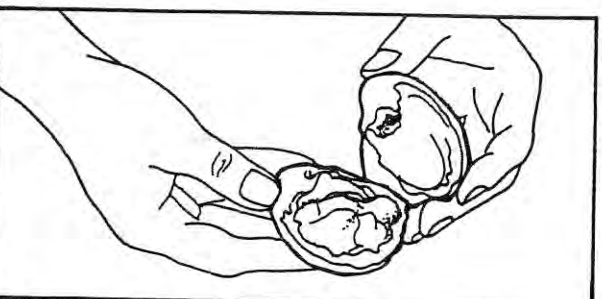
Inside are two muscles. Run the knife around the shell to sever both muscles.



Now insert the knife blade into the front of the shell and separate the two shells.



Scrape the meat free from the top and bottom shell.



Source: UNC Sea Grant Publication UNC-SG-88-02

UNCONTROLLED COPY

METHYL MERCURY IN TISSUE BY ALCOHOLIC POTASSIUM HYDROXIDE DIGESTION, ETHYLATION, PURGE AND TRAP, AND COLD VAPOR ATOMIC FLUORESCENCE SPECTROMETRY

EPA 1630

ALS-KELSO

SOP ID:	MET-1630T	Rev. Number:	2	Effective Date:	1/31/2013

Approved By: Technical Director – Jeff Coronado   Date: 12/27/12

Approved By: QA Manager – Suzanne LeMay   Date: 12/27/12

Approved By: Laboratory Director – Jeff Grindstaff   Date: 12/28/12

Proprietary

Issue Date:	__________	Doc Control ID#:	___________	Issued To:	___________

Standard Operating Procedure

For

METHYL MERCURY IN TISSUE BY ALCOHOLIC POTASSIUM HYDROXIDE DIGESTION, ETHYLATION, PURGE AND TRAP, AND COLD VAPOR ATOMIC FLUORESCENCE SPECTROMETRY

1. SCOPE AND APPLICATION

1.1. This procedure is used for the determination of mono-methyl Hg (MMHg) in fish tissues and other marine biota, based on EPA Method 1630. The analysis of tissues is performed on samples that have been homogenized, optionally lyophilized, and Teflon bomb digested with 25% KOH in Methanol (w/v).

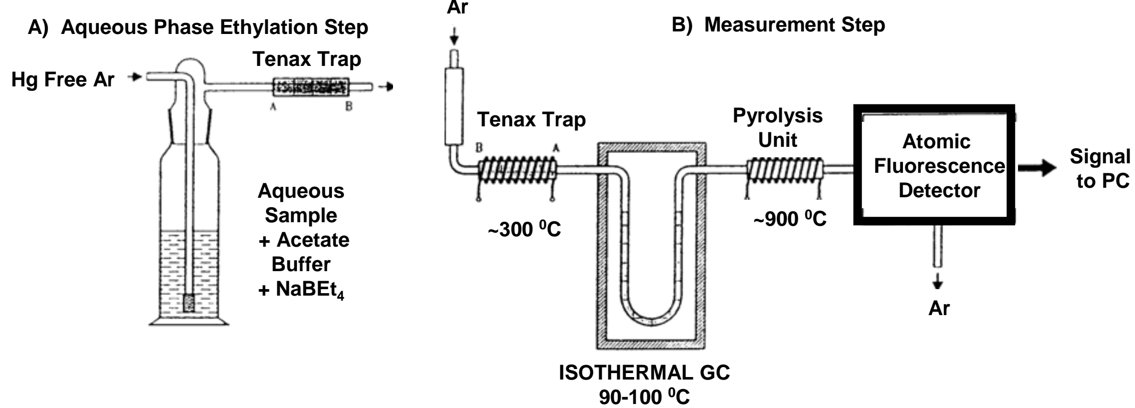
1.2. EPA Method 1630 is a cold vapor atomic fluorescence procedure used in determining the mono methyl mercury (MMHg) concentration in water. Portions of this method were adopted for the digested tissue samples. Method 1630 is designed for the measurement of MMHg in water ranging from 2 to 2000 (or more) total pg at the instrument.

1.3. Methyl mercury as defined in this method means all methyl mercury forms and species found in the digestate such as: CH_3Hg+, CH_3HgCl, CH_3HgOH, and CH_3HgS-R.

2. METHOD SUMMARY

Samples are collected using clean sample handling protocols into acid-cleaned Teflon™ bottles, or mercury free plastic bags. Freezing preserves samples until sample preparation is performed. Tissues are homogenized and freeze dried per client's request. Prior to analysis, samples are digested or leached in 25% KOH/Methanol until solubilized. An aliquot of the digestate is ethylated with $NaBEt_4$ in a bubbler. The resultant methyl ethyl mercury (MEHg) is purged via Argon onto a Carbotrap trap. The trapped MEHg is then thermally desorbed, swept with Argon into a GC Column for speciation, subsequently pyrolyzed to Hg^0, and detected by a Brooks Rand Model III cold-vapor atomic fluorescence spectrometer (CVAFS) for detection.

Proprietary



3. DEFINITIONS

3.1. **Analysis Sequence** - Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration followed by sample analyses interspersed with various quality control checks. The sequence is ≤ 20 tissue samples and concurrent QC checks.

3.2. **Bubbler (Ethylation) Blank** – A minimum of one Ethylation blank is run for each calibration curve. The ethylation blank is run on clean DIH_2O exactly as standards (i.e. with acetate buffer and ethylating reagent and purged onto a trap). The peak height or area of the ethylation blank (or mean of multiple ethylation blanks) is subtracted from the area of each standard to establish the mean calibration factor.

3.3. **Ethylating Agent** – Sodium Tetra Ethyl Borate or STEB of $NaBEt_4$.

3.4. **Dissolved Methyl Mercury** - All distillable CH3Hg forms and species found in the filtrate of an aqueous solution that has been filtered through a 0.45 micron filter.

3.5. **Methyl Mercury** - Mono Methylmercury = MMHg = Methyl Mercury = MeHg = CH_3Hg. All acid-distillable Hg, which, upon reaction with NaBEt4 yields methylethyl mercury. This includes, but is not limited to, CH_3Hg+, strongly organo-complexed CH_3Hg compounds, adsorbed particulate CH_3Hg, and CH_3Hg bound in microorganisms. In freshly collected samples, dimethyl mercury ($(CH_3)_2Hg$) will not be recovered as CH_3Hg, but in samples which have been acidified for several days, most $(CH_3)_2Hg$ has broken down to CH_3Hg. In this method, CH_3Hg and total recoverable CH_3Hg are synonymous.

3.6. **Analysis Sequence** - Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration followed by sample analyses interspersed with various quality control checks. The sequence is ≤ 20 samples and concurrent QC checks.

3.7. **Ethylation Blank** – A minimum of one Ethylation blank is run for each calibration curve. The ethylation blank is run on clean DIH_2O exactly as standards (i.e. with acetate buffer and ethylating reagent and purged onto a trap). The peak height or area of the ethylation blank (or mean of multiple ethylation blanks) is subtracted from the area of each standard to establish the calibration factors.

3.8. **Ethylating Agent** – Sodium Tetra Ethyl Borate or STEB or $NaBEt_4$.

3.9. **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.

3.9.1. 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.

3.9.2. 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,

Proprietary

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.

3.10. **Sample**

3.10.1. Field Sample - An environmental sample collected and delivered to the laboratory for analysis; a.k.a., client's sample.

3.10.2. Laboratory Sample - A representative portion, aliquot, or subsample of a field sample upon which laboratory analyses are made and results generated.

3.11. **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.

3.11.1. 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.

3.11.2. Drinking water - Any aqueous sample that has been designated a potable or potential potable water source.

3.11.3. Saline/Estuarine water - Any aqueous sample from an ocean or estuary or other salt-water source.

3.11.4. Nonaqueous Liquid - Any organic liquid with <15% settleable solids.

3.11.5. Animal tissue - Any tissue sample of an animal, invertebrate, marine organism, or other origin; such as fish tissue/organs, shellfish, worms, or animal material.

3.11.6. Solids - Any solid sample such as soil, sediment, sludge, and other materials with >15% settleable solids.

3.11.7. 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.

3.11.8. 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.

3.12. 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.

Proprietary

3.13. 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.

3.14. 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.

3.15. 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.

3.16. 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

3.17. 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.

3.18. 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.

3.19. 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.

3.20. 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.

3.21. **Ongoing Precision Recovery (OPR)** - A standard analyzed at the beginning and end of an analytical sequence. The spiked standard is carried through the whole method and is used to verify the ongoing validity of the method.

3.22. **Standard Reference Material (SRM)** - A sample with a certified concentration.

3.23. **Methyl Mercury** – Mono Methylmercury = MMHg = Methyl Mercury = MeHg = CH_3Hg

Proprietary

3.24. **Quality Control Sample (QCS)** - QCS solutions are made from a stock solution which is different from the stock used to prepare calibration standards and is used to verify the validity of the standardization.

3.25. **Ongoing Precision Recovery (OPR)** - A standard analyzed at specified intervals and used to verify the ongoing validity of the instrument calibration.

3.26. **Standard Reference Material (SRM)** - A sample of tissue with a certified concentration.

4. INTERFERENCES

4.1. Extreme care must be taken to avoid contamination when collecting and analyzing samples for trace methyl 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, dirt and dust. Even human contact can be a source of trace mercury contamination (e.g., mercury fillings, contaminated gloves).

4.2. Within the laboratory, interferences from contaminated reagents must be minimized. Ultrex 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 must be cleaned by wiping with a lint-free cloth or wipe soaked with reagent water.

4.3. Tissue digestate itself is an interference during the ethylation reaction. The use of uniform sample weights, the method of standard additions, and the use of the smallest sample aliquot necessary for accurate quantification can overcome this. For samples with greater than 0.1 g/g as methyl mercury, the sample and aliquot size can be sufficiently small so that no peak inhibition compared to deionized water is observed.

Proprietary

4.4. No HNO_3 or other oxidizing agents (Cl_2, BrCl, CrO_4, etc.) should be present in the sample, or CH_3Hg may be destroyed. Also, chlorine present in dilution or reagent water destroys both methyl mercury and the ethylating reagent.

4.5. BrCl will oxidize nearly all forms of mercury; therefore, pipettes used to aliquot BrCl or bubblers used/cleaned with BrCl should never be used for methyl mercury analysis.

4.6. The Ethylation portion of the methyl mercury analysis is very pH sensitive. Each digest (or type or subset), should be tested for pH before analysis, making sure not to place pH paper directly into sample. A pH of 4.5-5.0 (4.9 is ideal) can usually be achieved with the addition of 0.3 – 0.4 mL of the 2 M Potassium Acetate buffer. If the pH is too low, add more acetate buffer to achieve the desired range for the Ethylation reagent to work correctly. Because of the basic nature of tissue digests prepared with the KOH/methanol digestion, a larger amount (up to 0.6 mL) of acetate buffer may be needed. Also, it is recommended that aliquots of no more than 50 uL of the tissue digest be analyzed, unless otherwise instructed a senior analyst.

4.7. When analyzing samples for methyl mercury, bubblers should be tripled rinsed and new reagent water added between every sample.

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 Acid and Potassium Hydroxide are used in this method. These acids and bases are extremely corrosive and care must be taken while handling them. A face shield should be used while pouring acids. 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 associated risks. **High concentrations of MMHg can be fatal!**

5.5. ALS purchases a dilute standard solution of methyl mercury for this method. If primary solutions are prepared, they must be prepared in a fume hood with the utmost caution.

5.6. Hands should be washed thoroughly after each manipulation and before breaks.

5.7. 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.

5.8. Ethylating agent ($NaBEt_4$) is toxic, gives off toxic gases (triethylboron), and is spontaneously combustible. To discard unused portions of ethylating agent and empty bottles, place into a large beaker of ~10% 1N HCl in the hood, triethylboron will bubble off to the air where it is eventually oxidized to harmless boric acid. Leave the acid beaker in the hood indefinitely, or boil down to half the original volume to destroy residues before discarding as acid waste.

6. SAMPLE COLLECTION, PRESERVATION, AND STORAGE

6.1. All sample handling in the laboratory should be undertaken in a mercury-free clean bench, clean room, or cleanest area possible. Gloves protect analyst and sample from contamination.

6.2. Recommended containers for sample collection and storage are glass jars with Teflon-lined caps. When possible, sample containers should be shown to be mercury-free through pre-cleaning and analysis, or certificates of analysis.

Proprietary

6.3. Samples should be stored at -10 to -20˚C until analysis. The recommended holding time is 6 months for either raw or freeze-dried samples. The holding time is 1 year when frozen.

7. APPARATUS AND EQUIPMENT

7.1. Cold vapor atomic fluorescence spectrometer (CVAFS):
Brooks-Rand (Seattle, WA) Model III CVAFS, or equivalent.

7.2. Flowmeter, with needle valve capable of reproducibly keeping carrier gas flow at 60 mL/min.

7.3. Flow meter/needle valve capable of controlling and measuring gas flow rate to the purge vessel at 350 ± 50 mL/min.

7.4. Pyrex bubbler with 4 way Teflon stopcock, 220 mL
(Brooks-Rand, Seattle, WA, part no. AF-32 or equivalent)

7.5. Fluoropolymer fittings: Connections between components and columns are made using 6.4-mm O.D. fluoropolymer tubing and fluoropolymer friction-fit or threaded connectors.

7.6. Carbotrap® trap for Hg speciation. Silanized, blanked with ends plugged with Teflon. 1/4" O.D. x 10 cm length. (Brooks Rand AF-21-00 or equivalent)

Isothermal GC Unit: The GC column is ~ 0.3 M long borosilicate glass column tubing with 0.25-in. O.D. and 4-mm I.D. bore. The column is silanized, and packed with preconditioned 60/80 mesh 15% OV-3 on Chromasorb WAW-DMSC, held in place with silanized glass wool plugs. The column is held in a small temperature-controlled isothermal oven made from a heating mantle. (Supelco Inc. custom product or from Brooks Rand)

7.7. Pyrolytic Organo-mercury Breakdown Column: A 20-cm length of 7-mm O.D. by 4.5-mm I.D. quartz tubing with the central 10 cm packed with quartz wool. The column is wrapped with 1.5 m of 22-gage Nichrome wire that is electrically heated to about 750-900 °C (bright orange) with ~56 volts from an autotransformer.

7.8. Gold-coated sand trap or gold wire trap for removing Mercury from Argon line and for trapping mercury exiting the detector: A tube 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 with the ends plugged with quartz wool. (Brooks Rand, Ltd., Seattle, WA, Part No. AF-20 or equivalent).

7.9. 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.

7.10. Computer (Pentium or better) and Windows Mercury Guru software to record and integrate the signal from the spectrometer.

7.11. Pipettors: All-plastic pneumatic fixed-volume and variable pipettors in the range of 20 uL to 10.0 mL.

7.12. Analytical balance capable of weighing to the nearest 0.0001 g

7.13. Oven able to maintain temperature of 70 to 100º Celsius.

Proprietary

7.14. 50 mL Teflon Bombs (Savillex or equivalent).

8. STANDARDS, REAGENTS AND CONSUMABLE MATERIALS

8.1. 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.

8.2. 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.

8.3. Reagent water: Water in which mercury is not detected by 1631 total mercury method. 18-megaohm ultra-pure deionized water from a pre-purified (laboratory DI water) source.

8.4. 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.

8.5. Hydrochloric acid: trace-metal purified reagent HCl containing less than 1 pg/mL Hg.

8.6. Potassium Acetate: Neat, reagent grade. Blank Pre-tested and verified to be low in mercury.

8.7. Potassium Hydroxide: Neat, pellets Semiconductor grade (or shown low in Hg).

8.8. Sodium Tetraethyl Borate: 1-g vials. **Use STREM as supplier**.

8.9. Ethylating Agent (Sodium Tetraethyl Borate 1% Solution, STEB, $NaBEt_4$):

8.9.1. Rinse approximately 20 seven-milliliter Teflon vials with reagent water and arrange in a vial rack in a class-100 clean hood. (Or use amber HPLC auto-sampler vials.)

8.9.2. Pour 100 mL of reagent water into a 125-mL Teflon bottle and add 2.0 g of KOH pellets. Dissolve mixture and place in freezer until ice crystals just begin to form.

8.9.3. Turn on reagent water. Remove wax and tape seal from a 1.0-g ampoule of sodium tetraethyl borate. Open the ampoule and immediately transfer its contents to the 125-mL Teflon bottle, cap tightly, and give a few quick shakes.

8.9.4. Fill the now empty ampoule and its cap with reagent water and sink into a beaker containing water and HCl (the pure borate powder is pyrophoric and could spontaneously-combust when exposed to air if not stabilized by the addition of water).

8.9.5. Shake the borate solution again then transfer to the vials, leaving ample air space for freezing and capping tightly.

8.9.6. Place all the vials in the freezer standing upright.

Proprietary

8.9.7. If any doubt arises about the quality of the ethylating reagent, make a new batch, as old material often gives good results for reagent water spikes, but not for environmental samples. Do not use STEB solid or 1% solution if they have a yellow color.

8.10. Glacial Acetic Acid: Reagent grade. Blank Pre-tested and verified to be low in mercury.

8.11. 2M Acetate Buffer: 2 M Acetate Buffer:

8.11.1. Add 100 mL reagent water to a Teflon bottle.

8.11.2. Add 19.6 g of Potassium Acetate.

8.11.3. Add 11.8 mL of Glacial Acetic Acid.

8.11.4. Ethylate with 0.5 mL STEB (1% solution) and purge overnight with argon to remove trace MMHg This solution has an indefinite lifetime when stored in Teflon.

8.12. Argon (Ar): Use Grade 5.0 (ultra high purity grade) argon that has been further purified by the removal of mercury using a gold or iodated carbon trap that is located in line between the gas output and the analyzer gas input.

8.13. 25% Potassium Hydroxide/Methanol: 250 g of KOH is dissolved in methanol to make a final volume of 1.0 L. This solution has an indefinite lifetime when stored in a Teflon bottle at room temperature. **NOTE: Add KOH slowly as the reaction is highly exothermic.**

8.14. Stock methylmercury standard: Stock methylmercury standards (1.0 ug/mL= 1,000 ppm) are obtained from Brooks Rand Seattle. Two standards are purchased. The first is MeHgCl and the second (used for QCS), is from a second source and is MeHgOH. Both standards are sold as 1.00 ug/L in 0.5% HOAc and 0.2% HCL (v/v). These solutions are kept in the refrigerator and are stable until the expiration date. (one year)

8.15. MeHg standard A: Pipette 0.100 mL of the stock solution (1ug/mL = 1,000,000 pg/mL) to 100 mL volumetric flask containing ~ 50 mL DIH_2O, 0.5ml HOAc, and 0.2 ml HCL. QS with DIH_2O. The resulting solution contains 1000 pg/mL MeHg. Keep in a tightly closed Teflon bottle in the refrigerator. This expiration date is the same as the stock standard.

8.16. MeHg standard B: Pipette 10.0 mL of MeHg STD B (1,000 pg/mL) to 100 mL volumetric flask containing ~ 50 mL DIH_2O, 0.5ml HOAc, and 0.2 ml HCL. QS with DIH_2O. The resulting solution contains 100 pg/mL MeHg. Keep in a tightly closed Teflon bottle in the refrigerator. This expiration date is the same as the stock standard.

8.17. MeHg standard C: Pipette 1.0 mL of MeHg STD B (1,000 pg/mL) to 100 mL volumetric flask containing ~ 50 mL DIH_2O, 0.5ml HOAc, and 0.2 ml HCL. QS with DIH_2O. The resulting solution contains 10 pg/mL MeHg. Keep in a tightly closed Teflon bottle in the refrigerator. This expiration date is the same as the stock standard.

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 GC column damaged by chemicals or overheating.

9.3. Traps should be tracked by unique identifiers so that any trap producing poor results can be quickly recognized and discarded. Traps are best when blanked before each use in analysis.

9.4. Bubblers must be kept clean by at least triple rinsing with / storing in DIH_2O between runs. Make sure to fill and drain the dip tube during each rinse. Alternatively store in dilute HCl.

9.5. Occasionally move the Pyrolytic Coil off center (Right and Left) while blanking traps. This is done to remove mercury from the sides of the Pyrolytic tube.

9.6. Condition the GC column ~ every 2 to 3 months. **SLOWLY** ramp the GC oven up to 180 ^{0}C and condition for 12-16 hours. This purges impurities from the column. See the Model III manual for this procedure. **NOTE: Ramping too quickly causes the oven to overshoot the desired temperature. Going too high (200 ^{0}C) will ruin the column packing material**

9.7. Depending on usage the Mercury Source Lamp in the CVASS 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 prepared with the same reagents and analyzed within the same 12-hour shift. The analytical batch contains the following standards, samples, blanks, and QC Checks:

Method Blank 1
Method Blank 2
Method blank 3
OPR (used to demonstrate end to end analytical system stability)
QCS or SRM
up to 10 samples
Matrix Spike
Duplicate Matrix Spike
Up to 10 additional samples

Proprietary

OPR (used to demonstrate end to end analytical system stability)

For more than 20 samples in a 12 hour shift, the sequence is repeated.

Method blanks are analyzed in triplicate. The average of these values is used to correct the sample reading for the contribution of the reagents and the prep tools/containers to the measured methylmercury in unknown samples.

11.2. Sample Preparation

11.2.1. The sample is dissected and homogenized with acid-washed stainless steel tools.

11.2.2. The homogenized sample may be freeze dried if desired.

11.2.3. Approximately 0.5 to 1.0 g of the wet homogenate or 0.1 to 0.5 g of freeze dried material is weighed to the nearest tenth of a milligram into the 50 mL Teflon™ Bomb.

11.2.4. Unused homogenate is returned to the freezer.

11.2.5. Pipette 10.0 mL of the 25% KOH/methanol reagent to each sample/bomb.

11.2.6. The sample is then capped tightly with wrenches, shaken and placed in a 70 to 80 ^{0}C oven for 2 to 4 hours, or until all soft tissue is visibly made soluble.

11.2.7. The samples/bombs are occasionally shaken during the oven digest.

11.2.8. The samples/bombs are allowed to cool to room temperature before opening.

11.2.9. Using a pipette of 10.0 mL of methanol only, rinse the cap and the walls of the Teflon™ bomb to a 50 mL volumetric flask. Continue with DIH_2O to quantitatively transfer the sample. Use at least three rinses. QS the 50 mL volumetric flask with DIH_2O.

11.2.10. **NOTE changes in sample weight, final volumes, and/or dilutions may be needed to allow the sample to be detected at the AFS in a "typical" analytical range of 1 to 2000 pg MMHg at the instrument**.

11.2.11. Samples with undissolved solids are allowed to settle thoroughly prior to analysis. It may be desirable to centrifuge the material. It may also be useful to add a second 10.0 mL portion of KOH/Methanol and return to the oven to achieve a better digest. If more KOH is used, add this to extra method blanks to ensure continuity.

11.3. Ethylation Purge and Trap:

11.3.1. Add 50 mL DIH_2O to a clean bubbler.

11.3.2. Pipette 0.3 mL of acetate buffer to the bubbler.

11.3.3. Pipette 0.050 mL of digestate above to the bubbler.
(If more aliquot is desired, use caution in adjusting the pH and limiting the amount of

Proprietary

methanol, as these will interfere with ethylation.)

11.3.4. Pipette 0.040 mL of freshly thawed 1% NaBEt4 solution. Close the reaction vessel with the bubbler cap, clip the cap on, and swirl gently to mix.

11.3.5. Allow the contents of the bubbler to react for 17 min. The MMHg in the sample will be converted to volatile methylethyl mercury (MEHg).

11.3.6. After reaction, attach a Carbotrap ® trap to each bubbler with the 1/4" fluoropolymer fitting, and purge the sample with Argon (~200 mL/min) for 17 min.

11.3.7. The trap must be attached such that the gas from the bubbler enters the trap on side A (the crimped and numbered side of the trap).

11.3.8. Once the sample has been purged for 17 min, any adsorbed water must be dried from the trap. Switch the 4 way valve and purge argon through the trap only for 7 minutes to dry.

11.3.9. After the 7 minute drying, remove the traps and plug the ends with Teflon plugs. The sample is now ready for analysis. Trapped MEHg is quantitatively stable for 6 hours.

11.4. Thermal Desorption, GC-CVAFS analysis:

11.4.1. Ensure the instrument is warmed up with the following conditions:

- Ar flow to GC is on at 10 psi and 30-35 on the flow meter.
- GC oven is at 95 °C.
- PMT voltage is typically 640 to 660 Volts. Change this in order to achieve an offset as close to 50,000 counts as possible and very steady. (± 15 counts)
- Pyrolytic and Trap Coil controllers are turned on.
- Pyrolytic Coil is centered over glass wool in Pyrolytic Tube.
- A daily Measure Instrument Noise test was done (typically 36-50).

11.4.2. Remove the previous trap from the GC.

11.4.3. Attach next trap to the GC column using a 1/4" fluoropolymer friction fit connector, such that side A (the crimped and numbered side), is facing toward the GC.

11.4.4. Place the Nichrome wire heating coil around the trap, centered over, and extending beyond the packing material on side A.

11.4.5. Re-connect the argon gas to side B of the trap.

11.4.6. Allow at least 30 sec for the pressure in the system to equilibrate.

11.4.7. Using the mercury Guru software, apply power to the coil around the sample trap for 0.55 minutes (33 seconds using the software's automatic timer), to thermally desorb the ethylated mercury species from the sample trap into the GC column.

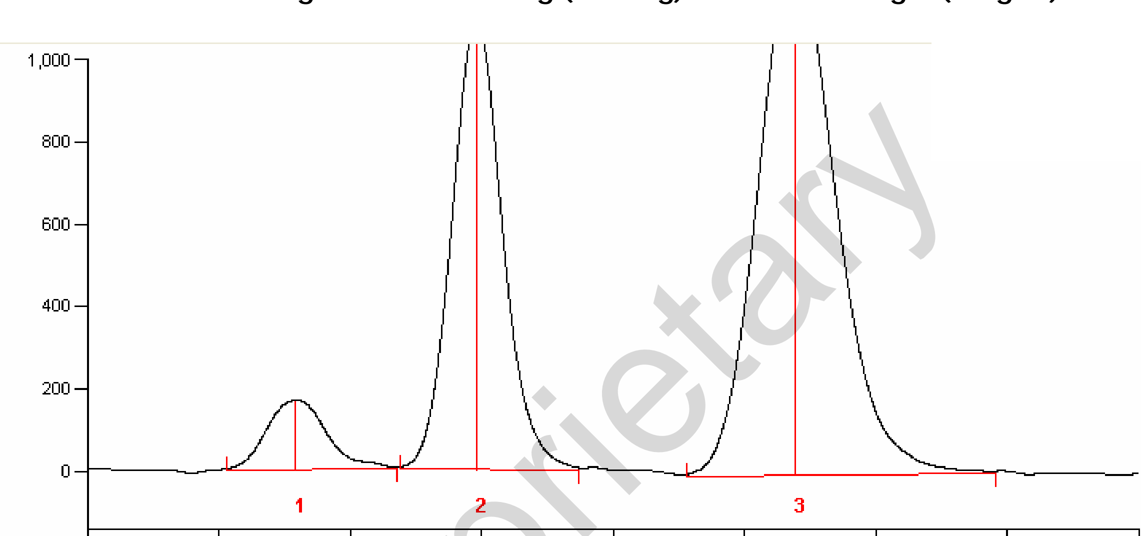
11.4.8. Three peaks should emerge during the analytical run. The first peak (~1 min) is Hg °, which is residual, and non-quantitative. The second peak to emerge (~2.5 min) is

Proprietary

methylethyl mercury (the ethylation product of methylmercury). This is the peak of interest. Following this (~4 min), is the peak for diethyl mercury ($(CH_3CH_2)_2Hg$), which is the ethylation product of Hg (II). If diethyl mercury ($(CH_3)_2Hg$), were present in the sample, it would appear as a second peak between Hg° and methylethyl mercury-not fully resolved from the Hg ° See Representative Chromatogram below:

Figure 1: A Representative Chromatogram for ~ 5 pg STD

Peak 1 Hg° Peak 2 MMHg (→MEHg) Peak 3 Hg II (→HgEt₂)



11.4.9. Allow the GC to run ~ 1 min beyond the point that the diethyl mercury (Hg(II)) peak returns to base line. Place the next trap in line and proceed with next sample analysis.

11.4.10. Peaks generated using this technique should be sharp and close to symmetrical. (Unfortunately, some tailing is typical with this system.) Methylethyl mercury elutes at approximately 2.5 min and has a width at half-height of ~ 20 sec. Earlier peaks (Hg °, $(CH3)_2Hg$) are sharper, while later peaks (diethyl mercury) are broader.

11.4.11. If only the (Hg°) appears, it usually signifies that either that the pyrolytic column is not on, or that $NaBEt_4$ was not added to the sample, or sample leaked during trapping.

11.4.12. Normally the Hg° peak is quite small. However, some Hg° is generated by thermal degradation of diethyl mercury during the desorption step. Thus, when samples contain a high concentration of Hg (II), both the Hg° and the diethyl mercury peaks will be bigger.

11.4.13. The ratio of the two peaks is indicative of the quality of the Carbotrap ® trap. As the trap degrades, the amount of thermal breakdown of organomercurials increases. Since the diethyl mercury is much more sensitive to thermal breakdown than the methyl-ethyl mercury, monitoring the latter peak can serve as an early warning for trap replacement. Generally, traps should be replaced any time the Hg° peak grows to be as large as the diethyl mercury peak. (Traps typically last 3 to 6 months.)

11.5. Calibration and standardization

11.5.1. The calibration contains five or more non-zero points and the results of the analysis of at least one bubbler (ethylation) blanks.

11.5.2. Using the procedure in Section 11.3 and 11.5, standards are analyzed by the addition of aliquots of Hg working standards A, B, or C (Section 8.13-8.15) directly into 50 mL of previously DIH_2O in the bubbler. A typical curve might contain 2, 5, 10, 50, 100, 500 pg for low end analysis, or for higher concentrations: 5, 50, 100, 500, 1000, 2000 pg.

11.5.3. For each calibration point, subtract the mean peak area of the bubbler (ethylation) blank(s) for the batch from the area of each standard. Calculate the calibration factors (CF) for Hg in each of the five to six standards as follows:

$$CF = \frac{(C_s)}{(A_{Corr})}$$

where C_s = Concentration of the standard
A_{corr} = bubbler (ethylation) blank corrected peak height (or area).

Calculate the percent relative standard deviation (%RSD) of the calibration factors over the six-point range.

11.5.4. Calibration criteria are as follows:

(a) There must be a minimum of five non-zero calibration points.
(b) Calculate the percent relative standard deviation (% RSD) for each standard.

11.5.5. Ongoing precision and recovery: Perform the ongoing precision and recovery test to verify calibration prior to and at the end of each 20 field sample analytical batch.

12. QA/QC REQUIREMENTS

12.1. Initial demonstration of capability:

12.1.1. Laboratory performance is compared to the established performance criteria 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 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 MMHg.

Proprietary

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 CAS SOP for *Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation* (ADM-MDL).

12.2.2. Calculate the average concentration found (X) in the sample concentration, and the standard deviation (s) of the concentrations for each analyte. Calculate the MDL for the 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 and in the SOP for *Sample Batches*. An analytical batch is a set of samples prepared 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 or SRM. In addition, there must be one MS/MSD pair analyzed for every 20 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 an OPR standard near the middle of the curve, before and after analysis of each analytical batch according to the procedure beginning in Section 11. Subtract the mean peak area of the bubbler (ethylation) blank from the area for the OPR to compute the concentration for the OPR.

12.3.1.2. 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, the concentration is not in the specified range, the analytical process is not in control. Correct the problem and repeat the OPR and recovery test.

12.3.2. Quality control sample (QCS): A QCS from a source different than the MeHg used to produce the standards (OPR and working standards), must be analyzed at the beginning of each analytical batch. The acceptance criteria for the QCS is 75 -125% Recovery. Alternatively, a reference material is used and the acceptance criterion is the known concentration range provided with the material.

12.3.3. Matrix spike (MS) and matrix spike duplicate (MSD):

Proprietary

12.3.3.1. To assess the performance of the method on a given sample matrix, a minimum of 1 MS/MSD set per batch of 20 samples is performed. A spike level of 5 to 10 pg total at instrument has been found to acceptable.

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 by the following equation:

$$P = 100 (A-B)/T$$

where:

A = Measured concentration of analyte after spiking
B = Measured concentration of analyte before spiking
T = True concentration of the spike

Compare the percent recovery with the QC acceptance criteria in Table 1.

If the MS/MSD results fail the acceptance criteria, and recovery for the OPR standard for the analytical batch is within the acceptance criteria in Table 1, an interference may be present. The result should be flagged and reported to ALS management.

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.

12.3.3.4. 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.

$$RPD = 200 \times |D1-D2| / (D1+ D2)$$

Where:

Dl = concentration of MMHg in the MS sample
D2 = concentration of MMHg 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 (Ethylation) Blank - A minimum of one Ethylation Blank is run for each calibration curve. The ethylation blank is run on clean DIH_2O exactly as standards (i.e. with acetate buffer and ethylating reagent and purged onto a trap). The peak height or area of the ethylation blank (or mean of 2 ethylation

Proprietary

blanks) is subtracted from the standards to establish the mean calibration factor.

12.3.4.2. Method Blank: During sample preparation, prepare three method blanks for every 20 tissue samples. Method Blanks are prepared by adding all reagents to a bomb with no sample and performing the entire analysis. The mean analyte concentration for the Method Blank (volume and weight corrected if needed), is subtracted from the concentration of the sample. (If all weights and volumes are equivalent, the mean Peak Height can be subtracted before calculating the sample concentrations.)

12.4. 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, REVIEW, AND REPORTING

13.1. 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.

13.2. Quantitation

13.2.1. Calculate the concentration of MMHg in each sample directly from the mean calibration factor (CFm) as follows:

$$\frac{\text{(PK Ht SAMPLE – PK Ht METH BLK) * (CFm) (Bomb Volume i.e. 50 mL)}}{\text{Aliquot Volume i.e. 0.050 mL * Tissue SAMPLE weight i.e. 0.2g * 1000 ng/pg}}$$

= ng MMHg / g tissue

NOTE: If different volumes and weights are used:

- Calculate the "at instrument" concentrations of the Sample and the Method blank first, using the proper weight / volume / dilution factor corrections.
- Calculate the difference of the Sample Concentration minus the Mean Method Blank concentration.
- Multiply this concentration by the Bomb Volume / (Aliquot volume * Sample Weight (g) * 1000 ng/pg)

13.3. If any sample result is over the calibration range, prepare and analyze a diluted sample using the appropriate dilution factor to bring within range.

13.4. The data packet for the sequence is submitted for review by supervisor or designee. The results are transferred to the appropriate report form located in the ALS network directory R:\ICP\WIP. Once the results are transferred, the report is reviewed.

13.5. Refer to the SOP for Laboratory Data Review Process for instructions for data review.

14. METHOD PERFORMANCE

Proprietary

14.1. This method was validated through single laboratory studies of accuracy and precision. Refer to the reference method for additional method performance data available.

14.2. The method detection limit (MDL) is established using the procedure described in the SOP for *Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation* (ADM-MDL). Method Reporting Limits are established for this method based on MDL studies and as specified in the ALS Quality Assurance Manual.

15. CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

15.1. Refer to the SOP for *Non Conformity 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.

15.2. Handling out-of-control or unacceptable data

15.2.1. 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.

15.2.2. 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

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

Proprietary

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 for standard procedures.

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 . Summaries of the IPR are reviewed and signed by the supervisor. Copies may be forwarded to the employee's training file.

17.2.4. 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.

Proprietary

18. METHOD MODIFICATIONS

18.1. Method 1630 is applicable to water matrix. This procedure has been modified to apply to soils or sediments.

19. REFERENCES

19.1. Bloom, N.S (1989) Determination of Picogram Levels of Methyl Mercury by Aqueous Phase Ethylation, Followed by Cryogenic Gas Chromatography with Cold Vapor Atomic Fluorescence Detection, Can. J. Fish. Aq. Sci. 46: 1131.

19.2. Bloom, N.S., M. Horvat, and C.J. Watras (1995) Results of the International Mercury Speciation Intercomparison Exercise, Wat. Air Soil Pollut. (in press).

19.3. Bloom, N.S. (1995) "Trace Metals and Ultra-Clean Sample Handling," Environ. Lab. 7, 20.

19.4. Bloom, N.S.; Colman, J.A.; and Barber, Lee. (1997) "Artifact Formation of Methyl Mercury during Aqueous Distillation and Alternative Techniques for the Extraction of Methyl Mercury from Environmental Samples." Journal of Analytical Chemistry 358:371-377.

19.5. EPA Method 1630: Methyl Mercury in Water by Distillation, Aqueous Ethylation, Purge and Trap, and Cold Vapor Atom Fluorescence Spectrometry. U.S. Environmental Protection Agency, Office of Water, Washington D.C., 1998.

19.6. EPA Method 1631, Revision E: Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry. U.S. Environmental Protection Agency, Office of Water, Washington D.C., 2002

19.7. Fischer R, Rapsomanikis S, Andreae MO. Determination of methylmercury in fish samples using GC/AA and sodium tetraethyl borate derivatization. Anal Chem. 1993 Mar 15;65(6):763-6.

19.8. Gill, G.A. and Fitzgerald, W.F. (1985) Mercury Sampling of Open Ocean Waters at the Picogram Level . Deep Sea Res. 32: 287.

19.9. Horvat, M.; Bloom, N.; and Liang, L. (1993) "A Comparison of Distillation with Other Current Isolation Methods for the Determination of Mercury Compounds in Low Level Environmental Samples, Part I: Sediments." Analytica Chimica Acta 281:135-152.

19.10. Leermakers M, Ph. Quevauviller, M. Horvat, W. Baeyens, (2005). Mercury in environmental samples: Speciation, artifacts and validation. Trends in Analytical Chemistry 24, No. 5: 383-393

19.11. Liang, L.; Bloom, N.S.; and Horvat, M. (1994) "Simultaneous Determination of Mercury Speciation in Biological Materials by GC/CVAFS After Ethylation and Room-Temperature Precollection." Clin. Chem. 40/4: 602-607.

20. CHANGES SINCE THE LAST REVISION

20.1. Updated SOP to ALS formatting;

20.2. Updated definitions section.

Proprietary

Table 1

1.1.1. Acceptance Criteria for Test Performance

Acceptance Criterion	Criteria
Method Reporting Limit	2.0 ng/g (DRY)
Method Detection Limit	0.7 ng/g (DRY)
Initial Precision and Recovery	
Precision (s)	± 31%
Average Recovery (X)	69-131%
Matrix Spike/Matrix Spike Duplicate	
Recovery	65-135 %
Relative Percent Difference	± 35%
Ongoing Precision and Recovery	67-133%

Proprietary

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

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: [signature] Date: 12/11/15
Department Manager/Technical Director – Jeff Coronado

Approved By: [signature] Date: 12/11/15
QA Manager – Carl Degner

Approved By: [signature] Date: 12/13/15
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
Signature	Title	Date
Signature	Title	Date
Signature	Title	Date

UNCONTROLLED COPY

TABLE OF CONTENTS

1.SCOPE AND APPLICATION 3
2.METHOD SUMMARY 3
3.DEFINITIONS 3
4.INTERFERENCES 6
5.SAFETY 6
6.SAMPLE COLLECTION, CONTAINERS, PRESERVATION AND STORAGE 7
7.STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS 8
8.APPARATUS AND EQUIPMENT 9
9.PREVENTIVE MAINTENANCE 10
10.RESPONSIBILITIES 11
11.PROCEDURE 11
12.QA/QC REQUIREMENTS 17
13.DATA REDUCTION AND REPORTING 21
14.CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA 22
15.METHOD PERFORMANCE 23
16.POLLUTION PREVENTION AND WASTE MANAGEMENT 23
17.TRAINING 23
18.METHOD MODIFICATIONS 24
19.REFERENCES 24
20.CHANGES SINCE THE LAST REVISION 24

UNCONTROLLED COPY

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.

UNCONTROLLED COPY

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.

UNCONTROLLED COPY

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.

UNCONTROLLED COPY

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.

UNCONTROLLED COPY

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.

UNCONTROLLED COPY

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□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 $SnCl_2$□$2H_2O$ 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.

UNCONTROLLED COPY

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.

UNCONTROLLED COPY

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

UNCONTROLLED COPY

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

UNCONTROLLED COPY

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

<u>Soil Run Manual System:</u>

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

UNCONTROLLED COPY

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.

UNCONTROLLED COPY

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

UNCONTROLLED COPY

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.

UNCONTROLLED COPY

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 $SnCl_.$

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:

UNCONTROLLED COPY

$$CF = \frac{(C_s)}{(A_{Corr})}$$

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

UNCONTROLLED COPY

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-

UNCONTROLLED COPY

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.

UNCONTROLLED COPY

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

UNCONTROLLED COPY

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):

UNCONTROLLED COPY

- 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

UNCONTROLLED COPY

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).*

UNCONTROLLED COPY

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.

UNCONTROLLED COPY

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

DETERMINATION OF ARSENIC SPECIES BY HYDRIDE GENERATION CRYOGENIC TRAPPING GAS CHROMATOGRAPHY

MET-1632

ALS-KELSO

SOP ID:	MET-1632	Rev. Number:	3	Effective Date:	02/15/2014

Approved By: Department Supervisor – Jeff Coronado Date: 1/13/14

Approved By: QA Manager – Suzanne LeMay Date: 1/13/14

Approved By: Laboratory Director – Jeff Grindstaff Date: 1/16/14

PROPRIETARY

Issue Date:	___________	Doc Control ID#:	___________	Issued To:	___________

Standard Operating Procedure

for

DETERMINATION OF ARSENIC SPECIES BY HYDRIDE GENERATION CRYOGENIC TRAPPING GAS CHROMATOGRAPHY

1. SCOPE AND APPLICATION

1.1. This Standard Operating Procedure (SOP) describes the procedure used for the analysis of Total Inorganic Arsenic (TIA) and Arsenic species by atomic absorption spectrophotometry based upon EPA 1632. This procedure describes both the preparation and analysis procedures used to determine the target analytes and reporting limits listed.

1.2. This procedure is used to determine the analytes of interest in aqueous samples. 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) and Method Detection Limits (MDLs) for target analytes are presented in Table 1. This method is designed for the measurement of arsenic species in the range of 0.01-50 ug/L.

