Final
Sampling and Analysis Plan/Quality Assurance Project Plan
2017-2018 Southern Ute Indian Tribe Wild Plant Sampling

Bonita Peak Mining District
San Juan County, Colorado

Prepared For:
United States Environmental Protection Agency, Region 8
Ecosystem Protection and Remediation – Program Support
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A. PROJECT MANAGEMENT

A.1 Approval Sheet

ESAT Team Manager:

4/10/18
Signature/Date
Mark McDaniel

ESAT Field Task Lead:

4/10/18
Signature/Date
Steven Auer

Region 8 Remedial Project Manager:

4/10/18
Signature/Date
Rebecca Thomas

Region 8 Remedial Project Manager:

4/10/18
Signature/Date
Robert Parker

Region 8 Risk Assessor:

4/2/2018
Signature/Date
Deborah McKean

Region 8 Task Order Contracting Officer Representative (TOCOR):

4/2/18
Signature/Date
Nicole Marotta

Region 8 Delegated Quality Assurance Authorizing Official:

4/2/18
Signature/Date
Charles Partridge

RECORD OF MODIFICATION

INSTRUCTIONS: This form is required anytime there are deviations in the field from, or modifications are made to any of the following documents: Sampling and Analysis Plan (SAP), Quality Assurance Project Plan (QAPP) Site-Specific Health and Safety Plan (Site HASP), and Standard Operating Procedures (SOPs). If deviations/modifications are made to multiple documents, complete a separate Record of Modification Form for each document.

Requestor: Deborah McKean

Title: Toxicologist

Name of Site/Field Event: 2018 Southern Ute Indian Tribe Wild Plant Sampling

Date of Modification: May 1, 2018

Modified Document (Provide Title and Document Number, if applicable): March 2018
DCN: 03072-5-04-F119-IN-0328

What was the modification?

The field activities described in the Sampling and Analysis Plan/Quality Assurance Project Plan for the 2017-2018 Southern Ute Indian Tribe Wild Plant Sampling implies that all sampling is conducted in one field season. This modification will define a phased approach for plant sampling activities. During the 2018 field sampling (and co-incident with other field activities for the BPMD studies), the field crews, with the support of ecologists familiar with plant identification, will survey the mining district and identify any plants listed in Table A.6-1 and record plant name, and GPS coordinate. In addition, information on quantity of plant at that location will also be recorded. The only plants collected during the 2018 field season and submitted for metals analysis will be sumac and willow plant tissues (as described in Section B.2.2.5) and Piñon pine nuts (as described in Section B 2.2.6) in Sampling and Analysis Plan/Quality Assurance Project Plan, 2017-2018 Southern Ute Indian Tribe Wild Plant Sampling. Survey and analytical data will then be evaluated to determine if additional plant collection activities are warranted in the subsequent year.

Justification or Reason for the Modification:

Quantities of plant material in different locations of the mining district is important to determine if any single species is found in the amount needed for chemical analysis.

Technical Review/Approval: _____ **Date:** 5/31/18

EPA Review/Approval: _____ **Date:** 5/22/2018

The original Record of Modification form is made as an attachment to the Sampling Activities Report (SAR) and a copy is sent to all individuals in the List of People to Notify.

(RPM or designee)

List of People to Notify:

Name	Title	Agency	Email Address
Dan Wall	Technical Support Unit Chief - TOCOR	USEPA	Wall.dan@epa.gov
Christina Progess	Remedial Project Manager for BPMD	USEPA	Progess.chistina@epa.gov
Steve Auer	Field Task Lead	ESAT	Auer.steve@epa.gov
Jamie Miller	Remedial Project Manager	USEPA	Miller.jamie@epa.gov

Document Revision Log

Revision	Date	Primary Changes
Draft Revision 0	5/17/2017	First draft SAP/QAPP drafted and delivered to EPA for review
Draft Revision 1	10/07/2017	EPA reviewer comments addressed and final draft SAP/QAPP delivered to EPA for final review
Final Revision 0	4/03/2018	Draft Revision 1 EPA review comments were addressed and SAP/QAPP subsequently finalized

A.2 Table of Contents

List of Tables

List of Figures

List of Appendices

List of Attachments

List of Abbreviations and Acronyms

ºC	Degree Celsius
%R	Percent Recovery
ARSG	Animas River Stakeholders Group
BLM	Bureau of Land Management
BPMD	Bonita Peak Mining District
CA	Corrective Action
CDPHE	Colorado Department of Public Health and Environment
CFR	Code of Federal Regulations
CLP	Contract Laboratory Program
COC	Chain of Custody
CPR	Cardiopulmonary Resuscitation
CPW	Colorado Parks and Wildlife
DQA	Data Quality Assessment
DQO	Data Quality Objective
DRMS	Division of Reclamation, Mining and Safety
EDD	Electronic Data Deliverable
EPA	United States Environmental Protection Agency
ESAT	Environmental Services Assistance Team
ID	Identification
LCS	Laboratory Control Spike
LCS/LCSD	Laboratory Control Spike/Laboratory Control Spike Duplicate
LIMS	Laboratory Information Management System
MSI	Mountain Studies Institute
MS/MSD	Matrix Spike/Matrix Spike Duplicate
NPL	National Priorities List
OSHA	Occupational Safety and Health Administration
PE	Performance Evaluation
PQL	Practical Quantification Limit
PSQ	Principal Study Question
QA	Quality Assurance
QAO	Quality Assurance Officer
QAPP	Quality Assurance Project Plan
QA/QC	Quality Assurance/Quality Control
QC	Quality Control
QMP	Quality Management Plan
RPD	Relative Percent Difference
RPM	Remedial Project Manager
RSL	Regional Screening Level
SAP	Sampling and Analysis Plan
SAR	Sampling Activities Report
SOP	Standard Operating Procedure
SUIT	Southern Ute Indian Tribe
TAL	Target Analyte List
TOCOR	Task Order Contracting Officer Representative
TU	Trout Unlimited

USFS	United States Forest Service
USFWS	United States Fish and Wildlife Service

A.3 Distribution List

The following is a distribution list of personnel that will receive a copy of the Sampling and Analysis Plan (SAP)/Quality Assurance Project Plan (QAPP) for the 2017-2018 Southern Ute Indian Tribe Wild Plant Sampling. Agency and contractor affiliations are also listed for each individual.