1.3. Hydride generation cryogenic trapping gas chromatography atomic absorption spectrophotometry (HG-CT-GC-AAS) is used for sensitive species-specific determination of arsenite [As(III)], monomethylarsonic acid [MMA], and dimethylarsinic acid [DMA]. It is also used for the determination of total inorganic arsenic [TIA]. Arsenate [As (V)] is determined as the difference between TIA and As(III): As(V) = TIA - As(III). The same analyzer may also be used for low level quantification of arsenic speciation in various solid digests and leachates.

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 ADM-DOD, *Department of Defense Projects - Laboratory Practices and Project Management* may supersede the requirements defined in this SOP.

2. METHOD SUMMARY

2.1. In this technique arsenic species are selectively volatilized from solution by controlling the pH of the sample solution and reducing them to their corresponding hydrides with borohydride. The arsines are purged from the solution by a helium gas flow and trapped in a liquid nitrogen cooled 'U' tube packed with 15% OV-3 on Chromasorb WAW-DMCS. Heating the column revolatilizes the arsines and allows for chromatographic separation based on boiling points. Once separated the arsines are carried into a quartz furnace with a hydrogen-air flame where they are atomized and detected via atomic absorption spectrophotometry.

PROPRIETARY

3. DEFINITIONS

3.1. Analysis Sequence - Samples analyzed in a set are referred to as an analysis sequence. The sequence begins with instrument calibration followed by samples, interspersed with calibration standards (Calibration Verifications, Calibration Blanks, 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. Quality Control Sample (QCS) - The QCS solution is made from a second source stock solution and is used to verify the validity of the calibration standards. This standard is also known as Independent Calibration Verification (ICV).

3.3. 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. Sample duplicates 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.

3.4. 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. Because water samples do not require digestion prior to analysis, method blank samples are equivalent to instrument blanks or calibration blanks.

3.5. Ongoing Precision Recovery (OPR) - A method blank spiked with known quantities of analytes, carried throughout the digestion procedure. This is also known as a laboratory Control Sample (LCS).

3.6. Continuing Calibration Verification (CCV) - is spiked reagent water (aqueous blank spike) and is used to determine that the instrument remains in control. This is also known as a calibration verification (CALVER).

3.7. Continuing Calibration Blank (CCB) - The continuing calibration blank is a volume of arsenic-free water (typically 50mL) analyzed in the same manner as samples. 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. This is also known as a calibration blank (CALBLK).

3.8. Initial precision and recovery (IPR) - Four aliquots of the ongoing precision and recovery standard analyzed to establish the ability to generate acceptable precision and recovery. IPR test are performed before a method is used for the first time and any time the methods or instrumentation is modified.

3.9. Field duplicates - two separate samples collected in separate sample bottles at the same time. Analysis of duplicates give a measure of the precision associated with sample collection and laboratory procedures.

PROPRIETARY

3.10. Dissolved Inorganic Arsenic - All Potassium Borohydride (KBH_4) reducible As^{+3} and As^{+5} found in aqueous solution filtrate after passing the sample through a 0.45 um filter.

3.11. Total Inorganic Arsenic - All KBH_4 reducible As^{+3} and As^{+5} found in a sample. Total inorganic arsenic and total recoverable inorganic arsenic are synonymous.

4. INTERFERENCES

4.1. Little interference from environmental matrices (i.e. salts, chlorides, nitrates, and organics) has been noted. However high levels of transition metals or noble metals will dramatically inhibit the formation of arsenic hydrides, most likely by consumption of the borohydride, or by co-precipitation of reduced arsenic with other reduced metals. This is particularly a problem with respect to high dissolved Fe and Mn in reducing ground water and mine adduct samples.

4.2. If water is allowed to condense in the trap, multiple and irregular peaks occur in the region where Me_2AsH elutes. This may be overcome by heating the transfer line between the chromatographic column and the furnace. It was also observed that free chlorine (as contained in bad batches of HCl) completely suppresses the signal for all hydrides during cryotrapping. Thus, HCl with obvious green/yellow color must be avoided, and, if in doubt, it can be tested for free chlorine with iodide solution (formation of red/brown iodine). Hydrochloric acid can be purged in the hood of its free chlorine with N_2 (500 mL min^{-1} for 1 hour).

5. SAFETY

5.1. Follow all ALS safety practices as described in the ALS Safety Manual.

5.2. Each chemical compound or reagent should be treated as a potential health hazard. Exposure to these chemicals should be reduced to the lowest possible level. A reference file of material safety data sheets is available to all personnel involved in these analyses.

5.3. Hydrochloric Acid is used in this method. Hydrochloric acid is extremely corrosive and care must be taken while handling it. A face shield should be used while pouring acids. Safety glasses, lab coat and gloves should always be worn while working with these solutions.

5.4. Potassium Borohydride (KBH_4) is used in this method. KBH_4 is a flammable solid, water reactive substance and is harmful if inhaled, ingested, or absorbed. Safety glasses, lab coat and gloves should always be worn while working with this solution.

5.5. Arsenic standards are used in this method. Arsenic is harmful if inhaled, ingested, or absorbed. ALS purchases dilute standard solutions for this method. If primary solutions are prepared, they must be prepared in a hood. Safety glasses, lab coat and gloves should always be worn while working with arsenic standards.

6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION, AND STORAGE

6.1. Aqueous samples must be collected in polyethylene or glass bottles washed with 4 N HCl using accepted clean techniques. Do not use HNO_3 cleaned bottles, as their surfaces are oxidizing with respect to As(III). Caution must be taken with glass as glass containers have been found to contaminate for arsenic.

PROPRIETARY

6.2. Sample preservation must be performed in the field to reduce changes in Arsenic speciation that may occur during transport and storage. It is very difficult to preserve the original As(III)/As(V) ratio. River water tends to spontaneously reduce As(V) to As(III) and freezing of water tends to induce oxidation of As(III) to As(V), except in the case of very rapid freezing (in liquid nitrogen). Water samples are acidified to pH <2 with hydrochloric acid (3 mL 6M HCL/L) and stored at 0-4°C from the time of collection until analysis. If As species are not target analytes, the samples may be preserved upon receipt by the laboratory. Store the preserved sample for a minimum of 48 hours to allow the As adsorbed on the container walls to completely dissolve in the acidified sample. Holding time is 28 days from the time of collection until the time of analysis.

The best storage scheme is to quick freeze samples to -196°C in liquid nitrogen and store at -80°C (on dry ice) until analysis.

6.3. Current evidence suggests that HCl acidification is suitable for preserving arsenic speciation and it is valuable in preventing the precipitation of Fe(OH)3 which can both oxidize and co-precipitate As.

6.4. Tissue and solid samples are collected in plastic 4 oz jars and stored at <-18°C or freeze dried and stored at room temperature for up to a year prior to digestion and analysis.

7. REAGENTS AND STANDARDS

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 increasing the MDL, or lowering the accuracy of the determination.

7.2. Reagent Water: 18 meg-Ω ultra pure deionized water starting from a pre-purified (distilled, R.O., etc.) source.

7.3. Arsenite (As(III)) Stock Solution: A 1,000 ppm stock solution is prepared by dissolving 0.173 g sodium meta-arsenite (>98.7%) in 100 mL of 0.1% ascorbic acid solution. This solution is kept refrigerated at 0°- 4° C in an amber bottle. This solution is stable and is good for 12 months. Arsenite 1,000 ppm stock solutions can also be purchased from Elements Inc., Shasta Lake, CA and Inorganic Ventures, Inc., Lakewood, NJ.

7.4. Arsenate (As(V)) Stock Solution: A 1,000 ppm stock solution is prepared by dissolving 0.416 g sodium arsenate (98.0%) in 100 ml reagent water. This solution is stable and is good for five years. Arsenate 1,000 ppm stock solutions can also be purchased from Elements Inc., Shasta Lake, CA and Inorganic Ventures, Inc., Lakewood, NJ

7.5. Monomethylarsonate (MMA) Stock Solution: A 1,000 ppm working standard is prepared by diluting 0.1866 g monomethylarsonic acid in 100 ml reagent water. This solution is good for five years. Monomethylarsonate stock solutions can also be purchased from High Purity Standards, Charleston, SC.

7.6. Dimethylarsinate (DMA) Stock Solution: A 1,000 ppm stock solution is prepared by dissolving 0.1842 g dimethylarsinic acid (98%) in 100 mL of water. This solution is good for five years. Dimethylarsinate stock solutions can also be purchased from High Purity Standards, Charleston, SC.

PROPRIETARY

7.7. Working Standard A: An intermediate solution containing 10 mg/L of As(III), MMA, and DMA is made from measured aliquots of the above stock solutions (7.3, 7.5, 7.6) and diluted to measured volume with reagent water. A working standard containing 100 ug/L of As(III), MMA, and DMA is prepared from the intermediate solution.

7.8. Working Standard B: An intermediate solution containing 10 mg/L of TIA (5 mg/L As(III) and 5 mg/L As(V)), MMA, and DMA is made from a measured aliquot of the above stock solutions (7.3, 7.4, 7.5, 7.6) and diluted to measured volume with reagent water. A working standard containing 100 ug/L of TIA, MMA, and DMA is prepared from the intermediate solution.

7.9. Quality Control Sample (QCS) -The QCS must be prepared from a source different from that used to produce the calibration standards.

7.10. TRIS Buffer: To prepare pH 6.2 buffer solution, 394 g tris(hydroxymethyl)-aminomethane hydrochloride and 2.5 g reagent grade sodium hydroxide are dissolved in water to make 1000 mL of solution. This solution is good for five years.

7.11. Borohydride Solution: A 10% solution is prepared by dissolving 10 g KBH_4 into reagent water and diluting up to 100 ml with reagent water. This solution is stable for up to 3 days when kept covered and stored in the refrigerator overnight. For low level TIA determinations, KBH_4 is preferable over $NaBH_4$ since it was found to contain lower As blanks, dissolves much faster in water, forms a clear solution and liberates much less hydrogen.

7.12. 6 M Hydrochloric Acid: Equal volumes of reagent grade HCl and water are mixed to give a 6 M solution. This solution must be checked before use and should not have any measurable Arsenic.

7.13. 2M Hydrochloric Acid: Reagent grade HCl and water are combined in a 1:6 ratio to give a solution approximately 2M in HCl.

7.14. 0.1% EDTA: To prepare 1.0 liter of 0.1% EDTA, 0.416g of EDTA are dissolved in water and diluted up to measured volume.

7.15. 0.1M Phosphoric Acid: To prepare 1.0 liter of 0.1M phosphoric acid solution, 6.8ml of 85% H_3PO_4 are dissolved in water and diluted up to measured volume.

7.16. 0.1M Trifluoroacetic Acid: To prepare 1.0 liter of 0.1M trifluoroacetic acid solution, 11.4 grams of 99.5%+ CF_3COOH are dissolved in water and diluted up to measured volume.

7.17. 5M Potassium Hydroxide: To prepare 1.0 liter of 5M KOH, 316.9g of 88.5% KOH are dissolved in water and diluted up to measured volume.

7.18. Air: Breathing quality non-flammable gas.

7.19. Helium: High purity non-flammable gas.

7.20. Hydrogen: Ultra-high purity flammable gas.

PROPRIETARY

7.21. Liquid Nitrogen (LN2): Industrial grade, low pressure liquid gas.

8. EQUIPMENT AND SUPPLIES

8.1. The apparatus needed for speciation analysis of arsenic is shown in principle in Figure 1. A 1 cm column of silanized glass wool is inserted between the purging vessel and the GC column to prevent transfer of water droplets and aerosols to the GC column, which causes irreproducible elution of DMA and gradual decomposition of the GC packing material. However, since Teflon and other plastics seem to absorb small quantities of methylated arsines, the aerosol separator should be avoided, and all connections should be as short as possible when analyzing small concentrations of MMA and DMA. The specific instruments and equipment used in this laboratory is listed below.

8.2. Reaction Vessel. The reaction vessel made by grafting an 1/4" O.D. by 15 mm long side-arm inlet onto a 90 ml "Midget Impinger" at a 45° upward angle, starting at a point 1.0 cm from the bottom of the impinger. The side arm may be fitted with silicone rubber septa (Ace Glass #9096-31) to allow injection of borohydride into the solution. Reaction vessels of other sizes may be used to facilitate the analysis of smaller or larger samples.

8.3. Cryogenic GC Trap and Column. The cryogenic GC trap is constructed from a 10 mm o.d., 4.1 mm i.d. borosilicate glass tubing about 30 cm long bent into a 'U' shape with appropriate dimensions to fit into a tall wide-mouth Dewar flask. The quartz is silanized prior to packing, to minimize active sites, using Sylon-CT (Supelco). The entire column is wrapped in 4 meters of 22 gauge nichrome wire (*ca.* 3 turns cm^{-1}) the ends of which are affixed to electrical contacts. The column is packed at the outlet half with pre-conditioned 15% OV-3 on 60/80 mesh Chromosorb WAW-DMCS (Supelco). The ends are plugged with silanized glass wool. The entire assembly is then preconditioned by heating to 190°C for 30 min with a helium flow of 150 ml min^{-1}. After this it is silanized by placing two 100 ul aliquots of Sylon-CT in the inlet end of the column and heating to 190°C after each for 15 min. This conditioning and silanization process may be repeated whenever broadening or a decrease in sensitivity of analyte peaks is observed.

8.4. Atomizer: The furnace/atomizer consists of a quartz tube (9.0 mm I.D.) with opposing 6 mm O.D. inlets perpendicular to the furnace. A mixture of hydrogen and the carrier gas are admitted via one inlet and air through the opposing inlet. The opposing inlets minimize flame noise and reduce the possibility of extinguishing the flame with surges of hydrogen gas created by the reaction mixture. To ignite the furnace, all gases are turned on and a flame is brought to the furnace ends. After the furnace has warmed up (*ca.* 5 min), a flat metal spatula is placed smoothly, first, over one end of the furnace and, then, the other. This restricts the flame to the center of the tube and reduces flame noise. Because the flame is invisible, check to see that the flame is still burning by placing a mirror near the furnace openings and inspecting for water vapor condensation. Furnace temperature can also be monitored using a thermo-couple thermometer, by gluing the sensor tip to the outside, center, of the furnace with silicone glue. Optimal gas flow rates and pressures are as follows:

PROPRIETARY

Gas	Flow Rate (ml/min)	Flow meter setting	Pressure (psi)
Helium	160	'40'	10
Hydrogen	370	'65'	20
Air	180	'45'	20

8.5. Detector: This work is performed using a Buck Scientific Atomic Absorption Spectrophotometer fitted with a Hollow Cathode Lamp. The 193.7 nm line is used with a 0.7 nm (low) slit width and without background correction.

8.6. Connections: To minimize active sites all connections are either Teflon friction-fit or threaded tubing connectors. All transfer lines are either 3.2 mm O.D. Teflon tubing or silanized 6 mm O.D. borosilicate glass. Teflon connections must be kept as short as possible when analyzing for low levels of methylated As species because they appear to be absorbed.

8.7. Recorder: A single channel USB chromatography data system is used to collect the instrument output.

PROPRIETARY

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. 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. Damage to the GC trap is indicated by irreproducibility, irregular peak shapes, and spurious additional peaks. Replace the GC trap as needed.

9.3. Sometimes a trap can be revitalized by injecting three 50uL aliquots of the silanizing agent "Silyl-8" to the column, held in an oven at 160°C while passing an inert gas through the column.

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 ADM-TRAIN, *ALS-Kelso Training Procedure*.

11. PROCEDURE

11.1. Sample Preparation

11.1.1. Record all sample preparation and sample information on the applicable benchsheet.

11.1.2. Tissue Samples

11.1.2.1. Tissue samples for total inorganic arsenic are prepared by weighing 1 gram into a glass scintillation vial. To each sample, add 20ml of 2M HCl. The samples are heated at 75-85°C overnight (16hrs), then shaken and allowed to cool. The supernatant liquid is analyzed.

11.1.2.2. Tissue samples for arsenic (III) are prepared by weighing 100 milligrams into a glass scintillation vial. To each sample, add 20mL of 0.1% EDTA. The samples are heated at 75-85°C overnight (16hrs), then shaken and allowed to cool. The supernatant liquid is analyzed.

11.1.2.3. Tissue samples for monomethylarsonate and dimethylarsinate are prepared in the same manner as total inorganic arsenic, see section 11.1.2.1.

11.1.3. Solid samples

11.1.3.1. Solid samples for total inorganic arsenic are prepared in the same manner as tissue samples for total inorganic arsenic, see section 11.1.2.1.

11.1.3.2. Solid samples for arsenic(III) are prepared by weighing 1 gram into a plastic centrifuge tube. To each sample, add 25mL of 0.1M H_3PO_4. The samples are shaken for 18-24 hours, centrifuged, and then the supernatant liquid transferred to a clean tube for analysis.

PROPRIETARY

11.2. Sample Analysis

11.2.1. Water Samples

11.2.1.1. Determination of Total Inorganic and Methylated Arsenic: Place 50 ml of aqueous sample (or sample extract containing between 5 and 25ng As, diluted to 50 ml with reagent water) and 2.0 mL of 6 M HCl in reaction vessel and replace top. At this time raise the Dewar flask filled with liquid nitrogen so that the trap is immersed and top off liquid nitrogen to completely fill the dewar. Turn 4-way valve such that bubbles are purging through the liquid, then slowly (30-45 seconds) inject a 1.25 mL aliquot of 10% KBH4 through the silicon rubber septum with a disposable 3 ml hypodermic syringe. Since all of the substantial As blank comes from the borohydride solution, it is essential that the injected amount is as reproducible as possible if low MDLs are to be met. Purge the system for 3 min (starting from the beginning of the injection), which is sufficient for quantitative recovery of all species. Longer purging times transfer more water vapor to the GC column, which causes faster column deterioration and has a negative influence on the determination of the methylated arsines, namely lower recoveries and worse reproducibility. At the end of purging, first turn the 4-way valve such that

gas is flowing around the bubbler, then, as simultaneously as possible, remove the liquid nitrogen from the GC column, start the chromatography data collector, and begin heating the GC column with 28 v (end temperature should be approximately 150°C). Arsines elute in order of their boiling points (AsH_3, $MeAsH_2$, Me_2AsH). A low broad "peak" following the Me_2AsH peak indicates the passage of the water vapor collected during purging.

11.2.1.2. Arsenite Determination: Arsenite is determined by varying the pH so that only As(III) reacts to form AsH3. The procedure is similar to that described above except that 2.0 ml of TRIS buffer is used in place of the 6 M HCl and only 0.75 mL of KBH4 is injected through the septum. In this case the KBH4 is injected quickly, as there will be little or no foaming. Prior to addition of KBH4 the sample pH is adjusted to 5-7 using 5M KOH. At this pH the separation of As(III) from As(V) is quantitative. If organo-arsenic compounds are present, they will be partially released - ignore these peaks.

11.2.2. Tissue Samples - tissue samples are analyzed in the same manner as aqueous samples, with the exception that only 2.0 ml of extract is added to the reaction vessel then diluted up to 50mls with reagent water.

11.2.3. Solid samples - solid samples are analyzed in the same manner as aqueous samples, with the exception that only 0.1mL of extract is added to the reaction vessel then diluted up to 50mls with reagent water.

11.2.4. An antifoam agent may be needed, particularly for tissue samples, to prevent foam from damaging the trap. If needed, a 1% solution of antifoam B is used.

11.3. Calibration and Standardization

11.3.1. Allow the AAS instrument to warm up for 15 minutes and the flame on for 30 minutes before beginning the calibration. Verify the wavelength is set to 193.7 nm with a 0.7 nm slit width.

11.3.2. A calibration curve is determined daily from analysis of 3 or more different amounts. The lowest calibration point must be equivalent to the MRL, or lower. The response factors determined from this curve must be less than 20% in order to continue with analysis. Quantitation is based on peak area.

11.3.3. For each point, subtract the peak area of the first instrument blank from the peak area of each standard. Calculate the response factors for each arsenic species in each of the standards as follows:

$RF = (A_x)/(C_x)$

where:
A_x = net peak area of the compound being measured.
C_x = Concentration of the compound being measured (ng).

11.3.4. Calculate the mean response factor $(\overline{RF_x})$ for each analyte from the calibration levels. Calculate standard deviation (SD) and the percent relative standard deviations (%RSD) for each analyte from the mean with:

PROPRIETARY

$$\%RSD = \frac{(SD)}{(\overline{RF_x})}100.$$

11.3.5. The % RSD should be less than 20% for each compound.

11.3.6. If the % RSD for any compound is 20% or less, linearity can be assumed over the calibration range, and the relative response factor for each analyte is used to quantitate sample analytes.

11.3.7. Calibration Verification (CCV) – The calibration curve is verified daily and every 10 samples and at the end of the analytical run. Recovery of the CCV standard must be within the limits listed in Table 2

11.3.8. Matrix spike and matrix spike duplicates (MS/MSD) – For each analytical sequence and every 10 samples (or fewer), spike at the level specified in section 12.3.6.1, and process in the same manner as the samples.

11.3.9. Calibration blanks – Analyze at least three calibration blanks per analytical sequence. One is required after calibration as well as after every CCV analysis. CCBs are equivalent to method blanks for water analysis only. For soils and other leachates 3 method blanks are required in addition to the CCBs.

11.4. An analytical sequence is as many samples as can be analyzed in a 24 hour period given the instrument remains in control. The analytical sequence contains the following standards, samples, and blanks, in order:

Water Run:

Total Inorganic and Methylated Arsenic or Arsenite Analytical Sequence:

Calibration Blank
Working Std A : 0.5 ng
Working Std A : 2.5ng (low point for DMA, otherwise not included)
Working Std A : 10 ng
Working Std A : 20 ng
Working Std A : 30 ng
Calibration Blank
Working Std B : CCV 10 ng
Calibration Blank
Up to 10 samples including QC (MS/MSD)
Working Std B : CCV 10 ng
Calibration Blank

Tissue or Soil Run:

Total Inorganic and Methylated Arsenic or Arsenite Analytical Sequence:

PROPRIETARY

Calibration Blank
Working Std A : *
Working Std A : 10 ng
Working Std A : 20 ng
Working Std A : 30 ng
Calibration Blank
Working Std B : CCV 10 ng
Calibration Blank
Ongoing Precision Recovery (OPR)
Method Blank 1
Method Blank 2
Method Blank 3
Up to 6 samples including QC (MS/MSD)
Working Std B : CCV 10 ng
Calibration Blank
Up to 10 samples including QC (MS/MSD)
Working Std B : CCV 10 ng
Calibration Blank

*Low point concentrations:

- As(III), tissue: 0.4ng or lower
- As(III), soil: 0.4ng or lower
- TIAs, soil: 0.5ng or lower
- TIAs, tissue: 2.0ng or lower
- MMA, tissue: 2.0ng or lower
- DMA, tissue: 4.0ng or lower
- MMA/DMA, soil: not performed

12. QUALITY CONTROL

12.1. Initial Precision and Recovery Validation

12.1.1. 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 water samples are spiked with the QCS solution, then prepared and analyzed.

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 (water or soil) 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/L, and the standard deviation of the concentrations (s) in µg/L for each analyte. Calculate the MDL for each analyte. Refer to CE-QA011, *Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantification.* The MDL study must be verified annually.

12.2.3. Limits of Quantification (LOQ)

IF THIS SOP IS ACCESSED ELECTRONICALLY, IT IS AN UNCONTROLLED COPY AND WILL NOT BE UPDATED.

PROPRIETARY

12.2.3.1. The laboratory establishes 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 or extract prepared at the lowest point calibration standard provides confirmation of the established sensitivity of the method. The LOQ recoveries must be within 50% of the true values to verify the data reporting limit. Refer to CE-QA011, *Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantification.*

12.2.4. 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.

12.3. Ongoing QC Samples required are described in the ALS-Kelso Quality Assurance Manual, in the SOP for Sample Batches, and in Method 1632. An analytical sequence must contain a calibration verification and a calibration blank every 10 samples. One MS/MSD pair must be analyzed for every 10 samples.

12.3.1. Method Blank

12.3.1.1. At least three method blanks are analyzed with every analytical sequence. 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.

12.3.1.2. Method Blanks for water samples are identical to calibration blanks.

12.3.2. Calibration blank - Analyze at least three calibration blanks per analytical sequence. One is required after calibration as well as after every CCV analysis. CCBs are equivalent to method blanks for water analysis only. For soils and other leachates 3 method blanks are required in addition to the CCBs. If the calibration blank exceeds the MRL, discontinue analysis, correct the problem, and recalibrate the instrument.

12.3.3. Calibration Verification - The calibration verification is verified daily and every 10 samples and at the end of the analytical run. Recovery of the CCV standard must be within the limits listed in Table 2

12.3.4. Ongoing and Precision Recovery (OPR) or Lab Control Sample (LCS)

12.3.4.1. CCV analysis is equivalent to the analysis of an aqueous OPR.

12.3.4.2. The OPR is composed of analyte-free water into which is spiked a number of appropriate target analytes. The OPR is designed to monitor the accuracy of the procedure. The concentration of the spike in the OPR matrix should be at 5 to 10 times the MRL or at levels specified by a project analysis plan.

PROPRIETARY

12.3.4.3. An OPR must be prepared and analyzed with every batch of 20 (or fewer) samples. Calculate the OPR recovery as follows:

$$\%R = X/TV \times 100$$

Where X = Concentration of the analyte recovered
TV = True value of amount spiked

12.3.4.4. The acceptance criteria are given in Table 2. If the OPR fails acceptance criteria, corrective action must be taken. Corrective action includes recalculation or reanalysis.

12.3.5. Quality Control Sample (QCS)

12.3.5.1. The QCS is designed to verify the validity of the calibration. The QCS is analyzed on a quarterly basis, and whenever the calibration standards are re-prepared.

12.3.5.2. The QCS is prepared from a source different from that used to produce the calibration standards. The lab may use Certified Reference Materials (CRMs) or prepare a spike solution obtained from a second source to make the QCS.

12.3.5.3. The determined mean concentration from three analyses of the QCS must be within ± 10% of its stated value.

12.3.6. Matrix Spike

12.3.6.1. A matrix spike (MS) and duplicate matrix spike (DMS) must be prepared and analyzed with every batch of 10 (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. The spike must contain analytes at one to five times the background levels in the parent sample. 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

12.3.6.2. Calculate Relative Percent Difference (RPD) as:

$$\%RPD = \frac{|R1 - R2|}{(R1 + R2)/2} \times 100$$

Where R1= Higher Result
R2= Lower Result

PROPRIETARY

12.3.6.3. Following analysis of the MS the percent recovery is calculated and compared to acceptance limits in Table 2. If the recovery is within control limits the results may be reported. If not, and the QCS is within control limits, this indicates that the matrix potentially biases analyte recovery. Verify 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.

13. CALCULATIONS, DATA REDUCTION, AND REPORTING

13.1. Calculate the concentration of each arsenic species in each sample directly from the mean calibration factor.

Aqueous Samples:

$$Concentration\,(\mu g/L) = \frac{(As - Ab)}{(RFx)(V)}$$

Where A_s = peak area for arsenic species in sample
A_b = peak area for arsenic species in blank
RF_x = Response Factor Mean
V = Volume of sample

13.2. The concentration of As(V) is calculated by subtracting the value for As(III) from that of total inorganic As.

13.3. Nonaqueous Samples:

$$Concentration\,(mg/Kg) = \frac{(As - Ab)(D)}{(RFx)(W)}$$

Where A_s = peak area for arsenic species in sample
A_b = peak area for arsenic species in blank
RF_x = Response Factor Mean
D = Dilution Factor
W = Weight of sample extracted in grams. The wet or dry weight may be used, depending upon the specific client requirements.

13.4. 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.

13.5. Reporting

13.5.1. Refer to ADM-RG, *Data Reporting and Report Generation* for reporting guidelines.

PROPRIETARY

13.5.2. Report results for each As species in ug/L or ug/g to three significant figures.

13.5.3. All sample results are reported blank corrected.

13.5.4. Reports are generated in the CAS LIMS by compiling the SMO login, sample prep database, instrument date, and client-specified report requirements (when specified). This compilation is then transferred to a file which Excel© uses to generate a report. The forms generated may be ALS standard reports, DOD, or client-specific reports. The compiled data from LIMS is also used to create EDDs.

13.5.5. As an alternative, reports are generated using Excel© templates located in R:\ICP\FORMS\Arsenic-1632. 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.

13.6. Data Review and Assessment

13.6.1. 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. 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.

PROPRIETARY

14. CORRECTIVE ACTION

14.1. Refer to CE-QA008, *Nonconformance and Corrective Action* 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. Documentation of a nonconformity must be done using a Nonconformity and Corrective Action Report (NCAR) when:

- 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 specified in Section 12.1. The method detection limit(s) and method reporting limit(s) were established for this method as specified in Section 12.2.

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.

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 ADM-TRAIN, ALS-Kelso Training Procedure.

17.2.1. 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. This procedure uses Potassium Borohydride (KBH_4) instead of Sodium Borohydride ($NaBH_4$) as stated in the method. See Section 7.11.

PROPRIETARY

18.2. The calibration blank is evaluated to the laboratory MRL. See Section 12.3.2.

18.3. In Section 11.2.1.1, the sample is purged for 3 minutes instead of 7 as stated in the method.

19. REFERENCES

19.1. *Chemical speciation of Arsenic in Water and Tissue by Hydride Generation Quartz Furnace Atomic Absorption Spectrometry,* Method 1632, Revision A, January 2001, U. S. Environmental Protection Agency Office of Water Engineering and Analysis Division.

19.2. Crecelius, E.A., Bloom, N.S., Cowan, C.E., and Jenne, E.A., *Speciation of Arsenic in Natural Waters and Sediments, Volume 2: Arsenic Speciation.* Final Report, prepared for Electric Power Research Institute, Palo Alto, CA by Battelle, Pacific Northwest Laboratories, Richland, WA, 1986.

20. CHANGES SINCE THE LAST REVISION

20.1. Sec. 11.1.2.3: Sample prep for MMAs and DMAs in tissue was changed to more closely follow EPA 1632. Tissue samples are prepped in the same manner as TIAs. The section has been revised to refer to section 11.1.2.1.

20.2. Section 12.3.2: Added calibration blank evaluation criterion.

20.3. Section 18: Added deviations from the reference method.

PROPRIETARY

Table 1

TARGET COMPOUNDS, MRLs, and MDLs

Analyte	Method Detection Limit			Method Reporting Limit		
	Water ug/L	Tissue mg/Kg	Soil mg/Kg	Water ug/L	Tissue mg/Kg	Soil mg/Kg
IA	0.003	0.007	0.08	0.02	0.02	0.2
As^{+3}	0.003	0.02	0.04	0.02	0.04	0.1
MMA	0.002	0.008	Tbd	0.02	0.02	Tbd
DMA	0.006	0.02	Tbd	0.05	0.04	Tbd

Table 2

QC ACCEPTANCE CRITERIA

Aqueous Samples

IPR			QCS/ICV	CCV/OPR	MS/MSD	
Analyte	RPD %	% Rec			% R	%RPD
IA	<25	60-140	90-110%	80-120%	50-150	<35
As^{+3}	<25	40-160	90-110%	70-130%	30-170	<35
MMA	<20	70-130	90-110%	80-120%	60-140	<25
DMA	<30	50-150	90-110%	70-130%	40-160	<40

Tissue and Soil Samples

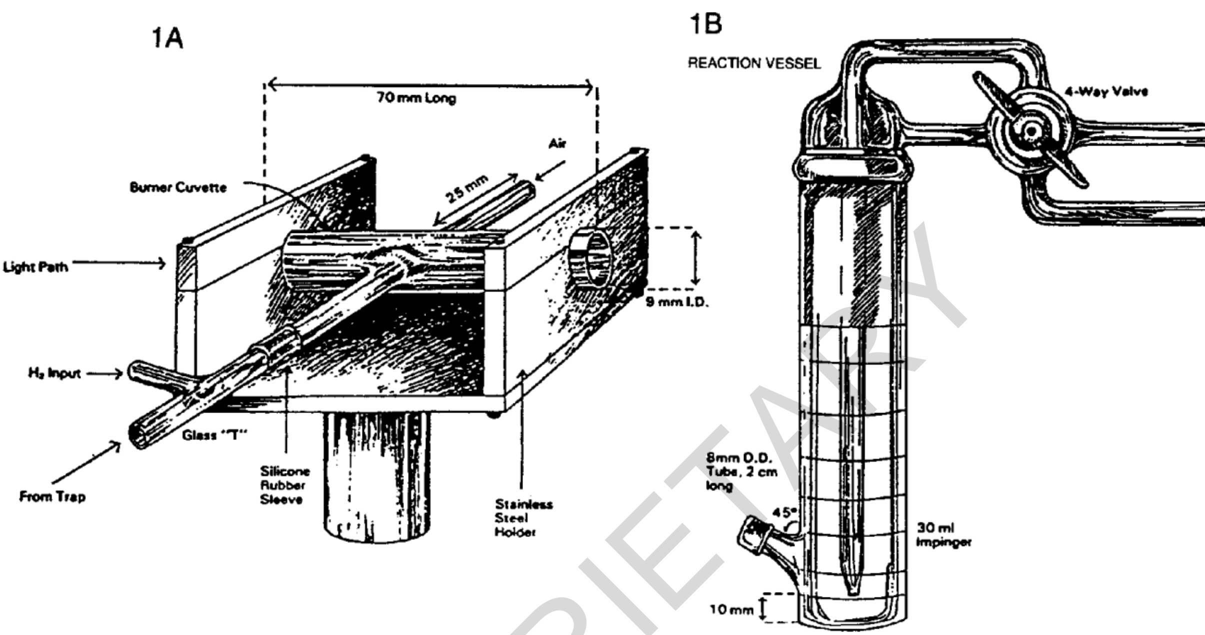
IPR			OPR	QCS/ICV	CCV/OPR	MS/MSD	
Analyte	RPD %	% Rec				% R	%RPD
IA	<25	60-140	50-150	90-110%	80-120%	50-150	<35
As^{+3}	<25	40-160	30-170	90-110%	70-130%	30-170	<35
MMA	<20	70-130	60-140	90-110%	80-120%	60-140	<25
DMA	<30	50-150	40-160	90-110%	70-130%	40-160	<40

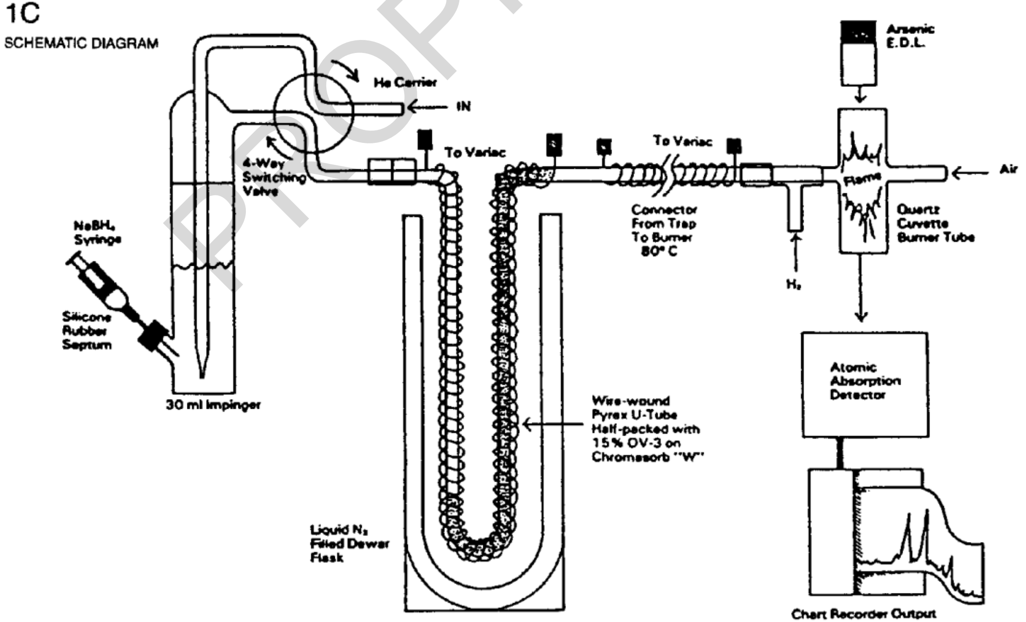
PROPRIETARY

IF THIS SOP IS ACCESSED ELECTRONICALLY, IT IS AN UNCONTROLLED COPY AND WILL NOT BE UPDATED.

Figure 1

Figure 1. Arsenic Speciation Apparatus: (a) Quartz Cuvette Burner Tube, (b) Reaction Vessel, and (c) Schematic Diagram





PROPRIETARY

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:	16
EFFECTIVE DATE:	1/01/2015

UNCONTROLLED COPY

DETERMINATION OF METALS AND TRACE ELEMENTS BY INDUCTIVELY COUPLED PLASMA-MASS SPECTROMETRY (METHOD 6020)

ALS-KELSO

SOP ID:	MET-6020	Rev. Number:	16	Effective Date:	1/01/2015

Approved By: [signature] Date: 12/9/14
Department Manager/Technical Director – Jeff Coronado

Approved By: [signature] Date: 12/9/14
QA Manager – Lee Wolf

Approved By: [signature] Date: 12/9/14
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

UNCONTROLLED COPY

TABLE OF CONTENTS

1.SCOPE AND APPLICATION 3
2.METHOD SUMMARY 3
3.DEFINITIONS 3
4.INTERFERENCES 5
5.SAFETY 6
6.SAMPLE COLLECTION, CONTAINERS, PRESERVATION AND STORAGE 6
7.STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS 6
8.APPARATUS AND EQUIPMENT 9
9.PREVENTIVE MAINTENANCE 9
10.RESPONSIBILITIES 10
11.PROCEDURE 10
12.QA/QC REQUIREMENTS 12
13.DATA REDUCTION AND REPORTING 16
14.CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA 17
15.METHOD PERFORMANCE 18
16.POLLUTION PREVENTION AND WASTE MANAGEMENT 18
17.TRAINING 18
18.METHOD MODIFICATIONS 19
19.REFERENCES 19
20.CHANGES SINCE THE LAST REVISION 19

UNCONTROLLED COPY

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.

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,

UNCONTROLLED COPY

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.

UNCONTROLLED COPY

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

UNCONTROLLED COPY

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

UNCONTROLLED COPY

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.

UNCONTROLLED COPY

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) solution 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.

UNCONTROLLED COPY

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. "OmniTrace Ultra" Concentrated Nitric Acid (EM Science # NX0408-2)

7.4.3. 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

UNCONTROLLED COPY

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. After the instrument has been placed in the "Operate" mode, begin completing the daily instrument log (see Attachments). Refer to the instrument manuals for the optimum settings for each instrument.

11.3. 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).

11.3.1. Multiplier Voltages

11.3.2. Gas Flows - Coolant Ar

11.3.3. The nebulizer and auxiliary flows are adjusted later as part of the optimizing procedure.

11.4. Optimization

11.4.1. Gas Flows

UNCONTROLLED COPY

11.4.1.1. Allow a period of not less than 30 minutes for the instrument to warm up.

11.4.1.2. Aspirate a mixed tune solution into the plasma and monitor the instrument output signal of In 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.

11.4.2. Tuning

11.4.2.1. 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.

11.4.2.2. 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.

11.4.2.3. 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.

11.4.2.4. Stability Check - Using the tune / mass calibration solution, perform a short-term stability check as per EPA Method 6020 or 6020A. The relative standard 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.

11.5. Analytical Run

11.5.1. 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.

11.5.2. 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

UNCONTROLLED COPY

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.

11.5.3. 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.

UNCONTROLLED COPY

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, 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.

UNCONTROLLED COPY

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 to the analytical isotopes via interpolation of the responses from nearest internal standard isotopes (Thermo instruments) or direct correlation of analyte to IS (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 125% when running method 6020, or 70-125% when running method

UNCONTROLLED COPY

6020A Revision 1. 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 reanalysis.

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. 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

UNCONTROLLED COPY

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 (Sec. 12.14) should be run on this sample. If both the matrix spike and the post digestion spike fail, then matrix effects are confirmed.

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 discussed above in section 12.6.2. 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:

$$\mu g / L\,(Sample) = C^{*} \; x \; Digestion\ Dilution\ Factor \; x \; Post\ Digestion\ Dilution\ Factor$$

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

UNCONTROLLED COPY

- 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.

UNCONTROLLED COPY

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

20. CHANGES SINCE THE LAST REVISION

20.1. Reformatted SOP to current ALS format.
20.2. Minor changes (correct typos and errors, etc.) throughout SOP.
20.3. Section 1 – revised to eliminate redundant language.
20.4. Section 7.2.7.2 – updated to replace Excell with Agilent
20.5. Section 7.3 – revised to list specific internal standards and concentrations.
20.6. Section 8.1 – updated instrument information.
20.7. Section 11.4.2.3 – revised to correct peak height %.
20.8. Sections 12.10 and 12.12 – revised to refer to DQO tables for QC limits
20.9. Section 16 – revised to include default language.
20.10. Section 17 – revised to include default language and be consistent with 200.8 SOP.
20.11. Table 1 – updated.
20.12. Attachments updated.

UNCONTROLLED COPY

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

UNCONTROLLED COPY

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

UNCONTROLLED COPY

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

UNCONTROLLED COPY

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

UNCONTROLLED COPY

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

UNCONTROLLED COPY

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

UNCONTROLLED COPY

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

UNCONTROLLED COPY

ALS Standard Operating Procedure

DOCUMENT TITLE:	DETERMINATION OF METALS AND TRACE ELEMENTS BY INDUCTIVELY COUPLED PLASMA ATOMIC EMISSION SPECTROMETRY (ICP)
REFERENCED METHOD:	EPA 200.7/6010C
SOP ID:	MET-ICP
REVISION NUMBER:	25
EFFECTIVE DATE:	01/01/2015

DETERMINATION OF METALS AND TRACE ELEMENTS BY INDUCTIVELY COUPLED PLASMA ATOMIC EMISSION SPECTROMETRY (ICP)

ALS-KELSO

SOP ID:	MET-ICP	Rev. Number:	25	Effective Date:	01/01/2015

Approved By: Department Supervisor/Technical Director – Jeff Coronado Date: 12/8/14

Approved By: QA Manager – Lee Wolf Date: 12/8/14

Approved By: Laboratory Director – Jeff Grindstaff Date: 12/8/14

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

1.SCOPE AND APPLICATION 3
2.METHOD SUMMARY 3
3.DEFINITIONS 3
4.INTERFERENCES 5
5.SAFETY 5
6.SAMPLE COLLECTION, CONTAINERS, PRESERVATION AND STORAGE 6
7.STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS 6
8.APPARATUS AND EQUIPMENT 8
9.PREVENTIVE MAINTENANCE 8
10.RESPONSIBILITIES 8
11.PROCEDURE 8
12.QA/QC REQUIREMENTS 11
13.DATA REDUCTION, REVIEW, AND REPORTING 14
14.CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA 15
15.METHOD PERFORMANCE 15
16.POLLUTION PREVENTION AND WASTE MANAGEMENT 16
17.TRAINING 16
18.METHOD MODIFICATIONS 16
19.REFERENCES 16
20.CHANGES SINCE THE LAST REVISION 17

DETERMINATION OF METALS AND TRACE ELEMENTS BY INDUCTIVELY COUPLED PLASMA ATOMIC EMISSION SPECTROMETRY (ICP)

1. SCOPE AND APPLICATION

1.1. This procedure describes the steps taken for the analysis of soil, sludge surface water and drinking water digestates using EPA methods 6010C, 200.7, and CLP ILM04.0 for a variety of elements. The digested samples and QC standards are all diluted in a similar acid matrix. A procedure is also given for calculation of hardness by Standard Methods 2340B.

1.2. The Method Reporting Limits (MRLs) for common elements are listed in Table 1. Equivalent nomenclature for MRL includes Estimated Quantitation Limit (EQL). Therefore, MRL=EQL. The reported MRL may be adjusted if required for specific project requirements, however, the capability of achieving other reported MRLs must be demonstrated. The Method Detection Limits (MDLs) that have been achieved are listed in Table 1. The MDL and MRL may change as annual studies are 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 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. A representative aliquot of sample is prepared as described in the applicable digestion SOP. The digestate is analyzed for the elements of interest using ICP spectrometry. The instrument measures characteristic emission spectra by optical spectrometry. The intensity of emission lines are monitored.

2.2. Final results are calculated using the digestion information and the results from the ICP analysis. Data is reported using standard ALS procedures and formats, or following project specific reporting specifications.

2.3. Deviations from the reference method(s): This SOP contains no deviations from the reference 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.

3.1.1. 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 injected 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. Non-aqueous 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 3.3.1 through 3.3.6. These can be such matrices as non-aqueous liquids, solvents, oil, etc.

3.3.8. Miscellaneous matrices – Samples of any composition not listed in 3.3.1 – 3.3.7. These can be such matrices as plant material, paper/paperboard, wood, autofluff, 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. 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.

3.7. 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.

3.8. Laboratory fortified Blank (LFB) - A laboratory blank that has been fortified with target analyte at the method reporting limit and used to determine if the laboratory can detect contaminants at the method reporting limit.

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 standard analyzed at specified intervals and used to verify the ongoing validity of the instrument calibration.

3.11. Instrument Blank (CCB) - The instrument blank (also called continuing calibration blank) is a volume of blank reagent of composition identical to the digestates. The purpose of the CCB is to determine the levels of contamination associated with the instrumental analysis.

4. INTERFERENCES

4.1. Interferences from contaminated reagents must be eliminated. The purity of acids must be established by the laboratory as being high enough to eliminate the introduction of contamination above the MRL (or above ½ the RL for DoD work).

4.2. Background emission and stray light can be compensated by background correction.

4.3. Spectral overlaps resulting in interelement contributions can be corrected for by using interelement correction factors. Interelement correction factors are established for each instrument and are maintained by the analyst at the workstation.

5. SAFETY

5.1. 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.

5.2. Hydrochloric, Nitric and Hydrofluoric Acids 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. Safety glasses, lab coat and gloves should be worn while working with the solutions.

5.3. High Voltage - The power unit supplies high voltage to the RF generator which is used to form the plasma. The unit should never be opened. Exposure to high voltage can cause injury or death.

5.4. UV Light -The plasma when lit is a very intense light, and must not be viewed with the naked eye. Protective lenses are in place on the instrument. Glasses with special protective lenses are available.

6. SAMPLE COLLECTION, CONTAINERS, PRESERVATION AND STORAGE

6.1. Samples are prepared using methods 3005A, 3010A, 3050, or CLPILM04.0 (ALS SOPs MET-3005A, MET-3010A, MET-3050, and MET-DIG). Samples are received in the ICP lab as completed digestates. Samples are stored in 50 mL plastic centrifuge tubes, 100 mL digestion vessels or in 100 mL volumetric flasks.

6.2. Water samples analyzed by EPA method 200.7 are preserved after arrival at the laboratory. These samples are held for a minimum of 24 hours and the pH verified to be <2 prior to digestion.

6.3. Soil samples are diluted prior to instrumental analysis by a factor of 2. This allows the method to meet the required 1 g of sample to 200 mL dilution during digestion.

6.4. Following analysis, digestates are stored until two weeks after all results have been reviewed and then brought to 3< pH<10 and disposed of through the sewer system.

7. STANDARDS, REAGENTS, AND CONSUMABLE MATERIALS

7.1. Standards Preparation

7.1.1. 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 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. Manufacturer's expiration dates are used to determine the viability of standards.

7.1.2. Calibration Standards

Calibration standards are prepared from commercially purchased single element 1000 ppm or 10,000 ppm stock standards as well as pre-mixed multi element stock standards. All standards are aliquoted using Class A volumetric pipettes, or calibrated

fixed and adjustable volume autopipetters. All dilutions are made in Class A volumetric glassware.

The standard mixes for each ICP system vary based on the requirements of each instrument. The composition of the ICAP 6500 standards are outlined in Table 2.

7.1.3. Continuing Calibration Verification (CCV) Standards

CCV standards are analyzed at the midpoint of the calibration. These standards are produced by making a two-fold dilution of each calibration standard. The CCV standards are then run in sequence during the analytical run.

7.1.4. Initial Calibration Verification (ICV) Standards

The ICV working standards are produced by direct dilution of two certified mixed stock solutions (QCP-CICV1 and QCP-CICV3 purchased from Inorganic Ventures or another qualified vendor and various single element stock solutions from sources different than the calibration standards. The composition of these standards is outlined in Table 3.

7.1.5. Interference Check Solutions (ICSA & ICSAB)

The ICSA and ICSAB working standards are produced by direct dilution of certified mixed stock solutions (CLPP-ICS-A and CLPP-ICS-B or equivalent.) Antimony is also added to the ICSAB solution from a 1000 ppm single element stock standard. The composition of these standards is outlined in Table 4.

7.1.6. CRI/Low Level Calibration Verification

The CRI, Low Level Initial Calibration Verification (LLICV), and Low Level Continuing Calibration Verification (LLCCV) are produced by diluting 1000 or 10000ppm single stock standards into a 100X intermediate standard and then diluted 1/100 to obtain the MRL level. Note: The level used is that of the normal MRL used for both instruments.

7.1.7. The solutions and materials used for the LCS and matrix spikes are described in the applicable digestion SOP.

7.1.8. Standard Log

The analyte, source, initial volume, final volume, final concentration and expiration date are recorded in a standard logbook kept in the ICP lab. The operator who prepares the standard must date and initial the entry in the standards logbook. The operator also places his initials and the date prepared on the standard container. In addition to working standards used in calibration, all other standards used in the analytical run such as ICVs, MRL standards, and other project or client specific standards shall be documented in the standard logbook.

7.2. High Purity Argon.

7.3. Capillary, rinse and peristaltic pump tubing.

7.4. 17 x 100mm polypropylene test tubes.

8. APPARATUS AND EQUIPMENT

8.1. Inductively Coupled Plasma Atomic Emission Spectrometer

8.1.1. Thermo Scientific ICAP 6500 (AES-03).
8.1.2. Thermo Scientific ICAP 6500 (AES-04).

8.2. Concentric nebulizers.

8.3. Microflow nebulizer for ICAP 6500.

8.4. Torches and injector tips for each ICP.

8.5. Cyclonic spray chambers for each instrument.

8.6. Water coolers for each ICP.

8.7. Argon Humidifiers for the ICAP 6500.

8.8. ESI SC4 DX Autosampler with Fast System for ICAP 6500.

8.9. Peristaltic Pumps for each Spectrometer.

8.10. RF Generators for each ICP (internal on the IRIS and ICAP 6500).

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. Torch, nebulizer, and spray chambers are cleaned as required. All instrument filters are vacuumed monthly. Dirty ICP torches and mixing chambers are soaked in aqua regia overnight, rinsed and placed in a clean dry area. The conical nebulizer is back flushed with acid or DI water as needed. The microflow nebulizer is not back flushed. Use the obstruction removal kit.

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. Training and proficiency is documented in accordance with the SOP *ADM-TRANDOC*.

11. PROCEDURE

11.1. Operating Parameters

11.1.1. For each Thermo Scientific ICAP 6500, the operating parameters are defined in the Method file. Default operating parameters are given in Tools/Options/New Method Parameters. However, each unique set of operating parameters is saved as a new file and the analyst must select and use the correct Method file for the application. Refer to the method files on the workstation for a listing of parameters for each file. The interelement correction factors to be used are established for the ICAP 6500 and are saved on the workstation also. Since these parameters change with method and correction factor updates, and due to the large amount of hardcopy printout for listing these parameters, it is not practical to include the parameters in this SOP.

11.2. Calibration/Standardization

11.2.1. ICAP 6500

11.2.1.1. Plasma is ignited and instrument is allowed to warm up for at least 30 minutes.

11.2.1.2. An internal standard is used for routine analyses on this instrument. Yttrium and Indium are used as internal standards. The internal standard solution is introduced into the analyzed solutions (standards, blanks, QC, samples, etc.) at 0.8 ug/mL for Y, and 1.6 ug/mL for In.

11.2.1.3. Run a peak check standard and adjust peaks as needed.

11.2.1.4. Standardize by running a Blank and a High Standard for each element in the analytical method. Analyst will initial and date the first page of the standardization.

11.2.2. Standardization is completed by analyzing an ICV for each analyte to be determined. For method 200.7 the result must be within ±5% of the true value. For method 6010B/C the result must be within ±10% of the true value. If the ICV fails when running method 6010C, either the calibration standards or the ICV must be prepared fresh and the instrument re-standardized. If the ICV fails when running methods 200.7 and 6010B only re-standardization is necessary.

11.2.3. Method 6010C also requires a LLICV be analyzed at the MRL level. The result must be within ±30% of the true value. The LLICV need not be made up with stock standards different than those of the calibration standards.

11.3. Analytical Run

11.3.1. Following standardization and ICV analysis, the remainder of the run is determined by what analytical method is being performed. These are listed below.

11.3.1.1. CLP ILM04.0: ICB, CCV, CCB, CRI, ICSA, ICSAB, CCV, CCB, routine samples. The CRI, ICSA, and ICSAB will be analyzed every 20 samples.

They will be labeled with an F indicating Final. Each set will be numbered in increasing order, i.e. ICSAF1, ICSAF2.

11.3.1.2. Methods 200.7 and 6010B/C: ICB, LLICV, CCV, CCB, CRI, ICSA, ICSAB, routine samples.

11.3.2. Evaluate the initial QC using the following criteria:

11.3.2.1. For methods 200.7 and 6010B/C, the following criteria apply:

- The ICB and CCB results are evaluated using method specified requirements. The following guidelines should also be used to determine acceptability:
- For 200.7, the result should be less than 3 times the standard deviation of the mean background signal.
- For method 6010B, the result should be less than the Method Detection Limit (MDL). In cases where the associated sample results are being reported to the Method Reporting Limit (MRL) the result may be greater than the MDL if the result does not adversely impact data quality.
- For method 6010C, the result should be less than the Lower Limit of Quantitation (LOQ).
- Where project specifications allow, the result may be over the MDL if the result does not adversely impact data quality.
- The CCV immediately following standardization must verify within ± 10% of the true values with a relative standard deviation of <5% from 2 replicate integrations for methods 6010B/C. For 200.7, the first CCV must verify within ± 5% with a RSD of <3% from 4 replicates. Calculate %RSD as follows:

$$\% RSD = \frac{StdDev_{CCV}}{Average_{CCV}} x\, 100$$

where: StdDevccv = Standard deviation of the replicate integrations
Averageccv = Average of the replicate CCV integrations

- The LLICV or CRI is a low level standard with concentrations at the RL. For DoD projects, the LLICV standard concentrations will be equal to the project RLs. For method 6010C the CRI results should be within 30% of the true value. For 200.7 and 6010B the LLICV/CRI results should be greater than the MDL and less than 2X the MRL. For method 6010C, the LLICV results should be ± 30% of the true value.

- The ICSA is run to check the validity of the Interelement Correction Factors (IECs).

Note: DoD QSM requires this to be run at the beginning of each analytical run.

- The ICSAB must be within 20% of the expected value for the CLPP-ICS-B elements and Sb.

11.3.2.2. The ICV, LLICV, ICB, CCV, CCB, CRI, and ICSAB must meet the criteria listed. Reanalyze any elements that fail.

11.3.2.3. For CLP, refer to SOW ILM04.0 for acceptance criteria.

11.3.3. Continuing Calibration Verification

11.3.3.1. CCVs are analyzed after every 10 samples and at the end of the analytical run. They must verify within ±10% of the expected value with a RSD of <10%.

11.3.3.2. CCBs are analyzed after every 10 samples and at the end of the analytical run. CCBs are evaluated as in section 11.3.2.1.

11.3.3.3. Method 6010C requires a LLCCV be analyzed at the end of each analysis batch. The LLCCV is at the MRL level and must verify within ±30% of the true value. Reanalyze any elements to be reported at low levels that are bracketed by the LLCCV if the standard fails.

11.3.4. If the CCV or CCB solutions fail, reanalyze any elements to be reported.

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 for each analyte must meet LCS criteria and the RSD< 30%.

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 replicates at a level near or below the MRL. Follow the procedures in Section 11 to analyze the samples. 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) 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. MDLs must be performed whenever there is a significant change in the background or instrument response.

12.2.3. A Limit of Detection (LOD) check must be performed after establishing the MDL and at least annually (quarterly if DoD) afterward. A blank is spiked with analytes at 2-4X the MDL and carried through the preparation and analytical procedure. The LOD is verified when the signal/noise ratio is > 3 for all analytes.

12.3. Limit of Quantitation Check(LOQ)/Lower Limit of Quantitation Check(LLQC)

For Method 6010C and drinking waters by method 200.7 a Lower Limit of Quantitation Check (LOQ/LLOQ) sample must be analyzed after establishing the MRL and at least annually (quarterly if DoD) afterward to demonstrate the desired detection capability. The LOQ/LLOQ sample is spiked at 1-2X the MRL and must be carried through the entire preparation and analytical procedure. Limits of quantitation are verified when all analytes are detected within 30% of their true value.

12.4. Linear Dynamic Range

The upper limit of the LDR must be established for each wavelength utilized. It must be determined from a linear calibration prepared in the normal manner using the established analytical operating procedure for the instrument. The LDR should be determined by analyzing at least three succeeding higher standard concentrations of the analyte until the observed analyte concentration is no more than 10% above or below the stated concentration of the standard. Determined LDRs must be documented and kept on file. The LDR which may be used for the analysis of samples should be judged by the analyst from the resulting data. Sample analyte concentrations that are greater than 90% of the determined upper LDR limit must be diluted and reanalyzed. The LDRs are verified semi-annually or whenever, in the judgment of the analyst, a change in analytical performance caused by either a change in instrument hardware or operating conditions would dictate they be redetermined.

12.5. Instrument Detection Limit

On a quarterly basis, the instrument detection limits for all analytes are determined as per procedures outlined in ILM04.0 (Section E, paragraph 10, 12 resp.). IDLs are determined using blanks and this data is kept on file.

12.6. Interelement Correction Factors

Semi-annually, instrument interferences are calculated as per ILM04.0 (Section E, paragraph 11) and Method 6010B/C. During the course of routine work, other interferences may be found. They are verified by the operator during the analytical run and data is manually corrected. Copies of this data are kept on file. Data can be manually corrected or automatically corrected using iTEVA software.

12.7. Internal Standard

Internal standard values are tracked by the instrument software. Values should remain within 60-125% of the value found in the calibration blank. If a sample is found to have and internal standard outside this value, the sample will be diluted to bring the internal standard into range.

12.8. 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 are:

12.8.1. Each sample preparation batch must have a method blank associated with it. The method blank result should be < MRL. If the method blank is found to be contaminated, it may be reported if the concentration in the associated samples is at least 20 times the amount found in the method blank for methods 200.7 and 6010B, otherwise redigest the batch. For Method 6010C, the method blank may be reported if the concentration in the associated samples is at least 10 times the amount found in the method blank.A contaminated method blank (MB) may also be reported if all of the associated samples are non-detect (ND).

Note: DoD QSM requires contamination in the MB be <1/2 the RL or < 1/10 any sample amount.

12.8.2. A Laboratory Control Sample (LCS) is digested one per batch, or per 20 samples. For soil samples, the recovery must fall within the ranges specified for the reference material. For CLP, use the prescribed limits for the SOW in use. If the LCS fails the acceptance criteria, redigest the batch of samples. For specifics on the preparation and composition of LCS samples refer to the appropriate digestion SOP.

12.8.3. A Duplicate sample is digested one per batch, or per 20 samples (i.e. 5%) for 6010B/C analysis, or per 10 samples (i.e. 10%) for 200.7 analyses. If the RPD is outside acceptance limits, either redigest the sample batch or flag the data appropriately, depending on the physical nature of the samples (e.g. non-homogenous).

12.8.4. A Laboratory fortified Blank (LFB) at the MRL is digested and analyzed with every batch of drinking water samples (method 200.7). The default acceptance criteria of 50-150% are to be used until sufficient data points are acquired to calculate in-house control limits.

12.8.5. A Matrix Spike sample is digested one per batch, or per 20 samples (i.e. 5%) for 6010B/C analysis, or per 10 samples (i.e. 10%) for 200.7 analyses. Where specified by project requirements, a matrix spike duplicate may be required. If the recovery is outside acceptance limits, either redigest the sample batch or flag the data appropriately, depending on the physical nature of the samples (e.g. non-homogenous). If the sample concentration is >4x the spike level, no action is required and data is flagged accordingly. For specifics on the preparation and composition of matrix spike solutions refer to the appropriate digestion SOP.

12.8.6. Acceptance criteria

12.8.6.1.Current ALS control limits and acceptance criteria for ongoing QC analyses are listed in the current ALS-Kelso DQO tables. Criteria are subject to change as statistical data are generated. The default method criteria may be used if

statistically generated criteria are broader or insufficient points are available for accurate statistical limits.

12.8.6.2. For all QC analyses, project-specific or program-specific (e.g. DOD) acceptance criteria may supersede ALS criteria. For analyses under the CLP SOW use the prescribed limits for the SOW in use.

12.8.7. Matrix Interference

12.8.7.1. When an analyst suspects that there may be any matrix interferences present, a post digestion spike may be performed. The recovery should be ± 20%.

12.8.7.2. If the post spike fails, a 1:5 serial dilution test shall be performed. The dilution should be within ± 10% of the original result.

12.8.7.3. A 1:5 serial dilution shall be performed for all Tier III or IV deliverables.

Note: DoD QSM recovery acceptance limits are 75-125%.

12.8.7.4. Post spikes for 6010C shall be performed for Tier III and Tier 1V.

12.9. Additional QC measures include control charting and compiling of QC data for generation of control limits.

12.10. CLP analyses are performed as per the QA/QC guidelines in the most current CLP SOW.

13. DATA REDUCTION, REVIEW, AND REPORTING

13.1. 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. The wavelengths used to quantify each metal are summarized in Table 5 for the IRIS and Table 6 for the ICAP6500.

Aqueous samples are reported in ug/L:

$$\mu g/L(Sample) = C^{*}\ x\ Digestion\ Dilution\ Factor\ x\ Post\ Digestion\ Dilution\ Factor \times 1000\ \mu g/mg$$

Solid samples are reported in mg/Kg:

$$mg/Kg\ \ (Sample) = C^{*}\ x\ Post\ Digestion\ Dilution\ Factor\ x\ \frac{Digestion\ Vol.(ml)}{Sample\ wt.(g)}\ x\ \frac{1L}{1000ml}\ x\ \frac{1000g}{1Kg}$$

C*= Concentration of analyte as measured at the instrument in mg/L.

13.2. If total hardness is to be reported, use Calcium and Magnesium results to calculate as follows. For reporting calcium hardness, use only the calcium portion of the equation.

$$Hardness, mg\ equivalent\ CaCO_3/L = 2.497\left[Ca, mg/L\right] + 4.118\left[Mg, mg/L\right]$$

13.3. A daily run log of all samples analyzed is maintained. All CLP data should be printed and stored after operator has checked for evenness of burns. A copy of this document will go with each package of Tier III or higher data run that day.

13.4. Data Review and Reporting

13.4.1. 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. The data is then placed in a work order file until complete. When the work order is complete, a report is generated. A final review is performed and the data is delivered to the project management department.

14. CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

14.1. Refer to the SOP for *Nonconformance 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) 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 3-10 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. Assist in the procedure under the guidance of an experienced analyst for approximately two weeks. 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 NELAP Initial Demonstration of Capability.

17.2. Training is documented following the *ALS Kelso, Training Procedure* (ADM-TRAIN) and the Corporate *Training Policy (*CE-QA003*).*

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. There are no known modifications in this laboratory standard operating procedure from the reference method.

19. REFERENCES

19.1. USEPA, Contract Laboratory Program, SOW #ILM04.0

19.2. Thermo Jarrell Ash ICAP61 Manual

19.3. USEPA, Test Methods for Evaluating Solid Waste, SW-846, 3rd Edition, Update III, Method 6010B, Revision 2, December 1996.

19.4. USEPA, Test Methods for Evaluating Solid Waste, SW-846, 3rd Edition, Update III, Method 6010C, Revision 3, February 2007.

19.5. USEPA, Methods for Determination of Metals in Environmental Samples, Supplement I, EPA/600/R-94/111, Method 200.7, Revision 4.4, May 1994.

19.6. *Hardness by Calculation, Method 2340B,* Standard Methods for the Examination of Water and Wastewater, 20th ed., 1998.

20. CHANGES SINCE THE LAST REVISION

20.1. Updated to current ALS format.
20.2. Revised internal document references from CAS to ALS
20.3. Minor typographical and format corrections.
20.4. Section 3– updated several definitions to standard definitions for SOPs.
20.5. Section 7.1.4 – corrected standard composition to reflect current practice.
20.6. Section 9.2 – revised to reflect current practice.
20.7. Section 11.3.2.1 – LL ICV criteria revised to reflect current practice.
20.8. Section 12.2.3 – LOD spike level corrected.
20.9. Section 12.4 – revised to reflect current practice (LDR semi-annual).
20.10. Sections 12.8.2. – 12.8.5 revised to remove outdated/redundant QC criteria and added new section 12.8.6.
20.11. Section 14 – updated to standard language.
20.12. Section 17 – updated to standard language.
20.13. Tables reference errors corrected and tables updated.

TABLE 1

Target Elements, Method Reporting Limits, and Method Detection Limits

METHOD	PREP METHOD	ANALYTE	MATRIX	MDL	MRL
0.7	EPA 3050B	Aluminum	Soil	0.5	2
200.7	EPA 3050B	Antimony	Soil	2	4
200.7	EPA 3050B	Arsenic	Soil	2	4
200.7	EPA 3050B	Barium	Soil	0.3	0.8
200.7	EPA 3050B	Beryllium	Soil	0.08	0.2
200.7	EPA 3050B	Bismuth	Soil	3	8
200.7	EPA 3050B	Boron	Soil	0.7	4
200.7	EPA 3050B	Cadmium	Soil	0.09	0.2
200.7	EPA 3050B	Calcium	Soil	1	4
200.7	EPA 3050B	Chromium	Soil	0.3	0.8
200.7	EPA 3050B	Cobalt	Soil	0.2	0.4
200.7	EPA 3050B	Copper	Soil	0.4	0.8
200.7	EPA 3050B	Iron	Soil	2	4
200.7	EPA 3050B	Lead	Soil	0.7	2
200.7	EPA 3050B	Lithium	Soil	0.6	4
200.7	EPA 3050B	Magnesium	Soil	0.2	2
200.7	EPA 3050B	Manganese	Soil	0.04	0.2
200.7	EPA 3050B	Molybdenum	Soil	0.2	0.8
200.7	EPA 3050B	Nickel	Soil	0.2	0.8
200.7	EPA 3050B	Phosphorus	Soil	3	8
200.7	EPA 3050B	Potassium	Soil	10	40
200.7	EPA 3050B	Selenium	Soil	2	4
200.7	EPA 3050B	Silver	Soil	0.3	0.8
200.7	EPA 3050B	Sodium	Soil	5	40
200.7	EPA 3050B	Strontium	Soil	0.05	0.2
200.7	EPA 3050B	Sulfur	Soil	4	8
200.7	EPA 3050B	Thallium	Soil	0.8	2
200.7	EPA 3050B	Tin	Soil	0.6	4
200.7	EPA 3050B	Titanium	Soil	0.2	0.4
200.7	EPA 3050B	Vanadium	Soil	0.3	0.8
200.7	EPA 3050B	Zinc	Soil	0.2	1

TABLE 1 – continued

METHOD	PREP METHOD	ANALYTE	MATRIX	MDL	MRL
200.7	MET-DIG (CLP)	Aluminum	Water	4	10
200.7	MET-DIG (CLP)	Antimony	Water	6	20
200.7	MET-DIG (CLP)	Arsenic	Water	5	10
200.7	MET-DIG (CLP)	Barium	Water	0.6	4
200.7	MET-DIG (CLP)	Beryllium	Water	0.5	1
200.7	MET-DIG (CLP)	Bismuth	Water	6	40
200.7	MET-DIG (CLP)	Boron	Water	4	20
200.7	MET-DIG (CLP)	Cadmium	Water	0.5	1
200.7	MET-DIG (CLP)	Calcium	Water	0.9	20
200.7	MET-DIG (CLP)	Chromium	Water	0.9	4
200.7	MET-DIG (CLP)	Cobalt	Water	1	2
200.7	MET-DIG (CLP)	Copper	Water	2	4
200.7	MET-DIG (CLP)	Iron	Water	3	20
200.7	MET-DIG (CLP)	Lead	Water	5	10
200.7	MET-DIG (CLP)	Lithium	Water	4	20
200.7	MET-DIG (CLP)	Magnesium	Water	0.3	5
200.7	MET-DIG (CLP)	Manganese	Water	0.3	1
200.7	MET-DIG (CLP)	Molybdenum	Water	0.9	4
200.7	MET-DIG (CLP)	Nickel	Water	0.6	4
200.7	MET-DIG (CLP)	Phosphorus	Water	6	40
200.7	MET-DIG (CLP)	Potassium	Water	60	200
200.7	MET-DIG (CLP)	Selenium	Water	9	20
200.7	MET-DIG (CLP)	Silicon	Water	20	200
200.7	MET-DIG (CLP)	Silver	Water	2	4
200.7	MET-DIG (CLP)	Sodium	Water	20	200
200.7	MET-DIG (CLP)	Strontium	Water	0.2	1
200.7	MET-DIG (CLP)	Sulfur	Water	20	40
200.7	MET-DIG (CLP)	Thallium	Water	4	10
200.7	MET-DIG (CLP)	Tin	Water	3	20
200.7	MET-DIG (CLP)	Titanium	Water	0.8	2
200.7	MET-DIG (CLP)	Vanadium	Water	1	4
200.7	MET-DIG (CLP)	Zinc	Water	0.6	4

TABLE 1 – continued

METHOD	PREP METHOD	ANALYTE	MATRIX	MDL	MRL
6010C	EPA 3050B	Aluminum	Soil	0.5	2
6010C	EPA 3050B	Antimony	Soil	2	4
6010C	EPA 3050B	Arsenic	Soil	2	4
6010C	EPA 3050B	Barium	Soil	0.3	0.8
6010C	EPA 3050B	Beryllium	Soil	0.08	0.2
6010C	EPA 3050B	Bismuth	Soil	3	8
6010C	EPA 3050B	Boron	Soil	0.7	4
6010C	EPA 3050B	Cadmium	Soil	0.09	0.2
6010C	EPA 3050B	Calcium	Soil	1	4
6010C	EPA 3050B	Chromium	Soil	0.3	0.8
6010C	EPA 3050B	Cobalt	Soil	0.2	0.4
6010C	EPA 3050B	Copper	Soil	0.4	0.8
6010C	EPA 3050B	Iron	Soil	2	4
6010C	EPA 3050B	Lead	Soil	0.7	2
6010C	EPA 3050B	Lithium	Soil	0.6	4
6010C	EPA 3050B	Magnesium	Soil	0.2	2
6010C	EPA 3050B	Manganese	Soil	0.04	0.2
6010C	EPA 3050B	Molybdenum	Soil	0.2	0.8
6010C	EPA 3050B	Nickel	Soil	0.2	0.8
6010C	EPA 3050B	Phosphorus	Soil	3	8
6010C	EPA 3050B	Potassium	Soil	10	40
6010C	EPA 3050B	Selenium	Soil	2	4
6010C	EPA 3050B	Silver	Soil	0.3	0.8
6010C	EPA 3050B	Sodium	Soil	5	40
6010C	EPA 3050B	Strontium	Soil	0.05	0.2
6010C	EPA 3050B	Sulfur	Soil	4	8
6010C	EPA 3050B	Thallium	Soil	0.8	2
6010C	EPA 3050B	Tin	Soil	0.6	4
6010C	EPA 3050B	Titanium	Soil	0.2	0.4
6010C	EPA 3050B	Vanadium	Soil	0.3	0.8
6010C	EPA 3050B	Zinc	Soil	0.2	1
6010C/AVS-SEM	EPA 821/R-91-100	Antimony	Soil	0.0008	0.003
6010C/AVS-SEM	EPA 821/R-91-100	Arsenic	Soil	0.002	0.005
6010C/AVS-SEM	EPA 821/R-91-100	Cadmium	Soil	0.00007	0.0002
6010C/AVS-SEM	EPA 821/R-91-100	Chromium	Soil	0.0004	0.002
6010C/AVS-SEM	EPA 821/R-91-100	Copper	Soil	0.0005	0.002
6010C/AVS-SEM	EPA 821/R-91-100	Lead	Soil	0.0005	0.001
6010C/AVS-SEM	EPA 821/R-91-100	Nickel	Soil	0.0003	0.001
6010C/AVS-SEM	EPA 821/R-91-100	Silver	Soil	0.0003	0.001
6010C/AVS-SEM	EPA 821/R-91-100	Zinc	Soil	0.0003	0.003

TABLE 1 – continued

METHOD	PREP METHOD	ANALYTE	MATRIX	MDL	MRL
6010C	MET-DIG (CLP)	Aluminum	Water	4	10
6010C	MET-DIG (CLP)	Antimony	Water	6	20
6010C	MET-DIG (CLP)	Arsenic	Water	5	10
6010C	MET-DIG (CLP)	Barium	Water	0.6	4
6010C	MET-DIG (CLP)	Beryllium	Water	0.5	1
6010C	MET-DIG (CLP)	Bismuth	Water	6	40
6010C	MET-DIG (CLP)	Boron	Water	4	20
6010C	MET-DIG (CLP)	Cadmium	Water	0.5	1
6010C	MET-DIG (CLP)	Calcium	Water	0.9	20
6010C	MET-DIG (CLP)	Chromium	Water	0.9	4
6010C	MET-DIG (CLP)	Cobalt	Water	1	2
6010C	MET-DIG (CLP)	Copper	Water	2	4
6010C	MET-DIG (CLP)	Iron	Water	3	20
6010C	MET-DIG (CLP)	Lead	Water	5	10
6010C	MET-DIG (CLP)	Lithium	Water	4	20
6010C	MET-DIG (CLP)	Magnesium	Water	0.3	5
6010C	MET-DIG (CLP)	Manganese	Water	0.3	1
6010C	MET-DIG (CLP)	Molybdenum	Water	0.9	4
6010C	MET-DIG (CLP)	Nickel	Water	0.6	4
6010C	MET-DIG (CLP)	Phosphorus	Water	6	40
6010C	MET-DIG (CLP)	Potassium	Water	60	200
6010C	MET-DIG (CLP)	Selenium	Water	9	20
6010C	MET-DIG (CLP)	Silicon	Water	20	200
6010C	MET-DIG (CLP)	Silver	Water	2	4
6010C	MET-DIG (CLP)	Sodium	Water	20	200
6010C	MET-DIG (CLP)	Strontium	Water	0.2	1
6010C	MET-DIG (CLP)	Sulfur	Water	20	40
6010C	MET-DIG (CLP)	Thallium	Water	4	10
6010C	MET-DIG (CLP)	Tin	Water	3	20
6010C	MET-DIG (CLP)	Titanium	Water	0.8	2
6010C	MET-DIG (CLP)	Vanadium	Water	1	4
6010C	MET-DIG (CLP)	Zinc	Water	0.6	4

TABLE 1 – continued

METHOD	PREP METHOD	ANALYTE	MATRIX	MDL	MRL
6010C	PSEP TISSUE	Aluminum	Tissue	0.3	1
6010C	PSEP TISSUE	Antimony	Tissue	0.5	2
6010C	PSEP TISSUE	Arsenic	Tissue	0.5	1
6010C	PSEP TISSUE	Barium	Tissue	0.07	0.4
6010C	PSEP TISSUE	Beryllium	Tissue	0.05	0.1
6010C	PSEP TISSUE	Boron	Tissue	0.8	2
6010C	PSEP TISSUE	Cadmium	Tissue	0.04	0.1
6010C	PSEP TISSUE	Calcium	Tissue	2	4
6010C	PSEP TISSUE	Chromium	Tissue	0.08	0.4
6010C	PSEP TISSUE	Cobalt	Tissue	0.07	0.2
6010C	PSEP TISSUE	Copper	Tissue	0.2	0.4
6010C	PSEP TISSUE	Iron	Tissue	1	2
6010C	PSEP TISSUE	Lead	Tissue	0.3	1
6010C	PSEP TISSUE	Lithium	Tissue	0.3	2
6010C	PSEP TISSUE	Magnesium	Tissue	0.6	2
6010C	PSEP TISSUE	Manganese	Tissue	0.03	0.1
6010C	PSEP TISSUE	Molybdenum	Tissue	0.2	0.4
6010C	PSEP TISSUE	Nickel	Tissue	0.2	0.4
6010C	PSEP TISSUE	Phosphorus	Tissue	2	4
6010C	PSEP TISSUE	Potassium	Tissue	9	20
6010C	PSEP TISSUE	Selenium	Tissue	0.9	2
6010C	PSEP TISSUE	Silicon	Tissue	4	20
6010C	PSEP TISSUE	Silver	Tissue	0.2	0.4
6010C	PSEP TISSUE	Sodium	Tissue	2	20
6010C	PSEP TISSUE	Strontium	Tissue	0.04	0.1
6010C	PSEP TISSUE	Thallium	Tissue	0.4	1
6010C	PSEP TISSUE	Tin	Tissue	0.3	2
6010C	PSEP TISSUE	Titanium	Tissue	0.08	0.2
6010C	PSEP TISSUE	Vanadium	Tissue	0.2	0.4
6010C	PSEP TISSUE	Zinc	Tissue	0.2	0.4

TABLE 1 – continued

METHOD	PREP METHOD	ANALYTE	MATRIX	MDL	MRL
6010C	1311/3010A	Antimony	TCLP	0.03	0.1
6010C	1311/3010A	Arsenic	TCLP	0.025	0.05
6010C	1311/3010A	Barium	TCLP	0.5	1
6010C	1311/3010A	Beryllium	TCLP	0.001	0.005
6010C	1311/3010A	Cadmium	TCLP	0.001	0.05
6010C	1311/3010A	Chromium	TCLP	0.01	0.05
6010C	1311/3010A	Cobalt	TCLP	0.0035	0.01
6010C	1311/3010A	Copper	TCLP	0.01	0.1
6010C	1311/3010A	Lead	TCLP	0.02	0.05
6010C	1311/3010A	Manganese	TCLP	0.0025	0.005
6010C	1311/3010A	Nickel	TCLP	0.0035	0.1
6010C	1311/3010A	Selenium	TCLP	0.025	0.1
6010C	1311/3010A	Silver	TCLP	0.004	0.05
6010C	1311/3010A	Thallium	TCLP	0.1	0.25
6010C	1311/3010A	Zinc	TCLP	0.1	1

TABLE 2 Standard A for ICAP 6500 ICP-OES					
Analyte	Source	Source Concentration (ppm)	Aliquot (mL)	Final Volume (mL)	Final Concentration (ppm)
Antimony	(1)	100	5	1000	0.5
Beryllium	(1)	100	5	1000	0.5
Boron	(1)	100	5	1000	0.5
Cadmium	(1)	100	5	1000	0.5
Calcium	Ca stock	1000	0.5	1000	1.0*
Chromium	(1)	100	5	1000	0.5
Cobalt	(1)	100	5	1000	0.5
Copper	(1)	100	5	1000	0.5
Iron	(1)	100	5	1000	0.5
Lead	(1)	100	5	1000	0.5
Magnesium	(1)	100	5	1000	0.5
Manganese	(1)	100	5	1000	0.5
Molybdenum	(1)	100	5	1000	0.5
Nickel	(1)	100	5	1000	0.5
Selenium	(1)	100	5	1000	0.5
Silver	(1)	100	5	1000	0.5
Tin	Elemental Stock	1000	0.5	1000	0.5
Thallium	(1)	100	5	1000	0.5
Titanium	(1)	100	5	1000	0.5
Vanadium	(1)	100	5	1000	0.5
Zinc	(1)	100	5	1000	0.5
Hydrochloric Acid	-	-	50	1000	5%
Nitric Acid	-	-	10	1000	1%

(1) Mixed Standard, QCS-26
* 0.5mL 1000ppm Ca added to 5mL QCS-26(100ppm Ca), 1000mL Final Volume

TABLE 3
ICP ICV Standards

ICV1 Solution

Analyte	Source	Source Concentration (ppm)	Aliquot (mL)	Final Volume (mL)	Final Concentration (ppm)
Aluminum	QCP-CICV-1	1000	2.5	500	5.0
Antimony	QCP-CICV-1	1000	1.25	500	2.5
Arsenic	QCP-CICV-3	500	2.5	500	2.5
Barium	QCP-CICV-1	1000	2.5	500	5.0
Beryllium	QCP-CICV-1	25	2.5	500	0.125
Cadmium	QCP-CICV-3	250	2.5	500	1.25
Calcium	QCP-CICV-1	2500	2.5	500	12.5
Chromium	QCP-CICV-1	100	2.5	500	0.5
Cobalt	QCP-CICV-1	250	2.5	500	1.25
Copper	QCP-CICV-1	125	2.5	500	0.625
Iron	QCP-CICV-1	500	2.5	500	2.5
Lead	QCP-CICV-3	500	2.5	500	2.5
Magnesium	QCP-CICV-1	2500	2.5	500	12.5
Manganese	QCP-CICV-1	250	2.5	500	1.25
Molybdenum	Elemental Stock	1000	1.0	500	2.0
Nickel	QCP-CICV-1	250	2.5	500	1.25
Potassium	QCP-CICV-1	2500	2.5	500	12.5
Selenium	QCP-CICV-3	500	2.5	500	2.5
Silver	QCP-CICV-1	125	2.5	500	0.625
Sodium	QCP-CICV-1	2500	2.5	500	12.5
Thallium	QCP-CICV-3	500	2.5	500	2.5
Titanium	Elemental Stock	1000	1.0	500	2.0
Vanadium	QCP-CICV-1	250	2.5	500	1.25
Zinc	QCP-CICV-1	250	2.5	500	1.25
Hydrochloric Acid	-	-	25	500	5%
Nitric Acid	-	-	5	500	1%

TABLE 4
ICP Interference Check Solutions

ICSA Solution

Analyte	Source	Source Concentration (ppm)	Aliquot (mL)	Final Volume (mL)	Final Concentration (ppm)
Aluminum	CLPP-ICS-A	5000	50	500	500
Calcium	CLPP-ICS-A	5000	50	500	500
Iron	CLPP-ICS-A	2000	50	500	200
Magnesium	CLPP-ICS-A	5000	50	500	500
Hydrochloric Acid	-	-	25	500	5%
Nitric Acid	-	-	5	500	1%

ICSAB Solution

Analyte	Source	Source Concentration (ppm)	Aliquot (mL)	Final Volume (mL)	Final Concentration (ppm)
Aluminum	CLPP-ICS-A	5000	50	500	500
Antimony	Elemental Stock	1000	0.5	500	1
Barium	CLPP-ICS-B	50	5	500	0.5
Beryllium	CLPP-ICS-B	50	5	500	0.5
Cadmium	CLPP-ICS-B	100	5	500	1
Calcium	CLPP-ICS-A	5000	50	500	500
Chromium	CLPP-ICS-B	50	5	500	0.5
Cobalt	CLPP-ICS-B	50	5	500	0.5
Copper	CLPP-ICS-B	50	5	500	0.5
Iron	CLPP-ICS-A	2000	50	500	200
Lead	CLPP-ICS-B	100	5	500	1
Magnesium	CLPP-ICS-A	5000	50	500	500
Manganese	CLPP-ICS-B	50	5	500	0.5
Nickel	CLPP-ICS-B	100	5	500	1
Silver	CLPP-ICS-B	100	5	500	1
Vanadium	CLPP-ICS-B	50	5	500	0.5
Zinc	CLPP-ICS-B	100	5	500	1
HCl	-	-	25	500	0.05
HNO3	-	-	5	500	0.01

TABLE 5
IRIS Analytical Wavelengths

Analyte	Wavelength	
Aluminum	237.3	
Antimony	206.8	
Arsenic	189.0	
Barium	233.5	
Beryllium	313.0	
Boron	249.7	
Cadmium	226.5	
Calcium	317.9	
Calcium	211.2	High Line
Chromium	267.7	
Cobalt	228.6	
Copper	324.7	
Iron	259.9	
Iron	271.4	High Line
Lead	220.3	
Lithium	670.7	
Magnesium	279.5	
Magnesium	202.5	High Line
Manganese	257.6	
Manganese	293.9	High Line
Molybdenum	202.0	
Nickel	231.6	
Phosphorus	214.9	
Potassium	766.4	
Selenium	196.0	
Silicon	251.6	
Silver	328.0	
Sodium	589.5	
Strontium	407.7	
Thallium	190.8	
Tin	189.9	
Titanium	323.4	
Vanadium	310.2	
Zinc	206.2	

TABLE 6
ICAP 6500 Analytical Wavelengths

Analyte	Wavelength	
Aluminum	167.0	Low Line
Aluminum	394.4	
Antimony	206.8	
Antimony	217.5	Alternate
Arsenic	189.0	
Barium	455.4	
Beryllium	234.8	
Boron	249.6	
Cadmium	226.5	
Cadmium	214.4	Alternate
Calcium	315.8	
Calcium	393.3	Low Line
Chromium	267.7	
Cobalt	230.7	
Cobalt	228.6	Alternate
Copper	327.3	
Copper	224.7	Alternate
Iron	259.9	
Lead	220.3	
Lithium	670.7	
Magnesium	279.0	High Line
Magnesium	279.5	Low Line
Magnesium	285.2	
Manganese	257.6	
Manganese	260.5	High Line
Molybdenum	202.0	
Nickel	221.6	
Nickel	231.6	Alternate
Phosphorus	214.9	
Phosphorus	178.2	Alternate
Potassium	766.4	
Selenium	196.0	
Silicon	251.6	
Silver	328.0	
Sodium	588.9	Alternate
Sodium	589.5	

TABLE 6
ICAP 6500 Analytical Wavelengths, continued

Analyte	Wavelength	
Strontium	407.7	
Thallium	190.8	
Tin	189.9	
Titanium	336.1	
Vanadium	292.4	
Zinc	206.2	
Zinc	213.8	Alternate

ALS Inc.