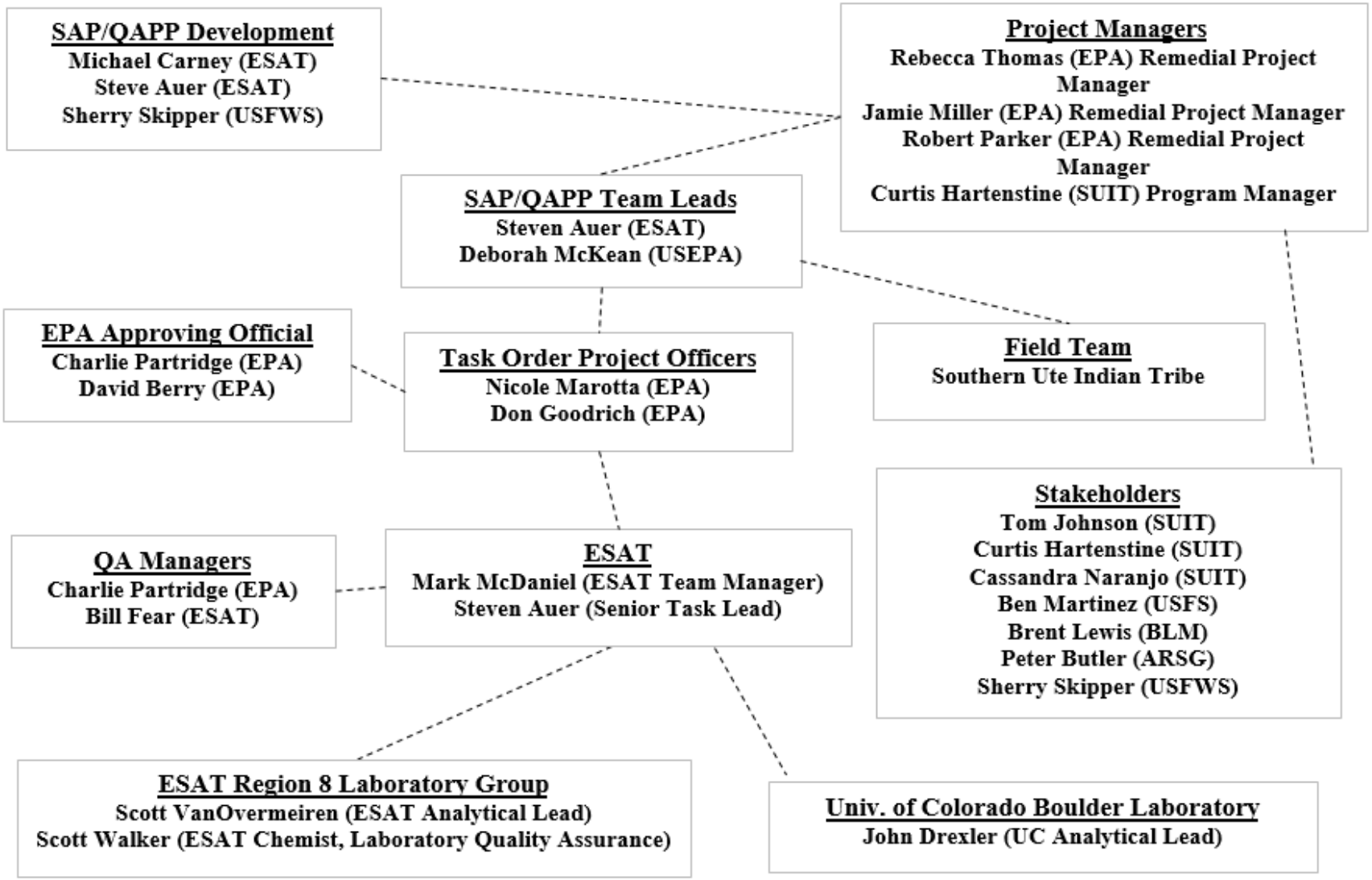
Rebecca Thomas	United States Environmental Protection Agency (EPA) Region 8 Remedial Project Manager (RPM)
Robert Parker	EPA Region 8 RPM
Joyel Dhieux	EPA Region 8 On-Scene Coordinator
Nicole Marotta	EPA Region 8 TOCOR
Dan Wall	EPA Region 8 Ecotoxicologist
Deborah McKean	EPA Region 8 Toxicologist
David Berry	EPA Region 8 Toxicologist
Andrew Todd	EPA Region 8 Ecotoxicologist
Brian Sanchez	EPA Region 8 Ecotoxicologist
Charles Partridge	EPA Region 8 Toxicologist
Brent Lewis	Bureau of Land Management (BLM)
Ben Martinez	United States Forest Service (USFS)
Sherry Skipper	United States Fish and Wildlife Service (USFWS)
Peter Butler	Animas River Stakeholders Group (ARSG)
Tom Johnson	Southern Ute Indian Tribe (SUIT) Environmental Programs Division
Curtis Hartenstine	SUIT Environmental Programs Division
Cassandra Naranjo	SUIT Environmental Programs Division

A.4 Project and Task Organization

The following is a list of involved project personnel involved with field sampling and chemical analyses processes, their respective agencies or contract affiliations, and key personnel project responsibilities.

Personnel	Organization	Project Responsibilities
Managers		
Rebecca Thomas Robert Parker	EPA	RPMs are responsible for project oversight/management, and document review, data quality and usability and corrective action (CA) administration; the Decision Maker
Nicole Marotta	EPA	ESAT TOCOR responsible for assessment and oversight of field sampling activities
Don Goodrich	EPA	Analytical TOCOR responsible for assessment and oversight of analytical activities
Deborah McKean	EPA	Human health risk assessor, responsible for human health data and sample collection activities
Mark McDaniel	ESAT	ESAT Team Manager responsible for staff oversight and document review
Sherry Skipper	USFWS	Technical Lead responsible for SAP/QAPP development
Delegated Quality Assurance Approving Officials (QAOs)		
Charles Partridge	EPA	EPA delegated quality assurance authorizing official
Bill Fear	ESAT	ESAT officer responsible for data validation and usability
Field Team		
Steve Auer	ESAT	Field Task Lead for EPA led field work, ESAT Field Quality Assurance (QA) Lead, Health and Safety Officer responsible for SAP/QAPP development, maintenance of the official approved SAP/QAPP, sample collection oversight, field documentation oversight, report review, and ensures field and laboratory procedures comply with this SAP/QAPP
Curtis Hartenstine	SUIT	Field Task Lead for SUIT led field work; SUIT Health and Safety Officer sample collection oversight and field documentation oversight
Laboratory Group		
Scott VanOvermeiren	ESAT	Analytical Task Lead responsible for sample intake, analysis and analytical report preparation
Scott Walker	ESAT	Analytical Support Lead responsible for sample intake, analysis, analytical report preparation, report review, ESAT laboratory QA management
John Drexler	Univ. of CO Boulder	Analytical support

Organizational Chart



A.5 Problem Definition/Background

The Bonita Peak Mining District (BPMD) is located in the upper Animas River watershed near the town of Silverton in San Juan County, Colorado. The watershed lies within and just outside of the extensively mineralized Silverton Caldera basin. Geologic resources in this basin contain large amounts of metals (Storosh, 2013). As such, the BPMD has been subject to both large and small mining operations in boom and bust fashion from 1871 to 1991. Years of mining operations and associated waste disposal practices have contaminated the local BPMD environment with metals (EPA, 2017a).

Due to releases of hazardous substances into the environment and potential human and ecological health impacts, EPA added 48 historic BPMD mines and mining-related sources to the National Priorities List (NPL) in April 2016 (EPA, 2016). Now designated as a Superfund site, EPA has the authority to investigate and remediate contamination from BPMD NPL sites to lessen or eliminate impacts to human health and the environment.

This SAP/QAPP was developed to define standard procedures, including quality principles and practices, for data collection to support human health risk assessment activities associated with the BPMD NPL sites, specifically, risks associated with metals exposure from wild plants that are culturally important to the SUIT. This information does not currently exist. Therefore, information on metals concentrations in wild plants needs to be collected. This SAP/QAPP provides guidance on wild plant sample collection, chemical analysis, and results reporting methods.

The wild plant sampling and analysis effort described in this SAP/QAPP will be split between the SUIT and EPA. Specifically, the SUIT will collect wild plant samples and EPA will analyze samples collected by the SUIT. Other than producing this SAP/QAPP, EPA will not be involved in field collection of plant tissue samples outside of the BPMD. This is in part due to inherent sensitivity of not revealing the locations of SUIT culturally important wild plant collection areas. Field data collection procedures provided in this SAP/QAPP are cognizant of the SUIT's need to keep plant collection locations private. Since EPA is analyzing samples, information regarding attributes and concentrations of metals in collected plant tissues will be known to EPA. This information is intended to be used in the future BPMD human health risk assessment without identifying locations of SUIT wild plant collection areas.

EPA may collect and analyze SUIT culturally important wild plants within the BPMD under the separate *2017-2018 Combined Risk Assessment Sampling Events* SAP/QAPP (EPA, 2017b). Data generated from this independent effort may be shared with SUIT members and used in the future BPMD human health risk assessment to estimate exposure to metals in human receptors.

This SAP/QAPP uses the Data Quality Objectives (DQOs) process to identify sampling objectives and associated assessment procedures (EPA, 1997). DQOs are used to ensure that the type, quantity, and quality of environmental data to be collected are suitable in supporting future BPMD risk assessments. This SAP/QAPP also provides detailed

information on how field data collection, plant sampling, and sample chemical analyses activities will be conducted. This data collection effort will accomplish the following study objectives:

- Determine concentrations of metals in culturally important wild plant species collected by the SUIT.
- Determine concentrations of metals in soils collected from plant sampling areas
- Determine the bioaccessibility of metals in plant tissues to human receptors.
- Collect exposure data to facilitate the development of the BPMD human health risk assessment.

A.6 Project and Task Description

This SAP/QAPP has been prepared in accordance with the EPA's *Requirements for Quality Assurance Project Plans QA/R-5* (EPA, 2001), *Guidance for Quality Assurance Project Plans QA/G-5* (EPA, 2002b), and *Guidance on Systematic Planning Using the Data Quality Objectives Process QA/G-4* (EPA, 2006). This SAP/QAPP is designed to guide collection and chemical analysis of plant tissue samples, including field Quality Assurance/Quality Control (QA/QC) samples.

The following data will be collected:

- Plant tissue samples collected by the SUIT from culturally important areas that will be chemically analyzed. **Table A.6-1** provides a list of plant species and sample types that will be considered. Samples will be analyzed for a comprehensive suite of total recoverable metals and mercury.
- Surficial soil samples collected from plant sampling areas that will be chemically analyzed for the same suite of total recoverable metals and mercury as plant tissue samples.
- Field data that documents species, sample type, collection, and location information. Field data will be recorded on project-specific field datasheets that are provided in this SAP/QAPP (**Attachment 1**).
- Plant species and sample type information documented on task-dedicated Chain of Custody (COC) forms. General sample collection locations will also be documented on COC forms. Specifically, whether sample collection sites are up- or downgradient from sources of BPMD contamination. This information will be recorded for each sample and is critical to sample analysis and the risk assessment process. As such, completed COC forms will be delivered to the ESAT Region 8 laboratory with respective samples. A blank copy of the project-specific COC form is provided in this SAP/QAPP (**Attachment 2**).

- Documentation of all deviations made to procedures described in this SAP/QAPP during sample collection, handling, and analysis. Deviations will be recorded using project-specific field and laboratory notebooks. Field notebooks that contain deviation information will be privy to both SUIT and EPA project personnel, as it is critical to sample analysis, data reporting, and the risk assessment process.

It is anticipated that sampling activities described in this SAP/QAPP will occur from the remainder of 2017 to the end of 2018. Chemical extraction analyses of collected samples will occur within 180 days for all metals with the exception of mercury which is 28 days. Chemical analysis and data reporting will likely occur over the 2017-2018 and 2018-2019 winter seasons.

Plant tissue sampling locations will be selected and accessed by the SUIT. Sampling locations will be in areas that are culturally important to SUIT. All potentially contaminated sampling locations will be within or downgradient from the BPMD. **Figure A.6-1** provides an overview map of the general BPMD assessment area (EPA, 2017a). Note that sampling locations that are not within the BPMD assessment area will be, by default, within the Animas River floodplain downgradient from Silverton, CO. Sampling locations should be as close to the BPMD as possible so that samples represent the greatest levels of BPMD-based metal exposure and to avoid interferences from other sources of metals contamination. As stated above, EPA may not be provided specific information on where plant samples are collected. EPA only needs confirmation that SUIT sampling occurs within the BPMD assessment area or the Animas River floodplain downriver of Silverton, CO. Section B.3.2 *Sample Custody, Shipping and Receiving* describes the sample location confirmation process that will be followed. Lastly, collocated soil samples will provide required critical information on the level of soil contamination at plant collection sites.

A.7 Quality Objectives and Criteria

This section discusses the DQO process and how it will be applied to this study. Specific areas addressed include the planning team and stakeholders, DQOs, and the parameter metrics precision, accuracy, representativeness, completeness, comparability, and sensitivity. SUIT wild plant sampling is intended to support the following goal:

- Collect samples of plants that are culturally important to the SUIT and collocated soils and analyze for metals so that exposure information can be incorporated into the future BPMD human health risk assessment.

A.7.1 Planning Team and Stakeholders

This section identifies members of the DQO planning team and their responsibilities. These individuals are primary decision makers and parties who may be impacted by the results of this study or who may use the data generated as a result of the DQO process.

Note that actual execution of field sample and data collection activities will be conducted solely by the SUIT or their representatives. However, sample information and chemistry

data obtained from field efforts will be used by EPA for future human health risk assessment activities. Given that EPA is the end user for data collected under this SAP/QAPP, EPA will lead the DQO planning teams and associated decision making.

A.7.1.1 DQO Planning Team

Table A.7-1 lists the DQO planning team members, their respective organizations, and affiliation with that organization. These individuals are responsible for working through the DQO process so that it is complete and sufficient in determining and achieving goals associated with wild plant and soil sampling, chemical analysis, and subsequent data uses.