Service Request Number(s):

TISSUE HOMOGENIZATION

Laboratory ID	Weight of Sample* (g)	Tare of Jar (g)			#Clams	

Comments:

* The weight of sample Is after homogenization.

Balance ID: Date Balance Checked:

Analyst:	Date:
Reviewed:	Date:

ALS Inc.

Service Request Number(s):
Analysis for:

ALIQUOT DATA

Service Request #		Wet Wt. (g)		Tare Wt. (g)		Matrix

Balance ID:	Date Balance Checked:
Analyst:	Date:
Reviewed:	Date:

Benchsheet

Service Request #:	**K1513658, KQ1514481**	**Run #:**	**475394**
Test:	**Frz Dry**	**Balance ID:**	**K-Balance-45**
Method:	**Frz Dry**		

Pan ID:	Lab Code:	Tare (g)	Wet Wt. (g)	Tare + Dry Wt. (g)	Dry Weight (g)	% Total Solids	RPD
	K1513658-001	16.974	5.277	20.025	3.05	57.8	
	K1513658-002	16.599	10.076	21.985	5.39	53.5	
	K1513658-003	16.581	5.483	20.260	3.68	67.1	
	K1513658-003DUP	16.554	5.570	20.315	3.76	67.5	<1
	K1513658-004	16.545	4.152	19.126	2.58	62.2	
	K1513658-005	16.558	4.955	17.829	1.27	25.7	
	K1513658-006	16.749	10.397	19.163	2.41	23.2	
	K1513658-007	16.506	8.225	18.601	2.10	25.5	
	K1513658-008	17.115	10.170	19.560	2.45	24.0	
	K1513658-009	16.750	10.148	18.881	2.13	21.0	
	K1513658-010	17.371	10.104	19.676	2.31	22.8	
	K1513658-011	17.119	2.733	17.997	0.878	32.1	
	K1513658-012	16.549	1.482	17.067	0.518	35.0	
	K1513658-013	17.087	7.470	19.216	2.13	28.5	
	K1513658-014	17.073	2.124	17.752	0.679	32.0	
	K1513658-015	17.126	2.857	18.241	1.12	39.0	

	FreezeDryer ID	Date In	Time In	Date Out	Time Out	Thermometer ID
FreezeDry		12/9/2015	16:50	12/11/2015	08:35	

Cal EQID	Cal Start Value	Cal End Value	Start Date	Start Time	End Date	End Time

Comments: Data entered 12-14-15 L.J. Reviiewed 12-14-15 K.L.

Preparation Information Benchsheet

Prep Run: 253122 **Prep Workflow**: MetDigTissMS **Status**: Draft **Prep Date**: 01/04/2016 06:10
Team: Metals **Prep Method**: PSEP Metals **Current Step**: Digestion
Analyst: KLINN **Rush/NPDES**: NPDES **Due Date**: 01/08/2016
Hold Date: 01/24/2016

Lab Code	Client ID	Bottle #	Initial Amt	Initial Basis	Final Volume	Spike Amt	Spike ID	Comments
KQ1600008-01	Method Blank							
KQ1600008-02	Lab Control Sample					0.3 mL 0.05 mL 0.05 mL 0.3 mL	84678 86136 86222 86223	
KQ1600008-03	Standard Reference Material					0.3 g	65838	
KQ1600008-04	Standard Reference Material					0.3 g	65841	
K1514654-001	15-01	.03						
K1514654-002	15-02	.03						
K1514654-003	15-03	.03						
K1514654-004	15-04	.03						
K1514654-005	15-05	.03						
K1514654-006	15-06	.03						
K1514654-007	15-07	.03						
K1514654-008	15-08	.03						
K1514654-009	15-09	.03						
K1514654-010	15-10	.03						
K1514654-011	15-11	.03						
K1514654-011: KQ1600008-05	Duplicate	.03						
K1514654-011: KQ1600008-06	Matrix Spike	.03				0.3 mL 0.05 mL 0.05 mL 0.3 mL	84678 86136 86222 86223	
K1514654-012	15-12	.03						
K1514654-013	15-13	.03						
K1514654-014	15-14	.03						
K1514654-015	15-15	.03						
K1514655-021	15-36	.03						
K1514655-022	15-37	.03						
K1514655-023	15-38	.03						
K1514655-024	15-39	.03						

25 Total Samples consisting of 19 Client Samples, 2 Client QC Samples, 4 Batch QC Samples associated with the current Prep Run.

Spiking Solutions

Name	Type	ID	Expires	Name	Type	ID	Expires
K-MET Alt 200.8 spiking solution	Spike	84678	1/12/2016	K-MET SS3	Spike	86222	7/13/2016
K-MET DORM-4	Spike	65838	1/6/2016	K-MET SS4	Spike	86136	5/23/2016
K-MET SS1	Spike	86223	7/21/2016	K-MET TORT-3	Spike	65841	1/6/2016

Preparation Materials

Preparation Hardware / Equipment

Preparation Steps

Step	Started	Finished	By	Assisted By	Training?	Comments
Digestion					N	

Comments

Review

Reviewed by: ______________________ Date: ____________

VISTA ANALYTICAL

QUALITY MANUAL

REVISION 23

EFFECTIVE DATE: DECEMBER 15, 2015

12/15/15

Andrew Patterson — Date

Technical Director

12/15/15

Martha M. Maier — Date

Acting Quality Assurance Manager

12/15/15

Martha M. Maier — Date

President / Laboratory Director

12/15/15

Melanee Schuld — Date

Director of HRMS

Vista Analytical Laboratory
1104 Windfield Way, El Dorado Hills, CA 95762
(916) 673-1520

TABLE OF CONTENTS

1. INTRODUCTION 6
1.1. POLICY 6
2. ORGANIZATION AND FACILITIES 8
2.1. MANAGEMENT RESPONSIBILITIES 8
2.2. QUALITY ASSURANCE MANAGER 9
2.3. LIMS ADMINISTRATOR 5
2.4. APPROVED SIGNATORIES 6
2.5. FACILITIES 11
3. QUALITY SYSTEM 13
3.1. QUALITY DOCUMENTS 13
3.2. USE OF QUALITY DOCUMENTS 13
3.3. DOCUMENT CONTROL 14
3.4. QUALITY ASSURANCE OBJECTIVES AND QUALITY CONTROL PROCEDURES 17
4. PURCHASING 26
4.1. QUALITY MATERIALS AND SERVICES 26
4.2. CONTROL OF QUALITY MATERIALS AND SERVICES 26
4.3. PROCUREMENT DOCUMENTS 26
5. SAMPLE CONTROL 27
5.1. RECEIPT OF MATERIALS 27
5.2. STORAGE, HANDLING, AND DISPOSAL 27
5.3. NOTIFICATION OF PROBLEMS 28
5.4. RECORDS 28
6. TRACEABILITY OF MATERIALS 32
6.1. VERIFICATION OF ITEMS DEVELOPED IN-HOUSE 32
6.2. CONTROL OF LABORATORY SAMPLES 32
6.3. STANDARDS AND REAGENTS TRACEABILITY 32
6.4. QUALITY CONTROL RECORDS 32
6.5. CERTIFICATES OF ANALYSIS 33
6.6. INSTRUMENTS AND EQUIPMENT 33
7. PROCESS CONTROL 34

7.1. INSTRUMENTS AND FACILITIES ... 34
7.2. PERFORMANCE AUDITS ... 34

8. LABORATORY INSTRUMENTATION ... 35

8.1. CALIBRATION STANDARDS AND INSTRUMENTS ... 35
8.2. CALIBRATION RECORDS ... 35

9. QUALITY RECORDS ... 37

9.1. DOCUMENTATION OF QUALITY RECORDS ... 37
9.2. QUALITY AND TECHNICAL RECORDS ... 37
9.3. RECORDS MANAGEMENT AND STORAGE ... 38

10. CORRECTIVE ACTION ... 39

10.1. CAUSES OF NONCONFORMANCE ... 39
10.2. CORRECTIVE ACTION ... 39
10.3. DOCUMENTATION ... 39

11. REPORTS ... 40

11.1. HANDLING AND STORAGE OF REPORTS ... 40
11.2. PACKAGING AND DELIVERY OF REPORTS ... 40
11.3. LABORATORY REPORT FORMAT AND CONTENT ... 41

12. PERFORMANCE AND SYSTEM AUDITS ... 44

12.1. SYSTEM AUDITS ... 44
12.2. MANAGEMENT REVIEWS ... 45
12.3. PERFORMANCE TESTING SAMPLES ... 45
12.4. EXTERNAL AUDITS ... 46
12.5. DATA AUDITS ... 46

13. TRAINING ... 48

13.1. INITIAL ON-SITE TRAINING ... 48
13.2. TRAINING PROGRAMS ... 48
13.3. TRAINING DOCUMENTATION ... 48

14. CLIENT SERVICES ... 50

14.1. ROUTINE SERVICES ... 50
14.2. CONTRACT REVIEW ... 50
14.3. RESPONSES TO CLIENT AUDITS, INQUIRIES, AND COMPLAINTS ... 51

15. STATISTICAL TECHNIQUES 52

15.1. STATISTICAL PROCESS CONTROL PROCEDURES 52

16. SUBCONTRACTING 53

17. DATA INTEGRITY AND ETHICS 54

APPENDIX

Vista Organization Chart

Key Resumes

List of Certifications

Quality Manual Revision Log

FOREWORD

The Quality Manual (QM) describes the Quality System implemented at Vista Analytical Laboratory in El Dorado Hills, California. The policies and procedures outlined in this QM are designed and developed to comply with the established NELAP Standards. It is the intent of Vista to meet or exceed the Quality Assurance/Quality Control (QA/QC) requirements set by ISO 17025, NELAP, the USEPA or other appropriate governmental or private entities to assure that all analytical data generated are scientifically valid, defensible, comparable, and of known acceptable precision and accuracy.

The QM shall be amended to reflect any changes to Vista's capability, location or Quality System. The Quality Assurance Manager is responsible for the maintenance and annual review of the QM.

1. INTRODUCTION

Vista Analytical Laboratory in El Dorado Hills, CA was established in 1990 and is a privately owned California corporation. Vista provides state-of-the-art mass spectrometry services to chemical manufacturers, environmental engineering firms, and the pulp and paper industry as well other industrial and governmental clients. Vista operates with the intent of providing data of the highest quality with responsive service in a short turnaround time.

Vista has an expanding national and international client base attributable to its reliable reputation in performing difficult trace level analyses. Vista's expertise lies in the analysis of semivolatile organic compounds such as Polychlorinated Dioxin/Furans (PCDD/PCDF), Perfluorinated compounds, Polynuclear Aromatic Hydrocarbons (PAHs), Polychlorinated Biphenyls (PCBs), Polychlorinated Naphthalenes (PCNs), Hexachlorobenzene (HCB), Polybrominated Diphenyl Ethers (PBDEs), and Pesticides.

1.1. Policy

It is the policy and commitment of Vista to meet the specific quality requirements and to satisfy the needs of the client, the regulatory authorities or organizations providing recognition, such as the EPA, TNI Standards, ISO/IEC 17025, and/or the Department of Defense (DoD) Quality Systems Manual (QSM), throughout data generation and process operations. A Quality System has been established to achieve this policy. The system encompasses all of the applicable elements of the established TNI Standard. It is Vista's intent to provide full compliance with this Quality System throughout all phases of client services and to ensure that only an acceptable final product is presented to the client.

1.1.1. It is Management's responsibility to instill a commitment to continuously improve the quality standards throughout the company, and to ensure each employee has a clear understanding of the Quality System.

- Quality is the responsibility of all Vista employees.
- All Vista employees must comply with all QA/QC procedures as it pertains to their function.
- All employees shall be accountable for the quality of their individual assignments and functional responsibilities.
- Employees shall be responsible for reporting any non-conformance to Management or the QA Manager.

- The laboratory shall have sufficient personnel with necessary education training, technical knowledge and experience for the assigned positions.

1.1.2. Management is responsible to ensure personnel are free from any commercial, financial, and other undue pressures, which might affect the quality of work.

1.1.3. All Vista employees shall be confident in their independence of judgment and maintain integrity at all times.

2. ORGANIZATION AND FACILITIES

The management staff of Vista consists of a President/Laboratory Director, Technical Director, and QA Manager.

The relationships between technical operations, quality management, and support services of Vista Analytical Laboratory are shown in the organizational chart presented in the Appendix.

2.1. Management Responsibilities

2.1.1. President / Laboratory Director

The President manages the production scheduling and client management for the laboratory, is responsible for final review and interpretation of analytical data and final reports, and also serves as Laboratory Director. Should the Laboratory Director be absent for more than 15 consecutive calendar days, the Quality Assurance Manager or the Technical Director shall be designated acting Laboratory Director. In the absence of the Laboratory Director for more than 65 calendar days, all laboratory Accrediting Bodies shall be notified in writing by the QA Manager or Technical director. The Laboratory Director has the authority and is responsible for:

- Ensuring that the laboratory is current with all regulations that affect operations and the information be communicated to all laboratory personnel by the Technical Director and/or QA Manager;
- Overseeing the develop and implementation of the QA program which assures that all data generated will be scientifically sound, legally defensible and of known quality;
- Maintaining sufficient personnel, equipment and supplies to achieve production goals;
- Ensuring that personnel are free from commercial, financial, and other undue pressures which might affect the quality of their works;
- Being responsible for certifying the qualifications and training of the analysts;

- Reviewing all laboratory SOPs and training materials prior to their implementation and ensures that all approved SOPs are implemented and adhered to; and
- Being responsible for the review and negotiation of client contracts.

2.1.2. Technical Director

The Technical Director is responsible for implementing the scientific procedures and best practices needed for the optimal performance of the laboratory. The Technical Director reports directly to the Laboratory Director. Should the Technical Director be absent for more than 15 consecutive calendar days, the Laboratory Director shall be designated acting Technical Director. The Technical Director has the authority and is responsible for:

- Identifying, resolving, and documents analytical problems encountered in the laboratory;
- Overseeing all method development work ;
- Being involved in the hiring and training of chemists and analysts;
- Providing technical expertise in evaluating potential work prior to bid submittal;
- Supporting QA and Operations with revisions of laboratory SOPs; and
- Providing support to QA for internal and external audit responses.

2.2. Quality Assurance Manager

The QA Manager is responsible for managing the QA activities of the entire laboratory. The QA Manager reports directly to the Laboratory Director and is also the Deputy Laboratory Director. The QA Manager serves as the focal point for QA/QC and is responsible for the oversight and/or review of quality control data. When QA oversight is necessary, the QA Manager functions must be independent from the laboratory operations. Should the QA Manager be absent for more than 15 consecutive calendar days,

Technical Director shall be designated acting QA Manager. The QA Manager has the authority and is responsible for:

- Implementing, maintaining, and improving the quality system;
- Ensuring that all personnel understand their contributions to the quality system;
- Ensuring communication takes place at all levels within the laboratory regarding the effectiveness of the quality system;
- Evaluating the effectiveness of training;
- Review and update the Quality Manual as needed to remain current, at least annually;
- Using available tools, such as internal and external audits and surveillance results, control charts, proficiency testing results, data analysis, corrective and preventive actions, customer feedback, and management reviews in efforts to monitor trends and continually improve the quality system; and
- Notifying management of deficiencies in the quality system and monitors corrective actions.

The QA Manager works with management to ensure that the Vista QM and associated SOPs are followed as written and maintains a position that is free from outside influence in order to evaluate the data and perform all other QA Manager responsibilities objectively.

2.3. LIMS Administrator

A key support staff position is the Laboratory Information Management System (LIMS) Administrator. The LIMS Administrator is responsible for assisting in developing and maintaining all LIMS-related programming and assists with other IT development and maintenance needs. The LIMS Administrator reports directly to the Laboratory Director. Should the LIMS Administrator be absent, internal laboratory support can be provided by the Principal or the Promium Help Desk shall be contacted for assistance. The LIMS Administrator has the authority and is responsible for:

- Implementing and maintaining the LIMS system;

- Providing LIMS system support, technical assistance and training to all laboratory personnel;
- Setting-up and managing LIMS system user accounts and security privileges;
- Managing and documenting change control for the LIMS system;
- Investigating and resolving LIMS system errors, correct user errors;
- Creating documentation of SOPs and training materials;
- Communicating changes, enhancements and modifications to the LIMS system to appropriate laboratory personnel;
- Developing data backup and recovery processes related to the LIMS system; and
- Communicating with IT vendors to resolve issues.

2.4. Approved signatories

2.4.1. Approved signatories include the Laboratory Director, the Technical Director, the QA Manager, the Principal and the Director of HRMS.

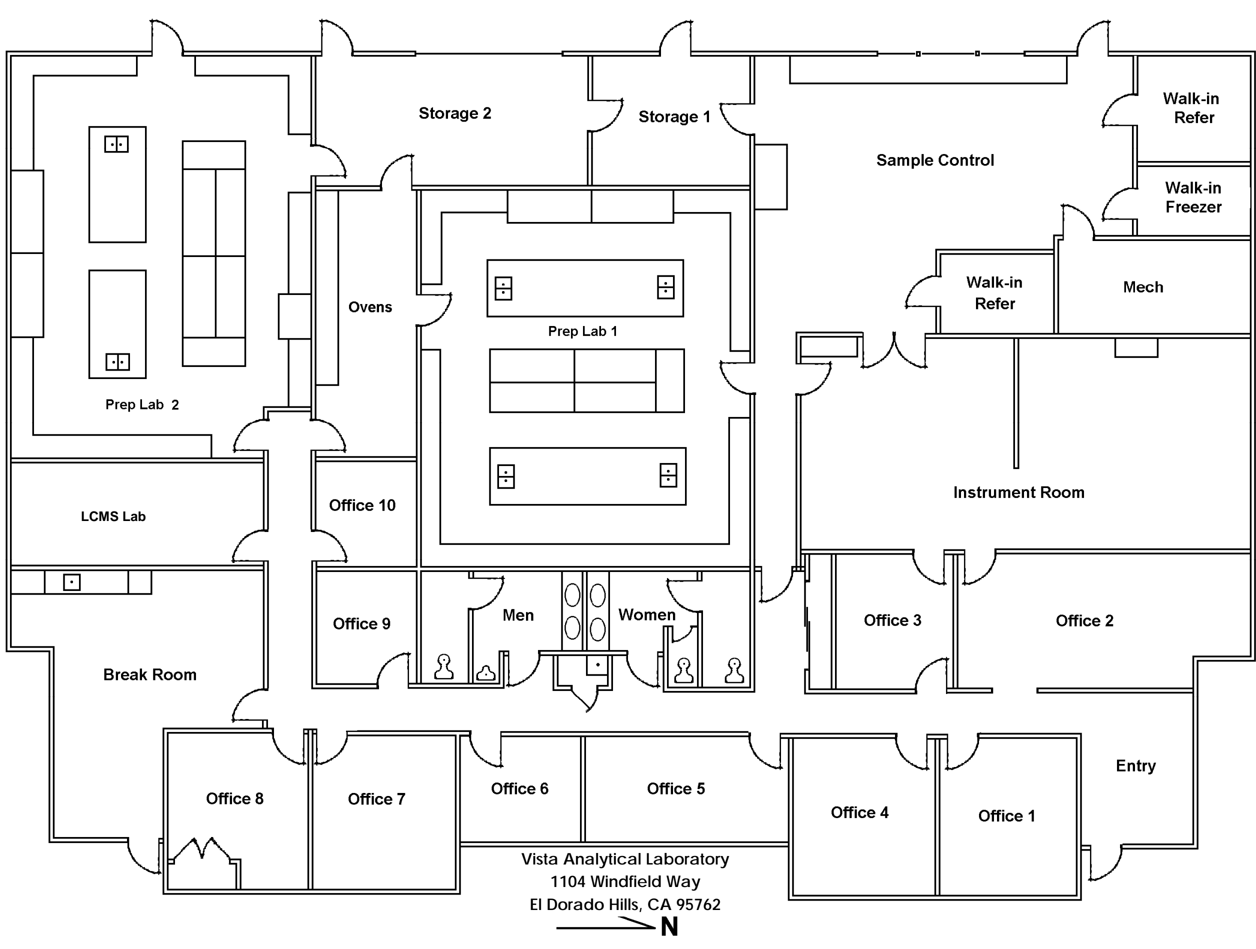
2.5. Facilities

2.5.1. Vista Analytical Laboratory operates from El Dorado Hills, CA. The facility consists of 9,000 square feet.

2.5.2. The facility has been constructed and maintained to ensure that results are not invalidated or do not adversely affect the required accuracy of measurement.

2.5.3. Layout – 1104 Windfield Way, El Dorado Hills, CA

Vista
Analytical Laboratory


Storage 2
Storage 1
Walk-in Refer
Sample Control
Walk-in Freezer
Ovens
Walk-in Refer
Mech
Prep Lab 1
Prep Lab 2
Office 10
Instrument Room
LCMS Lab
Office 9
Men
Women
Office 3
Office 2
Break Room
Entry
Office 8
Office 7
Office 6
Office 5
Office 4
Office 1
Vista Analytical Laboratory
1104 Windfield Way
El Dorado Hills, CA 95762
N

3. QUALITY SYSTEM

The Quality System applies to Vista Analytical Laboratory.

The company's Quality System is designed to comply with the applicable requirements of NELAP Standard and to satisfy the needs of the client or organization providing recognition. All policies, systems, and procedures are documented to assure quality of the data. Personnel shall familiarize themselves with quality documentation and implement the policies and procedures in their work.

Senior Management will review the effectiveness and suitability of the Quality System at least annually. The reviews shall address issues that impact quality. The results of the reviews shall be used to design and implement improvements to the system. The reviews include reports from management and supervisory personnel, recent internal audits, external audits, proficiency testing, client feedback, and corrective action reports. The QA Manager will maintain records of the review meeting, findings, and corrective actions.

3.1. Quality Documents

3.1.1. The Quality System is outlined and documented in the Quality Manual and supporting quality documents. The documented quality system assures that services provided to clients comply with specified quality criteria.

3.1.2. The Quality Manual contains Quality Policies covering the applicable requirements of the NELAP quality standard.

3.1.3. Program specific quality criteria are specified in the Quality Assurance Program Plan (QAPP).

3.1.4. Procedural activities that affect quality are described in more detail in the Standard Operating Procedures (SOPs).

3.2. Use of Quality Documents

3.2.1. Management will review and approve all quality documents prior to issuance. All quality documentation shall be communicated to, understood by, available to, and implemented by the appropriate personnel.

3.2.2. A QAPP or other project-specific requirements submitted by the client will be reviewed to determine whether they are within the scope of the Analytical Procedures. Any discrepancies will be

discussed with the client and documented prior to commencement of the project.

3.2.3. The Quality Manual will be understood and implemented throughout the company. The QAPP and SOPs will be understood and implemented throughout applicable operations.

3.2.4. Quality documents shall be periodically reviewed to ensure continuing suitability and compliance with applicable requirements. The Quality System will be reviewed on an ongoing basis and revised as needed to ensure that it effectively encompasses the company's quality criteria. The QA Manager will maintain the Quality Manual. Revisions to the Quality Manual may be made by replacing individual policies or the entire manual.

3.2.5. Any departures from policies or planned activities that affect quality will be approved by management prior to occurrence.

3.2.6. The QAPP will be maintained by the designated responsible manager, or the QA Manager. Revision may be made to individual sections of the entire plan.

3.2.7. Standard Operating Procedures will be maintained as designated in the specific SOP with revisions being made on an as needed basis.

3.3. Document Control

Standard Operating Procedures (SOPs) or any documents that specify quality requirements or otherwise affect quality are Controlled Documents. All controlled documents will be prepared, issued and revised in accordance with the applicable SOPs. The SOPs are presented in Table 3.1.

3.3.1. Procedures are established to control and maintain the issue, distribution, and revisions of all controlled documentation. The Standard Operating Procedures SOP is SOP 3.

3.3.2. Appropriate documents shall be made available at all locations where operations essential to the effective functioning of the laboratory are performed.

3.3.3. Complete and current copies of the controlled documents shall be made available upon issuance, and obsolete copies will be removed from all points of issue or use. The controlled document copies will be stamped, in red, as an "Official QA Copy".

3.3.4. All original controlled documents are archived by QA Manager.

3.3.5. A master list will be used to ensure that the correct revision of each SOP is available for use, and that obsolete revisions are removed from service. Each controlled document has an associated revision number and effective date to enable tracking of current revisions.

3.3.6. Document changes are reviewed and approved by the appropriate personnel.

3.3.7. Documents are periodically reviewed and, where necessary, revised to ensure continuing suitability and compliance with applicable requirements. The Quality Manual will be revised as needed and reviewed annually.

3.3.8. QA Manager will maintain records of revisions for Controlled Documents and the QAPP.

Table 3.1 List of Standard Operating Procedures

SOP #	Title
1	Laboratory Security
2	Laboratory Audits
3	Standard Operating Procedures
5	Data Collection, Reporting, and Archival
6	Corrective Actions
7	Control Charts
8	Method Detection Limits
9	Manual Integrations
10	Instrument Maintenance Logbooks and Schedule
11	Laboratory Support Instrument Calibration
12	Sample Receiving and Sample Control Procedures
13	Consignment Tracking
14	Bottle Order Preparation
15	Reagents and Standards – Preparation, Handling, and Documentation
16	Sample Preparation and Analysis of PUF Samples for PCDD/PCDFs/PCBs by EPA Method TO-9A
17	Preparation and Shipping of Air Sampling Media for in Field Use
18	Sample Preparation of MM5 Train for Analysis of PAHs by Method CARB 429
19	Sample Preparation of MM5 Train for Analysis of PCDD/PCDFs by Methods CARB 428 and Method 23 or Method 0023A
20	Sample Preparation and Analysis of Sampling Trains and PUFs and PUF/XAD2 for Analysis of PCBs by Modified Method 1668

Table 3.1 List of Standard Operating Procedures	
SOP #	**Title**
21	Sample Preparation and Analysis of Sampling Trains and PUFs and PUF/XAD for Analysis of PBDEs by Modified Method 1614 (Draft)
22	Preparation of Surface Wipes
23	Polychlorinated Dibenzo Dioxin/Furans by USEPA Method 8280A
24	Polychlorinated Dibenzo Dioxin/Furans by USEPA Method 8290
25	Tetrachlorodibenzodioxin in Aqueous Samples by Modified USEPA Method 613
26	Polychlorinated Dibenzo Dioxin/Furans by Method 1613B
28	Sample Analysis of Pentachlorobenzene/Hexachlorobenzene/ Hexachlorobutadiene by Modified Method 1625B
30	Preparation and Analysis of Polybrominated Dibenzo-Dioxin/Furans by Modified EPA Method 8290
31	Analysis of Polychlorinated Biphenyls (PCBs) by Method 1668A/C
32	Preparation and Analysis of Various Matrices for Polybrominated Diphenyl Ethers (PBDE) by EPA Method 1614
33	Preparation and Analysis of Polychlorinated Naphthalenes (PCN) by Modified EPA Method 1668A
34	Preparation and Analysis Using Modified Method 8290 for PCDD/PCDFs and Modified Method 1668A For Coplanar/Mono-Ortho PCBs
35	Glassware Preparation
36	Sample Preparation of MM5 Train for Analysis of PCDDs/PCDFs/PCBs/PAHs by Methods 0023A/1668/CARB 429
37	NCASI 551: Procedures for the Preparation and Isomer Specific Analysis of Pulp and Paper Industry Samples for 2,3,7,8-TCDD And 2,3,7,8-TCDF
38	Computer Systems
39	Preparation and Analysis of Pesticides by Method 1699
40	Preparation and Analysis of Water Using Modified Method 1694 for Pharmaceuticals and Personal Care Products
41	Extraction and Analysis of Hexachloroxanthene by HRMS
42	Computer Software Validation
44	Incremental Subsampling
45	EPA Method 1668 Contract/Government Sites
46	Sample Preparation and Analysis of PUF Samples For PAH By Modified Method TO-13
47	Sample Preparation and Analysis of PAHs Based On A Modified EPA 8270 / 1625 Method
49	Preparation and Analysis for the Determination of Perfluorinated Compounds
50	Preparation and Analysis of Aqueous and Solid Matrices for the Determination of Hexachlorophene
51	Client Custom SOP - Confidential
52	Sample Preparation – DRAFT

Table 3.1 List of Standard Operating Procedures	
SOP #	**Title**
53	Preparation and Analysis Solid Matrices for the Determination of 2,4,6,8-Tetrachlorodibenzothiophene
58	1-4 Dioxane by Modified EPA Method 522
59	Bligh-Dyer Extraction of Lipids
60	Fish Sample Preparation

3.4. Quality Assurance Objectives and Quality Control Procedures

Quality assurance objectives employed at Vista provide routine mechanisms of ongoing control and evaluation of measurement data quality. The quality control (QC) procedures routinely followed evaluate method performance in terms of accuracy and criteria specified by the method or protocol.

3.4.1. Accuracy and precision

Accuracy and precision objectives for HRMS analyses are listed in Table 3.2. Vista's internal quality control procedures include the analysis of method blanks, duplicate samples, laboratory control samples, and matrix spikes.

3.4.2. Definitions

3.4.2.1. **Accuracy:** Accuracy is the nearness of a measurement to the true or theoretical value. Accuracy is assessed by determining recoveries from laboratory control samples, matrix spikes or by comparing values obtained from reference samples.

3.4.2.2. **Analytical Batch:** An analytical batch is a maximum set of 20 samples of the same matrix that are analyzed together using the same method, reagents, and standards. QC results associated with individual analytical batches such as ongoing precision and recovery samples, laboratory control samples, method blanks, matrix spike samples, and duplicate samples are evaluated together to assess data quality. Each batch will be assigned a unique batch number, which will be used to associate sample results with quality control data. All samples associated with a particular batch must be extracted on the same day.

3.4.2.3. **Clean-up Recovery Standard:** A clean-up recovery standard is a reference substance that is an isotopically labeled analyte that is added to the sample extract prior to any clean-up procedures. This standard is used to quantitatively assess losses occurring throughout the clean-up process.

3.4.2.4. **Control/Warning Limits:** Warning and control limits are limits used in laboratory control charts tracking average recovery and relative percent difference. For a Means Chart, typical warning and control levels are ± 2 and ± 3 standard deviations (s) from the central line (i.e., average mean recovery), respectively. Similarly, the warning and control limits for a RPD Chart are usually set at + 2s and + 3s above the mean RPD, respectively.

3.4.2.5. **Detection Limit (DL):** The lowest concentration of an analyte within an environmental matrix that a method or equipment can detect.

3.4.2.6. **Duplicate Sample (DS):** Duplicate samples are two separate aliquots taken from the same source. Duplicate samples are analyzed independently to assess laboratory precision.

3.4.2.7. **Estimated Maximum Possible Concentration (EMPC):** The EMPC is calculated when the response has a S/N in excess of 2.5, but the ion abundance criteria are not met.

3.4.2.8. **Internal Standards (IS):** An internal standard is a reference substance that is an isotopically labeled analyte which is added to the sample prior to extraction and used in the quantitation and identification of native analytes.

3.4.2.9. **Laboratory Control Sample (LCS):** A laboratory control sample is prepared by adding a known quantity of native standards to an interferent free matrix.

3.4.2.10. **Limit of Detection (LOD):** An estimate of the minimum amount of an analyte that can be reliably detected. An LOD is analyte, matrix specific and laboratory

3.4.2.11. **Limit of Quantitation (LOQ):** The minimum concentration of an analyte that can be reported. The LOQ shall be set at or

above the concentration of the lowest initial calibration standard.

3.4.2.12. **Method Blank (MB):** A method blank is a sand, XAD or deionized water preparation that is free of native analyte or interferents that has been prepared and analyzed using the same procedures followed for the rest of the analytical batch. The method blank is used to determine the level of background laboratory contamination, if present.

3.4.2.13. **Method Detection Limit (MDL):** The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero in the matrix tested. MDLs follow 40 CFR Part 136, Appendix B.

3.4.2.14. **Method Quantitation Limit (MQL):** The method quantitation limit is defined as the quantity of native analyte that corresponds to the lowest concentration of the calibration curve. The Method Quantitation Limit is also known as the Reporting Limit.

3.4.2.15. **Matrix Spike (MS/MSD):** A matrix spike sample is prepared by adding a known quantity of native standards to a sample matrix prior to extraction. Matrix spike concentration levels will vary according to the matrix encountered and study objectives.

3.4.2.16. **Native Standard:** A native standard is a reference substance that is a non-isotopically labeled analyte. Native standards are used in conjunction with internal standards to determine response factors and quantitatively assess accuracy.

3.4.2.17. **Ongoing Precision and Recovery (OPR):** A laboratory blank spiked with known quantities of analytes. The OPR is analyzed exactly like a sample. Its purpose is to assure that the results produced by the laboratory remain within the specified limits.

3.4.2.18. **Precision:** Precision is the agreement between a set of replicate measurements. Relative Percent Difference (RPD) is used as the principal measure of precision and is based on the analysis of duplicate quality control samples.

3.4.2.19. **Pre-Spike Standards:** A pre-spike standard is an isotopically labeled analyte that is spiked into an MM5 resin cartridge or PUF prior to sampling. The recoveries of pre-spike standards provide a measure of the air sampling efficiency for native analytes.

3.4.2.20. **Quality Control Sample:** Quality control samples are analyzed to access the various aspects of the analytical process in order to monitor quality within the laboratory. The most frequently used QC samples are method blanks, duplicates, matrix spikes, matrix spike duplicates and LCS pairs.

3.4.2.21. **Recovery Standard:** A recovery standard is a reference substance that is an isotopically labeled analyte which is added to the sample extract after clean-up and prior to injection. This standard is used to quantitatively assess the absolute recoveries of the internal and clean-up recovery standards.

3.4.2.22. **Resin QC:** A resin QC is an XAD-2 preparation that is analyzed to assess possible background contamination originating from the resin.

3.4.2.23. **Reporting Limit:** See Method Quantitation Limit.

3.4.2.24. **Signal-to-Noise Ratio:** Dimensionless measure of the relative strength of an analytic signal to the average strength of background instrument noise.

3.4.3. Calculations

3.4.3.1. Percent Recovery (%R): Percent recovery is a measure of accuracy and is calculated according to the following expression:

$$\%R = \frac{(Amount\ \ Found)}{(Amount\ \ Spiked)}\ X\ 100$$

3.4.3.2. Relative Percent Difference (RPD): Percent Recovery (%R) from duplicate LCS or matrix spike analyses are used to calculate RPD using the following expression:

$$RPD = \frac{|\%\ R_1 - \%\ R_2\,|}{\left(\frac{(\%\ R_1 + \%\ R_2\,)}{2}\right)} \times 100$$

3.4.3.3. Similarly, the RPD for duplicate sample analyses, is calculated using the sample concentration (C), as follows:

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

3.4.3.4. Relative Standard Deviation (RSD): Also known as the coefficient of variation.

$$RSD = \frac{SD}{Mean} \times 100$$

3.4.4. Quality Control Procedures

3.4.4.1. Method Blanks:

A method blank is run with each analytical batch or 20 samples (whichever is less) per method and matrix type.

For any method involving the determination of native 2,3,7,8-substituted isomers, the levels measured in the method blank must be less than the MQL, or ten times lower than the concentration found in samples within the analytical batch, unless otherwise mandated by project or client requirements.

All samples within an analytical batch are re-extracted and analyzed if the method blank associated with that batch does not meet internal standard recovery criteria or contamination limits specified above. Otherwise, the data is qualified appropriately.

3.4.4.2. Ongoing Precision and Recovery/Laboratory Control Samples

A single OPR or a pair of LCS is analyzed with every batch of clients' samples.

If the recoveries of the OPR/LCS do not fall within the acceptable control range for accuracy or if the RPD falls outside the specified precision limit established by the method, then all samples within the analytical batch are re-extracted and analyzed. If the OPR/LCS recoveries are not within the acceptable control range and the analytes are not detected in the samples, then the Analyst, Project Manager

and/or QA Manager will use professional judgment to assess the effect on the sample data. A decision will be made to re-extract the affected batch or qualify the data that is reported.

3.4.4.3. Matrix Spike and Duplicate Sample Analyses

An MS, MS/MSD, or duplicates are analyzed upon client request, method requirements, or at the discretion of the Project Manager.

If the RPD from duplicate samples or MS/MSD exceeds criteria in Table 3.2, corrective action will be taken as directed in the method, unless there is demonstrated matrix effect.

3.4.5. Quality Control Charts

Quality control data are calculated as needed by the QA Manager and distributed to the Laboratory Director for review if necessary. A set of current QC control charts is maintained in QA Manager. Original copies of the QC charts and any associated tabular data are stored in QA Manager. QC control charts are available upon written request of clients or regulatory agencies or may be reviewed during facility audits.

Table 3.2 Accuracy and Precision Objectives

DATA ACCEPTANCE/REJECTION CRITERIA Precision/Accuracy and QC Requirements					
METHOD	**Method Blank**	**Internal Standard Recovery Limits**	**OPR Recovery Limits**	**Duplicate Sample Analysis**	**MS/MSD**
EPA 8280/ 8280A	One/extraction batch ≤ML, report in ng/g or ng/L ≤5% regulatory limit or amount in sample	25-150%	70-130%	By client request RPD ≤50%	By client request RPD ≤50%
EPA 8290/ 0023A	One/extraction batch Run between calibration std and 1st sample	40-135%	70-130%	By client request RPD≤25%	By client request RPD≤20%
EPA 23	One/extraction batch Run between calibration std and 1st sample	Surrogate 70-130% IS Tetra-Hexa 40-130% Hepta-Octa 25-130%	70-130%	Not applicable	Not applicable
T0-9A	One/extraction batch Run between calibration std and 1st sample	Surrogate 70-130% IS Tetra-Hexa 50-120% Hepta-Octa 40-120%	70-130%	Not applicable	Not applicable
EPA 613	One/extraction batch	25-150%	70-130%	By client request RPD ≤25%	10% of samples or 1/month RPD ≤20%

Table 3.2 Accuracy and Precision Objectives

DATA ACCEPTANCE/REJECTION CRITERIA Precision/Accuracy and QC Requirements					
METHOD	**Method Blank**	**Internal Standard Recovery Limits**	**OPR Recovery Limits**	**Duplicate Sample Analysis**	**MS/MSD**
EPA 1613A EPA 1613B	One/extraction batch after OPR Must be ≤ 1/3 of minimum level (10 pg/L or regulatory compliance level whichever is greater).	Method Tables 7 and Table 7A	Method Tables 6 and 6A	By client request RPD ≤25%	By client request RPD ≤20%
EPA 1614	Method Blank ≤ML; ≤1/3 regulatory limit or amount in sample	Tetra-Hepta: 30-140% Tetra-Hepta: 25-150% Samples Deca: 20-200%	Tetra-Hepta: 50-150% Deca: 40-200%	By client request RPD ≤50%	By client request RPD ≤50%
EPA Method 1625	One/extraction batch	Method Table 8	Method Table 8	By client request RPD ≤25%	By client request RPD ≤20%
EPA 1668A/C PCBs	One/extraction batch ≤ 10X amount in sample	Samples 25-150% OPR Recovery per SOP 31	OPR Recovery per SOP 31	By client request RPD ≤50%	By client request RPD ≤20%
Modified 1668A (PCN)	One/extraction batch	30-140% 25-150% Samples	50-150%	By client request RPD ≤25%	By client request RPD ≤20%
EPA Method 1699	One/extraction batch	Method Table 5	Method Table 5	By client request RPD ≤25%	By client request RPD ≤20%
NCASI 551	Method Blank IS & RS Recovery >40%	40-120% or S/N >10:1, if %R is >20% "H" Qualifier	70-130%	By client request RPD ≤25%	By client request RPD ≤20%

Table 3.2 Accuracy and Precision Objectives

DATA ACCEPTANCE/REJECTION CRITERIA Precision/Accuracy and QC Requirements					
METHOD	**Method Blank**	**Internal Standard Recovery Limits**	**OPR Recovery Limits**	**Duplicate Sample Analysis**	**MS/MSD**
CARB 428 PCB's	One/extraction batch ≤ 10X amount in sample	40-120% or S/N >10:1	60-140%	Not applicable	Not applicable
CARB 428 Dioxin/Furan	One/extraction batch Must be ≤ ML	Surrogates= 60-140% IS= 40-120% or S/N >10:1	60-140%	Not applicable	Not applicable
CARB 429	One/extraction batch ≤ 5% amount in sample	50-150% or S/N >10:1 "H" Qualifier	Field Spikes 50-150%	By client request RPD ≤25%	Not applicable
Modified Method 1694	One/extraction batch	Method Table 12	Method Table 12	By client request RPD ≤25%	By client request RPD ≤20%
EPA Method 537	One/extraction batch	Samples: 5–153% OPR: 5-168%	50-120%	By client request RPD ≤50%	By client request RPD ≤20%

4. PURCHASING

4.1. Quality Materials and Services

Materials and services that affect the quality of the company's services will be designated as quality material and services. Purchases shall be made only from approved suppliers (based on historical experience or quality certifications).

4.2. Control of Quality Materials and Services

Quality Materials and Services and, where appropriate, potential suppliers' Quality Systems, shall be evaluated to ensure that specified quality requirements are met. Any purchased equipment and consumable materials, whenever possible, shall be inspected, calibrated, or otherwise verified as complying with any standard specifications relevant to the calibrations or tests concerned prior to use. Records of actions taken to check compliance shall be maintained.