A.7.1.2 Decision-Making Authority

The decision maker has the ultimate authority for making final decisions based on the recommendations of the DQO team. The Decision Maker for field activities is Rebecca Thomas or her designee, Jamie Miller or Robert Parker; the EPA RPM. The Decision Maker for laboratory activities will be Don Goodrich; the EPA DOA. Decision Makers are responsible for ensuring that the DQO process is complete and sufficient in determining and achieving goals associated with wild plant and soil collections, chemical analysis, and subsequent data uses.

A.7.1.3 Stakeholders

Stakeholders are parties who may be affected by the results of the study or persons who may later use the data resulting from the DQO process. **Table A.7-2** lists the impacted organizations and stakeholders, and the individuals that are representing those organizations.

A.7.2 Data Quality Objectives

The DQO process specifies project decisions, the data quality required to support those decisions, specific data needed, data collection requirements, and analytical techniques necessary to generate the specified data quality. The process also ensures that the resources required to generate the data are justified. The DQO process consists of the following seven steps:

1. State the problem;
2. Identify the goal of the study;
3. Identify the information inputs;
4. Define the boundaries of the study;
5. Develop the analytic approach;
6. Specify performance or acceptance criteria; and
7. Develop the plan for obtaining data.

During the first six steps of the process, the planning team develops decision performance criteria to help with the data collection design. The final step of the process involves

developing the plan to obtain the required data. The following sections discuss these steps and their application to this project.

A.7.2.1 Step 1: State the Problem

The BPMD contains numerous mining-related features (e.g., mine shafts, tailings piles, waste rock piles, adits, drainage tunnels, former smelter sites, slag piles, etc.) which release metals into the BPMD environment. When released into the environment, metals may be dissolved in water or adsorbed to soils, sediments, and suspended particulates. These materials are either carried downstream via the gulches and creeks into the Animas River or settle down along the way and becomes incorporated into the surrounding upland, floodplain, and river substrates. High-flow conditions during the annual snowmelt or rain events may entrain metals-laden particles and move them further downstream or deposit them along the banks of the waterways.

Plants could be exposed to metals via direct contact with contaminated water, sediments, and soils. If concentrations of metals in exposure media are great enough to be toxic, plants would not survive. When concentrations are lower than toxicity thresholds, plants could be present. When present, plants actively or passively assimilate metals from exposure media into their tissues. Assimilation rates and associated thresholds differ between plant species and environmental conditions. Metals can also differentially accumulate in different plant parts (i.e. leaves, stems, fruit, or roots) for the same species.

Humans can be exposed to metals when ingesting plants grown in metals-impacted areas. The SUIT collect and uses a wide variety of culturally important wild plant species. Wild plant uses range from a food sources and medicine to raw construction materials. The SUIT may be exposed to metals via ingestion when using plants during any of these uses. As such, information on metals concentrations in wild plant species used by the SUIT are useful in evaluating human exposure and associated health risks. Currently there is no information on concentrations of metals in SUIT plant species to evaluate such risks. Therefore, plant tissue chemistry data from plants traditionally used by the SUIT are needed.

A.7.2.2 Step 2: Identify the Goals of the Study

The purpose of this step is to define the Principal Study Questions (PSQs) that this study will attempt to resolve. The PSQs help determine appropriate data inputs and potential alternative actions. PSQs can be used to develop estimation statements to resolve the problem. Estimation statements are applicable to PSQ outcomes and may not lead to specific decisions, or the information may be used to gain a greater understanding of existing data.

There are three PSQs associated with this sampling effort. These PSQs are provided below with respective, corresponding estimation statements:

PSQ 1: *What are the concentrations of metals in SUIT culturally important plant species that are potentially exposed to BPMD contamination sources?*

Wild plant tissue sampling and associated metals analyses will be used to determine metals concentrations in plants utilized by the SUIT. Plant tissue metals concentrations will subsequently be used as inputs to estimate exposure and potential risk to SUIT members in the future BPMD human health risk assessment.

PSQ 2: *What are the concentrations of metals in soils where SUIT culturally important plant species are collected?*

Collocated soil samples and associated metals analyses will be used to determine metals concentrations in SUIT plant sampling areas. Soil metals concentration information is required to assess sampled plants exposure. This is particularly important since plant sampling locations will not likely be known to the EPA. Soil metals concentrations may also be helpful in assessing uptake of metals in sampled plants.

PSQ 3: *If metals are detected in sumac and willow plant tissues, are they bioavailable to humans*?

SUIT traditionally use their teeth to strip the bark off of sumac and willow branch bark when constructing woven basketry. A specialized chemical analysis will be used to determine bioaccessibility of metals in sumac and willow bark to determine if stripping bark with teeth poses a risk. This analysis replaces the typical low pH (<2.0) sample digestion process with a process that replicates a typical pH value (~7.0) of human saliva. Sample digestions will be analyzed for lead and arsenic. Data will be used to estimate exposure and potential risk to SUIT basket makers. Note that bark samples will also be analyzed using the typical <2.0 pH digestion to provide a range of potential *exposure.*

A.7.2.3 Step 3: Identify Information Inputs

The purpose of this step is to identify the data required to answer the PSQs. The primary information and decision inputs will be data generated from field observations and chemical analyses of culturally important plant species collected from locations potentially contaminated with BPMD-derived metals. The information inputs are summarized below.

Actions for field parameters associated with sample collection activities will include:

- Determining sampling locations;
- Identifying plant species that will be sampled;
- Collecting plant and soil samples;
- Documenting sample and sampling location information on task-dedicated field datasheets;
- Documenting deviations from this SAP/QAPP in a dedicated field notebook; and
- Recording all required sample information on respective COC forms.

Analytical laboratory required actions for plant tissue samples will include:

- Documenting receipt of samples;

- Determining whether or not sample handling and condition are within acceptable limits for chemical analysis;
- Sieving bulk soil to obtain a <2.0-micron fraction (#10 mesh sieve) for chemical analysis (when not done in the field);
- Peeling bark from sumac and willow branches (when not done in the field);
- Sumac and willow bark samples will be sent to Dr. John Drexler at the University of Colorado Boulder for bioaccessibility sample extraction analysis;
- Homogenizing heterogeneous samples;
- Storing samples under acceptable conditions until ready for chemical analysis;
- Chemically analyzing plant tissue samples for Target Analyte List (TAL) metals and mercury; and
- Reporting results and any deviations using appropriate data dissemination sources.

A.7.2.4 Step 4: Define the Boundaries to the Study

The objective of this step is to define the spatial and temporal components of the study area. This includes defining the scale of inference that identifies the types and numbers of samples to be collected and where samples will be collected. Temporal components are related to the anticipated timing of sample collection. Implementing this step helps ensure that the data are representative of the population or media under consideration.

Scale of inference

Only wild plants that are culturally important and used by the SUIT will be collected. A list of specific plant species is provided in **Table A.6-1**. This species list was developed using information on culturally important plants provided to EPA by the SUIT. The original SUIT plant list only included common names. After receiving the list, EPA matched common plant names to specific plant species. EPA researched and identified all potential species matching SUIT plant common names that would occur in Southwestern Colorado, Northwestern New Mexico, or Southeastern Utah. It is important to note that some SUIT plant common names were associated with more than one plant species. When this occurred, included plant species were often of the same genus. A number of other resources were also consulted when developing the species lists; including SUIT members and USFS stakeholders.

In addition to specific plant species, sampling will be specific to parts of each plant. **Table A.6-1** provides specific parts of each plant that will be collected. These include entire plants, roots, seeds, and berries that match cultural uses of each species. Cultural uses and plant parts were selected using information provided in the original SUIT plant list. Additional literature-based information sources were also consulted to identify plant uses. These sources included Brett (2003) and McBeth (2010) that provided species-specific accounts of Ute Indian Tribes plant use. Again, plant uses and respective plant parts were also reviewed by SUIT members.

Although this SAP/QAPP lists plants to a species level, this level of identification may not be achievable in the field nor is it required to achieve study objectives. That said, effort will be made to identify plants to a species level. At a minimum, plants will be identified to genus. This is the minimum taxonomic level that corresponds to the original list of plant common names and uses. One exception to this would be *Pinus* spp. This genus is common between three of the original common plant names and uses; piñon pine seeds, bristlecone pine seeds, and ponderosa pine cambium, seeds, cones, and resin. However, *Pinus* spp. are easily identified to the species level and doing so should be achievable.

Plants will be sampled from collection areas used by the SUIT. Sampling areas will also coincide with locations that may be contaminated with metals from the BPMD Superfund site. Reference sampling areas, not associated with BPMD contamination, should also be considered. Sampling areas that are within the BPMD assessment area (**Figure A.6-1**) or in the Animas River floodplain down river of Silverton, CO will be considered potentially contaminated with metals. Reference sampling areas will be in collection areas outside of the BPMD assessment area and Animas River floodplain. Reference sampling areas should be located in areas that are not likely contaminated. These areas may be located in non-impacted tributaries to the Animas River that are located outside of the BPMD. Determination of the total number of sampling areas will be made at the discretion of the SUIT. However, at least one potential BPMD-impacted area and one reference area will be sampled.

Temporal limits of the study question

Wild plant samples will be collected in the remainder of 2017 or spring, summer, and fall of 2018. This should provide the level of specification required to characterize current exposure conditions. Plant collections should coincide with the same season in which the SUIT traditionally collect wild plants. For most species of interest, this occurs when leafy plants are young and tender, roots and tubers have developed, and fruit and nuts are ripe. **Table A.6-1** provides the general seasons when plants are typically harvested. Sampling should be scheduled around these times throughout the 2017 and 2018 growing seasons.

A.7.2.5 Step 5: Develop the Analytic Approach

Wild plant tissue and collocated soil samples will be analyzed for TAL metals and mercury (**Table A.7-3**). Previous ecological risk assessments have shown that these metals and metalloids are associated with and may be present in areas downgradient from BPMD sources. A number of the metals and metalloids of interest are also known to accumulate in plant tissues.

Bioaccessibility samples will be analyzed for lead and arsenic by Dr. John Drexler at the University of Colorado Boulder Laboratory for Environmental and Geological Studies. Only sumac and willow bark samples will be analyzed for lead and arsenic bioaccessibility.

Analytical results will be used to determine risks associated with plant exposure in human receptors. Exposure estimates will be calculated using methods described in guidance for conducting human health risk assessments under the Superfund program.

The risk assessment process consists of four primary steps: Data evaluation, exposure assessment, toxicity assessment and risk characterization. Data will be evaluated for quality assurance and concentrations will be compared to conservative screening levels to identify contaminants of concern for the risk assessment. High end and central tendency exposures will be estimated for all current and future land uses and receptors identified at the site. These site-specific exposures will be compared to toxicity values for both cancer and non-cancer endpoints. The primary source for these toxicity values is EPA's Integrated Risk Information System database (http://www.epa.gov/iris/). Secondary sources include the EPA's National Center for Environmental Assessment provisional toxicity values, California Environmental Protection Agency and Agency for Toxic Substances and Disease Registry toxicity values. These site-specific exposures are compared to toxicity values to derive quantitative estimates of cancer and non-cancer risks. Estimates of quantitative risks should be carefully evaluated against the uncertainties associated with the sampling, analysis, and risk assessment.

A.7.2.6 Step 6: Specify Performance or Acceptance Criteria

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

Table A.7-3 provides the analytical laboratory Practical Quantitation Limits (PQLs) for each of the TAL metals and mercury. Although the range of metals concentrations in plant tissue and soil samples are unknown at this time, stated PQLs should be sensitive enough to detect each target analyte above respective toxicity thresholds (see Section A.7.3 for more details).

Sample collection processes will be consistent with Standard Operating Procedures (SOPs) and guidance provided in this SAP/QAPP to minimize the potential for false positive or negative errors associated with field sampling. When applicable, this includes collecting duplicate samples and implementing decontamination procedures. For laboratory analyses of samples, QA/QC steps, such as the use of laboratory controls, Matrix Spike/Matrix Spike Duplicate (MS/MSDs), and blank samples will be consistent with ESAT Region 8 reporting requirements.

Field duplicate samples will be used to determine sampling precision and the correlation between samples. According to the EPA (2017c) *National Functional Guidelines for Inorganic Superfund Methods Data Review*, a control limit of 35% for solid matrices for the Relative Percent Difference (RPD) shall be used for original and duplicate sample values that exceed five times the contract-required quantitation limit. Note, that these requirements are laboratory guidelines which may not apply to all field situations such as analysis of duplicate plant tissues samples. However, all duplicate sample RPDs will be calculated and reported. RPD values will be calculated using the following equation:

$$\text{RPD} = 100 \times \frac{|\text{Sample Result} - \text{Duplicate Result}|}{0.5(\text{Sample Result} + \text{Duplicate Result})}$$

Data collection, sample processing, chemical analyses, and reporting will follow steps and requirements described in EPA-approved SOPs. Appropriate QA/QC measures will be in place (*e.g.*, collection of field duplicates, laboratory splits, calibration data) as specified in this QAPP to reduce the risk of sampling and analytical error. Implementing these measures will result in a reliable analytical data set suitable for use in the future BPMD human health risk assessment. Analytical data will be evaluated in accordance with precision, accuracy, representativeness, completeness, and comparability parameters through use of laboratory control samples, calibration data, and results of MS/MSD samples. These parameters are presented **Tables A.7-4 and A.7-5**.

A.7.2.7 Step 7: Develop a Plan for Collecting Data

This SAP/QAPP includes all information and documentation materials needed to collect, chemically analyze, and report results associated with wild plant tissue and soil samples. Data collection will be largely based on a judgmental sampling design as described in Guidance for Choosing a Sampling Design for Environmental Data Collection QA/G-5S (EPA, 2002a).