4.3. Procurement Documents

Procurement documents will clearly specify all information and requirements necessary to ensure that the correct materials and services are purchased and received. Any discrepancies between request and contracts shall be resolved before any work commences. Request and contracts shall be reviewed to determine the effect of financial, legal and time schedule aspects. Any amendments to the request or contract after work has commenced shall require another review process.

5. SAMPLE CONTROL

Samples and other material received from clients shall be handled and maintained in accordance with laboratory SOPs.

5.1. Receipt of Materials

5.1.1. Samples and materials received from clients, and any other materials received from an outside source in the regular course of business, will be inspected upon receipt to insure that they meet specified quality requirements. All conditions, including any abnormalities or departures from standard conditions, shall be recorded according to SOPs.

5.1.2. Immediately after inspection samples will be logged into the laboratory computer system. A unique laboratory identification number is assigned to each sample at the time of login. This unique laboratory identification allows the sample to be controlled and tracked during storage, handling, and disposal.

5.1.3. Other materials will be properly identified upon verification that they meet specified quality requirements.

5.2. Storage, Handling, and Disposal

5.2.1. Samples and materials received from clients will be stored and handled in a manner that ensures the integrity and quality characteristics are maintained.

5.2.1.1. All samples are stored away from all standards; reagents, food, or any other potentially contaminating sources in such a manner as to prevent cross contamination.

5.2.2. Samples, sample extracts, and any other sample preparation fractions are stored according to the conditions specified by preservation protocols or according to the appropriate test method.

5.2.3. Samples are stored for a minimum of 90 days. If the client provides any relevant instructions regarding sample storage, then the samples are stored according to the client's request.

5.2.4. Samples will be disposed of in a manner that:

- Protects the environment

- Complies with applicable regulatory requirements
- Complies with any project specific requirements

5.2.5. Excess materials will either be returned to the client, or disposed of in accordance with the applicable SOPs.

5.2.6. Access to laboratories and sample storage facilities will be restricted to authorized personnel to further ensure that sample integrity is maintained.

5.2.7. Ambient conditions will be monitored in storage facilities and laboratories where control of those conditions is necessary to maintain the integrity of the sample.

5.3. Notification of Problems

Clients or suppliers will be notified if the integrity of their samples or materials is jeopardized either upon receipt or while in the possession of the company.

5.4. Records

Records of all procedures to which a sample is subjected to while in the laboratory shall be maintained. Chain of custody records shall establish an intact, continuous record of the physical possession, storage, and disposal of all samples.

Table 5 Sample Containers, Preservatives and Maximum Holding Times

Method	Sample Type	Maximum Holding Times	Container Type	Preservation
EPA Method 8280	Aqueous	Extraction: 30 days (1) Analysis: 45 days (2)	Amber Glass	< 6 °C
	Solid	Extraction: 30 days (1) Analysis: 45 days (2)	Glass Container	≤ 6 °C
EPA Method 8290	Aqueous	Extraction: 30 days (1) Analysis: 45 days (2)	Amber Glass	< 6 °C dark
	Solid	Extraction: 30 days (1) Analysis: 45 days (2)	Glass Container	< 6 °C dark
	Fish/Tissue	Extraction: 30 days (1) Analysis: 45 days (1)	Glass Container	< -10 °C dark
EPA Method 1613B	Aqueous	Extraction: 1 year (1) Analysis: 1 year (2)	AGB	0 – 6 °C (3) dark
	Solid Fish/Tissue	Extraction: 1 year (1) Analysis: 1 year (2)	AGJ	< 6 °C dark (6) < -10 °C dark (7)
EPA Method 1614	Aqueous (3)	Extraction: 1 year (1) Analysis: 1 year (2)	AGB	0 – 6 °C (3) dark
	Solid Fish/Tissue	Extraction: 1 year (1) Analysis: 1 year (2)	AGJ	< 6 °C dark < -10 °C dark (7)
Modified EPA Method 1625	All samples	Extraction: 7 days (1) Analysis: 40 days (2)	Amber Glass Containers	0 – 6 °C (3) dark
EPA Method 1668A/C	Aqueous	Extraction: 1 year (1) Analysis: 1 year (2)	AGB	0 – 6 °C (3) dark
	Solid Fish/Tissue	Extraction: 1 year (1) Analysis: 1 year (2)	AGJ	< 6 °C dark (6) < -10 °C dark (7)
EPA Method 1694	Aqueous	Extraction: 7 days (1) Analysis: 40 days (2)	Amber Glass Containers	< 6 °C dark

Table 5 Sample Containers, Preservatives and Maximum Holding Times

Method	Sample Type	Maximum Holding Times	Container Type	Preservation
EPA Method 1694	Solid	Extraction: 7 days (1) Analysis: 40 days (2)	Amber Glass Containers	< 6 °C dark < -10 °C dark (7)
EPA Method 1699	Aqueous (3)	Extraction: 7 days (1) Analysis: 40 days (2)	AGB	< 6 °C dark
	Solid Fish/Tissue	Extract/Analyze: 1 year	AGJ	< 6 °C dark < -10 °C dark (7)
Modified EPA Method 537	Aqueous Solid	Extraction: 14 days (1) Analysis: 28 days (2)	Polypropylene or HDPE	≤ 10 °C Receipt (8) < 6 °C dark (8)
EPA Method 613	Aqueous	Extraction: 7 days (1) Analysis: 40 days (2)	AGB	6 °C (3) dark
EPA Method 23	MM5 Train	Extraction: 30 days (1) Analysis: 45 days (2) Trap Prep: 30 days	Train and/or AGB	Adsorbents on ice (6)
EPA Method T0-9A (4)	PUF	Extraction: 7 days (1) Analysis: 40 days (2) PUF Prep: 30 days	PUF	< 6 °C
CARB Method 428 (4)	MM5 Train	Extraction: 30 days (1) Analysis: 45 days (2) Trap Prep: 30 days	Train and/or AGB	0 – 6 °C dark (5)
CARB Method 429	MM5 Train	Extraction: 21 days (1) Analysis: 40 days (2) Resin QC Date: 21 days	Train and/or AGB	< 6 °C dark
NCASI 551 (4)	All Samples	NA	NA	< 6 °C
PCN	Aqueous	Extraction: 1 year (1) Analysis: 1 year (2)	AGB	0 – 6 °C (3) dark < -10 °C dark (7)
	Solid Fish/Tissue	Extraction: 1 year (1) Analysis: 1 year (2)	AGJ	< -10 °C dark (7)

(1) From collection
(2) From extraction
(3) If residual chlorine is present sodium thiosulfate is added as per the method
(4) Holding times set by Vista Analytical Laboratory

(5) Recommended by Vista Analytical Laboratory
(6) From collection until laboratory receipt
(7) Solid matrices not extracted within 21 days will be stored <-10 ^{o}C
(8) Preserved in the field with Trizma

6. TRACEABILITY OF MATERIALS

Procedures for identifying, controlling, and tracking items purchased from vendors, items developed in-house, samples received from clients, and client reports are detailed in SOPs.

Purchased materials and supplies will be checked to confirm that they meet quality specifications.

6.1. Verification of Items Developed In-house

6.1.1. Items developed in-house such as computer programs, equipment, and procedures, will be tested to verify that they meet the intended objectives. Test records will be maintained so that client reports can be traced to specific items.

6.2. Control of Laboratory Samples

6.2.1. Each sample will be assigned a unique laboratory ID number that will be used to track the sample as it is processed through the laboratory. This unique ID number is also used to associate the analytical results with the sample.

6.2.2. Samples will be batched for analysis. Each batch will be assigned a unique batch number that will be used to associate sample results with quality control data.

6.3. Standards and Reagents Traceability

6.3.1. Documented procedures shall exist for the purchase, reception, and storage of consumable materials used for the technical operations within the laboratory. Certificate of Analysis records for all standards shall be retained by QA Manager. Reagent and standard preparation documentation shall indicate traceability to purchased stock or neat compounds, reference to method of preparation, date of preparation, expiration date, and preparer's initials.

6.4. Quality Control Records

Records will be maintained to trace calibration standards and instrument calibration data to NIST or USEPA standards as appropriate. If NIST or USEPA standards are not available other standards will be used which are acceptable to specific project requirements. In compliance to A2LA, external calibrations

and verifications, these must be recorded in a calibration certificate or report and must include an endorsement by the recognized Accreditation Body's symbol (or otherwise makes reference to accredited status by a specific, recognized accreditation body); and an indication of the type of entity that is accredited (e.g., via an accreditation certificate number, inclusion of "calibration laboratory" with the symbol, etc.); and the measurement uncertainty.

In-house calibrations must be performed as per SOP 11.

6.4.1. Each instrument will be assigned a unique ID number. Records will be maintained to document the performance and maintenance of each instrument.

6.4.2. Records will be maintained to identify the individuals responsible for preparing calibration standards, analyzing samples, and reviewing analytical data.

6.4.3. Quality control records will be maintained to demonstrate that individual test procedures have been verified. Individual analytical results will be traceable to these quality control records.

6.5. Certificates of Analysis

6.5.1. All client reports and certificate of Analysis will be uniquely identified. Where appropriate, contract or purchase order numbers will be referenced on client reports. When requested, test procedures will be referenced on Certificates of Analysis.

6.6. Instruments and Equipment

6.6.1. All measuring operations and testing equipment effecting accuracy or validity of tests shall be calibrated and verified before being put into service and on a continuing basis.

7. PROCESS CONTROL

Analytical procedures and other processes that directly affect the quality of services will be conducted under controlled conditions using SOPs that are written at a level of detail appropriate to the complexity of the process.

Personnel will be properly trained before being given responsibility for an analytical procedure or other process that directly affects the quality of a service.

7.1. Instruments and Facilities

7.1.1. Analytical instruments will be maintained in a condition, which will ensure that they are able to meet specified operating conditions.

7.1.2. Laboratory facilities will be designed to meet specific operating conditions, and maintained in a condition, which will ensure that the operating conditions are consistently met.

7.1.3. Results of quality control checks will be recorded.

7.2. Performance Audits

7.2.1. The laboratory shall ensure the quality of results provided to clients by implementing checks to monitor the quality of the laboratories analytical activities.

7.2.1.1. Internal QC procedures.

7.2.1.2. Participation in proficiency testing or other interlaboratory comparisons.

7.2.1.3. Use of certified reference materials.

8. LABORATORY INSTRUMENTATION

All laboratory instrumentation and testing equipment used by the company will be maintained and calibrated in accordance with SOPs to verify proper operation. Table 8 details a list of current laboratory instrumentation for analysis.

Instrumentation will be placed into service dependent upon the capability of achieving the accuracy required and shall comply with relevant specifications to the instrument.

Authorized personnel shall operate laboratory instrumentation and testing equipment.

Instrumentation and equipment will be used in a manner that ensures that measurement uncertainty is known and consistent with specified quality requirements.

Methods and intervals of calibration specified for each instrument will be based on the individual operating characteristics of the instrument and the quality requirements of the analytical procedure.

8.1. Calibration Standards and Instruments

8.1.1. Calibration and verification procedures will use standards and instruments, whenever applicable, that are traceable to recognized national or international standards. Where traceability to national standards does not exist, the basis for the calibration will be documented.

8.1.2. Prior to use, laboratory instrumentation and testing equipment shall be calibrated or calibration verified and checked to establish that it meets the laboratory's specification requirements and complies with the relevant standard specifications.

8.1.3. Where applicable, reference standards and instrumentation will be checked periodically between calibration and verification procedures.

8.2. Calibration Records

8.2.1. Except for procedures requiring reanalysis, calibration prior to each analysis and previous calibration data will be reviewed when an instrument is out of calibration to determine whether or not the analytical results are acceptable.

8.2.2. Instruments that are unable to maintain calibration or not operating properly will be taken out of service. Instruments will

not be placed back into service until they have been repaired and verified to be operating properly.

8.2.3. The records for each test or calibration shall contain sufficient information to indicate whether specified quality or process parameters are achieved. Each instrument will be assigned a unique ID number. Records will be maintained to document the performance and maintenance of each instrument.

Table 8 Instrument List

Name	ID	Acquired
Waters Autospec Ultima High Resolution Mass Spectrometer	VG-7	2001
Waters Autospec Ultima High Resolution Mass Spectrometer	VG-8	2001
Waters Autospec Ultima High Resolution Mass Spectrometer	VG-9	2004
Waters Autospec Premier High Resolution Mass Spectrometer	VG-10	2008
Waters Autospec Ultima High Resolution Mass Spectrometer	VG-11	2015
Waters Micromass Quattro Premier XE Tandem Quadrupole Mass Spectrometer (QT™Q) MS/MS	Q-1	2008
AB SCIEX API 4000 Triple Quadrupole Mass Spectrometer	Q-2	2015

9. QUALITY RECORDS

Procedures for identification, collection, indexing, access, filing, storage, maintenance and disposal of quality and technical records shall be in accordance with SOPs. Quality records shall include internal audits and management reviews as well as records of corrective actions and preventative actions. Technical records include original observations, calculations and derived data, calibration records and a copy of final report.

9.1. Documentation of Quality Records

9.1.1. Quality records will be generated in accordance with the specification of applicable procedures, programs, and contracts. These records will be maintained to demonstrate that specified quality requirements are met, and that the Quality System is functioning successfully.

9.1.2. Quality records of subcontractor services which affect the quality of the company's services will be required to meet the conditions of this section.

9.1.3. Documents will be clean and legible, and will reference back to the specific activities or procedures to which they apply.

9.2. Quality and Technical Records

9.2.1. Quality and technical records shall be conducted in accordance with SOPs.

9.2.2. History of all samples must be traceable and readily understood through the documentation.

9.2.3. Instruments may not be used in analytical procedures unless maintenance and calibration records indicate that specified quality requirements are achieved. The results of instrument maintenance and calibration inspections will be clearly identified either on the instrument or in maintenance and calibration documents

9.2.4. Work must pass specified quality requirements before it will be released to the succeeding step in the process or, finally, to the clients. The results of quality control checks on work processes will be documented in a manner that clearly indicates the status of the work to the responsible personnel.

9.2.5. Individuals authorized to conduct instrument maintenance and calibration procedures and quality control checks will be identified in the documentation.

9.3. Records Management and Storage

9.3.1. The laboratory shall retain on record all original observations, calculations and derived data, calibration records and a copy of report for a minimum of five years. This applies to both manual and electronic data.

- Individual records will be reviewed and noted if storage requirements longer than five years are required based on client, project or state specific regulations.

9.3.2. Records must provide sufficient information for an adequate audit trail that produces the same results for the sample analytical data. The sample from receipt to analysis must be readily understood through documentation.

9.3.3. All records shall be safely stored, held secure and in confidence to the clients. NELAP related records shall be available to the accrediting authority

9.3.4. All records shall be archived and protected from fire, theft, loss, and environmental deterioration. Any access to archived information shall be documented in the Archive Access Log

9.3.5. Quality documents will be stored in a manner that protects them from loss, damage, unauthorized alterations, and held in confidence to the client.

9.3.6. Documents will be indexed and filed in a manner that allows them to be readily retrieved. Clients will be provided access to records that document the quality of work done for them.

9.3.7. If the laboratory were to transfer ownership, the procedures on handling documents would remain the same. The transfer would ensure that the procedures in place prior to transfer show little significant change for client ease into transition.

9.3.8. If the laboratory were to go out of business, the laboratory would contact the client with the option of how they would like to proceed with their data. All data would be handled according to client or Vista approval for proper destruction or safekeeping.

10. CORRECTIVE ACTION

Nonconforming conditions are when any aspect of the quality system or technical operations does not conform to procedures or to client requirements. Nonconforming conditions have an adverse effect to the quality specifications and are handled in accordance with SOPs. If a nonconformance occurs, where necessary, the client shall be notified.

The applicable SOPs provide instructions for determining the root cause of nonconforming conditions, designing and implementing corrective action, and evaluating the effectiveness of the corrective action.

10.1. Causes of Nonconformance

Procedures will be implemented to determine the root cause of nonconformance conditions, and the corrective action will be designed to eliminate the root cause and prevent reoccurrence.

10.2. Corrective Action

10.2.1. Corrective actions are taken immediately, together with any decision about the acceptability of the nonconforming work. When nonconforming work is identified, the Laboratory Director and Quality Assurance Manger work together to investigate the source of the nonconformance. Either manager may halt work and withholding test reports, as necessary. Work shall not resume until the Laboratory Director has authorized the resumption of work.

10.2.2. Procedures that result in or allow nonconformance conditions will be revised. If necessary, new procedures will be written.

10.2.3. The revised or new procedures will be implemented and evaluated to ensure that the corrective action steps taken effectively eliminate the nonconformance conditions.

10.3. Documentation

10.3.1. Results of root cause analyses and corrective action steps implemented to eliminate nonconformance conditions will be documented and reported to appropriate levels of management in accordance with laboratory SOPs. Records of corrective actions are maintained by QA Manager.

11. REPORTS

Handling, storage, packaging, and, when applicable, delivery of client reports will be conducted in accordance with SOPs to ensure that specified quality requirements and confidentiality of the reports are maintained. The reports shall include all the information requested by the client or required by the method used. Reports may also include electronic data. Electronic data will follow the same criteria as reports. Any information not reported to the client shall be readily available in the laboratory.

11.1. Handling and Storage of Reports

11.1.1. Reports and files will be handled in a manner that ensures that client confidentiality is maintained, and that the reports are protected from loss, damage, or unauthorized alterations.

11.1.2. All reports and files will be coded for ease of identification and retrieval.

11.1.3. File cabinets and storage rooms will be designed to protect filed copies of reports from loss, damage, or unauthorized alterations.

11.1.4. Computer files will be backed up to electronic storage media and stored in a manner that protects them from loss, damage, or unauthorized personnel.

11.1.5. The condition of reports and files in storage will be periodically evaluated to ensure that there is no deterioration, and that the reports remain readily accessible to authorized personnel.

11.1.6. NELAP related records shall be made available to the accrediting authority, and shall be maintained for a minimum of five years.

- Individual records will be reviewed and noted if storage requirements longer than five years are required based on client, project or state specific regulations.

11.2. Packaging and Delivery of Reports

11.2.1. Client reports will be inspected prior to delivery to ensure that they meet specified quality requirements. Then the reports will be packaged for delivery to the client in a manner that ensures protection while in transit.

11.2.2. When required by specific contractual stipulations, the company will assume responsibility for protection of client reports while en route to the client.

11.3. Laboratory Report Format and Content

All laboratory reports shall include, at least, the following information:

11.3.1. A title, indicating the nature of the document (i.e. Test Report, Laboratory Results);

11.3.2. Name and address of the laboratory, location analysis was conducted if different from the address of the laboratory, and a phone number with name of a contact person;

11.3.3. Unique identification of the report and of each page, and the total number of pages. It must be clear that discrete pages are associated with a specific report, and that the report contains a specified number of pages;

11.3.4. NELAP accredited logo and a statement certifying that the report meets all requirements of NELAP and cannot be reproduced;

11.3.5. Name and address of client, where appropriate and project name if applicable;

11.3.6. Description and unambiguous identification of the tested sample including the client identification code;

11.3.7. Identification of test results derived from any sample that did not meet NELAP sample acceptance requirements such as improper container, holding time, or temperature;

11.3.8. Date of receipt of sample, date and time of sample collection, date(s) of performance test, and time of sample preparation and/or analysis if the required holding time for either activity is less than or equal to 72 hours;

11.3.9. Identification of the test method used, or unambiguous description of any non-standard method used;

11.3.10. If the laboratory collected the sample, reference to sampling procedure;

11.3.11. Any deviations from, additions to or exclusions from the test method, and any non-standard conditions that may have affected

the quality of results, and including the use and definitions of data qualifiers

11.3.12. Measurements, examinations and derived results, supported by tables, graphs, sketches and photographs as appropriate, and any failures identified; identify whether data are calculated on a dry weight or wet weight basis, identify the reporting units

11.3.13. A signature and title, or an equivalent electronic identification of the person(s) accepting responsibility for the content of the report, and date of issue;

11.3.14. Clear identification of all test data provided by outside sources, such as subcontracted laboratories, clients, etc.

- The original report from subcontracted laboratories should be included in the client laboratory report.

11.3.15. Reports shall, when required, include a statement of compliance/non-compliance with requirements and/or specifications, including identification or test results derived from any sample that did not meet NELAP sample acceptance requirements such as improper container, holding time, or temperature.

11.3.16. Additional information, which may be required by specific methods, clients or groups of clients.

11.3.17. After issuance of the report, the report remains unchanged.

11.3.18. Any report that requires amending must clearly state that the report has been revised. The amended report must also meet the requirements set forth within the most recent TNI standard.

DATA QUALIFIERS & ABBREVIATIONS

B	This compound was also detected in the method blank.
D	Dilution
E	The associated compound concentration exceeded the calibration range of the instrument.
H	Recovery was outside laboratory acceptance limits.
I	Chemical Interference
J	The amount detected is below the Lower Calibration Limit of the instrument.
P	The amount reported is the maximum possible concentration due to possible chlorinated diphenylether interference.
*	See Case Narrative
Conc.	Concentration
DL	Sample-specific estimated detection limit
MDL	Method Detection Limit as determined by 40 CFR 136, Appendix B
EMPC	Estimated Maximum Possible Concentration
M	Estimated Maximum Possible Concentration (CA Region 2)
NA	Not applicable
RL	Reporting Limit – concentrations that correspond to low calibration point
ND	Not Detected
TEQ	Toxic Equivalency

Unless otherwise noted, solid sample results are reported in dry weight. Tissue samples are reported in wet weight.

12. PERFORMANCE AND SYSTEM AUDITS

Performance, System, and External audits are conducted to verify conformance with Vista's quality assurance program, to determine the effectiveness of the QA program, and to continually improve Vista's data quality.

12.1. System Audits

12.1.1. Internal audits (facility audits) of activities affecting the quality of the company's services will be conducted by the QA Department on a regular schedule in accordance with laboratory SOPs. Internal audits are performed periodically and at least annually. The QA Manager is trained and qualified as an auditor who, wherever possible, is independent of the activities being audited. Other QA department members are trained prior to conducting internal audits. Internal audits verify that operations continue to comply with the requirements of the quality system and NELAP standards.

12.1.2. It is the responsibility of the QA Manager to plan and organize audits based on a predetermined schedule or as requested by management.

12.1.3. SOPs and checklists will be used to focus the internal audit on specific activities of the area to be audited.

12.1.4. Personnel will not be allowed to audit activities for which they are responsible or in which they are directly involved, unless it is demonstrated that an effective, nonbiased, audit can be performed.

12.1.5. Results of internal audits will be documented by the audit team and submitted to the manager(s) in charge of the audited area and the management of the QA Manager.

12.1.6. Appropriate corrective action steps will be promptly taken to address any deficiencies or areas for improvement identified by the internal audit. Laboratory management shall ensure that these actions are within the agreed time frame.

12.1.7. If client work was affected by any found deficiencies, laboratory management will notify the client, in writing, within five business days.

12.1.8. All records of internal facility inspections and responses will be maintained by the QA Manager.

12.2. Management Reviews

12.2.1. Management shall review the quality system annually to evaluate its continuing suitability and effectiveness, make any necessary changes or improvements, and follow up on previous recommendations.

12.2.2. The review includes reports from managerial and supervisory personnel, the outcome of recent internal audits, assessments by external bodies, the results of interlaboratory comparisons or proficiency tests, any changes in the volume and type of work undertaken, feedback from clients, corrective and preventative actions, in-depth monitoring of data integrity, and other relevant factors.

12.2.3. The management review procedure including scheduling is available on the Laboratory network.

12.3. Performance Testing Samples

12.3.1. Performance testing samples are conducted as single blind assay samples. A performance testing sample (PT), purchased from an independent contractor, and is analyzed twice a year. The acceptable result for the PT sample is unknown until after the experimental result is reported to the contractor. Other externally originated PTs are analyzed when supplied by the client as either a single blind or as a double blind sample and are scheduled through the laboratory as routine samples. All performance testing samples are handled in the same manner as real environmental samples including staff, method, procedures, equipment, facilities, and frequency.

12.3.2. The samples shall be analyzed and the results returned to the PT Provider no later than 45 calendar days from the opening of the study.

12.3.3. When analyzing a PT sample, the same calibration, laboratory quality control and acceptance criteria, sequence of analytical steps, number of replicates and other procedures are employed as used when analyzing routine samples.

12.3.4. No PT sample, or a portion of a PT sample, shall be sent to another laboratory for any analysis

12.3.5. No PT sample or portion of a PT sample shall be received from another laboratory for any analysis

12.3.6. Vista Analytical Laboratory management or staff shall not communicate with any individual at another laboratory concerning the PT sample or attempt to obtain the assigned value of any PT sample from their PT Provider.

12.3.7. Vista shall maintain copies of all written, printed, and electronic records, resulting from the analysis of any PT sample for five years or for as long as is required by the applicable regulatory program, whichever is greater. All of these laboratory records shall be made available to the assessors during on-site audits of the laboratory.

12.3.8. In the event that the laboratory receives test results that are "unacceptable", the likely cause is investigated, identified, and resolved. A Corrective Action PT sample, for which the laboratory shall report only the analytes for which corrective action was required, will then be analyzed. Documentation of the corrective action as well as the corrective action PT scores will then be submitted to the applicable accrediting authorities. The laboratory will provide A2LA the results of the Proficiency Testing and any related corrective action(s) within 14 days of receipt. Follow up results (if required) will be provided within 45 days. Form F104 will accompany all correspondence.

12.4. External Audits

12.4.1. External audits are performed on an on-going basis by clients, regulating agencies (State and Federal), or other third party auditors. These audits are pre-scheduled with the client and QA Manager to ensure that the appropriate laboratory personnel are available to address all audit inquiries. All deviations or deficiencies noted during the audit are to be addressed in the time frame provided by the auditor.

12.5. Data Audits

12.5.1. Data audits at Vista utilize a three tier data review system involving the Laboratory Director, Project Managers and the QA Manager.

12.5.2. Tier 1. In the initial phase, the analyst, defined as the instrument operator, reviews the raw data for correct analyte identification and integration, completes final data calculations, enters the data, signs and dates the raw data, and submits the package to a Director or Project Manager for review. In the case of anomalies, the analyst may be required to prepare a corrective action / preventative action report (CAPA) discussing the potential causes for the problems encountered as well as the recommended corrective action. The analyst will make any necessary corrections and/or reanalyze the sample, review, sign and date the raw data and any CAPAs (if applicable). The Project Manager after review of the data will approve all final datasheets.

12.5.3. Tier 2. The second tier review requires the Project Manager, signing the cover letter of the final report, to review and approve the data package. The Project Manager examines the chain of custody request, sample receipt information, analytical data for completeness and assesses whether the package as a whole meets the data quality objectives set by the client. The Project Manager is required to discuss or explain any data anomalies in the text of the case narrative.

12.5.4. Tier 3. The third tier review is performed by the QA Manager. The QA Manager will audit a minimum of 10% of the data packages and review all aspects of the data package covered during the first and second tier reviews. The QA Manager review may result in a request to the Laboratory Director for additional information regarding the data set and if necessary, re-analysis of selected samples.

13. TRAINING

Training assessments and all related training documentation shall be conducted in accordance with SOPs.

13.1. Initial On-Site Training

13.1.1. The training requirement of each employee will be assessed periodically to ensure the competency of their job responsibilities that career development objectives are being met, and that general-purpose educational opportunities are being utilized. The training program shall be relevant to the present and anticipated tasks of the laboratory.

13.1.2. Previous training, education, and experience will be considered when evaluating the training needs of each employee.

13.1.3. Manuals, texts, SOPs, journals, analytical methods and in-house Analytical Procedures are available for all new trainees, with on the job training performed by senior staff.

13.2. Training Programs

13.2.1. Job related training will be provided through regularly scheduled in-house seminars and courses, university courses, conferences and seminars, and one-on-one on the job tutorials.

13.2.2. Specified performance criteria must be successfully met while under supervision before personnel will be made responsible for activities that affect the quality objectives of the company.

13.3. Training Documentation

13.3.1. Training records will be maintained in each individual's training file. These records will be readily available to supervisors to ensure that employees have demonstrated capability prior to performing activities for which they are responsible. Employees are responsible for keeping their training file up-to-date. The training files shall maintain records of competence, education and professional qualifications, training, skills and experience of all technical personnel, including contracted personnel.

13.3.2. Evidence on file demonstrating each employee has read and understood the current version of in-house quality documents (QM, QAPP, SOPs).

13.3.3. Documentation of training courses.

13.3.4. Documentation of continued proficiency at least once per year.

14. CLIENT SERVICES

Routine client service as well as responses to client inquires, audit reports, recommendations, and complaints will be handled in accordance with SOPs.

14.1. Routine Services

14.1.1. Each client will be assigned a Project Manager who will be responsible for ensuring that the needs of the client are clearly understood and communicated to the appropriate areas of the company.

14.1.2. The Project Manager reviews all new work to ensure that it has the appropriate facilities and resources before commencing such work. Once the Project Manager accepts the new work, an acknowledgement letter is sent to the client for confirmation.

14.1.3. Clients will be given the opportunity to verify that the company's services conform to specified requirements. Regardless of whether or not client verifications are conducted, the Quality System will be responsible for ensuring that all services conform to specified requirements.

14.1.4. As the client's representative, the Project Manager will be responsible for ensuring that the client's needs are met. The Project Manager will maintain good communication, advice and guidance in technical matters, and opinions and interpretations based on results.

14.1.5. All client data are managed and maintained with the utmost care and diligence to ensure that the protection of clients' confidential information and proprietary rights are a primary concern.

14.2. Contract Review

14.2.1. For all analytical services to be provided, contract review is performed by the Contract Administrator and Project Manager. . Sales and client services personnel are responsible for implementing and documenting contract review. Client requirements are defined and documented in the written quote or contract.

14.2.2. For routine projects, a review by Technical Sales is considered as adequate. Technical Sales confirms that the laboratory can meet the client's data quality objectives, turn-around time, reporting requirements and required certification for the work.

14.2.3. For large or new projects, the proposal is given to the Laboratory Director for review. The Laboratory Director will, if required, request input from the Technical Director, Quality Assurance Manager, and Technical Sales in preparing a response to the proposal.

14.3. Responses to Client Audits, Inquiries, and Complaints

14.3.1. The QA Manager will be responsible for coordinating responses to client audits.

14.3.2. Complaints received from clients or other parties regarding data or laboratory activities will be directed to the appropriate project manager and reported to the Technical Director or Laboratory Director.

14.3.3. If a corrective action(s), which may require completion of a CAPA (corrective action / preventative action report), is taken, this will be documented and archived with the appropriate project data.

14.3.4. All complaints will be documented and records of actions in response to any complaints will be maintained.

14.3.5. If a complaint raises doubt regarding the laboratory's policies or compliance with NELAP or other standards, those areas shall be promptly reviewed or audited by the laboratory QA Manager.

15. STATISTICAL TECHNIQUES

Statistical techniques used to monitor the performance of activities that directly affect quality objectives will be conducted in accordance with SOPs.

15.1. Statistical Process Control Procedures

15.1.1. Statistical Process Control (SPC) will be used to monitor analytical procedure performance indicators such as accuracy and precision, and process performance indicators such as turnaround time and Nonconformance reports.

15.1.2. Results of SPC analyses will be used to improve processes that affect quality objectives.

16. SUBCONTRACTING

16.1. Vista Analytical may subcontract services, or may refer a client directly to another lab, for a particular analysis. Subcontracted laboratories are held responsible for the implementation of their own QM and meeting their data quality objectives.

16.2. Clients shall be notified prior to subcontracting any portion of their testing to another laboratory.

16.3. Services requiring NELAP accreditation will only be subcontracted to a laboratory with NELAP accreditation.

16.4. For DoD clients, subcontractor laboratories must have records to demonstrate their compliance with ISO/IEC 17025 for the subcontracted work in question, documented compliance with DoD QSM requirements, must be approved by the specific DoD laboratory approval process, must demonstrate the ability to generate acceptable results through the analysis of proficiency testing samples, and must receive project-specific approval from the DoD client before any samples are analyzed.

16.5. For services associated with projects outside of California, individual state accreditations may need to be met.

16.6. Vista Analytical shall retain records demonstrating that the above requirements have been met. Original reports received from a subcontracted laboratory will be included with the clients test report.

17. DATA INTEGRITY AND ETHICS

Vista Analytical Laboratory expects employee compliance with all laboratory SOPs and applicable regulatory guidelines and standards. Vista encourages participation in cooperative and educational efforts designed to promote and inform laboratory personnel of the necessity of active compliance.

17.1. Vista does not condone and will not tolerate the fraudulent manipulation or falsification of data, intentional non-compliance, gross negligence, or any other unethical conduct. Employees who are aware of, or reasonably suspicious of, any case fraudulent or unethical conduct shall notify the Technical Director, Laboratory Director, or QA Manager. Allegations of unethical conduct may be reported anonymously and will be fully investigated under the direction of the QA Manager. The investigation will be performed in a confidential manner until a full investigation or other appropriate actions have been completed and the issues clarified.

17.2. Any employee who knowingly manipulates and/or falsifies data or documents or engages in any unethical conduct is subject to immediate release from employment and other serious consequences.

17.3. Vista Analytical Laboratory provides mandatory initial and annual or as needed, Laboratory Ethics and Data Integrity refresher training to all employees. Topics covered are approved by management, documented in writing, and provided to all trainees.

17.3.1. Training topics include:

- Quality System requirements
- Personnel training requirements
- Vista Analytical Laboratory Ethics policy
- Examples of actions that are strictly prohibited
- Other breaches of data integrity
- Pertinent SOPs and other quality documents
- Potential consequences of misconduct
- Confidential mechanism for reporting allegations
- Investigation procedures and documentation
- “No-fault” policy to encourage personnel to come forward and report any fraudulent activities.

17.3.2. All employees sign an ethics statement and documentation of training attendance that demonstrates they have participated and understand their obligations related to data integrity. This sheet is maintained in individual training records.

17.4. Upon hire, new employees are required to read and sign a confidentiality statement. This signed statement is maintained in personnel files.

The content of the SOP is the confidential property of Vista Analytical Laboratory, Inc. and may not be copied, modified, retransmitted, or used for any purposes other than those that have been determined by associated contractual agreements without Vista's written authorization.

SOP 26	Revision: 15	Supersedes: Rev14
POLYCHLORINATED DIBENZO DIOXIN/FURANS BY USEPA METHOD 1613B		
Analyst Review:		
Management:		
Quality Assurance:		
Effective Date: 20 October, 2015		

Revision	Description of Revision
0	Renumbered.
1	Added criteria for ending standard in Section 13.4.1. Inclusion of MS/MSD or SD for DOD QSM clients in Section 12.3.1. Updated penta-CDD ion ratio in Table 1.
2	Removed the Ending Standard Requirement from Section 13.4; not method required.
3	Combined the preparation and analytical SOPs for this method. Update company name.
4	Embellish homogenization section
5	A daily reporting level LFB will be analyzed for Arizona compliance samples (16.4), added 9.3.1, MDL in section 11.1, added equipment to 8.27
6	Cleanup column pre-elutions, bulleting; reagent kilning times and temperatures
7	Table 7 was modified to move 1,3,4,6,8-PeCDF into the first descriptor window. Added clarification to Table 1. Descriptor switch clarification has been added. Changed DI to HPLC water; minor edits.
8	Referenced instrument maintenance logs
9	Revised Penta-CDD ion ratio windows. DB-5 to ZB-5MS, added ^{13}C-1,2,3,4,6,9-HxCDF as recovery standard.
10	Added Restek Rtx – Dioxin Column as an additional analytical column.
11	Added Section 10.3: Storing frozen samples. Added Section 12.3: Thawing frozen samples.

12	Added Section 4.11. Revised duplicate samples acceptance criteria in section 11.5. Updated Table 6 to WHO 2005 TEFs.
13	Revised Table 3, Table 7, Table 9.
14	Section 14.3.6 revised the elution amount. Section 4.5 Revised the extraction time from 16 hours to 18 hours. Section 14.1.4 Revised the reference point. Revised Section 12.5.9.
15	Added Table 10 (Quality Control Requirements).

1. PURPOSE

1.1 This procedure describes the preparation and analytical techniques used for the analysis of samples for polychlorinated dibenzodioxin and furans (PCDD/PCDF) by EPA Method 1613B.

2. SCOPE

2.1 All differences between Method 1613B and actual laboratory techniques have been developed to reduce interferences and increase sensitivity.

2.2 Data determined to be out-of-control from criteria stated within this SOP, is handled according to procedures addressed within the applicable section.

Compound	CAS Registry No.*
2,3,7,8-Tetrachlorodibenzo-p-dioxin (TCDD)	1746-01-6
1,2,3,7,8-Pentachlorodibenzo-p-dioxin (PeCDD)	40321-76-4
1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)	39227-28-6
1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin (HxCDD)	57653-85-7
1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin (HxCDD)	19408-74-3
1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin (HpCDD)	35822-46-9
1,2,3,4,5,6,7,8-Octachlorodibenzo-p-dioxin (OCDD)	3268-87-9
2,3,7,8-Tetrachlorodibenzofuran (TCDF)	51207-31-9
1,2,3,7,8-Pentachlorodibenzofuran (PeCDF)	57117-41-6
2,3,4,7,8-Pentachlorodibenzofuran (PeCDF)	57117-31-4
1,2,3,4,7,8-Hexachlorodibenzofuran (HxCDF)	70648-26-9
1,2,3,6,7,8-Hexachlorodibenzofuran (HxCDF)	57117-44-9
1,2,3,7,8,9-Hexachlorodibenzofuran (HxCDF)	72918-21-9
2,3,4,6,7,8-Hexachlorodibenzofuran (HxCDF)	60851-34-5
1,2,3,4,6,7,8-Heptachlorodibenzofuran (HpCDF)	67562-39-4
1,2,3,4,7,8,9-Heptachlorodibenzofuran (HpCDF)	55673-89-7
1,2,3,4,5,6,7,8-Octachlorodibenzofuran (OCDF)	39001-02-0
Total Tetrachlorodibenzo-p-dioxin (TCDD)	41903-57-5
Total Pentachlorodibenzo-p-dioxin (PeCDD)	36088-22-9
Total Hexachlorodibenzo-p-dioxin (HxCDD)	34465-46-8
Total Heptachlorodibenzo-p-dioxin (HpCDD)	37871-00-4
Total Tetrachlorodibenzofuran (TCDF)	55722-27-5
Total Pentachlorodibenzofuran (PeCDF)	30402-15-4
Total Hexachlorodibenzofuran (HxCDF)	55684-94-1
Total Heptachlorodibenzofuran (HpCDF)	38998-75-3

*Chemical Abstract Service

3. SUMMARY OF METHOD

3.1 This procedure uses HRGC/HRMS for detection and quantitation of PCDDs/PCDFs, isotope dilution techniques, and internal standard calibration.

3.2 All differences between the method and actual laboratory techniques have been developed to reduce interferences and increase sensitivity.

3.3 Detection limits are sample-specific and congener-specific.

4. MODIFICATIONS

4.1 Determination of Particle Size (1mm) is not performed because all aqueous samples are filtered through SPE for 1613B.

4.2 The internal standard (IS) is not spiked directly into the sample bottle. The IS is spiked into a test tube containing methanol. Then the test tube is poured into the sample bottle.

4.3 Disk Preparation is different because the filters used are purchased pre-cleaned. Vista treats the filters with methanol then HPLC water.

4.4 For tissue extraction: Vista equilibrates the sample and sodium sulfate mix for 30 minutes, not 18 – 24 hours.

4.5 For tissues only: Vista soxhlet extracts for 18 hours

4.6 Vista does not analyze MB immediately after OPR but injects a solvent blank immediately after the OPR to demonstrate freedom from contamination.

4.7 The IS solution may be mixed with acetone or methanol depending upon the type of extraction.

4.8 The rotovap temperature depends on the solvent used.

4.9 Tetradecane is the final extract solvent.

4.10 Cleanups include Acid Partition, Acid Base Silica Gel/Acid Alumina column then a Florisil Column or Charcoal Column, if necessary, for all matrices except fish.

4.11 ^{13}C-OCDF is used as an internal standard for OCDF.

5. CONTAMINATION AND INTERFERENCES

5.1 Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines which may cause misinterpretation of chromatographic data. All of these materials must be demonstrated to be free from interferents under the conditions of analysis by running laboratory method blanks.

5.2 The use of high purity reagents and solvents helps to minimize interference problems.

5.3 Contamination of the laboratory will be minimized by conducting most of the manipulations in a hood.

5.4 Interferants co-extracted from the sample will vary considerably from matrix to matrix.

5.5 If interferences are encountered that would cause the detection limit or reporting limit to be raised, then certain cleanup procedures must be selected to aid the analyst in their elimination.

6. DEFINITIONS

6.1 Definitions are presented in the Glossary.

7. SAFETY

7.1 Dioxins: 2,3,7,8-TCDD isomer has been found to be acnegenic, carcinogenic, and teratogenic in laboratory animal studies.

7.2 Procedures shall be carried out in a manner that protects the health and safety of all Vista employees, including the appropriate use of Personal Protective Equipment and engineering controls.

7.3 Each chemical compound should be treated as a potential health hazard. Exposure to these compounds should be reduced to the lowest possible level. Only highly trained personnel thoroughly familiar with handling and cautionary procedures and the associated risks should handle all compounds or reagents.

7.4 Each chemical compound should be handled in well-ventilated, controlled access laboratories.

7.5 Additional health and safety information can be obtained from material safety data sheets (MSDSs) available to all personnel involved in these analyses.

7.6 In the event of a known or potential compromise to the health and safety of a Vista associate, all work must stop and the incident reported immediately to management.