A.7.3 Criteria, Action Limits, and Laboratory Detection Limits

Table A.7-3 provides PQLs for each target analyte that will be measured in plant tissue and soil samples. Tissue and soil PQLs were compared to available EPA (2017d) resident soil Regional Screening Level (RSL) values to determine if they are low enough for use in future BPMD human health risk assessments. When available, RSLs are provided for each target analyte in **Table A.7-3**. All PQLs are well below RSLs, indicating that analytical methods are expected to be sensitive enough for use in analyzing samples collected under this SAP/QAPP. However, some solid matrices can be difficult to analyze and may result in PQLs that exceed those provided in **Table A.7-3**. In the event that actual PQLs exceed the screening criteria, a RPM will evaluate the data to determine if the DQOs were met and determine the impact or limitations on the project.

A.7.4 Precision, Accuracy, Representativeness, Completeness, Comparability, and Sensitivity

This section describes how data generated during the course of this project will be validated. Documentation of the data evaluation effort will be in the form of the worksheets prepared during validation. These worksheets will be included as an appendix to a Sampling Activities Report (SAR) associated with this sampling and analysis effort.

The future SAR will identify issues with sample collection and analyses that may affect data usability or require that the data be qualified. The future SAR will also discuss all precision, accuracy, representativeness, completeness, comparability, and sensitivity parameter results from the data validation and overall usability of the data for project objectives. Any biases associated with results of these parameters will also be discussed.

Biases refer to the systematic or persistent distortion of a measurement process that causes errors in one direction. The extent of bias will be determined by evaluating the laboratory initial calibration/continuing calibration verification, Laboratory Control Spike/Laboratory Control Spike Duplicate (LCS/LCSD), blank spikes, and MS/MSD samples.

The data and associated biases will be assessed for the following criteria:

- *Precision* – The measure of agreement among repeated measurements of the same property under identical or substantially similar conditions that is expressed as the RPD between the sample pairs. An acceptable RPD is ≤ 35% for plant tissue and soil samples (EPA, 2017c).

    - Field duplicates: RPD criteria met?
    - Laboratory duplicates: RPD criteria met?
    - Method of standard dilution performed and criteria met?
    - MSD: RPD criteria met? (If applicable)

- *Accuracy* – The measure of how close measured values are to the true values being measured. Accuracy analyses are helpful in identifying systematic errors associated with sampling and analysis methods.

    - MS/MSDs: Are Percent Recovery (%R) criteria met?
    - LCS/LCSD samples: Are %R criteria met?
    - Initial and continuing calibration recoveries met?
    - Interference check sample recoveries met?
    - Inductively coupled plasma serial dilution recoveries met?

- *Representativeness* – The measure of the degree to which data accurately and precisely represent a characteristic of a population parameter, variations at a sampling point, a process condition, or an environmental condition.

    - Sampling procedures and design: criteria met?
    - Holding times and preservation: criteria met?
    - Custody: all COC forms complete and provided in data package?

- *Completeness* – A measure of the amount of valid data obtained from a measurement system. The actual percentage of completeness is less important than the effect of completeness on the data set. Completeness will be assessed by the total number of samples collected versus the amount of samples planned.

    - The number of valid analytical results is comparable with the number determined necessary during establishment of DQOs.

- *Comparability* – The qualitative term that expresses the confidence that two data sets can contribute to common interpretation and analysis. Comparability is used

to describe how well samples within a data set, as well as two independent data sets, are interchangeable.

- Data compares with similar analysis and data sets?
- Sample collection methods comparable to similar data sets?
- Laboratory analytical methods comparable to similar data sets?

- *Sensitivity* – The ability to discriminate between small differences in analyte concentrations related to the rate of change in response when there is a small change in stimulus; this is reflected in the calibration curve. The detection limits of the field and laboratory methods are within the range of previous detections found at the BPMD Superfund site.

    - Did chemical analyses meet or exceed PQLs documented in **Table A.7-3**?

Uncertainty of validated data will be evaluated by the RPM or her designee to determine if the DQOs were met. In the event that the DQOs were not met, they will be reviewed to determine if they are achievable and may be revised if necessary, and the data may be further evaluated to determine the impact to the project.

A.8 Special Training and Certifications

All field data and sample collection personnel should have experience conducting work described in this SAP/QAPP. All personnel associated with activities described in this SAP/QAPP must read this SAP/QAPP and understand materials and requirements presented herein. This includes all SUIT, EPA, and ESAT Region 8 laboratory personnel.

If plant and soil sampling is conducted within the BPMD Superfund site, then all field personnel will have the Occupational Safety and Health Administration (OSHA) *40-hour Health and Safety Course for Hazardous Waste Site Worker Training* certification prior to sampling BPMD plants. In addition to this OSHA training requirement, at least one person in each field team will be trained in first aid and adult Cardiopulmonary Resuscitation (CPR). The SUIT Field Health and Safety Officer, Curtis Hartenstine, will be responsible from adhering to the BPMD Superfund site health and safety requirements.

EPA does not have the authority to enforce health and safety training requirements for SUIT field sampling personnel outside of the BPMD Superfund site. If EPA personnel or subcontractors become involved with field data and sample collection activities, then they will be required to adhere to the standard Region 8 health and safety training requirements.

All EPA laboratory personnel and subcontractors will have completed the OSHA *40-hour Health and Safety Course for Hazardous Waste Site Worker Training* in accordance with Sections (e) and (p) of OSHA 29 Code of Federal Regulations (CFR) 1910.120 and maintain this certification with annual eight-hour Hazardous Waste Site Operations Refresher Training as required by Sections (e) and (q) of OSHA 29 CFR 1910.120.

All EPA field personnel and subcontractors will have completed American Red Cross Standard First Aid and adult CPR training and maintain this certification annually for adult CPR and every two years for Standard First Aid. The ESAT and EPA Health and Safety Managers are responsible for ensuring that all field staff complete the training requirements as required by OSHA.

ESAT Region 8 laboratory staff will have all of the necessary training and accreditations needed to chemically analyze plant tissue samples for TAL metals and mercury.

TechLaw and each agency will provide training for their respective employees. The training documentation for ESAT personnel is stored in SharePoint, while training documentation for EPA personnel is maintained by the individual.

A.9 Documentation and Records

Sample collection, handling, and analysis documentation will be recorded using field datasheets, field notebooks, COC forms, and ESAT Region 8 laboratory sample inventory and analysis files. Field data will be generated and maintained as described in this section. ESAT laboratory records will be generated and retained according to procedures described in the Final Revision 2 Quality Management Plan (QMP; ESAT, 2015) and EPA (2017c).

Field data will be recorded on wild plant sampling project-specific field datasheets at the time of data collection. A blank copy of this datasheet is provided in **Attachment 1**. The SUIT Field Team Manager, Curtis Hartenstine or his designee, will retain original copies of all field datasheets generated under this effort. At the end of each sampling day, all datasheets should be photocopied and/or scanned, and originals saved in a secure location. Completed field datasheets may or may not be shared with EPA. Electronically stored data should be backed up on a dedicated computer or network after each day's sampling activities. Electronic files should also be saved on at least two computers or regularly backed up network to avoid unintentional loss of data.