8. APPARATUS AND MATERIALS

8.1 Analytical Balances, capable of reading to 0.01g and 0.0001 g

8.2 Aquacheck strips and pH strips

8.3 Centrifuge, Centrifuge bottles, 1L, 750 mL & 250 mL size. Centrifuge with rotor and cups for 1L, 750 mL & 250 mL bottles

8.4 Crimp top autoinjector vials plus caps and crimp tool

8.5 Drying Oven, VWR Model 1320 or equivalent

8.6 Electrothermal electromantle six sample and 500 & 1000 mL capacity

8.7 Funnels, 100 mm

8.8 Glass columns, 160 mm x 11 mm and 200 mm x 15 mm

8.9 Glass wool

8.10 Grinder

8.11 High Pressure Filtration Apparatus (HPFA)

8.12 Organomation 24-Station N-Evaporator

8.13 Pre-cleaned Glass fiber thimbles - coarse

8.14 Rotary evaporator

8.15 Round bottom flasks: 50, 100, 250, and 500 mL

8.16 Separatory funnels, typically 250 mL to 2000 mL size

8.17 Soxhlet/Dean-Stark (SDS) Extractor

8.18 Teflon boiling chips

8.19 Test tubes plus Teflon lined caps, 16 mm x 125 mm

8.20 Vials, Glass conical,

8.21 Volatile Organic Analysis (VOA) vials, 40 mL

8.22 C_{18} SPE discs

8.23 SPE glassware, HiVac pump, SPE Manifold

8.24 Whatman GF/C, GF/D, and GF/F filters

8.25 Wiretrol II Precision Disposable Micropipettes

8.26 Zymark TurboVap II plus 250 mL tubes with 1 mL stems or equivalent

8.27 EquipmentCTC Autosampler Model A200S.

8.28 Alpha Station 500.

8.29 Neslab HX200, HX300 or HX500 Water Cooler.

8.30 HP 6890F Gas Chromatograph

8.31 60 meter ZB-5MS GC column, 0.25 mm i.d., 0.25 μm film (J&W Scientific) or equivalent

8.32 30 meter DB-225 GC column, 0.25 mm i.d., 0.25 μm film (J&W Scientific) or equivalent

8.33 60 meter SP-2331 GC column, 0.25 mm i.d., 0.25 μm film or equivalent

8.34 60 meter Rtx-Dioxin2 GC column, 0.25 mm i.d., 0.25 μm film (Restek) or equivalent

8.35 Waters Autospec Ultima Magnetic Sector High Resolution Mass Spectrometer.

8.36 Injection vial inserts, 100 µL (Sun International or equivalent)

9. REAGENTS, SOLVENTS AND STANDARDS

9.1 Reagents (Highest purity available)

9.1.1 Acid Silica Gel, 44%

9.1.2 Activated Silica Gel, kilned for ~5 hours at 550°C, granular

9.1.3 Anhydrous sodium sulfate (Na_2SO_4), kilned for ~5 hours at 550°C, granular

9.1.4 Basic Silica Gel, 33%

9.1.5 Carbopack B/Celite 18%

9.1.6 Florisil

9.1.7 Acid Alumina

9.1.8 Hydrochloric acid, concentrated

9.1.9 Hydromatrix

9.1.10 Ottawa sand, kilned for ~5 hours at 550°C

9.1.11 Sodium hydroxide (NaOH), 10N

9.1.12 Sulfuric acid (H_2SO_4), concentrated

9.1.13 Ultra-pure nitrogen gas

9.1.14 Water, HPLC

9.2 Solvents

9.2.1 Tetradecane (C_{14})

9.2.2 Toluene

9.2.3 Ethanol

9.2.4 Hexane

9.2.5 Methanol

9.2.6 Methylene chloride (DCM)

9.2.7 Acetone

9.3 Standards

9.3.1 All analytical standards are obtained from a certified vendor. See SOP 15 and the current spike sheet for more information.

10. COLLECTION, PRESERVATION, AND HANDLING

10.1 Amber glass bottles and jars must be used for collection,

10.2 All samples must be maintained at $\leq$ 6ºC. Fish/tissue samples are maintained at <-10ºC.

10.3 If applicable, samples to be stored frozen are checked for adequate headspace prior to storage in case expansion occurs during freezing.

10.4 If there is residual chlorine in aqueous samples, add 80 mg of sodium thiosulfate per liter. If pH >9, adjust the pH to 7-9 with sulfuric acid.

10.5 Samples must be extracted within one year of sample collection. Samples must be analyzed within one year from extraction.

11. QUALITY CONTROL

11.1 Each time a modification is made to this method and the detection limit will be affected by the change, the laboratory is required to demonstrate that the MDL (40 CFR Part 136, Appendix B) is lower than one-third the regulatory compliance level or one-third the ML in the method, whichever is higher.

11.2 Method Blank (MB): Method blank is a matrix preparation that is free of native analyte that has been prepared and analyzed using the same procedures followed for the rest of the analytical batch. Simulate as close as possible the matrix to be extracted. See Table 10 for more information.

11.3 Ongoing Precision and Recovery Samples (OPR): An ongoing precision and recovery sample is prepared by adding a known quantity of native standards to an interferant free matrix and used to assess method performance (precision and recovery). See Table 10 for more information.

11.3.1 A 10 µL aliquot containing 200 pg Cl4 DD/DF, 1000 pg Cl5-Cl7 DD/DF & 2000 pg Cl8 DD/DF is used for spiking.

11.4 Matrix Spike (MS/MSD): A matrix spike sample is prepared by adding a known quantity of native standards to a sample matrix prior to extraction. See Table 10 for more information.

11.4.1 A 10 µL aliquot containing 200pg Cl_4 DD/DF, 1000pg Cl_5-Cl_7 DD/DF & 2000pg Cl_8 DD/DF is used for spiking.

11.5 Duplicate Samples: Duplicate samples are two separate aliquots taken from the same source. Duplicate samples are analyzed independently to assess laboratory precision. See Table 10 for more information.

12. SAMPLE PREPARATION

12.1 Residual Chlorine Determination

12.1.1 Obtain an Aquacheck strip and place it directly into a small amount of sample in a disposable weigh boat. Move the strip back and forth for 30 seconds.

12.1.2 Check the color on the strip against the color chart on Aquacheck container.

12.1.3 If there is chlorine present, add 80 mg of sodium thiosulfate to sample bottle.

12.1.4 Record procedure on extraction benchsheet.

12.2 pH Determination

12.2.1 Obtain a pH strip and place it directly into a small amount of sample in a disposable weigh boat. Move the strip back and forth for 30 seconds.

12.2.2 Check the color on the strip against the color chart on the pH container.

- If the pH is greater than 9, then the pH must be adjusted to 7-9 with sulfuric acid.

12.3 Thawing Frozen Samples

12.3.1 Remove the sample from the freezer and place under the hood under ambient temperature.

- If there is a concern that the sample container may break, place the container in a pre-cleaned secondary container.

12.3.2 Once the sample has thawed completely, proceed with sample preparation.

12.4 Compositing/Homogenization (Fillets)

12.4.1 Remove any obviously extraneous materials and homogenize sample prior to sub-sampling for extraction. Homogenization may be as simple as shaking the container vigorously by hand to crushing, chopping, and use of a mechanical grinder.

- Each whole fish filet is cut into dorsal/ventral strips ~2cm wide and shuffled prior to placing through a grinder to ensure proper homogenization unless the client requests otherwise. Once each fish fillet is homogenized, the entire ground fish filet is mixed with the other designated ground fish filets to make a composite. The entire composite is placed in an amber glass jar for extraction.

- By client request, whole fish may be either homogenized or filleted prior to homogenization. Skin on the fillets may be removed by client request.

- All grinding parts and components are cleaned prior to homogenization and between each sample. Wash with soap and water and rinse with HPLC water. Solvent rinse in the following order: Acetone → toluene → hexane → methylene chloride

12.5 % Solids Determination

12.5.1 “ZERO” or “TARE” the balance.

12.5.2 Place a weigh boat on the balance and record the weight as “Boat Weight”.

12.5.3 Add a portion of the sample (approximately 2 – 10 g) to the weigh boat and record the weight as “Wet Wt. + Boat Wt.”

12.5.4 Place the weigh boat plus sample in an oven at 110±5°C for at least overnight.

12.5.5 Remove the weigh boat plus sample from the oven and allow to come to room temperature.

12.5.6 “ZERO” or “TARE” the balance.

12.5.7 Place the weigh boat plus sample on the balance and record the weight as “Residue + Boat Wt.”

12.5.8 Calculate the percent solids by the following formula:

$$\%Solids = \frac{(Residue\ Wt. + Boat\ Wt.) - (Boat\ Wt.)}{(Wet\ Wt. + Boat\ Wt.) - (Boat\ Wt.)} X 100$$

12.5.9 For aqueous samples, samples with greater than 1% solids will be high pressure filtered and extracted as a solid.

12.5.10 Compositing (Soils)

- By client request

12.5.11 Samples are individually homogenized, if necessary, with a clean spoon, spoonula or spatula.

12.5.12 Weigh out approximately 50 grams, or amount designated by the client, from each individual sample and place into a pan.

12.5.13 Repeat the homogenization for each sample.

12.5.14 Place each individual sample into a new, separate container. Record the weight of each sample on the benchsheet.

12.5.15 Retain the original sample containers with remaining sample. The new container is given a new sample ID number and then processed through the appropriate extraction.

13. EXTRACTION PROCEDURES

13.1 Aqueous Samples

13.1.1 Record the combined weight of the bottle, cap and sample for each sample to be extracted. After the sample has been removed from the bottle, allow it to drain overnight and reweigh it and the cap to determine the amount of sample extracted.

13.1.2 For the method blank (MB) and OPR(s), transfer ~1 liter of HPLC water into a one liter bottle for each.

- Add the appropriate volume of Internal Standard (IS) solution to a test tube containing ~1 mL of methanol. Quantitatively transfer to the aliquot of matrix with small portions of the solvent used. Add the appropriate volume of Native Standard (NS) solution to a test tube containing the IS/solvent and then quantitatively transfer to the aliquot of matrix assigned as an OPR, MS or MSD. Allow the spiked samples to equilibrate for at least 1 hour before extraction.

13.1.3 Place the SPE disk on the extraction base and attach the funnel with vacuum off.

13.1.4 Add ███ mL of methanol to the funnel.

13.1.5 Draw, by vacuum, approximately half of the methanol through the disk. Do not let the disk go dry!

13.1.6 Add ███ mL of HPLC water.

13.1.7 Draw, by vacuum, approximately half of the liquid through the disk. Do not let the disk go dry!

13.1.8 Transfer the contents of each bottle to the funnel with several portions of HPLC water, being sure to transfer as much of any particulates that may be present as practical.

13.1.9 When the liquid level nears the SPE disk, rinse the inside walls of the funnel with HPLC water.

13.1.10 Continue to draw air through the SPE disk for at least a minute after all of the liquid has passed through the disk.

13.1.11 Assemble the SDS glassware, using toluene as the extraction solvent, place a portion of glass wool in the bottom of the soxhlet body followed by the SPE disk.

13.1.12 Extract for at least 16 – 24 hours.

13.1.13 Add CRS and 100-200 μL of tetradecane to the extract.

13.1.14 Exchange the extract into hexane and concentrate to <10 mL.

- See Appendix for alternate extractions.

13.2 Soil, Sediment, Solids, Clay

13.2.1 Samples are individually homogenized with a clean spoon, spoonula or spatula. Weigh the sample (nominal 10 g dry weight equivalent) directly into an analyte-free thimble, carefully breaking up any large lumps of sample.

- Add the appropriate volume of IS and NS solutions directly to the aliquot of matrix.

13.2.2 Assemble the SDS apparatus, and add a fresh charge of toluene to the receiver and reflux flask. Apply power to the heating mantle to begin refluxing. Wrap soxhlet with bubble foil to increase temperature.

13.2.3 Reflux the sample for a total of 16-24 hours. Cool and disassemble the apparatus.

13.2.4 Add CRS and 100-200 µL C_{14} to the extract.

13.2.5 Concentrate the extracts from the particles to the C_{14} using the rotary evaporator. Exchange with 50 – 100 mLs of hexane.

13.3 Sludge Samples

13.3.1 Weigh the sample (approximately 2 - 5 g dry weight equivalent) directly into an analyte free thimble.

13.3.2 If % solids is too low to accommodate a 10 g sample size into thimble, fill thimble half full of Hydromatrix.

13.3.3 Tare weight of thimble then fill thimble full of sludge. Record weight of sludge, spike and then transfer immediately to soxhlet.

13.3.4 SDS extract for 16 – 24 hours with toluene.

13.3.5 Add CRS and 100-200 µL C_{14} to the extract.

13.3.6 Concentrate the extracts from the particles to the C_{14} using the rotary evaporator. Exchange with 50 – 100 mLs of hexane.

13.4 Tissue

13.4.1 Weigh approximately 25 g of well ground fish/tissue with at least 60-70 g of pre-cleaned Na_2SO_4 in a beaker. (If 10 g of tissue are used, then 30-40 g of Na_2SO_4 is used.)

13.4.2 Stir frequently to remove any lumps. Re-stir and wait ~30 minutes before transferring to thimble.

13.4.3 Add, as appropriate, the IS and NS.

13.4.4 Soxhlet extract for 18-24 hours with 1:1 DCM:hexane.

13.4.5 Add the Cleanup Recovery Standard (and tetradecane if no percent lipids analysis will be performed) to the extract and concentrate to < 100 mL.

13.4.6 If % Lipids are to be determined proceed to the next step. Otherwise proceed with the appropriate cleanup procedures.

13.5 % Lipids

13.5.1 Transfer to a 250 mL mixing cylinder, reduce the extract to a convenient volume (typically 100 mL) using 1:1 DCM:hexane and mix well.

13.5.2 Transfer 10% of the solution to an aluminum dish that has been pre-weighed on an analytical balance.

13.5.3 Allow the extract to air dry completely and then place in a 50±5°C oven overnight.

13.5.4 When the aliquot is dry, allow to cool to room temperature, re-weigh the dish on an analytical balance and record the weight. Calculate the % lipids using the following equation:

- $$\% \text{lipids} = \frac{\text{lipid residue weight}}{10\% \text{ of sample weight}} \times 100$$

13.5.5 Using the remaining 90% of the extract, proceed with the appropriate cleanup procedures.

14. CLEANUP PROCEDURES

14.1 Acid/Base partitioning (ABP) or Acid Partitioning (AP)

14.1.1 Using hexane, adjust the extract volume to ~100 mL and transfer to the separatory funnel.

14.1.2 Carefully add ~50 mL of concentrated H_2SO_4 to a separatory funnel.

14.1.3 Shake for approximately 30 seconds with periodic venting, allow the layers to separate (centrifugation may be necessary) and discard the acid layer. Add ~50 mL of HPLC water to the separatory funnel. Shake for approximately 30 seconds with periodic venting and discard the aqueous layer. Repeat if sample still contains color.

14.1.4 If base partitioning is appropriate, proceed to step 14.1.5, otherwise proceed to step 14.3.6.

14.1.5 Add ~ 50 mL of 10N NaOH to the separatory funnel. Shake approximately 2 minutes with periodic venting, allow the layers to separate (centrifugation may be necessary) and discard the NaOH layer. Add ~ 50 mL of HPLC water to the separatory funnel. Shake for approximately 2 minutes and discard aqueous layer.

14.1.6 Pass the organic layer through Na_2SO_4 (pre-rinsed with 20 - 40 mL aliquots of hexane) then concentrate to C_{14}.

14.2 Acid Base Silica Gel/Acid Alumina (ABSG/AA)

14.2.1 Prepare the columns as depicted in Figure 1.

14.2.2 All traces of solvents other than hexane must be removed from the extract.

14.2.3 Adjust extract volume to <10 mL.

14.2.4 Rinse the ABSG column with ~[redacted] mL hexane and the AA column with ~[redacted] mL DCM and then [redacted] mL of hexane, discard the eluate.

14.2.5 Position the ABSG column so that it elutes directly onto the Acid Alumina column.

14.2.6 Transfer the extract to the ABSG column with [redacted] portions of hexane, discard the eluate.

14.2.7 When the extract reaches the sodium sulfate, add [redacted] mL of hexane.

14.2.8 When all of the ABSG eluate has passed through the Acid Alumina column, remove the ABSG column, discard all the eluates.

14.2.9 Elute the Acid Alumina column with [redacted] mL of [redacted]% DCM:hexane, collect the eluate.

14.2.10 Concentrate the eluate appropriately.

- Add 100-200 µL tetradecane only if continuing to Florisil cleanup, except tissues skip to Charcoal.

14.3 Florisil (F)

14.3.1 Prepare the column as depicted in Figure 2.

- After removing the florisil jar from the oven, allow the florisil to cool ~ 10 minutes before weighing it out.

14.3.2 Concentrate extract to C14 to ensure all DCM has been removed from the extract. Adjust extract volume to < 5 mL.

14.3.3 Rinse the column with ~ mL of DCM, then mL of hexane, discard the eluate.

14.3.4 Transfer the extract to the column with portions of hexane, discard the eluate.

14.3.5 Elute the column with mL of hexane, discard eluate.

14.3.6 Elute the column with of DCM, collect the eluate.

14.3.7 Concentrate the eluate appropriately.

14.4 Charcoal Column (CC)

14.4.1 Prepare the column as depicted in Figure 3. Pack the column tightly.

14.4.2 Rinse the column with the “A” end up with mL of toluene.

14.4.3 Flip the column 180° (the “B” end should be up).

14.4.4 Rinse the column with at least mL of DCM:MeOH. Discard eluate.

14.4.5 Rinse the column with at least mL of DCM: hexane, discard eluate.

14.4.6 Transfer the extract to the column with DCM: hexane.

14.4.7 Rinse the column with mL of DCM:hexane, discard eluate.

14.4.8 Rinse the column with L of DCM:MeOH, discard eluate.

14.4.9 Flip the column 180° (the “A” end should be up).

14.4.10 Elute the column with mL of toluene into a 50 mL round bottom.

14.4.11 Do not add tetradecane.

14.4.12 Cautiously concentrate to near dryness – not dry. Exchange ██ with ██ mL hexane.

15. ADJUST TO FINAL VOLUME

15.1 Using hexane, quantitatively transfer the concentrated eluate to a conical vial that contains the Recovery Standard (RS) and 10 µL of tetradecane.

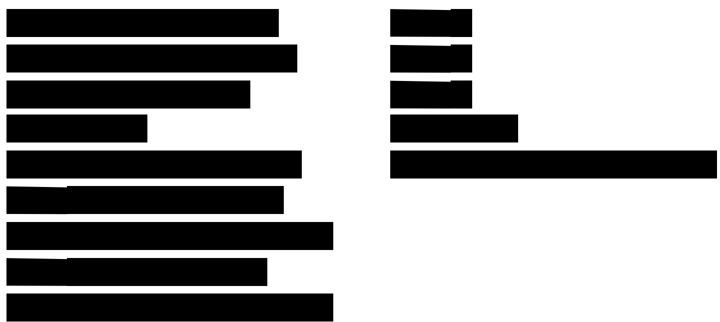
15.2 Using nitrogen blow down, concentrate to the tetradecane.

15.3 Rinse the walls of the conical with hexane, concentrate down to the tetradecane.

15.4 Using a 10-20 µL Wiretrol, transfer the tetradecane to an insert in a crimp top autoinjector vial and then cap.

16. GC/MS ANALYSIS

16.1 Establish the necessary operating conditions. The following GC operating conditions are for guidance and adjustments may be required.



16.2 Tuning

16.2.1 The reference compound perfluorokerosene (PFK) provides the required lock masses and is used for tuning the mass spectrometer.

16.2.2 The lock-mass ion from PFK is dependent on the masses of the ions monitored within each descriptor, as shown in Table 7. Each descriptor will be monitored in succession as a function of GC retention time to ensure that all PCDDs and PCDFs are detected.

- Note: The elution order of the ZB5MS column is such that the descriptor switch to the second homologous series occurs after the elution 1,3,4,6,8-PeCDF.

  See Table 10 for more information.

16.2.3 The mass spectrometer must be operated in a selected ion monitoring (SIM) mode with a total cycle time of 1 second or less. The suggested ions to monitor for PCDD/Fs and Chlorinated Diphenyl Ethers (CDPEs) are listed in Table 7. CDPEs are known interferences for the PCDFs.

- If CDPEs are detected in a sample, then the data must be qualified appropriately.

16.3 Initial Calibration

16.3.1 An initial calibration curve is created to demonstrate the linearity of the HRMS system over the calibration range. An initial calibration is repeated whenever a new set of spiking calibration standards are created or whenever the continuing calibration falls outside the acceptance criteria. See Table 10 for more information.

- The absolute retention time of $^{13}C_{12}$-1,2,3,4-TCDD must exceed 25.0 minutes on the ZB-5MS column and 15 minutes on the DB-225 column.

16.3.2 Inject 1-2 µL of each of the six calibration solutions containing all 16 2,3,7,8-substituted isomers including the Column Performance Check Solution (CPSM) and the first and last eluting isomers of each homologue group. The following criteria must be met:

- The first and last PCDD/PCDF eluters must be verified in accordance with Table 2.
- The chromatographic peak separation between 2,3,7,8-TCDD and the closest eluting isomers must be separated with a valley of ≤25%.
- Seventeen internal standards and three recovery standards are used to improve quantitation.
- For TCDD/TCDF, two internal standards and two recovery standards are used for quantitation.

16.3.3 An initial calibration curve is accepted if the criteria in Table 10 is met.

16.3.4 All initial instrument calibrations are verified with a standard (CS3) from a second manufacturer or lot.

16.3.5 The calibration range for the various types of matrices are as follows:

	Effluent/Aq. (pg/L)	Solid (pg/g)	Waste (pg/g)	Oil (pg/g)	Wipe (pg/sample)	Tissue (pg/sample)
Cl_4	5.0-4,000	0.50-400	0.50-400	0.50-400	5.0-4,000	0.2-160
Cl_5-Cl_7	25-20,000	2.5-2,000	2.5-2,000	2.5-2,000	25-20,000	1.0-1,800
Cl_8	50-40,000	5.0-4,000	5.0-4,000	5.0-4,000	50-40,000	2.0-1,600

16.4 Continuing Calibration

16.4.1 A verification (VER) standard from the initial calibration curve (CS3) containing the column performance standard mix (CPSM) is injected at the beginning of an analytical 12-hour sequence. See Table 10 for acceptance criteria.

16.5 Qualitative Determination

16.5.1 To identify a chromatographic peak as a PCDD or PCDF (either an unlabeled or a labeled compound), it must meet the following criteria:

16.5.2 The signals for the two exact m/zs being monitored (Table 7) must be present and must maximize within ±2 seconds of one another.

16.5.3 The signal-to-noise ratio (S/N) of each of the two exact m/zs must be ≥2.5:1 for a sample extract.

16.5.4 The ion abundance ratios must be within the limits established for the homologous series (Table 1).

16.5.5 The absolute retention times for non-2,3,7,8-substituted congeners must be within the corresponding windows set by the CPSM.

16.5.6 The absolute retention times for 2,3,7,8-substituted congeners must be within -1 to +3 seconds of the isotopically labeled standard.

16.6 Quantitative Determination

16.6.1 Quantitate the PCDD and PCDF peaks from the response relative to the appropriate internal standard.

16.6.2 Any sample in which the 2,3,7,8-TCDF is identified at or above the CS1 in Table 3 (Method Quantitation Limit) by analysis on the primary column must be confirmed on a secondary column.

- The lowest concentration of 2,3,7,8-TCDF identified on the two GC columns will be reported.

16.6.3 For 2,3,7,8-substituted congeners that are not identified, calculate a detection limit.

- Note: to calculate Totals EDL/EMPC, the EDL or EMPC corresponding to the highest EDL/EMPC of each individual congener in each homolog is reported.

16.6.4 If a chromatographic peak of a PCDD/F saturates the detector, a dilution of the extract must be analyzed.

See Table 10 for more information.

17. CALCULATIONS

17.1 The concentrations for the PCDD or PCDF compounds are calculated by using the formula:

$$C_x = \frac{(A^1_N + A^2_N)(Q_{IS})}{(A^1_{IS} + A^2_{IS})(RRF_N)(S)}$$

Where:

C_x = *Concentration of unlabeled PCDD/PCDF congeners (or group of coeluting isomers within an homologous series) in pg/g,*

A^1_N, A^2_N = *The areas of the primary and secondary m/z's for the native compound.*

A^1_{IS}, A^2_{IS} = *The areas of the primary and secondary m/z's for the internal standard.*

Q_{IS} = *Quantity, pg, of the internal standard.*

RRF_N = *Calculated relative response factor for the analyte.*

S = *Weight or volume of the sample.*

17.2 The Toxicity Equivalent Concentration (TEQ) is calculated using the following formula:

$$TEQ = (C_X) \times (TEF)$$

$$TEQ = (EMPC_X \text{ or } DL) \times (TEF)$$

$$TEQ_{min} = \sum(C_X) \times (TEF)$$

$$TEQ_{max} = \sum(EMPC_X \text{ or } DL) \times (TEF)$$

Where:

C_X = Concentration of unlabeled PCDD/PCDF congeners
$EMPC_X$ = Estimated maximum possible concentration of unlabeled PCDD/PCDF congeners

DL = Detection limit of unlabeled PCDD/PCDF congeners

TEF = Toxicity Equivalence Factor, either from the WHO list or I-89 list

- The 2,3,7,8-TCDD toxicity equivalents of PCDD/Fs present in the sample are calculated according to the above-mentioned equation. A 2,3,7,8-TCDD toxicity equivalent factor is assigned to each of the sixteen 2,3,7,8-substituted PCDD/Fs and to OCDD/F listed in Table 6.

17.3 The detection limits can be calculated using the following formula:

$$DL = \frac{(2.5)(H_N)(Q_{IS})}{(H_{IS})(S)(RRF_N)}$$

Where:
DL = *Detection Limit,*
H_N = *Noise height (peak to peak),*
H_{IS} = *Peak height of the internal standard,*
Q_{IS} = *Quantity, in pg, of the internal standard added to the sample before extraction*
S = *Weight or volume of the sample, and*
RRF_N = *Calculated relative response factor for the analyte.*

17.4 Internal standard recoveries are calculated by using the formula:

$$\%Rec = \frac{(A^1_{IS} + A^2_{IS})(C_{RS})}{(A^1_{RS} + A^2_{RS})(C_{IS})(RRF_{IS})} \times 100$$

Where:
A^1_{IS}, A^2_{IS} = *Areas of the primary and secondary m/z's for the internal standard.*
A^1_{RS}, A^2_{RS} = *Areas of the primary and secondary m/z's for the recovery standard.*
C_{IS} = *Concentration of the internal standard.*
C_{RS} = *Concentration of the recovery standard.*
RRF_{IS} = *Calculated relative response factor for the internal std. analyte.*

17.5 Relative Response Factors can be calculated using the following formula:

$$RRF = \frac{(A^1_N + A^2_N)(C_{IS})}{(A^1_{IS} + A^2_{IS})(C_N)}$$

Where:
A^1_N, A^2_N = *Areas of the primary and secondary m/z's for the native compound*

A^1_{IS}, A^2_{IS} = *Areas of the primary and secondary m/z's for the labeled compound.*
C_{IS} = *Concentration of the internal standard in the calibration standard.*
C_N = *Concentration of the native compound in the calibration standard*

17.6 RRF for unlabeled analytes (RRFN) and for labeled analytes (RRFIS):

$$RRF_N = \frac{(A_X)(Q_{IS})}{(Q_X)(A_{IS})} \qquad RRF_{IS} = \frac{(A_{IS})(Q_{RS})}{(Q_{IS})(A_{RS})}$$

Where:
A_X = *Sum of the integrated ion abundances of the quantitation ions for the unlabeled analyte*
A_{IS} = *Sum of the integrated ion abundances of the quantitation ions for the labeled standards*
A_{RS} = *Sum of the integrated ion abundances of the quantitation ions for the labeled recovery standards*
Q_{IS} = *Quantity of internal standard injected (pg)*
Q_{RS} = *Quantity of recovery standard injected (pg)*
Q_X = *Quantity of unlabeled PCDD/PCDF analyte injected (pg)*

17.7 Estimated Maximum Possible Concentration

$$EMPC = \frac{(A_X)\ (Q_{IS})}{(A_{IS})\ (W)\ (RF_N)}$$

Where:
A_X = *Sum of the area of the smaller peak and of the other peak area calculated using the theoretical chlorine isotope ratio*
A_{IS} = *Sum of the integrated ion abundances of the quantitation ions for the labeled internal standards*
Q_{IS} = *Quantity, in pg, of the internal standard added to the sample before extraction*
W = *Weight of the sample or volume of aqueous sample*
RF_N = *Calculated mean relative response factor for the analyte*

- The 2,3,7,8-TCDD toxicity equivalents of PCDD/Fs present in the sample are calculated according to the above-mentioned equation. A 2,3,7,8-TCDD toxicity equivalent factor is assigned to each of the sixteen 2,3,7,8-substituted PCDD/Fs and OCDD/F (Table 6).

18. METHOD PERFORMANCE

18.1 Method performance was validated and performance specifications were developed using data from EPA's international interlaboratory validation study (See References) and the EPA/paper industry Long-Term Variability Study of discharges from the pulp and paper industry (58 FR 66078).

19. POLLUTION PREVENTION

19.1 The solvent evaporation techniques used in this method are amenable to solvent recovery, and the laboratory shall recover solvents wherever feasible.

19.2 Standards should be prepared in volumes consistent with laboratory use to minimize disposal of standard.

20. WASTE MANAGEMENT

20.1 Waste generated in the procedure must be segregated and disposed according to the facility hazardous waste procedures. Safety officer should be contacted if additional information is required.

20.2 The laboratory waste management is in compliance with all federal, state, and local regulations to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations.

21. REFERENCES

21.1 USEPA Method 1613, Revision B, Dated October 1994.

21.2 Telliard, William A., McCarty, Harry B., and Riddick, Lynn S. “Results of the Interlaboratory Validation Study of USEPA Method 1613 for the Analysis of Tetra- through Octachlorinated Dioxins and Furans by Isotope Dilution GC/MS,” Chemosphere, 27, 41-46 (1993).

21.3 “Results of the International Interlaboratory Validation Study of USEPA Method 1613”, October 1994, available from the EPA Sample Control Center operated by DynCorp Viar, Inc., 30 N Lee St, Alexandria, VA 22314, 703-519-1140.

21.4 SOP 10 - Instrument Maintenance Logbooks and Schedule.

Appendix

II.

Vista
Analytical Laboratory

Figure 1
Acid Base Silica Gel/Acid Alumina (ABSG/AA)

beaker

Figure 2

Florisil

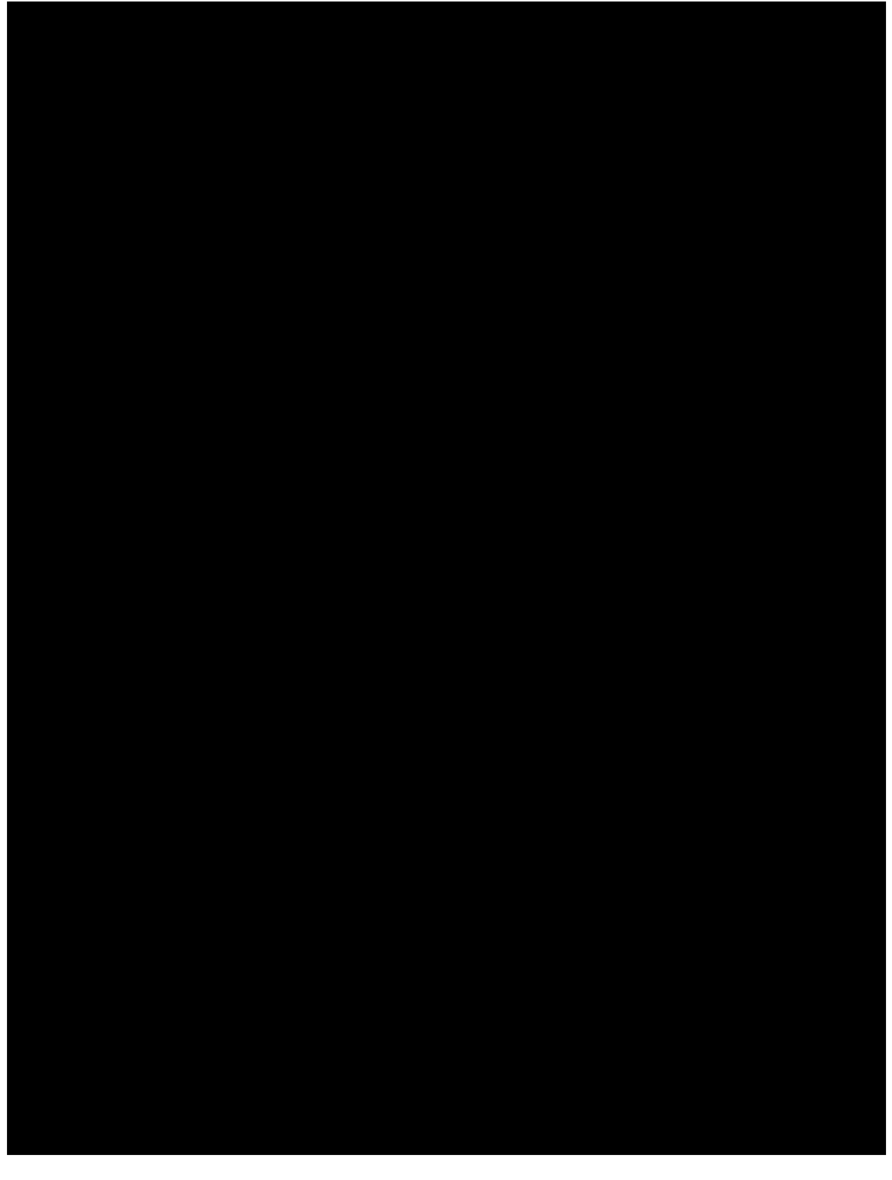


Figure 3

Column

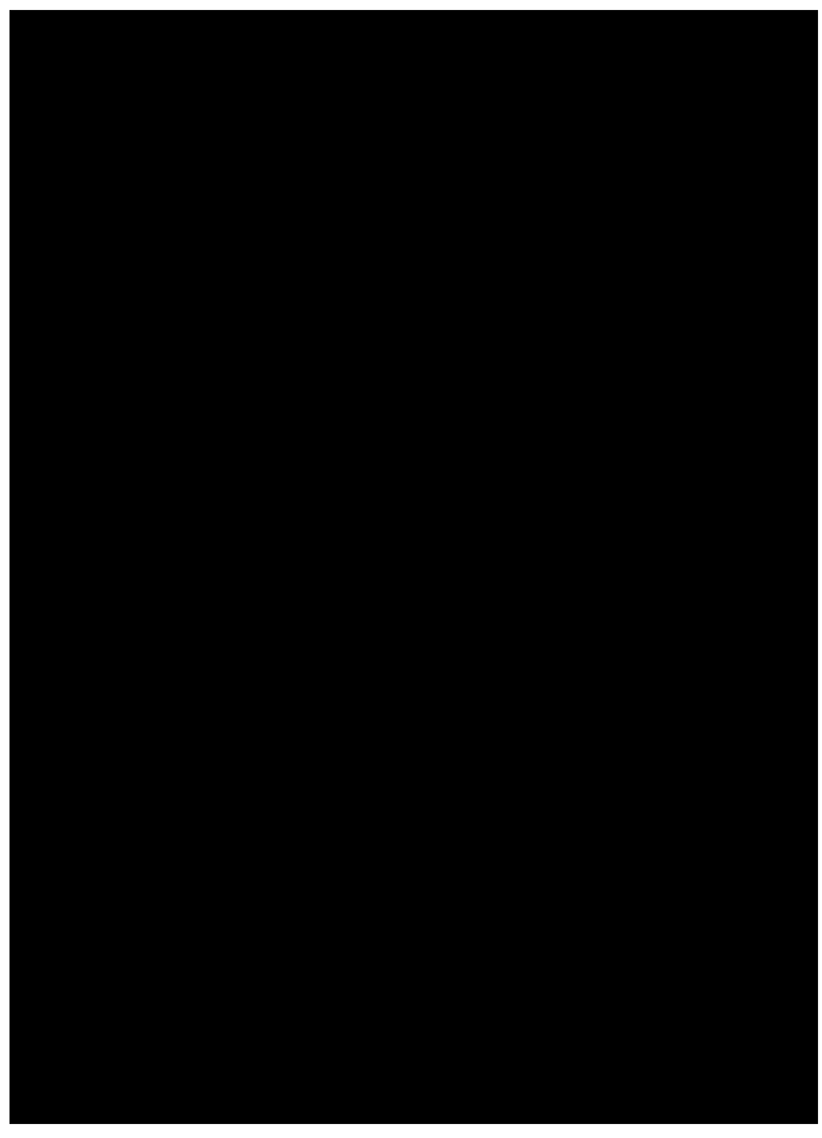


Figure 4

PCDD/DF Flow Chart

Add CRS , ~100 uL C14
Exchange 2X with 50-100 mL hexane
Conc to C14
(Adjust to 100mL Hexane for AP)

TCDD/F Effluents only:

ABSG/AA

Conc to <10 mL
Transfer to conical
containing RS & 10 uL C_{14}
Conc to C_{14}

PCDD/DF Analysis

Average samples:

ABSG/AA

Add ~100 uL C_{14}
Conc to C_{14}

F

Conc to <10 mL
Transfer to conical
containing RS & 10 uL C_{14}
Conc to C_{14}

PCDD/DF Analysis

Very complex samples:

A/P

Conc to C_{14}

ABSG/AA

Add ~100 uL C_{14}
Conc to C_{14}

F

Conc to <10 mL
Transfer to conical
containing RS & 10 uL C
Conc to C_{14}

PCDD/DF Analysis

Table 1

Theoretical Ion Abundance Ratios and Their Control Limits for PCDDs and PCDFs

Number of Chlorine Atoms	Ion Type	Theoretical Ratio	Control Limits [1]	
			Lower	Upper
4 [2]	M/M+2	0.77	0.65	0.89
5 (CDD) [5]	M/M+2	0.63	0.54	0.72
5 (CDF)	M+2/M+4	1.55	1.32	1.78
6	M+2/M+4	1.24	1.05	1.43
6 [3]	M/M+2	0.51	0.43	0.59
7	M+2/M+4	1.05	0.88	1.20
7 [4]	M/M+2	0.44	0.37	0.51
8	M+2/M+4	0.89	0.76	1.02

(1) Represents ± 15% windows around the theoretical ion abundance ratios

(2) Does not apply to $^{37}Cl_4$-2,3,7,8-TCDD (cleanup standard)

(3) Used for ^{13}C-HxCDF

(4) Used for ^{13}C-HpCDF

(5) Modified to mitigate PCB interference

Table 2

Retention Time Window Defining Solution and Isomer Specificity Test Standard

Primary Column	First Eluter	Last Eluter
TCDF	1,3,6,8-	1,2,8,9-
TCDD	1,3,6,8-	1,2,8,9-
PeCDF	1,3,4,6,8-	1,2,3,8,9-
PeCDD	1,2,4,7,9-	1,2,3,8,9-
HxCDF	1,2,3,4,6,8-	1,2,3,7,8,9-
HxCDD	1,2,4,6,7,9-	1,2,3,7,8,9-
HpCDF	1,2,3,4,6,7,8-	1,2,3,4,7,8,9-
HpCDD	1,2,3,4,6,7,9-	1,2,3,4,6,7,8-

TCDD Specificity Test Standard	TCDF Isomer Specificity Test Standard
1,2,3,7 + 1,2,3,8-TCDD	2,3,4,7-TCDF
2,3,7,8-TCDD	2,3,7,8-TCDF
1,2,3,9-TCDD	1,2,3,9-TCDF

Table 3

Calibration Curve Concentration (pg/µL)

Native CDDs and CDFs	CS0	CS1	CS2	CS3*	CS4	CS5
2,3,7,8-TCDD	0.25	0.5	2	10	40	300
2,3,7,8-TCDF	0.25	0.5	2	10	40	300
1,2,3,7,8-PeCDD	1.25	2.5	10	50	200	1500
1,2,3,7,8-PeCDF	1.25	2.5	10	50	200	1500
2,3,4,7,8-PeCDF	1.25	2.5	10	50	200	1500
1,2,3,4,7,8-HxCDD	1.25	2.5	10	50	200	1500
1,2,3,6,7,8-HxCDD	1.25	2.5	10	50	200	1500
1,2,3,7,8,9-HxCDD	1.25	2.5	10	50	200	1500
1,2,3,4,7,8-HxCDF	1.25	2.5	10	50	200	1500
1,2,3,6,7,8-HxCDF	1.25	2.5	10	50	200	1500
1,2,3,7,8,9-HxCDF	1.25	2.5	10	50	200	1500
2,3,4,6,7,8-HxCDF	1.25	2.5	10	50	200	1500
1,2,3,4,6,7,8-HpCDD	1.25	2.5	10	50	200	1500
1,2,3,4,6,7,8-HpCDF	1.25	2.5	10	50	200	1500
1,2,3,4,7,8,9-HpCDF	1.25	2.5	10	50	200	1500
OCDD	2.5	5	20	100	400	3000
OCDF	2.5	5	20	100	400	3000
Labeled Compounds						
^{13}C-2,3,7,8-TCDD	100	100	100	100	100	100
^{13}C-2,3,7,8-TCDF	100	100	100	100	100	100
^{13}C-1,2,3,7,8-PeCDD	100	100	100	100	100	100
^{13}C-1,2,3,7,8-PeCDF	100	100	100	100	100	100
^{13}C-2,3,4,7,8-PeCDF	100	100	100	100	100	100
^{13}C-1,2,3,4,7,8-HxCDD	100	100	100	100	100	100
^{13}C-1,2,3,6,7,8-HxCDD	100	100	100	100	100	100
^{13}C-1,2,3,4,7,8-HxCDF	100	100	100	100	100	100
^{13}C-1,2,3,6,7,8-HxCDF	100	100	100	100	100	100
^{13}C-1,2,3,7,8,9-HxCDF	100	100	100	100	100	100
^{13}C-2,3,4,6,7,8-HxCDF	100	100	100	100	100	100
^{13}C-1,2,3,4,6,7,8-HpCDD	100	100	100	100	100	100
^{13}C-1,2,3,4,6,7,8-HpCDF	100	100	100	100	100	100
^{13}C-1,2,3,4,7,8,9-HpCDF	100	100	100	100	100	100
^{13}C-OCDD	200	200	200	200	200	200
^{13}C-OCDF	200	200	200	200	200	200
Cleanup Recovery Standard						
$^{37}Cl_4$-2,3,7,8-TCDD	0.25	0.5	2.0	10	40	200
Recovery Standard						
^{13}C-1,2,3,4-TCDD	100	100	100	100	100	100
^{13}C-1,2,3,4-TCDF	100	100	100	100	100	100
^{13}C-1,2,3,4,6,9-HxCDF	100	100	100	100	100	100

* Calibration Verification Solution

Table 4
Acceptance Criteria for Performance Tests

CDD/CDF	Conc. (ng/mL)	IPR		OPR (ng/mL)	VER (ng/mL)
		SD (ng/mL)	X (ng/mL)		
2,3,7,8-TCDD	10	2.8	8.3-12.9	6.7-15.8	7.8-12.9
2,3,7,8-TCDF	10	2.0	8.7-13.7	7.5-15.8	8.4-12.3
1,2,3,7,8-PeCDD	50	7.5	38-66	35-71	39-65
1,2,3,7,8-PeCDF	50	7.5	43-62	40-67	41-60
2,3,4,7,8-PeCDF	50	8.6	36-75	34-80	41-61
1,2,3,4,7,8-HxCDD	50	9.4	39-76	35-82	39-64
1,2,3,6,7,8-HxCDD	50	7.7	42-62	38-67	39-64
1,2,3,7,8,9-HxCDD	50	11.1	37-71	32-81	41-61
1,2,3,4,7,8-HxCDF	50	8.7	41-59	36-67	45-56
1,2,3,6,7,8-HxCDF	50	6.7	46-60	42-65	44-57
1,2,3,7,8,9-HxCDF	50	6.4	42-61	39-65	45-56
2,3,4,6,7,8-HxCDF	50	7.4	37-74	35-78	44-57
1,2,3,4,6,7,8-HpCDD	50	7.7	38-65	35-70	43-58
1,2,3,4,6,7,8-HpCDF	50	6.3	45-56	41-61	45-55
1,2,3,4,7,8,9-HpCDF	50	8.1	43-63	39-69	43-58
OCDD	100	19	89-127	78-144	79-126
OCDF	100	27	74-146	63-170	63-159
$^{13}C_{12}$-2,3,7,8-TCDD	100	37	28-134	20-175	82-121
$^{13}C_{12}$-2,3,7,8-TCDF	100	35	31-113	22-152	71-140
$^{13}C_{12}$-1,2,3,7,8-PeCDD	100	39	27-184	21-227	62-160
$^{13}C_{12}$-1,2,3,7,8-PeCDF	100	34	27-156	21-192	76-130
$^{13}C_{12}$-2,3,4,7,8-PeCDF	100	38	16-279	13-328	77-130
$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	100	41	29-147	21-193	85-117
$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	100	38	34-122	25-163	85-118
$^{13}C_{12}$-1,2,3,7,8,9-HxCDD	100	41	29-147	21-193	85-118
$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	100	43	27-152	19-202	76-131
$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	100	35	30-122	21-159	70-143
$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	100	40	24-157	17-205	74-135
$^{13}C_{12}$-2,3,4,6,7,8-HxCDF	100	37	29-136	22-176	73-137
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	100	35	34-129	26-166	72-138
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	100	41	32-110	21-158	78-129
$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	100	40	28-141	20-186	77-129
$^{13}C_{12}$-OCDD	200	95	41-276	26-397	96-415
$^{13}C_{12}$-OCDF	200	95	41-276	26-397	96-415
$^{37}Cl_4$-2,3,7,8-TCDD	10	3.6	3.9-15.4	3.1-19.1	7.9-12.7

Table 4A

Acceptance Criteria for Performance Tests (Tetra Compounds only)

CDD/CDF	Conc. (ng/mL)	IPR		OPR (ng/mL)	VER (ng/mL)
		SD (ng/mL)	X (ng/mL)		
2,3,7,8-TCDD	10	2.7	8.7-12.4	7.3-14.6	8.2-12.3
2,3,7,8-TCDF	10	2.0	9.1-13.1	8.0-14.7	8.6-11.6
$^{13}C_{12}$-2,3,7,8-TCDD	100	35	32-115	25-141	85-117
$^{13}C_{12}$-2,3,7,8-TCDF	100	34	35-99	26-126	76-131
$^{37}Cl_4$-2,3,7,8-TCDD	10	3.4	4.5-13.4	3.7-15.8	8.3-12.1

Table 5

Labeled Compound Recovery in Samples

CDD/CDF	Conc. (ng/mL)	Labeled Recovery (ng/mL)	% Recovery
$^{13}C_{12}$-2,3,7,8-TCDD	100	25-164	25-164
$^{13}C_{12}$-2,3,7,8-TCDF	100	24-169	24-169
$^{13}C_{12}$-1,2,3,7,8-PeCDD	100	25-181	25-181
$^{13}C_{12}$-1,2,3,7,8-PeCDF	100	24-185	24-185
$^{13}C_{12}$-2,3,4,7,8-PeCDF	100	21-178	21-178
$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	100	32-141	32-141
$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	100	28-130	28-130
$^{13}C_{12}$-1,2,3,7,8,9-HxCDD	100	32-141	32-141
$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	100	26-152	26-152
$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	100	26-123	26-123
$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	100	29-147	29-147
$^{13}C_{12}$-2,3,4,6,7,8-HxCDF	100	28-136	28-136
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	100	23-140	23-140
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	100	28-143	28-143
$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	100	26-138	26-138
$^{13}C_{12}$-OCDD	200	34-313	17-157
$^{13}C_{12}$-OCDF	200	34-313	17-157
$^{37}Cl_4$-2,3,7,8-TCDD	10	3.5-19.7	35-197

Table 5A

Labeled Compound Recovery in Samples (Tetra Compounds only)

CDD/CDF	Conc. (ng/mL)	Labeled Recovery (ng/mL)	% Recovery
$^{13}C_{12}$-2,3,7,8-TCDD	100	31-137	31-137
$^{13}C_{12}$-2,3,7,8-TCDF	100	29-140	29-140
$^{37}Cl_4$-2,3,7,8-TCDD	10	4.2-16.4	42-164

Table 6

Toxic Equivalency Factors

Analyte	I-89[1]	WHO-2005[2]
2,3,7,8-TCDD	1	1
1,2,3,7,8-PeCDD	0.5	1
1,2,3,4,7,8-HxCDD	0.1	0.1
1,2,3,6,7,8-HxCDD	0.1	0.1
1,2,3,7,8,9-HxCDD	0.1	0.1
1,2,3,4,6,7,8-HpCDD	0.01	0.01
OCDD	0.001	0.0003
2,3,7,8-TCDF	0.1	0.1
1,2,3,7,8-PeCDF	0.05	0.03
2,3,4,7,8-PeCDF	0.5	0.3
1,2,3,4,7,8-HxCDF	0.1	0.1
1,2,3,6,7,8-HxCDF	0.1	0.1
2,3,4,6,7,8-HxCDF	0.1	0.1
1,2,3,7,8,9-HxCDF	0.1	0.1
1,2,3,4,6,7,8-HpCDF	0.01	0.01
1,2,3,4,7,8,9-HpCDF	0.01	0.01
OCDF	0.001	0.0003

[1] Taken from “Interim Procedures for Estimating Risks Associated with Exposures to Mixtures of Chlorinated Dibenzo-p-Dioxin and –Dibenzofurans (CDDs and CDFs) and 1989 Update”, (EPA/625/3-89/016, March 1989).