A project-specific field notebook will be used to document any deviations from this SAP/QAPP or applicable SOPs. The SUIT field team manager will be responsible for maintaining and retaining the field notebook. At the end of each sampling day, all notebook entries should be photocopied and/or scanned, and originals saved in a secure location. A copy of each sampling day's any new field notebook entries will be emailed to the ESAT TOCOR Nicole Marotta (marotta.nicole@epa.gov), ESAT Field Team Manager Steve Auer (auer.steven@epa.gov), and EPA RPM Jamie Miller (miller.jamie@epa.gov). The original field notebook will be retained by the SUIT field team manager. Copies will be maintained by ESAT Region 8 laboratory in perpetuity.

COC forms will be used to document project-critical sample attributes and will be used to relinquish samples from sample collectors to ESAT Region 8 laboratory staff. A blank copy of the project-specific COC form is provided in **Attachment 2**. The original COC form will accompany all respective samples when sending to the analytical laboratory.

This also includes bioaccessibility samples that will be sent to Dr. Drexler for chemical analysis. Prior to packaging COC forms with samples, SUIT personnel should photocopy and/or scan completed COC forms for their records. ESAT Region 8 laboratory will retain all original hard-copy COC forms and copies of COC forms sent to Dr. Drexler in a dedicated and secure location in perpetuity. More information on COC forms are provided in Section B.3.2 *Sample Custody, Shipping and Receiving*.

A peer review of the data package will also be produced by the ESAT laboratory. This data package will reflect the review for 100% of reported versus raw data. The final report of the data validation will be in a standard Contract Laboratory Program (CLP) format, including all laboratory and instrument QA/QC results.

The EPA TOCOR, Nicole Marotta, will receive the most current copy of the approved version of this SAP/QAPP. The TOCOR will also be responsible for distributing a final version of this SAP/QAPP to all personnel that are listed in **Table A.7-1** and **Table A.7-2**. The ESAT Task Lead will be responsible for maintaining an official, approved version of this SAP/QAPP and associated supporting documents.

B. DATA GENERATION AND ACQUISITION

This section describes data generation and acquisition activities associated with the sampling efforts described above, including process design, sampling and analytical methods, sample handling and custody, Quality Control (QC), equipment, and data use and management.

B.1 Sampling Process Design

BPMD sourced metals contamination has potential to accumulate in wild plants that are used by the SUIT. Plant tissue metals concentration data is required to estimate exposure and effects to SUIT members that utilize culturally important plants. The sampling effort described in this SAP/QAPP was designed to obtain information on metals concentrations in culturally important SUIT plant species to support future BPMD human health risk assessments.

Wild plant tissue sampling will only include plant species and respective plant parts that are culturally important and used by the SUIT. **Table A.6-1** provides a list of each plant species and respective parts that will be sampled. Since wild plant collection areas are culturally important to SUIT members, locations of collection areas will only be known to SUIT sampling personnel. EPA does not have any information for specific collection areas and does not require that level of detail to satisfy sampling objectives. However, EPA requires confirmation as to whether samples were collected from a potential BPMD impacted or reference area. This confirmation will be documented on the project-specific COC form as described in Section B.3.2.

EPA does not anticipate that every species of plant and plant part listed in **Table A.6-1** will be sampled. However, sufficient effort should be made to collect one composite sample of each plant species observed at each collection site. For sites with abundant

culturally important plant cover over a diverse landscape, more than one composite sample should be collected. At least one composite soil sample should be collected at each collection site where plants are also sampled.

Plants will be sampled from areas selected by the SUIT. Sampling areas may coincide with locations that may be contaminated with metals from the BPMD. This includes sampling areas that are within the BPMD (**Figure A.6-1**) or in the Animas River floodplain downgradient from Silverton, CO. Reference sampling areas not associated with BPMD contamination should also be considered. Reference sampling areas will be up gradient from BPMD or in non-impacted tributaries or watersheds. Determination of the total number of sampling areas will be made at the discretion of the SUIT. However, at least one potentially impacted and one reference area should be sampled.

Field duplicate samples will be collected at a rate of 1 duplicate sample per 10 field samples. This applies to both plant and soil samples. Given that more than a few plant samples will likely be collected at each plant collection site, duplicate sampling rates will be specific to plant or soil samples. A duplicate soil sample will be required if fewer than 10 soil samples are collected throughout the course of this project.

Wild plant samples will be collected in the remainder of 2017 or spring, summer, and fall of 2018. Plant collection should coincide with the same season in which the SUIT traditionally collect wild plants. For most species of interest, this occurs when leafy plants are young and tender, roots and tubers have developed, and fruits and nuts are ripe. **Table A.6-1** provides the general seasons when plants are harvestable. Sampling should be scheduled around these times throughout the 2018 growing season.

Plant samples will be sent to the ESAT Region 8 laboratory as soon after collection as possible. Samples will be in custody of the samplers and stored under appropriate preservation conditions prior to being sent to the ESAT Region 8 laboratory for analysis. Completed COC forms will be used to relinquish samples to the laboratory. All of the sample information specified on the project-specific COC form is critical to the goals of this project.

B.1.2 Nature of Data Collected

A variety of data will be collected during the 2017-2018 BPMD sampling events. Some of those data will be critical to achieve the established DQOs and project objectives, while others will be collected primarily for informational purposes or to supplement critical data, as shown below:

Data Type	Purpose
Plant tissue samples (analyzed for total recoverable metals including mercury and in-vitro bioavailability)	Critical
Collocated soil samples (analyzed for total recoverable metals and mercury)	Critical
COC forms with sample information	Critical

Field notebook that documents deviations made from this SAP/QAPP or applicable SOPs	Critical
Sample collection information documented on field datasheets	Critical
Photographs, photo log, and other ancillary field observations	Informational

B.1.3 Data Variability

Environmental data is inherently variable. However, sampling, sample handling, and sample analysis methods described herein are designed to reduce data variability. Efforts to reduce variability include using the same sampling equipment and methods throughout the project, sending samples to the laboratory as soon after collection as possible, and utilizing composite sampling strategies. Samples should also be collected within a discrete window of time by the same personnel. This is particularly important when collecting field duplicate samples.

An assessment of the variability associated with sample collection and laboratory analyses will be conducted using methods described in Section A.7.4. The final SAR will report results from accuracy and precision criteria testing analyses. Any uncertainties or biases that are identified will also be reported and discussed in the final SAR.

B.2 Sampling Methods

This section describes actual field-based plant tissue and soil sampling methods, applicable SOPs and work plans, and necessary equipment and support facilities that will be used for this project. Information provided herein, will supplement requirements stated in the *General Field Sampling Protocols* (SOP FLD-12.00) provided in **Appendix A** of this SAP/QAPP. SUIT field staff will be responsible for adhering to sample collection and handling requirements described in detail in this section. **Attachment 3** provides lists of the required sampling equipment.