[2] Taken from “The 2005 World Health Organization Re-evaluation of Human and Mammalian Toxic Equivalency Factors for Dioxin and Dioxin-like Compounds”. (Van den Berg et al)

Table 7

Descriptors, Exact M/Zs, M/Z Types, & Elemental Compositions of the CDDs/CDFs

Descriptor	Exact M/Z[1]	M/Z Type	Elemental Composition	Substance[2]
1	292.9825	LOCK	C_7F_{11}	PFK
	303.9016	M	$C_{12}H_4{}^{35}Cl_4O$	TCDF
	305.8987	M+2	$C_{12}H_4{}^{35}Cl_3{}^{37}ClO$	TCDF
	315.9419	M	${}^{13}C_{12}H_4{}^{35}Cl_4O$	TCDF[3]
	317.9389	M+2	${}^{13}C_{12}H_4{}^{35}Cl_3{}^{37}ClO$	TCDF[3]
	319.8965	M	$C_{12}H_4{}^{35}Cl_4O_2$	TCDD
	321.8936	M+2	$C_{12}H_4{}^{35}Cl_3{}^{37}Cl$	TCDD
	327.8847	M	$C_{12}H_4{}^{37}Cl_4O_2$	TCDD[4]
	330.9792	QC	C_7F_{13}	PFK
	331.9368	M	${}^{13}C_{12}H_4{}^{35}Cl_4O_2$	TCDD[3]
	333.9339	M+2	${}^{13}C_{12}H_4{}^{35}Cl_3{}^{37}ClO_2$	TCDD[3]
	375.8364	M+2	$C_{12}H_4{}^{35}Cl_5{}^{37}ClO$	HxCDPE
	339.8597	M+2	$C_{12}H_3{}^{35}Cl_4{}^{37}ClO$	PeCDF
	341.8568	M+4	$C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O$	PeCDF
2	339.8597	M+2	$C_{12}H_3{}^{35}Cl_4{}^{37}ClO$	PeCDF
	341.8568	M+4	$C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O$	PeCDF
	351.9000	M+2	$C_{12}H_3{}^{35}Cl_4{}^{37}Cl_2O$	PeCDF
	353.8970	M+4	$C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O$	PeCDF[3]
	366.9792	LOCK	C_9F_{13}	PFK
	353.8576	M	$C_{12}H_3{}^{35}Cl_4{}^{37}ClO_2$	PeCDD
	355.8546	M+2	$C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O_2$	PeCDD
	365.8970	M	${}^{13}C_{12}H_3{}^{35}Cl_4{}^{37}ClO_2$	PeCDD[3]
	367.8949	M+2	${}^{13}C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O_2$	PeCDD[3]
	409.7974	M+2	$C_{12}H_3{}^{35}Cl_6{}^{37}ClO$	HpCDPE
3	373.8208	M+2	$C_{12}H_2{}^{35}Cl_5{}^{37}ClO$	HxCDF
	375.8178	M+4	$C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O$	HxCDF
	383.8639	M	$C_{12}H_2{}^{35}Cl_5{}^{37}ClO$	HxCDF[3]
	385.8610	M+2	$C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O$	HxCDF[3]
	389.8157	M+2	$C_{12}H_2{}^{35}Cl_5{}^{37}ClO_2$	HxCDD
	391.8127	M+4	$C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O_2$	HxCDD
	380.9760	LOCK	C_9F_{15}	PFK
	401.8559	M+2	${}^{13}C_{12}H_2{}^{35}Cl_5{}^{37}ClO_2$	HxCDD[3]
	403.8530	M+4	${}^{13}C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O_2$	HxCDD[3]
	430.9729	QC	C_9F_{17}	PFK
	445.7555	M+4	$C_{12}H_2{}^{35}Cl_6{}^{37}Cl_2O$	OCDPE
4	407.7818	M+2	$C_{12}H_2{}^{35}Cl_6{}^{37}ClO$	HpCDF
	409.7789	M+4	$C_{12}H_2{}^{35}Cl_5{}^{37}Cl_2O$	HpCDF
	417.8253	M	${}^{13}C_{12}H{}^{35}Cl_7O$	HpCDF[3]
	419.8220	M+2	${}^{13}C_{12}H{}^{35}Cl_6{}^{37}ClO$	HpCDF[3]
	423.7766	M+2	$C_{12}H{}^{35}Cl_6{}^{37}ClO_2$	HpCDD
	425.7737	M+4	$C_{12}H{}^{35}Cl_5{}^{37}Cl_2O_2$	HpCDD
	430.9728	LOCK	C_9F_{17}	PFK
	435.8169	M+2	${}^{13}C_{12}H{}^{35}Cl_6{}^{37}ClO_2$	HpCDD[3]

Table 7

Descriptors, Exact M/Zs, M/Z Types, & Elemental Compositions of the CDDs/CDFs

Descriptor	Exact M/Z[1]	M/Z Type	Elemental Composition	Substance[2]
	437.8140	M+4	${}^{13}C_{12}H^{35}Cl_5{}^{37}Cl_2O_2$	HpCDD[3]
	479.7165	M+4	$C_{12}H^{35}Cl_7{}^{37}Cl_2O$	NCDPE

Table 7

Descriptors, Exact M/Zs, M/Z Types, & Elemental Compositions of the CDDs/CDFs

Descriptor	Exact M/Z[1]	M/Z Type	Elemental Composition	Substance[2]
5	441.7428	M+2	$C_{12}{}^{35}Cl_7{}^{37}ClO$	OCDF
	442.9728	LOCK	$C_{10}F_{17}$	PFK
	443.7399	M+4	$C_{12}{}^{35}Cl_6{}^{37}Cl_2O$	OCDF
	457.7377	M+2	$C_{12}{}^{35}Cl_7{}^{37}ClO_2$	OCDD
	459.7348	M+4	$C_{12}{}^{35}Cl_6{}^{37}Cl_2O_2$	OCDD
	469.7779	M+2	${}^{13}C_{12}{}^{35}Cl_7{}^{37}ClO_2$	OCDD[3]
	471.7750	M+4	${}^{13}C_{12}{}^{35}Cl_6{}^{37}Cl_2O_2$	OCDD[3]
	453.7831	M+2	${}^{13}C_{12}{}^{35}Cl_7{}^{37}ClO$	OCDF[3]
	455.7802	M+4	${}^{13}C_{12}{}^{35}Cl_6{}^{37}Cl_2O$	OCDF[3]
	513.6775	M+4	$C_{12}{}^{3}5Cl_8{}^{37}Cl_2O$	DCDPE

[1] Nuclidic masses used:

H = 1.007825 C = 12.00000 ${}^{13}C$ = 13.003355 F = 18.9984
O = 15.994915 ${}^{35}Cl$ = 34.968853 ${}^{37}Cl$ = 36.965903

[2] TCDD = Tetrachlorodibenzo-*p*-dioxin
PeCDD = Pentachlorodibenzo-*p*-dioxin
HxCDD = Hexachlorodibenzo-*p*-dioxin
HpCDD = Heptachlorodibenzo-*p*-dioxin
OCDD = Octachlorodibenzo-*p*-dioxin
HxCDPE = Hexachlorodiphenyl ether
OCDPE = Octachlorodiphenyl ether
DCDPE = Decachlorodiphenyl ether
TCDF = Tetrachlorodibenzofuran
PeCDF = Pentachlorodibenzofuran
HxCDF = Hexachlorodibenzofuran
HpCDF = Heptachlorodibenzofuran
OCDF = Octachlorodibenzofuran
HpCDPE = Heptachlorodiphenyl ether
NCDPE = Nonachlorodiphenyl ether
PFK = Perfluorokerosene

[3] Labeled compound.

[4] There is only one m/z for ${}^{37}Cl$-2,3,7,8,-TCDD (cleanup standard).

Table 8
Quantitation Limits

CDD/CDF	QL Aqueous (pg/L)	QL Solid (pg/g)	QL Tissue (pg/g)
2,3,7,8-TCDD	5.0	0.50	0.2
2,3,7,8-TCDF	5.0	0.50	0.2
1,2,3,7,8-PeCDD	25.0	2.5	1.0
1,2,3,7,8-PeCDF	25.0	2.5	1.0
2,3,4,7,8-PeCDF	25.0	2.5	1.0
1,2,3,4,7,8-HxCDD	25.0	2.5	1.0
1,2,3,6,7,8-HxCDD	25.0	2.5	1.0
1,2,3,7,8,9-HxCDD	25.0	2.5	1.0
1,2,3,4,7,8-HxCDF	25.0	2.5	1.0
1,2,3,6,7,8-HxCDF	25.0	2.5	1.0
1,2,3,7,8,9-HxCDF	25.0	2.5	1.0
2,3,4,6,7,8-HxCDF	25.0	2.5	1.0
1,2,3,4,6,7,8-HpCDD	25.0	2.5	1.0
1,2,3,4,6,7,8-HpCDF	25.0	2.5	1.0
1,2,3,4,7,8,9-HpCDF	25.0	2.5	1.0
OCDD	50.0	5.0	2.0
OCDF	50.0	5.0	2.0

Table 9

Relative Retention Times

CDD/CDF	Relative Retention Time
2,3,7,8-TCDF	0.999-1.003
2,3,7,8-TCDD	0.999-1.002
1,2,3,7,8-PeCDD	0.999-1.002
1,2,3,7,8-PeCDF	0.999-1.002
2,3,4,7,8-PeCDF	0.999-1.002
$^{13}C_{12}$-2,3,7,8-TCDF	0.923-1.103
$^{13}C_{12}$-2,3,7,8-TCDD	0.976-1.043
$^{37}Cl_{4}$-2,3,7,8-TCDD	0.989-1.052
$^{13}C_{12}$-1,2,3,7,8-PeCDF	1.000-1.425
$^{13}C_{12}$-2,3,4,7,8-PeCDF	1.011-1.526
$^{13}C_{12}$-1,2,3,7,8-PeCDD	1.000-1.567
1,2,3,4,7,8-HxCDF	0.999-1.001
1,2,3,6,7,8-HxCDF	0.997-1.005
1,2,3,7,8,9-HxCDF	0.999-1.001
2,3,4,6,7,8-HxCDF	0.999-1.001
1,2,3,4,7,8-HxCDD	0.999-1.001
1,2,3,6,7,8-HxCDD	0.998-1.004
1,2,3,7,8,9-HxCDD	1.000-1.019
1,2,3,4,6,7,8-HpCDF	0.999-1.001
1,2,3,4,7,8,9-HpCDF	0.999-1.001
1,2,3,4,6,7,8-HpCDD	0.999-1.001
OCDD	0.999-1.001
OCDF	0.999-1.001
$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	0.975-1.001
$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	0.979-1.005
$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	1.002-1.072
$^{13}C_{12}$-2,3,4,6,7,8-HxCDF	1.001-1.020
$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	1.002-1.026
$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	1.007-1.029
$^{13}C_{12}$-1,2,3,7,8,9-HxCDD	1.014-1.038
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	1.069-1.111
$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	1.098-1.192
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	1.117-1.141
$^{13}C_{12}$-OCDD	1.085-1.365
$^{13}C_{12}$-OCDF	1.091-1.371

Table 10. - Quality Control Requirements For DIOXIN/FURANS by USEPA Method 1613					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Instrument Tuning	Beginning and end of each shift	The instrument is tuned to the minimum required resolving power of 10,000 (10% valley) at a reference signal close to m/z 303.9016. The mass resolution check is achieved before any analysis is performed for each 12-hour clock. An appropriate lock mass will be monitored for each descriptor and shall not vary by more than ± 20% throughout the respective retention time window. The suggested ions to monitor for PCDD/Fs and Chlorinated Diphenyl Ethers (CDPEs) are listed in Table 7. CDPEs are known interferences for the PCDFs.	If the resolution of 10,000 is not achieved then all samples analyzed within the 12-hour clock must be re-analyzed. If the lock mass varies by more than +20%, the data must be evaluated to determine whether the batch shall be re-extracted or the data are qualified appropriately If CDPEs are detected in a sample, then the data must be qualified appropriately Dilute and re-analyze	Not applicable	
Initial Calibration (ICAL)	An initial calibration is repeated whenever a new set of spiking calibration standards are created or whenever the continuing calibration falls outside the acceptance criteria	The absolute retention time of $^{13}C_{12}$-1,2,3,4-TCDD must exceed 25.0 minutes on the ZB-5MS column and 15 minutes on the DB-225 column. The first and last PCDD/PCDF eluters must be verified in accordance with Table 2. The chromatographic peak separation between 2,3,7,8-TCDD and the closest eluting	If this criteria are not achieved, a new initial calibration curve must be re-injected or prepared	Not applicable	All initial instrument calibrations are verified with a standard (CS3) from a second manufacturer or lot.

Table 10. - Quality Control Requirements For DIOXIN/FURANS by USEPA Method 1613					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Initial Calibration (ICAL)		isomers must be separated with a valley of ≤25%. The signal to noise ratio (s/n) exceeds 10:1 for all ions monitored. The ion abundance ratio measurements are within ± 15% of the theoretical ratio. The %RSD for the mean response factors must be within ±20% for the native standards and within ±30% for internal standards.			
Initial Calibration Verification (ICV)/Continuing Calibration	Prior to running samples/with every batch	The chromatographic peak resolution between 2,3,7,8-TCDD and the closest eluting isomers must be resolved with a valley of ≤25%. If the resolution requirement is not met, then the GC column is trimmed, inner cleaned, or column changed and the extracts re-analyzed. The first and last PCDD/F eluters are verified to be within the eight homologue retention time windows. The concentrations and % recoveries for the native and labeled compounds respectively for the mid-range standard are within the limits of Table 4 (or Table 4A for	If the ratios do not meet the acceptance criteria, then the instrument must be recalibrated and the affected samples should be reanalyzed. If the s/n ratio is not met, then associated extracts should be re-analyzed. If the retention times are not obtained, the associated extracts should be re-analyzed. If the retention times are not obtained, the extracts should be re-analyzed.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Refer to laboratory flagging criteria and project requirements.	Arizona compliance samples will have a LFB analyzed at the low point of the calibration curve daily.

Table 10. - Quality Control Requirements For DIOXIN/FURANS by USEPA Method 1613					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Initial Calibration Verification (ICV)/Continuing Calibration		2,3,7,8-TCDD/F only). The ion ratios are within the criteria listed in Table 1. The signal to noise ratio (s/n) exceeds 10:1 for all ions monitored. The retention times shall be within ±15 seconds of the retention times obtained during initial calibration for ^{13}C-1,2,3,4-TCDD and ^{13}C-1,2,3,4,6,9-HxCDF recovery standards. The relative retention times of the unlabeled and labeled PCDDs and PCDFs shall be within the limits given Table 9.			
Method Blank (MB)	With every analytical batch or 20 samples, whichever is less, per matrix type	≤ minimum level or one-third of the regulatory compliance limit, whichever is greater; Or ≤ minimum level for each congener	If the amount found is greater than the minimum level or one-third the regulatory compliance limit, whichever is greater; or if any potentially interfering compound is found in the blank at or above the minimum level for each congener, the data must be evaluated to determine whether the batch shall be re-extracted or the data is qualified appropriately. If there is evidence of contamination within the	Refer to flagging criteria and project requirements.	

Table 10. - Quality Control Requirements For DIOXIN/FURANS by USEPA Method 1613					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Method Blank (MB)			MB, then the source of the contamination must be located. The data must be evaluated to determine whether the batch shall be re-extracted or the data is qualified appropriately		
Matrix Spike (MS)	By client request or to fulfill state agency requirements	The relative percent difference between MS/MSD samples should be ≤25%	If the criteria are not met, the data must be evaluated to determine whether the samples shall be re-extracted or the data are qualified appropriately.	Not applicable	Projects performed pursuant to the guidelines established by the DOD QSM shall contain an associated Matrix Spike per preparatory batch. A Matrix Spike Duplicate or Laboratory Duplicate shall also be analyzed per preparatory batch for these projects
Matrix Spike Duplicate (MSD) or Matrix Duplicate (MD)	Per client request only or to fulfill state agency requirements	The relative percent difference between MS/MSD samples should be ≤25%	If the criteria are not met, the data must be evaluated to determine whether the samples shall be re-extracted or the data are qualified appropriately.	Not applicable	For DoD projects: Refer to QSM limits. MSD or MD: RPD of all analytes ≤ 20% (between MS and MSD or sample and MD).
Internal Standards	Added to every sample	Recovery of each internal standard versus the recovery standard must be within the limits established in Table 4. Recovery of each internal standard versus the recovery standard for the analysis of TCDD/TCDF only must be within the limits established in Table 4A.	Recoveries below the limits may be accepted if the signal to noise is >10:1. If the signal to noise is not >10:1, samples must be re-extracted and re-analyzed or the data must be qualified.	Not applicable	

Table 10. - Quality Control Requirements For DIOXIN/FURANS by USEPA Method 1613					
QC Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**	**Flagging Criteria**	**Comments**
Internal Standards		The %RSD for the mean response factors must be within ±30% for internal standards.			
Ongoing Precision and Recovery (OPR)	OPR must be analyzed prior to samples With every analytical batch per matrix Analyze the method or solvent blank immediately after analysis of the OPR to demonstrate freedom from contamination	For each native and labeled compound, compare the concentration with the limits for ongoing accuracy in Table 4. Labeled compound recoveries should meet the criteria in Table 5.	If IS is low and native compounds are in, assess the data and determine whether to re-extract or narrate. If native compounds are out, re-extract.	Not applicable	

Glossary

Analyte — A CDD or CDF tested for by this method. The analytes are listed in Table 1.

Calibration Standard (CAL) — A solution prepared from a secondary standard and/or stock solutions and used to calibrate the response of the instrument with respect to analyte concentration.

Calibration Verification Standard (VER) — The mid-point calibration standard (CS3) that is used in to verify calibration. See Table 3.

CDD — Chlorinated Dibenzo-p-Dioxin — The isomers and congeners of tetra- through octa-chlorodibenzo-p-dioxin.

CDF — Chlorinated Dibenzofuran — The isomers and congeners of tetra- through octa-chlorodibenzofuran.

CS0, CS1, CS2, CS3, CS4, CS5 — See Calibration standards and Table 3.

Field Blank — An aliquot of reagent water or other reference matrix that is placed in a sample container in the laboratory or the field, and treated as a sample in all respects, including exposure to sampling site conditions, storage, preservation, and all analytical procedures. The purpose of the field blank is to determine if the field or sample transporting procedures and environments have contaminated the sample.

GC — Gas chromatograph or gas chromatography.

HPLC — High performance liquid chromatograph or high performance liquid chromatography.

HRGC — High resolution GC.

HRMS — High resolution MS.

IPR — Initial precision and recovery; four aliquots of the diluted PAR standard analyzed to establish the ability to generate acceptable precision and accuracy. An IPR is performed prior to the first time this method is used and any time the method or instrumentation is modified.

Laboratory Blank — See method blank.

Laboratory Control sample (LCS) — See ongoing precision and recovery standard (OPR).

Laboratory Reagent Blank — See method blank.

May — This action, activity, or procedural step is neither required nor prohibited.

May Not — This action, activity, or procedural step is prohibited.

Method Blank — An aliquot of reagent water that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with samples. The method blank is used to determine if analytes or interferences are present in the laboratory environment, the reagents, or the apparatus.

Minimum Level (ML) — The level at which the entire analytical system must give a recognizable signal and acceptable calibration point for the analyte. It is equivalent to the concentration of the lowest calibration standard, assuming that all method-specified sample weights, volumes, and cleanup procedures have been employed.

MS — Mass spectrometer or mass spectrometry.

Must — This action, activity, or procedural step is required.

OPR — Ongoing precision and recovery sample (OPR); a laboratory blank spiked with known

quantities of analytes. The OPR is analyzed exactly like a sample. Its purpose is to assure that the results produced by the laboratory remain within the limits specified in this method for precision and recovery.

PAR — Precision and recovery standard; secondary standard that is diluted and spiked to form the IPR and OPR.

PFK — Perfluorokerosene; the mixture of compounds used to calibrate the exact m/z scale in the HRMS.

Preparation Blank — See method blank.

Primary Dilution Standard — A solution containing the specified analytes that is purchased or prepared from stock solutions and diluted as needed to prepare calibration solutions and other solutions.

Quality Control Check Sample (QCS) — A sample containing all or a subset of the analytes at known concentrations. The QCS is obtained from a source external to the laboratory or is prepared from a source of standards different from the source of calibration standards. It is used to check laboratory performance with test materials prepared external to the normal preparation process.

Reagent Water — Water demonstrated to be free from the analytes of interest and potentially interfering substances at the method detection limit for the analyte.

Relative Standard Deviation (RSD) — The standard deviation times 100 divided by the mean. Also termed "coefficient of variation."

RF — Response factor.

RSD — See relative standard deviation.

SDS — Soxhlet/Dean-Stark extractor; an extraction device applied to the extraction of solid and semi-solid materials.

SICP — Selected ion current profile; the line described by the signal at an exact m/z.

SPE — Solid-phase extraction; an extraction technique in which an analyte is extracted from an aqueous sample by passage over or through a material capable of reversibly adsorbing the analyte. Also termed liquid-solid extraction.

Stock Solution — A solution containing an analyte that is prepared using a reference material traceable to EPA, the National Institute of Science and Technology (NIST), or a source that will attest to the purity and authenticity of the reference material.

TCDD — Tetrachlorodibenzo-p-dioxin.

TCDF — Tetrachlorodibenzofuran.

VER — See calibration verification standard.

SOP 26, Revision 13 / SOP 31, Revision 12	Amendment
22. POLYCHLORINATED DIBENZO DIOXIN/FURANS BY USEPA METHOD 1613B **PREPARATION AND ANALYSIS OF POLYCHLORINATED BIPHENYLS (PCBS) BY METHOD 1668A/C**	
Management: Martha Maier	
Quality Assurance: Meglech Tet	
Effective Date: 10/20/2015	

Description of Amendment
Requirements for extraction and analysis of samples from the State of Wisconsin: All samples from the State of Wisconsin shall be analyzed only on instruments with a valid LOD study for that parameter, matrix, and method, i.e., DoD instruments. All samples from the State of Wisconsin shall be extracted with a measurement of precision, such as an MS/MSD, LCSD, or sample duplicate, per batch and precision assessed. All reportable values for samples from the State of Wisconsin shall be within the range of calibration. Concentrations shall not be "E" qualified.

% Lipids for Extraction Set B5G0068

Procedures:

WO: 1500600 | Matrix: Tissue | Chemist: ______

Analysis: Percent Lipids | Prep Date: ______

Vista Sample ID	Sample Wt. Equiv.	Rnd Bottom Wt.	Rnd Bottom & Residue Wt.	Residue Wt.	% Lipids	Comments:
1500600-01						

Notes:

Sample Wt Equiv = $\frac{\%\text{Sample Wt Used} * \text{Sample Wt}}{100}$

% Lipids = $\frac{\text{Residue Wt} * 100}{\text{Sample Wt Equiv}}$

Balance ID ______

PREPARATION BENCH SHEET

Matrix: Tissue

B5H0121

Chemist: ____________________

Method: 1613 Full List

Prep Date/Time: 21-Aug-15 09:02

Prepared using: HRMS - Soxhlet

C	VISTA Sample ID	Sample Amt. (g)	IS/NS CHEM/WIT DATE	CRS CHEM/WIT DATE	ABSG CHEM/ DATE	AA CHEM/ DATE	Florisil CHEM/ DATE	Charcoal CHEM/ DATE	RS CHEM/WIT DATE
☐	B5H0121-BLK1								
☐	B5H0121-BS1								
☐	1500742-01								
☐	1500742-02								
☐	1500742-03								
☐	1500742-04								
☐	1500742-05								
☐	1500742-06								
☐	1500742-07								
☐	1500742-08								
☐	1500742-09								
☐	1500742-10								
☐	1500742-11								
☐	1500742-12								

IS Name	NS Name	CRS Name	RS Name	Cycle Time	APP: SEFUN SOX SDS	Check Out:
PCDD/F ____________	PCDD/F ____________	PCDD/F ____________	PCDD/F ____________	Start Date/Time	SOLV: ____________	Chemist/Date: ____________
PCB ____________	PCB ____________	PCB ____________	PCB ____________	____________	Other ____________	Check In:
PAH ____________	PAH ____________	PAH ____________	PAH ____________	Stop Date/Time	Final Volume(s) ________	ChemistDate: ____________
____________	____________	____________	____________	____________	________	Balance ID: __________

Comments:

PREPARATION BENCH SHEET

Matrix: Tissue

B5G0057

Chemist: ______________________

Method: 1668C Full List

Prep Date/Time: 14-Jul-15 15:41

Prepared using: HRMS - Soxhlet

C	VISTA Sample ID	Sample Amt. (g)	IS/NS CHEM/WIT DATE	CRS CHEM/WIT DATE	ABSG CHEM/ DATE	AA CHEM/ DATE	Florisil CHEM/ DATE	Charcoal CHEM/ DATE	RS CHEM/WIT DATE
☐	B5G0057-BLK1								
☐	B5G0057-BS1								
☐	1500600-01								

IS Name	NS Name	CRS Name	RS Name	Cycle Time	APP: SEFUN SOX SDS	Check Out: Chemist/Date: ______________
PCDD/F _______________	PCDD/F _______________	PCDD/F _______________	PCDD/F _______________	Start Date/Time	SOLV: _______________	
PCB _________________	PCB _________________	PCB _________________	PCB _________________	____________	Other _________________	Check In: ChemistDate: ______________
PAH _________________	PAH _________________	PAH _________________	PAH _________________	Stop Date/Time	Final Volume(s) ________	Balance ID: ___________
_________________	_________________	_________________	_________________	____________	________	

Comments:

Appendix C – Addendum

UCR Benthic Macroinvertebrate Tissue Sampling – Addendum to ALS SOPs for Tissue Processing

UCR Benthic Macroinvertebrate Tissue Sampling – Addendum to ALS SOPs for Tissue Processing

This addendum provides detail for the processing of mussels and crayfish that fall outside of ALS Environmental/Kelso-WA (ALS) general standard operating procedures (SOP). This addendum provides detailed descriptions for processing tissue samples in support of the UCR Benthic Macro-invertebrate Tissue Study and supersedes the general SOPs.

Note: The quality assurance project plan (QAPP) for the project in combination with the following comments supersedes the general SOPs maintained at ALS.

Crayfish Compositing, Dissection, Homogenization, and Sub-sampling

Preliminary Freezing of Samples: If crayfish are frozen in the field prior to shipping to ALS the potential exists for internal organs to rupture. Normally, tissue samples destined for analysis of individual organs or body parts are not frozen prior to dissection, which is consistent with general United States Environmental Protection Agency (EPA) guidelines for the sampling and analysis of biological tissue. If the crayfish are frozen prior to shipment to the laboratory, care will be taken to look for ruptured organs, and separation of material from the affected organs will be done as completely as possible. However, a fully quantitative separation in this case will not be possible without further jeopardizing the integrity of the samples.

Laboratory Freezing of Samples & Conservation Liquid: All samples will be packaged in the field in sealed containers such that no additional water is allowed to enter the container. The samples will be damp from the site water rinse, but free of extraneous water. The samples will be frozen at -20°C upon receipt at the laboratory, as they are continuously maintained in sealed containers. When thawing, any frost or ice that forms inside the sealed container will be allowed to thaw and will be included with the sample homogenates. Additional moisture will not be introduced during the entire process, so conservation of liquid is maintained from field to final homogenate.

Rinsing of Whole Samples: The general sample processing SOP (MET-TISP) states that samples will be rinsed in the laboratory with deionized water (DIW) to removed extraneous material (e.g., sediment, biogenic material, etc.), however, for this project an equivalent step will be performed in the field. Organisms will be rinsed in the field with site water as needed, as specified in the QAPP. The majority of residual water will be removed prior to packaging the samples for shipment, and therefore further rinsing at the laboratory is not necessary.

Sample Login and Initial Preparation of Composites: Individual crayfish will be received at the laboratory with unique identifiers and will be logged as individual samples (i.e. a unique laboratory code is assigned to each uniquely identified sample). Samples for the study will arrive

before finalization of the sample compositing plan. Samples will be logged in and properly stored until the compositing plan is received and samples can be processed.The individual samples identified for each composite sample will be combined at the time dissection and/or homogenization occurs.

Stepwise Details – Compositing, Dissection, and Homogenization:

- The individual samples that make up each composite sample will be identified on the ALS internal work order.
- Prepare certified clean glass containers that will be used for retaining the samples during storage and throughout the freeze-drying process.
- After labeling the containers with the associated sample identifications, record the tare weight of each in the original record log.
- Place the crayfish identified as belonging to the composite onto a Teflon cutting board lined with aluminum foil (dull side in contact with the sample). No DIW rinse is performed.
- Using a sharp filet knife, cut the carapace to expose the internal organs and remove the stomach with a scoopula, taking care to not rupture it. Refer to Figure 1 for anatomical detail.

  Note 1: Inspect the stomach to determine if rupture occurred during preliminary freezing in the field or at the laboratory.

  Note 2: If the stomach is ruptured, separate the residual stomach contents and add that material to the container designated for the carapace + stomach component.

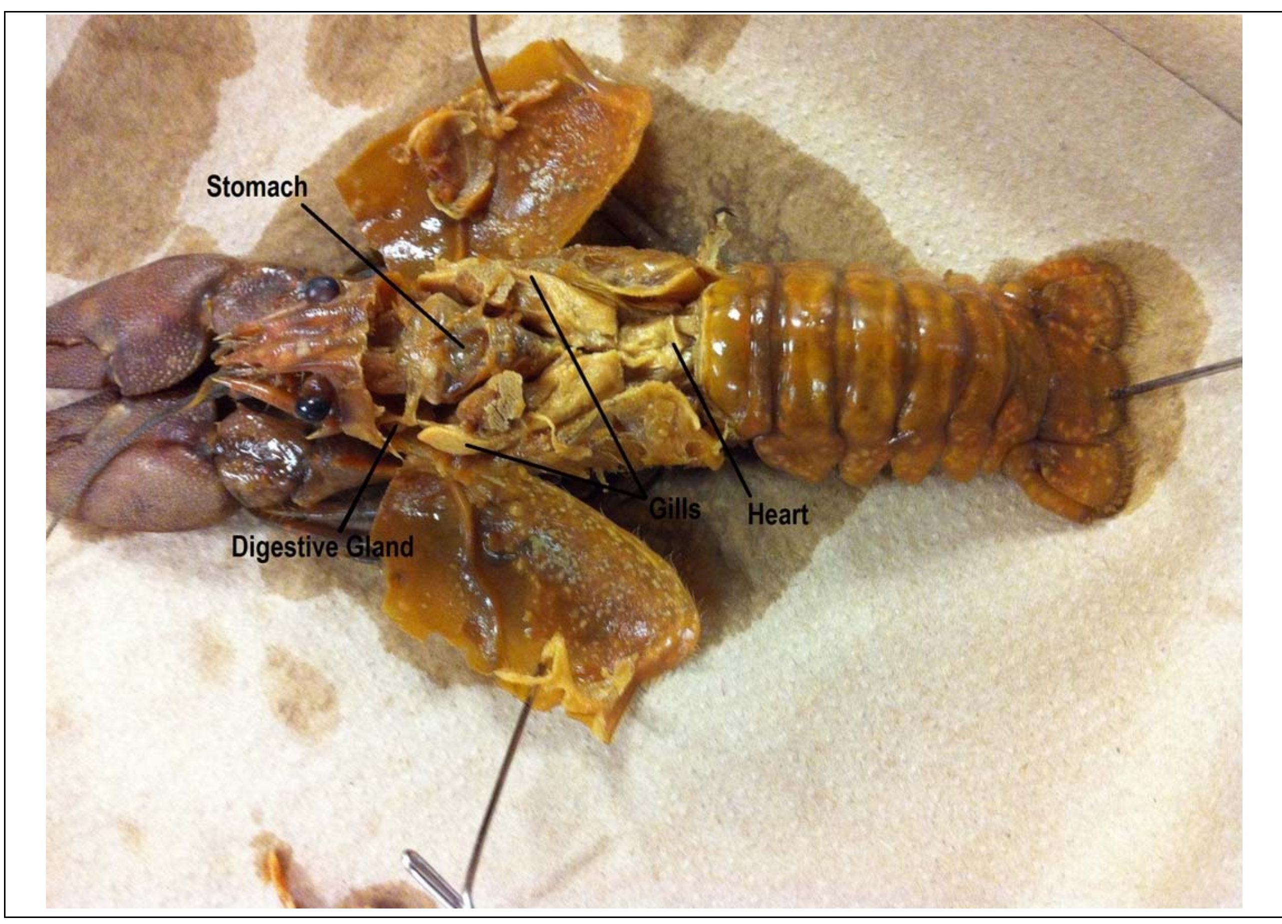


Figure 1. Anatomical detail of crayfish

- Place stomach in the associated tared container.
- Remove the entire carapace from the crayfish, including the portion containing the muscle tissue (after extending the original cut). Separate the tail from the carapace. Use the filet knife in combination with a scoopula or other utensil to separate the shell material from the soft tissue.
- Combine the carapace (excluding the tail) with the stomach in the associated tared container.
- Repeat the process for all individual samples that are identified as components to the composite sample.
- Record the total weight (of the composite) to three significant figures for the stomach+carapace+tare. Calculate the net weight of the stomach+carapace and record in the original record log.

- Transfer the remaining tissue (whole sample minus carapace+stomach, including the head, claws, tail, and shell around claws and tail) to the associated tared container. Carefully remove the foil covering and pour any residual liquid into the same container.
- Record the total weight (of the composite) to three significant figures for the residual tissue+tare. Calculate the net weight of the residual tissue and record in the original record log.
- Cap each container and transfer to a -20°C freezer and allow overnight storage to assure complete freezing.
- When ready to commence freeze-drying, follow normal ALS SOP.
- After the freeze drying process is complete, homogenize the sample with a laboratory blender commensurate with the size of the sample. Special care must be taken to obtain a uniform homogenate of dry material, as the carapace and other shell-like material will require additional mechanical blending to obtain a relatively fine meal.

Stepwise Details – Preparation of Sub-aliquots and/or Splits:

- Determine if the available mass of freeze-dried material is sufficient for all tests requested by referencing Table A7-3 of the QAPP or Table A2 of Appendix A.

  Note: The values listed in the QAPP and Appendix A are expressed as "wet weight" basis. Convert the available freeze-dried mass to equivalent wet weight basis when performing the evaluation.

  Eq. Wet Weight Mass, g = (Dry Mass of Sample, g) ÷ (% Total Solids/100)

- If insufficient mass is available to satisfy the requirements in the tables, consult with the chemistry laboratory project manager (ALS).
- If sufficient mass is available, prepare aliquots for Vista Analytical (Vista) (Dioxin/Furan and PCB Congeners).
- Sub-aliquots are prepared by thorough mixing of the homogenous freeze-dried material, spreading it out across the base of the container, and removing portions in a like manner as done when performing the incremental sampling method (i.e. remove sub-aliquots by sub-sampling the entire depth to avoid bias from differences in particle size).
- A subset of samples will be identified as "splits" for the purpose of creating "Field Splits" and/or "EPA Splits" where sufficient mass is available. The splits will be created from the freeze-dried and homogenized material as per the sub-aliquots for Vista.

Mussel Compositing, Dissection, Homogenization, and Sub-sampling

Preliminary Freezing of Samples: The integrity of mollusks identified for whole body analysis (minus shell) is not at risk when freezing prior to shucking.

Rinsing of Whole Samples: The general sample processing SOP (MET-TISP) states that samples will be rinsed in the laboratory with DIW to removed extraneous material (e.g., sediment, biogenic material, etc.), however, for this project an equivalent step will be performed in the field. Organisms will be rinsed in the field with site water as needed, as specified in the QAPP. The majority of residual water will be removed prior to packaging the samples for shipment, and therefore further rinsing at the laboratory is not necessary.

Sample Login and Initial Preparation of Composites: Individual mussels will be received at the laboratory with unique identifiers and will be logged as individual samples (i.e. a unique laboratory code is assigned to each uniquely identified sample). The individual samples identified for each composite sample will be combined at the time shucking and homogenization occurs.

Stepwise Details – Compositing, Dissection, and Homogenization:

- The individual samples that make up each composite sample will be identified on the ALS internal work order.
- Prepare certified clean glass containers that will be used for retaining the samples during storage and throughout the freeze-drying process.
- After labeling the containers with the associated sample identifications, record the tare weight of each in the original record log.
- Place the mussels identified as belonging to the composite onto a Teflon cutting board lined with aluminum foil (dull side in contact with the sample). No DIW rinse is performed.
- Transfer any residual fluid in the original sample container to the container designated for freeze-drying of the soft tissue.
- While holding the sample over the tared container, use a sharp filet knife and sever the muscles holding the two shell components together, open the shell, allow residual liquid to drain into the tared container, and separate the soft tissue from the shell as completely as possible.
- Place all soft tissue in the associated tared container, combining it with residual fluids previously transferred from the original container and drained during the initial shucking process.

- Repeat the process for all individual samples that are identified as components to the composite sample.
- Record the total weight (of the composite) to three significant figures for the soft tissue+tare. Calculate the net weight of the soft tissue and record in the original record log.
- Cap each container and transfer to a -20°C freezer and allow overnight storage to assure complete freezing.
- When ready to commence freeze-drying, follow normal ALS SOP.
- After the freeze drying process is complete, homogenize the sample with a laboratory blender commensurate with the size of the sample. Special care must be taken to obtain a uniform homogenate of dry material.

Stepwise Details – Preparation of Sub-aliquots and/or Splits:

- Determine if the available mass of freeze-dried material is sufficient for all tests requested by referencing Table A7-3 of the QAPP or Table A2 of Appendix A.

  Note: The values listed in the QAPP and Appendix A are expressed as "wet weight" basis. Convert the available freeze-dried mass to equivalent wet weight basis when performing the evaluation.

  Eq. Wet Weight Mass, g = (Dry Mass of Sample, g) ÷ (% Total Solids/100)

- If insufficient mass is available to satisfy the requirements in the tables, consult with the ALS project manager.
- If sufficient mass is available, prepare aliquots for Vista (Dioxin/Furan and PCB Congeners).
- Sub-aliquots are prepared by thorough mixing of the homogenous freeze-dried material, spreading it out across the base of the container, and removing portions in a like manner as done when performing the incremental sampling method (i.e. remove sub-aliquots by sub-sampling the entire depth to avoid bias from differences in particle size).
- A subset of samples will be identified as "splits" for the purpose of creating "Field Splits" and/or "EPA Splits" where sufficient mass is available. The splits will be created from the freeze-dried and homogenized material as per the sub-aliquots for Vista.

Primary Staff for Crayfish & Mussel Preparation

- **Jeff Coronado—**Manager – Metals Group, Tissue Preparation, Soil/Sediment Preparation
  27 years of experience at the Kelso (WA) laboratory including expertise with all phases of metals analysis and associated sample preparation, including oversight of the tissue preparation laboratory responsible for all biota received in Kelso and distributed internally and externally for inorganic and organic analytical chemistry. Experience with thousands of different biota species and associated sample preparations ranging from micro to macro applications.

- **Theresa Caron—**QA - Staff Biologists for Consultation of Dissections
  25 years of experience the Kelso (WA) laboratory including expertise in all phases of tissue preparation. Since 2000 has been part of the QA Unit in Kelso, but continues to act as the consulting biologist on staff for dissections as needed. From 1991 to 2000 performed tissue dissections, homogenization, and freeze-drying as a part of overall responsibilities. Experience in the preparation of thousands of different biota species.

- **Lance Jording—**Supervisor Metals Sample Preparation, Tissue Preparation, Soil/Sediment Preparation
  26 years of experience in sample preparations for metals (all matrices), tissue preparation for subsequent organic and inorganic testing, sediment/soil preparations for subsequent organic and inorganic testing, elutriations, and specialized laboratory procedures for project specific applications. Oversees day to day operation of the tissue preparation laboratory, including extensive experience with thousands of different biota species and multiple applications to analytical chemistry.

APPENDIX D

CULTURAL RESOURCES COORDINATION PLAN

CONTENTS

LIST OF MAPS .... **D-iv**

LIST OF TABLES .... **D-iv**

ACRONYMS AND ABBREVIATIONS .... **D-v**

UNITS OF MEASURE .... **D-vi**

1 INTRODUCTION **D-1-1**

1.1 BACKGROUND D-1-1

1.2 CULTURAL SETTING D-1-2

2 OVERVIEW OF LAWS AND REGULATIONS **D-2-1**

2.1 FEDERAL LEGISLATION AND REGULATIONS D-2-1

2.1.1 National Historic Preservation Act of 1966, as Amended through 1992 (16 USC 470-470w) D-2-1

2.1.2 Archeological Resources Protection Act of 1979 (16 USC 470aa-470ll) D-2-6

2.1.3 Native American Graves Protection and Repatriation Act (25 USC 3001-3013) D-2-7

2.1.4 American Indian Religious Freedom Act (42 USC 1996) D-2-8

2.2 PRESIDENTIAL EXECUTIVE ORDERS D-2-8

2.2.1 Executive Order 11593. Protection and Enhancement of the Cultural Environment D-2-8

2.2.2 Executive Order 13007. Indian Sacred Sites D-2-9

2.2.3 Executive Order 13175. Consultation and Coordination with Indian Tribal Governments D-2-9

2.3 TRIBAL LEGISLATION AND REGULATIONS D-2-9

2.3.1 Confederated Tribes of the Colville Reservation. Colville Tribal Law and Order Code Chapter 4-4, Cultural Resources Protection D-2-9

2.4 STATE LEGISLATION AND REGULATIONS D-2-10

2.4.1 Revised Code of Washington (RCW) Chapter 27.44, Indian Graves and Records D-2-10

2.4.2 RCW Chapter 27.53, Archaeological Sites and Resources D-2-10

2.4.3 RCW Chapter 68.60, Abandoned and Historic Cemeteries and Historic Graves D-2-11

2.4.4 RCW Chapter 43.21C, State Environmental Policy Act D-2-11

3 PROPOSED SAMPLING PROGRAM D-3-1

4 COORDINATION PLAN D-4-1

4.1 GENERAL CONSULTATION FRAMEWORK D-4-1

4.2 CULTURAL RESOURCE PROCEDURES IN THE SAMPLING PROCESS D-4-2

4.2.1 Archaeological Monitoring in the Sampling Program D-4-2

4.2.2 Curation D-4-5

4.2.3 Reporting D-4-6

4.3 CONFIDENTIALITY D-4-6

5 REFERENCES D-5-1

6 GLOSSARY OF TERMS D-6-1

Attachment D1. Protocols for Inadvertent Discoveries

LIST OF MAPS

Map D1. Proposed Macroinvertebrate Sampling Areas

LIST OF TABLES

Table D1. Sampling Locations

ACRONYMS AND ABBREVIATIONS

ACHP	Advisory Council on Historic Preservation
APE	area of potential effects
ARPA	Archeological 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
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
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 for sampling activities associated with the Macroinvertebrate 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 sediment 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 benthic macroinvertebrate 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 Washington State and includes approximately 150 river miles of the Columbia River extending from the U.S.-Canada border to the Grand Coulee Dam and those areas in proximity to such 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. 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

[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.

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.

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 the 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 and 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 already noted, 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 Colvile. 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 and therefore complying with the NHPA requirements is the responsibility of EPA. The Archeological 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 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 archeological 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's 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 using National Register 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 Council 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 Council will not review "No Adverse Effect" determinations. However, the Council 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 Council'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 Council when adverse effects are found and should invite the Council to participate in the consultation when circumstances as outlined within 36 CFR 800.6(a)(1)(i)(A)-(C) exist. A consulting party may also request the Council to join the consultation.

   When resolving adverse effects without the Council, 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 Council. 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 *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 Comprehensive Environmental Response, Compensation and Liability Act (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 on 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 Archeological 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 archeological 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 (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" (lands held in

[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."

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 descendant(s) 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,

---

[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.

with 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 on Historic Preservation 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 impacting 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 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 History and Archaeology Department. The code defines the procedure for the issuance of permits and the duties 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 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 historical 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 proposed sampling areas is provided in Table D1; with a detailed description of sampling techniques provided in this Quality Assurance Project Plan (QAPP). As indicated in the QAPP, mussel and crayfish tissue samples will be collected from a total of eight sampling areas in both Site and reference areas (Map D1). 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 chief 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 resource(s). 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 both known locations of cultural resources and locations where no cultural resources are presently recorded.

4.1 GENERAL CONSULTATION FRAMEWORK

Implementation of the RI/FS constitutes an "undertaking" as defined in the NHPA and 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 sections.

In addition, the UCR Cultural Resources Working Group recommended to 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 assure 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) and Indian burials and human remains (as defined in NAGPRA) during macroinvertebrate tissue sampling and associated activity that could result in ground disturbance.

4.2.1.1 Notification of Planned Macroinvertabrate Tissue Sampling

Teck American Incorporated (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/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, macroinvertebrate tissue 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 mussel sampling 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 D1 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

In light of the sampling method to be employed in the field for the collection of crayfish, i.e., bait traps deployed in the water and weighed down by brick, the UCR Cultural Resources Working Group is not requiring that an archaeologist or Tribal cultural monitor be present during crayfish sampling. However, for crayfish sampling in Sampling Area 5, the Spokane Tribe might seek to touch base with the crayfish crew prior to sampling in order to give the crew a heads up on where they need to be more careful with the bait traps due to the sensitivity of the area. EPA will help coordinate any such communication between the Spokane Tribe and the crayfish sampling crew.

With respect to the collection of mussels, the Working Group requires that a cultural/archaeological monitor be present on-site with each field crew conducting mussel sampling. The monitor will visually examine the beach prior to collection and will make judgement calls in the field concerning the collection of mussels so that no cultural resources are disturbed. For example, no rocks are to be moved/removed from cairns and samples are not to be collected in known burial/cemetery areas.

If artifacts or likely archaeological deposits are observed, the monitor will document/isolate the site, e.g., record the location of the materials and photograph the materials in place in such a manner to provide information on provenience.

The monitor will document his/her observations on a daily basis, including field notes and photographs that record the location, 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 monitor in response to any archaeological discoveries. A standardized archaeological monitoring form may be substituted for the field notes referenced above.

All monitors 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 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. Any direction, however, that may impact cultural resources monitoring and/or the protocol outlined in this Appendix must be brought to the attention of EPA immediately.

In the event that the Cultural Resources Working Group cannot provide a monitor(s) for some of the mussel sampling areas, the Working Group will consider using qualified back-up monitor(s), as needed. TAI may propose to EPA back-up monitor(s) with accompanying credentials. EPA will share any proposal that it receives with the Cultural Resources Working Group for its consideration. The Working Group will have final approval of any proposed back-up archaeological/cultural monitor including which sampling area(s) the back-up monitor(s) may be assigned to.

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 representatives can arrive to assess the find.

Other conditions for responses to discoveries of archaeological materials may be defined in the permit(s) 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 D1).

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 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.

---

[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 5.3, "Confidentiality."

5 REFERENCES

Kennedy, D.I.D., and R.T. Bouchard. (1998). Northern Okanagan, Lakes and Colville. In: *Handbook of North American Indians*, Vol.12, W.C. Sturtevant, general editor, Smithsonian Institution, Washington, DC.

McKay, K.L., and N.F. Renk. (2002). Currents and Under currents: An Administrative History of Lake Roosevelt National Recreation Area.

NPS (National Park Service). 1983 (with updates). Archeology and historic preservation: secretary of the interior's standards and guidelines [as amended and annotated]. National Park Service, Department of Interior. Available at: http://www.nps.gov/history/local-law/arch_stnds_9.htm

TAI (Teck American Incorporated). 2013. Upper Columbia River final quality assurance project plan for the Phase 2 sediment study. Appendix B. Technical memorandum, Upper Columbia River geographical information system-based surface sediment bed mapping. Prepared by Exponent and HDR HydroQual for Teck American Incorporated, Spokane, WA.

USEPA (United States Environmental Protection Agency). 1989. CERCLA compliance with other laws manual: Part II. Clean Air Act and other environmental statutes and state requirements. U.S. Environmental Protection Agency, Region 10, Seattle, WA.

USEPA. 2006. Settlement agreement for implementation of remedial investigation and feasibility study at the Upper Columbia River Site. June 2, 2006. U.S. Environmental Protection Agency, Region 10, Seattle, WA.

Westat. 2012. Upper Columbia River Site Remedial Investigation and Feasibility Study Tribal Consumption and Resource Use Survey. Final Report. Prepared for the USEPA, Region 10. June 22, 2012.

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, the protohistoric can be generally defined as from the late 1600s until late 1700s.

TABLE

Table D1. Sampling Areas

Sampling Area	Abbreviation	Location Type	HHRA	BERA	Approximate River Miles	Description of Location	Rationale for Sampling Area
Area 1	A1	Site		X	733 to 739	Near Northport	Location provides spatial coverage of the UCR for the BERA; overlaps with targeted fish sampling area for Reach 1 in 2009. In the USFWS survey, no crayfish were collected at survey locations in this area. Mussels were reported at some of the USFWS survey locations; with the exception of four mussels at one location, all were reported to be dead.
Area 2	A2	Site		X	718 to 725	Stretch of UCR from China Bend boat launch to North Gorge	Location provides spatial coverage of the UCR for the BERA; overlaps with targeted fish sampling area for Reach 2 in 2009. During the USFWS survey, only two crayfish were collected from the survey locations in this area. Mussels were collected from all survey locations in this area; dead mussels were reported at all locations other than the China Bend boat launch, where approximately half of the mussels were reported to be living.
Area 3	A3	Site		X	700 to 706	Near Kettle Falls	Location provides spatial coverage of the UCR for the BERA; overlaps with targeted fish sampling area for Reach 3 in 2009. During the USFWS survey, Crayfish were successfully collected at some locations; the most successful of which was the Kettle Falls location. The highest number of mussels in the entire UCR was reported at the Hayes Island location, which is only exposed during more extreme draw-down years. All mussels reported in survey locations from Reach 3 were reported to be dead.
Area 4	A4	Site		X	671 to 678	Stretch of UCR from Daisy to Gifford	Location provides spatial coverage of the UCR for the BERA; overlaps with targeted fish sampling area for Reach 4 in 2009. During the USFWS survey, crayfish were successfully collected in traps at the Daisy, Cloverdale, and Gifford sampling locations. Mussels were collected at most of the survey locations in this area; all were reported to be dead.
Area 5	A5	Site	X	X	639 to 643	Upstream of the mouth of the Spokane River (east side of river only)	Location reported as a possible source of mussels and crayfish by Spokane Tribe. Provi des spatial coverage of the UCR for the BERA. Located just upstream of targeted fish sampling area for Reach 5 in 2009. Extent of the area is based on potential human beach access locations north of the mouth of the Spokane River.
Area 6	A6	Site	X	X	613 to 618	Near mouth of the Sanpoil River (north side of river only)	Location reported as a source of crayfish in the tribal survey (Westat 2012). Provides spatial coverage of UCR for the BERA. Located just upstream of Reach 6 fish sampling area. Extent of the area is based on potential human beach access locations near the mouth of the Sanpoil River.
Rebecca Lake	RL	Reference	X	X	NA	Lake located north of the Grand Coulee Dam	Location noted to be a popular crayfish collection location in the tribal surve y (Westat 2012); some individuals also reported collecting mussels from this location.
Sanpoil River	SR	Reference	X	X	NA	Near the Sanpoil Campground and mouth of Silver Creek.	Location noted to be a popular mussel collection location in the tribal survey (Westat 2012); the Sanpoil River was also noted as a popular crayfish collection area.

Notes:

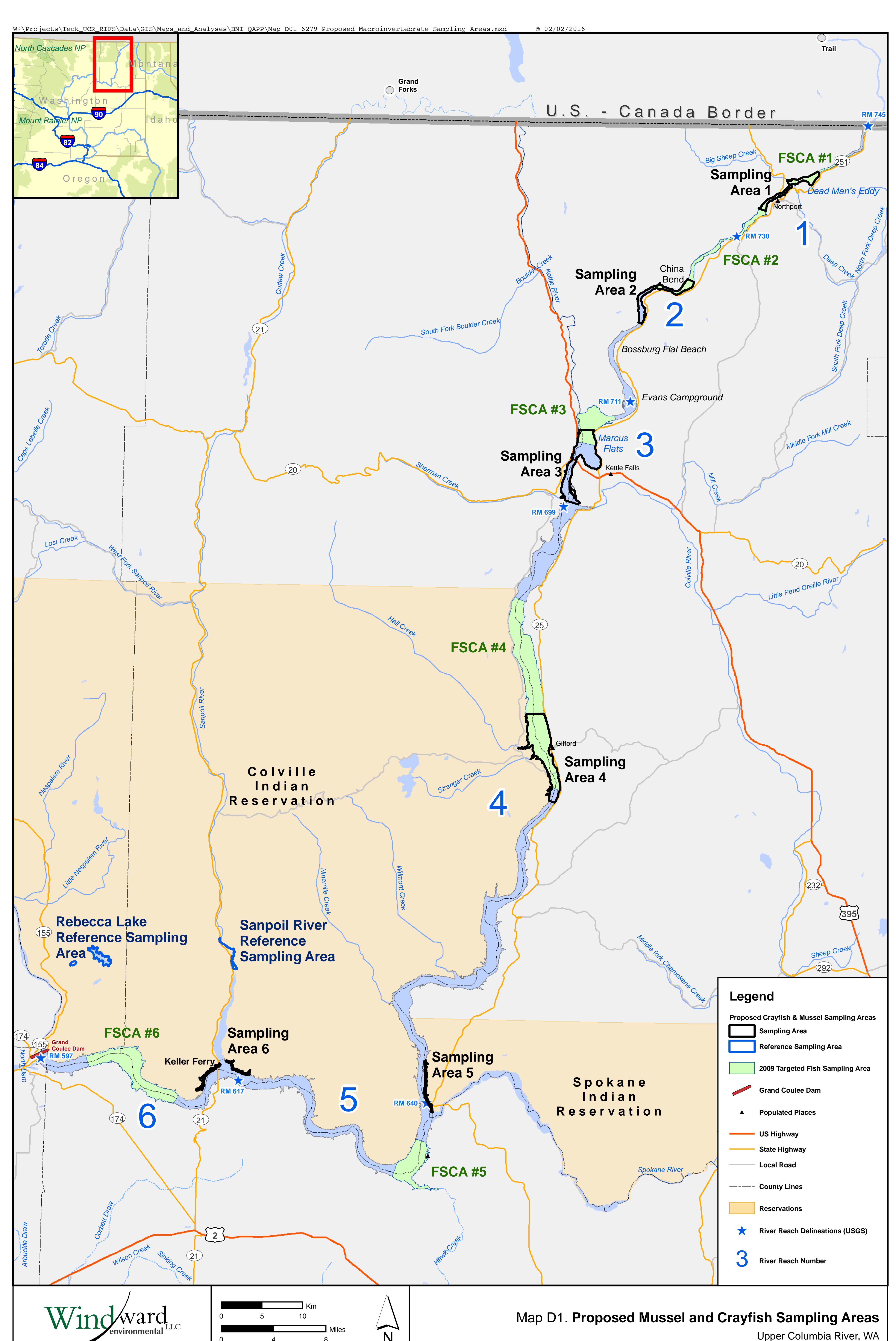
If sample collection at Rebecca Lake is not successful, nearby Buffalo Lake may also be used as a reference location.

BERA - baseline ecological risk assessment

HHRA - human health risk assessment

USFWS - U.S. Fish and Wildlife Service

MAP



Map D1. **Proposed Mussel and Crayfish Sampling Areas**

Upper Columbia River, WA

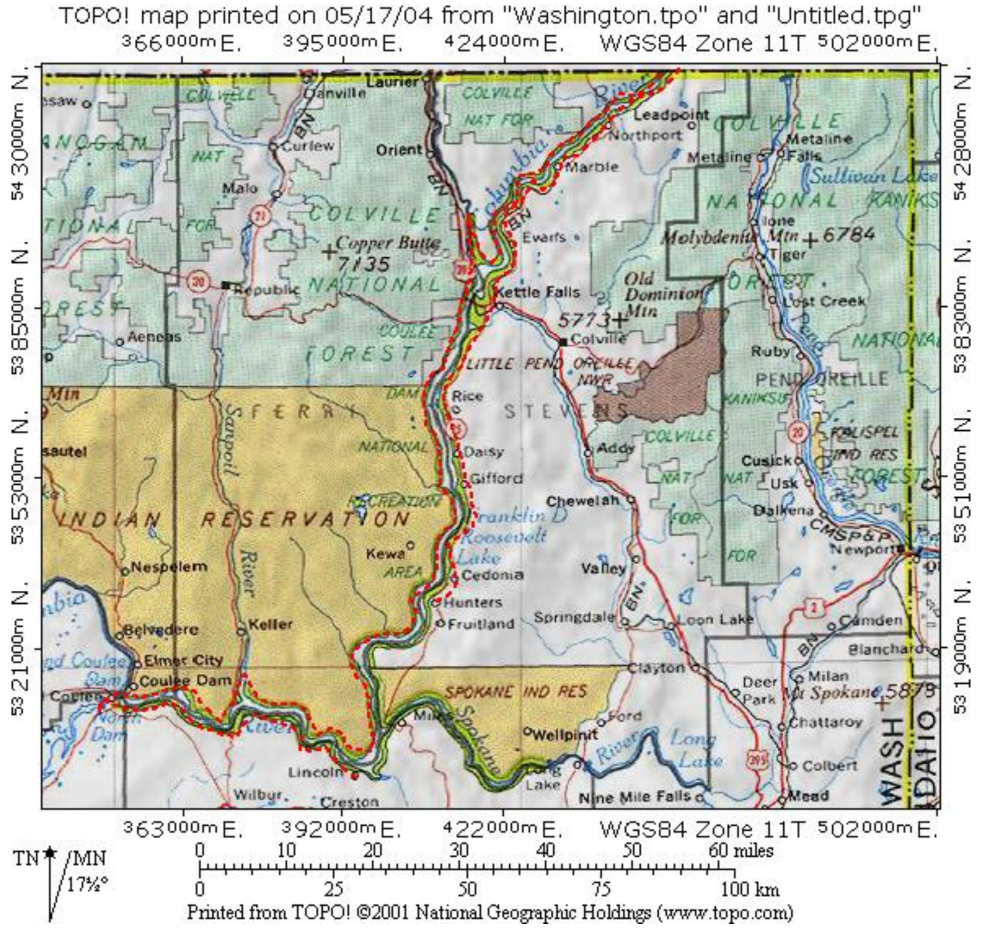
ATTACHMENT D1

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 Jon Meyer, Tribal Archaeologist is the alternate contact at (509) 634-2691 (office) or (509) 631-2130 (cell) and at jon.meyer@colvilletribes.com. In the event that neither Mr. Moura or Mr. Meyer cannot be contacted, then Arrow Coyote, CCT Senior Archaeologist will be contacted at (509) 634-2736 (office) or (509) 634-1280 (cell) and atmailto: arrow.coyote@colvilletribes.com. Mr. Meyer 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 or (509) 631-0306, and his FAX is (509) 738-3108, and internet address is keith_holliday@nps.gov.

Derek Beery, Power Office Archaeologist, is Reclamation's contact. His phone number is (509) 633-9233 [desk], (509) 237-4477 [cell phone] FAX 633-9138, and internet address ismailto: dbeery@usbr.gov. If Derek Beery is not available, contact Sean Hess, Regional Archaeologist (208) 378-5316, FAX (208) 378-5305, and internet address is 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 <u>within the boundaries of lands managed by the National Park Service/Lake Roosevelt National Recreation Area (Figure 1)</u>, 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.

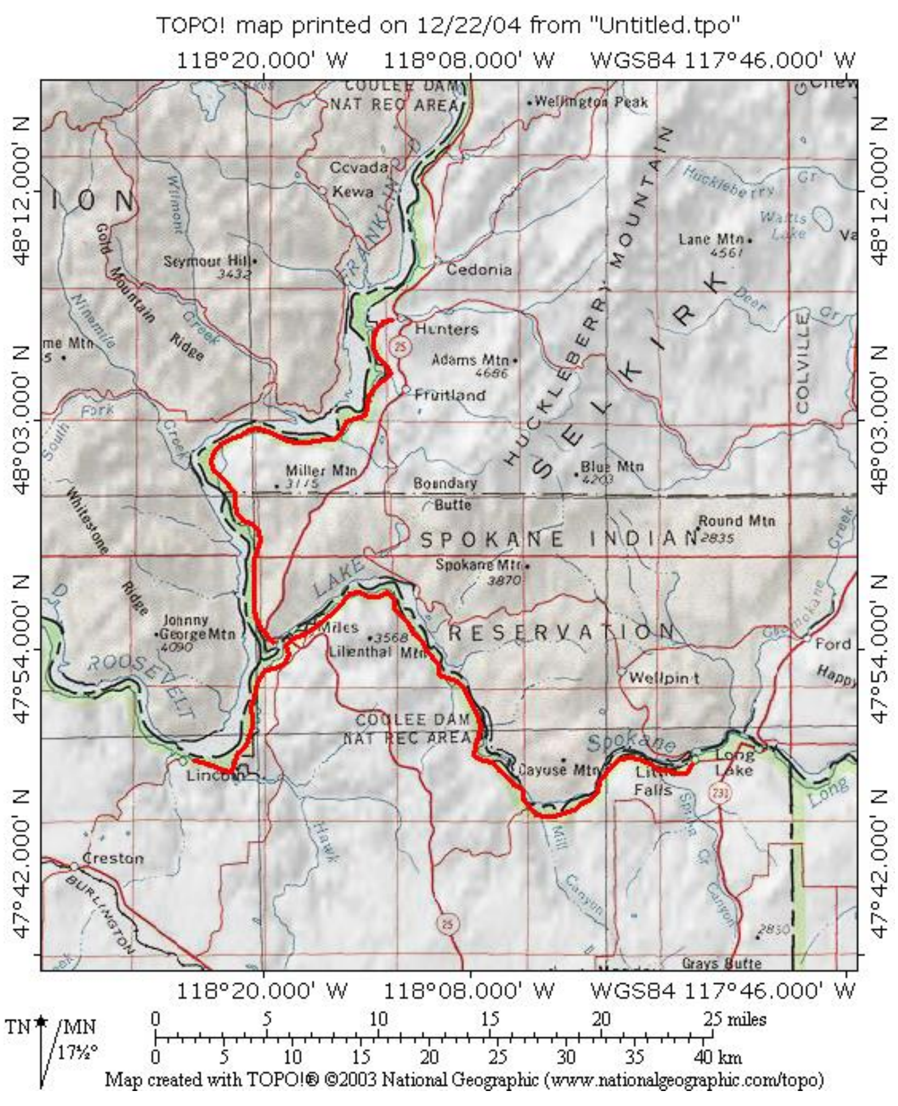


Figure 1. Lake Roosevelt National Recreation Area Shoreline Areas Managed by the National Park Service and Bureau of Reclamation

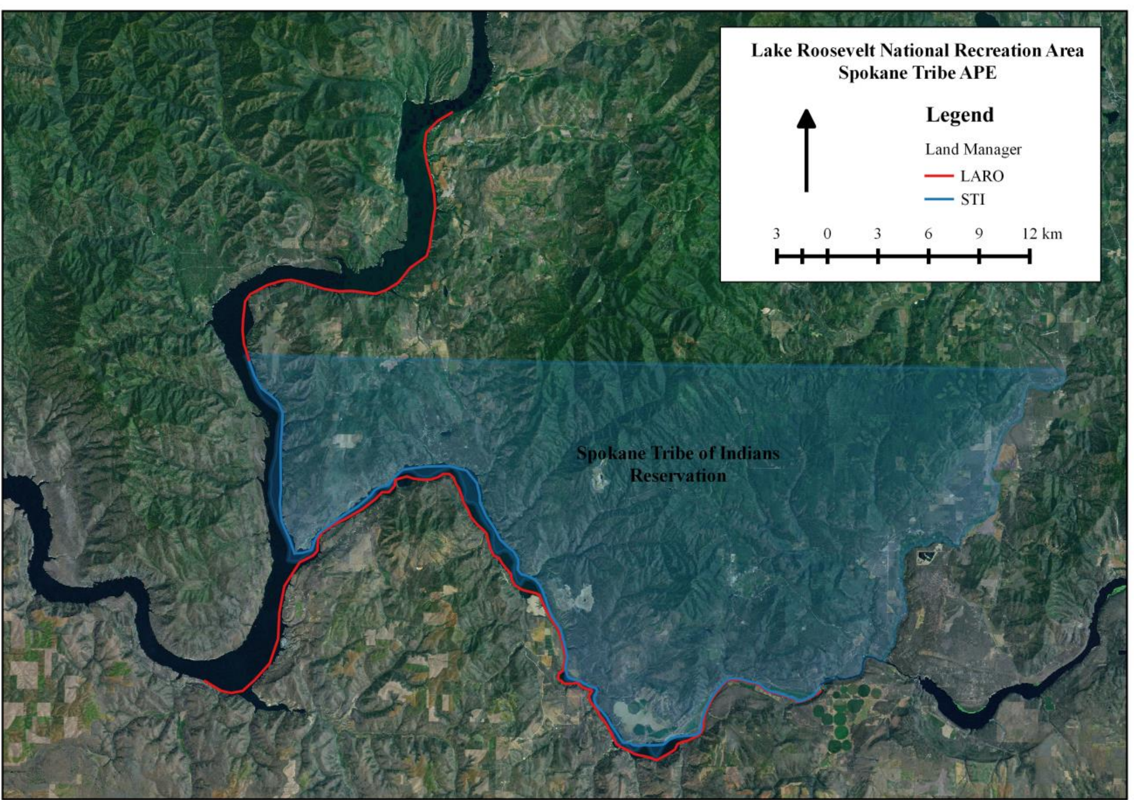


Figure 2. Spokane Tribe of Indians Reservation Land Not Covered by NPS Protocol

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 Internet 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 internet address is "dbeery@usbr.gov." If Derek Beery is not available, contact Sean Hess, Regional Archaeologist (208) 378-5316, FAX (208) 378-5305, and internet 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 internet 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.

Spokane Tribe of Indians
P.O. Box 100-Wellpinit, WA 99040
Tel 509-458-6500, Fax 509-458-6575

Century of Survival 1881-1981: Procedure for the Inadvertent Disturbance or Discovery of Spokane Human Remains and Cultural Resources

Introduction

Because many ground-disturbing processes, both natural and cultural, have the effect of prompting the destruction of evidence of Spokane Tribal heritage, it is the policy of the Spokane Tribe of Indians (hereafter "Spokane Tribe") to leave Spokane human remains and cultural resources in place and undisturbed. Purposeful disturbance of these resources without proper permit and consultation and/or approval of the Spokane Tribe is a violation of federal, Tribal, State, and/or local law. The National Historic Preservation Act (NHPA) and the Native American Graves Protection and Repatriation Act (NAGPRA) require that federal agencies take responsibility for damage to or loss of human burials caused by the project actions or that occur on off-reservation lands under the management jurisdiction. The Spokane Tribe has been delegated the federal authority as a Tribal Historic Preservation Office for Reservation lands pursuant to Section 101 (d)(2) of the National Historic Preservation Act.

Geographic Area of Applicability

This procedure for the inadvertent disturbance or discovery of Spokane human remains and cultural resources applies to all lands within the boundaries of the Spokane Indian Reservation and is advisory for all lands within the Spokane Tribe's aboriginal territory, as determined in proceedings before the Indian Claims Commission. For the purposes of cultural resource management, the ceded territory is bounded by and includes the Columbia River on the west, the Canadian border to the north, the Idaho border to the east, Rosalia to the southeast, Rosalia to the southeast, and Ritzville to the southwest.

Procedure

In cases of inadvertent disturbance or discovery of Spokane human burials or cultural resources, the following procedure is to be followed:

1. Upon inadvertent disturbance or discovery of human burials or cultural resources, any action(s) affecting the burials or resources shall immediately be halted.
2. The person(s) making the discovery shall immediately notify the appropriate office of the coroner or police. Upon a determination of the appropriate death investigation authority that the location of the remains is not the result of a crime, the following procedures shall apply:
    a) The entity making such disturbance or discovery shall notify the landowner, occupant, or manager. If the land occupant or manager is notified in lieu of the landowner, the occupant or manager will immediately notify the landowner. The entity making the disturbance or discovery will immediately notify the Spokane Tribal Historic Preservation Office, Wellpinit, Washington, in person or by telephone (at 509-258-4315), or by fax (at 509-248-6965), of the disturbance or discovery. The entity is advised to keep written documentation of such contact.
    b) The entity making the disturbance, or discovery will exert its best effort to protect such remains and/or objects until the landowner and/or land occupant or manager arrives to protect these remains and/or objects. Within 24 hours of notification, the

landowner shall supply protection for such remains and/or objects, until disposition or control of such remains and objects has been implemented.

3. The Spokane Tribal Historic Preservation Officer or designated representative(s) shall inspect in person the affected sited, human remains, or cultural resources, and shall determine, if possible evidence at the site, oral history, and/or existing records, the cultural affiliation of such site, human remains, and/or objects, until disposition or control of such remains and objects has been implemented.

   a. If the exposed human remains or cultural resources are clearly Native American and have known lineal descendants or owners, the Spokane Tribal Historic Preservation Officer shall then have the opportunity to make disposition or to take control of such human remains and/or associated funerary objects.

   b. If the exposed human remains and /or associated funerary objects are clearly prehistoric or non-modern Native American in origin and have no known lineal descendants, or if the lineal descendants decline the disposition or control, the Spokane Tribe, as the Indian Tribe which has the closest cultural affiliation and aboriginally occupying the are, claims ownership of such human remains and associated funerary objects, as they choose.[1] The Tribe's ownership and right to disposition and control of the human remains and/or associated funerary objects refers to the entire burial, to the extent it can be recovered, and does not allow in any case for separation of part of an individual's remains from other parts or from their associated funerary objects.

   c. If the exposed human remains and/or associated funerary objects are historic and non-Native American in origin, the Spokane Tribal Historic Preservation Officer will notify the Washington State Historic Preservation Officer (SHPO), Disposition and control over such burials will be determined the SHPO.

   d. If the exposed human remains and/or associated funerary objects are of uncertain or unidentifiable cultural identity, but clearly non-modern in origin, the Spokane Tribal Historic Preservation Officer will use reasonable means, such as professional consultation, to obtain a determination of the responsibility of the entity disturbing such remains. After cultural identity has been satisfactorily determined, the disposition or control of such remains and /or objects shall follow as otherwise provided in this procedure.

   e. If the exposed human remains and/or associated funerary objects are modern or possibly modern in origin, regardless of cultural affiliation, the Spokane Tribal Historic Preservation Officer will notify the local law enforcement authorities. Disposition and control over such burials will be determined by the law enforcement authorities.

---

[1] For the purposes of this procedure, modern is here defined as less than 50 years old; non-modern is defined as 50 years of age or older. For human remains, the age of such remains is defined as beginning at the death of the individual, to the present.

4. Within 48 hours of notification, the entity with right of disposition and control shall notify the landowner concerning plans for disposition and control over such objects. Actual disposition and control shall be implemented as soon as possible, although may be delayed is so agreed by the landowner and the entity with right of disposition and control, or is the extent of the damage or other circumstances require delay in disposition and control.

The entity performing any action which inadvertently disturbs or damages Spokane human remains or cultural resources shall be responsible for costs of inspection of the damage or disruption by Tribal staff; removal, reburial, and/or restoration of the site; identification of resources. Costs may include but are not limited to staff, equipment, supplies, laboratory costs, and travel. If the entity performing the action which inadvertently disturbs or damages such resource is not also the land owner, such entity is responsible for reimbursing the land owner for costs incurred by the land owner as a direct result of this procedure. In no case shall the required associated with the action or resources involved.

The Spokane Tribal Historic Preservation Office shall make best effort to minimize the costs associated with Inadvertent Disturbance or Discovery, especially when the entity involved fully cooperates with preservation and protection efforts; however, appropriate project undertaking funding shall ensure that sufficient measures are taken to complete the activities described in these procedures.

An entity solely reporting human remains or cultural resources to the Spokane Tribe, provided they have not damaged or disturbed such resources, or caused or been responsible for damage or disturbance of such resources, shall not be responsible for any additional costs under this section.

Relationship to Other Applicable Laws

Full compliance with all aspects of this procedure shall be considered by the Spokane Tribe as full and complete consultation and cooperation with the Spokane Tribe, as required by law, for the purposes of Inadvertent Disturbance and Discovery of human remains and cultural resources.

Limitations

Compliance with this procedure for a particular disturbance or discovery does not constitute consultation and cooperation with the Spokane Tribe on other disturbances or discoveries.

Notification of the Spokane Tribe under this procedure does not release the entity from responsibility for violations of federal, Tribal, state or local law.

Violations

Any entity discovering or disturbing any Spokane human remains or cultural resources who does not follow the procedure described here, shall be considered in violation of this procedure. Such action shall be considered deliberate and causing unauthorized damage to the affected resource; this action is subject to prosecution under applicable federal, Tribal, state and/or local laws.

Recovery of Eroding Human Remains

When approval from the appropriate authorities is given for the collection of scattered human remains or recovery of exposed and immediately endangered remains, standard professional practices will be used to ensure that all associated remains and grave goods are recovered, and that the location is documented to assist future monitoring or management practices. However, those making the recovery shall not open up areas around the burial or discovery with the intention of discovering additional burials and materials or to learn more about the site context. Excavations of this sort are strictly for the salvage of eroding or disturbed burials.

The methods for documentation are to be consistent with practices employed by the Spokane Tribe, including collection of locational data, controlled excavation of the burial pit, and screening of the pit fill.

A professional archaeologist shall be in the field with the burial recovery crew at all times, and shall participate in the documentation of burials in all aspects where their involvement does not violate traditional custom or practices. If permitted by the Spokane, to scale map documentation of excavated features (i.e., distribution of remains and grave goods in the burial pit) is recommended.

The project entity is responsible for the preparation of a site plan map that shows the locations of surface-visible cultural features, significant topographic features, and other information needed to relocate the site in subsequent years for management purposes.

Photographs shall be taken that show the location of excavated burials in relation to identifiable landmarks. Human remains will not be visible in the photograph if not approved by the Spokane Tribe; this authorization will be decided on a case by case basis. The location of un-recovered remains or each excavated grave will be documented on a 7.5' USGS quadrangle topographic map. GPS measurement of location is required.

Associated artifacts and grave goods may be subjected to examination and documentation if that is approved by the Spokane Tribe. Permission from the Spokane Tribal Business Council for examination and documentation of Native American burials and grave good, beyond that required to determine if the remains are Native American in origin, shall be gained in writing and a copy of the written approval shall be provided to the contracting professional investigation of the burial(s).

If the remains are Euro-American in ancestry, standard non-destructive analysis shall be completed of remains and any associated grave goods or mortuary materials.

All grave goods shall be stored with the appropriate skeletal remains.

Any recovered remains will be boxed according to Tribal standards (appropriate size and material to be decided by Tribal Elder in consultation); the contracted investigator will retain and protect the burials in their custody until repatriation occurs or, if such would prove necessary after completion of NAGPRA consultations, the Tribe notifies them to deliver the burial(s) to another location. We anticipate that, after completion of notification processes defined in NAGPRA, Native American remains would be repatriated to Spokane Tribe in Wellpinit, Washington.

Coordination

The Tribal Historic Preservation Officer, is the primary contact for the Spokane Tribe for notification purposes as well as consultation on matters of cultural patrimony. The phone number is (509) 258-4315, or FAX (509) 258-6965. The THPO shall be immediately notified whenever a human burial or scattered human remains are found on any Reservation or ceded land location.

Definitions

Cultural Resources

Cultural resources include (but not by way of limitation): archeological, historic, traditional, and ethnographic resources older than 50 years or originating more than 50 years ago. These include artifacts, features, and sites; pictographs and petroglyphs; traditional cultural properties; sacred sites and continuing practices; traditional gathering areas and resources; the Spokane and Columbia rivers; oral histories, myths, and stories; traditional ceremonies (separate from those practiced at historic sites), gatherings, and activities; and recordings of these in various formats. Those cultural resources specifically excluded from this definition are burial sites, human remains, and associated funerary objects, which possess certain qualities for the Spokane People that are not to be disclosed or discussed in this context.

To further expand this operational definition of cultural resources, three categories of property types should be noted; ancestral lifeways, property is usually an archaeological resource that contains material remains or physical evidence of past human life or activities, including the record of the effects of human activities on the environment. They are capable of revealing scientific and/or humanistic information through archaeological research. For the purposes of the Spokane Tribe, these sites are those that can be dated as originating prior to contact, that is, A.D. 1730.

An historic property may also be archeological in nature, but is better delimited by the time period of contact between the Spokane(e) Peoples and Euro-Americans, that is, between 1730 and 1950. This transitional period and the material culture generated may provide useful insights on assimilation and cultural resistance. In the long run, these contrasts will offer broader cultural and chronological reconstructions, documenting significant events, occupations or activities, and/or structures or landscapes whether extant or vanished, apart from the value of any existing structure or landscape.

Additional cultural properties are those associated with cultural practices or beliefs of a living community that are rooted in that community's history or are important in maintaining its cultural identity. These may also include traditional resource areas, those which traditionally support subsistence or other consumptive or ceremonial use of natural resources. Use can be on-site and visible, inferred from effects, or off-site and referenced in traditional narratives. Traditional ceremonial use may also involve sites, structures, each with their own special local names; as such they are eligible for listing in the National Register Historic Places.

Damage to Cultural Resources

Any intentional or unintentional disturbance to any cultural resource which has not been authorized by the Spokane Tribal Council as appropriate for that resource is considered damage. Damage to cultural resources includes (but not by way of limitation) looting, vandalism, disturbance , or displacement of any artifact, human remains or associated cultural objects, cultural features or sites, sacred sites, or burial sites; collection of non-modern artifacts (older than 50 years) from the surface of the ground; painting, drawing, carving, or other defacement of pictographs or petroglyphs; digging or disturbance in cultural sites; disturbance, clearing, or spraying pesticides in traditional gathering areas; handling of Spokane burial remains or associated objects by non-Tribal members; and desecration of burial grounds.

Entity or Person

For the purposes of the procedure "entity or person" shall mean an individual, corporation, partnership, trust, institution, association, or any other private entity or any officer, employee, agent, department, or instrumentality of the United States, of any Native American Tribe, and/or of any State or political subdivision thereof.

Objects of Cultural Patrimony

For the Spokane Tribe these objects include (not by way of limitation) Spokane Elders' oral histories, myths, stories; burial remains and associated objects of individuals without known descendants; objects associated with cemeteries and sacred sites; and the recordings in any and all media of these classes of objects.