B.2.1 Sampling Location Selection

A judgmental sampling design as described in *Guidance on Choosing a Sampling Design for Environmental Data Collection QA/G-5S* (EPA, 2002a) will be used to identify actual plant sample collection locations. Since this data will be used to assess risk from SUIT plants, sample locations should be those that are or could be used by the SUIT.

Sampling location selection will consider whether or not the collection areas are downgradient from BPMD contamination sources or in reference areas not impacted from BPMD contamination. Reference sampling locations are needed to determine potential impacts from BPMD contamination. Ideally, a series of downgradient sampling areas will be selected to represent a range of potential contamination. The SUIT will make final determination of the plant and soil collection locations and total number of sampling areas.

B.2.2 Sampling for Plant Tissue

This section provides details on how plant tissue samples will be physically collected.

All plant tissue samples will be composites made from subsamples collected from individual plants of the same species, taxon, or functional group from each sampling area. The number of subsamples in each composite sample will range from a minimum of 5 to a maximum of 30 and be determined in the field. This determination will consider the size of the area under investigation. Larger areas will require more subsamples or more than one composite sample. Subsampling will also consider the mass of each plant. Larger plants may need to be clipped into smaller pieces and homogenized before filling the sample container with subsamples. Only clean, decontaminated, stainless-steel knives and non-metallic cutting boards, mixing bowls, and mixing tools will be used to clip and homogenize subsamples.

New 1-gallon resealable plastic freezer bags, 1 liter wide-mouth HDPE bottles, 8 oz wide-mouth glass jars or brown paper bags can be used as sample containers. All sample containers will be new.

Each composite plant sample will consist of a minimum of 50 grams of plant tissue. For larger whole-plants or root samples, a composite sample representing at least five individual subsamples weighing a total of 200 grams will be collected for analysis. An absolute minimum of 20 grams wet-weight of willow bark is required. This sample mass should be enough to make two 10 gram samples needed to conduct in-vitro bioaccessibility and total metals analyses.

Instructions for the different sampling techniques that will be used to collect all culturally important plant samples are provided below.

B.2.2.1 Entire plant

Five subsamples will be collected from each plant to create one sample. Culturally important plant species are listed in **Table A.6-1**. Each entire plant sample must weigh at least 200 grams wet-weight.

First, put on new pair of nitrile gloves and obtain a decontaminated hand trowel for small plants or spade shovel for larger plants. Use the trowel or shovel to loosen the soil around the base of the plant. When the ground is sufficiently loosened, grab the base of the plant with your hand and pull up with a gentle twisting motion to remove the entire plant. Effort should be made to harvest equal proportions of the above and below ground biomass when collecting species that do not have definable root ball, propagate through rhizomes or grow in clonal colonies. Use a clean, decontaminated soft bristle brush to remove loose soil from the roots and stem base. Thoroughly wash all remaining soil off of the roots or bulb with deionized or reverse osmosis water. Also wash off the stem and leaves with deionized water. It may be more efficient to submerge the entire plant or just the foliage in a non-metallic bowl of fresh deionized or reverse osmosis water. Once all visible soil is removed from the roots and foliage has been washed, pat dry with a clean paper towel and put into sample container. If needed, plants can be clipped or cut into small pieces with a decontaminated Teflon coated or stainless steel knife on a clean, decontaminated, non-metallic cutting board before putting it into the sample container.

B.2.2.2 Roots and bulbs

Select SUIT plant tissue types will be processed into root and bulb composite samples using methods provided herein. Each root and bulb plant sample will be at least 200 grams wet-weight. Note that some of these plant types will also be sampled for other plant parts such as leaves and flowers. For these plants, root-specific samples will be collected and leaves and flowers will be sampled separately as described in Section B.2.2.3.

Remove the entire plant from the ground as described above for the entire plant sampling. Remove loose soil from the roots with a soft bristle brush. Set the entire plant on a non-metallic cutting board and clip off the root or bulb with a ceramic or stainless steel knife. Thoroughly wash all remaining soil off of the roots or bulb with deionized or reverse osmosis water. Once clean, pat dry with a clean paper towel and place into sample container. When compositing larger roots and bulbs, they may need to be cut into smaller pieces in order to be placed in the sample container. This step will be done after they are thoroughly washed and dried. Cleaned roots and bulbs should be processed on a decontaminated cutting board then put into the sample container.

B.2.2.3 Leaves, needles, flowers, buds, fruits, berries, seeds, acorns, and whole pine cones

Many SUIT plants will be sampled for or combinations of leaves, needles, flowers, buds, fruits, berries, seeds, acorns, and whole pine cones. If leaves, flowers, and buds occur for a single plant type and are present, they can be combined into a single plant type-specific composite sample. Fruits, seeds, acorns, and pine cones will not be combined with any other plant parts.

If needed, position a ladder next to the sampling area. Next, put on new pair of nitrile gloves and obtain a decontaminated non-metallic bowl. Using gloved hands, pick sampled vegetation from the plant(s) and place into the bowl. Once enough mass has been collected, thoroughly wash all of the collected vegetation using deionized or reverse osmosis water. Once clean, pat dry with a clean paper towel and put into sample container. If collecting fruits, nuts, or cones from the ground, the same methods will be used, except that samplers may need to brush off any soil before washing with deionized or reverse osmosis water.

B.2.2.4 Whole stems with leaves

Many SUIT plant samples will consist of whole stems with leaves (**Table A.6-1**). Each whole stems with leaves composite sample will be at least 200 grams and be composed of subsamples collected throughout each sample collection area.

First, put on new pair of nitrile gloves and obtain a newly decontaminated pair of stainless-steel garden sheers or similar stainless-steel cutting tool and a non-metallic bowl. Working on one sample type at a time, use the cutting tool to clip off whole stems with leaves. For grasses, the sample will represent the upper ¾ of the aboveground

biomass; including any flowers or seeds that may be present. For herbs and sedges, the sample will represent young tender branches off of the main stem; including petioles, leaves, buds, and seeds. Tender young stems and shoots of shrubs and trees will be targeted and will include any leaves, buds, and catkins.

Whole stem with leaves subsample clippings will be placed into the bowl of deionized or reverse-osmosis water and carefully prodded to dislodge any soil, dust, or insects. The vegetation will then be removed from the water bath using gloved hands and placed on clean paper towels. Use more paper towels to pat dry. Next, transfer the vegetation sample to a new, labeled sample container. Repeat the process with a new bowl of water until enough sample mass is collected.

B.2.2.5 Sumac and willow stems and outer bark

The SUIT sumac and willow samples will consist of cleaned stems with bark and bark only samples. Methods described herein will be used to collect these two sample types.

First, put on new pair of nitrile gloves and obtain a newly decontaminated pair of stainless steel garden sheers or similar non-metallic or stainless steel cutting tool. Also set out a clean, decontaminated cutting board. Use the cutting tool to remove stems from the plant. Only collect a few stems at one time to avoid having to place them on the ground while they are processed. Process stems using a gloved hand to pick off all leaves and smaller twigs from the stem. Rinse off the stem with deionized or reverse osmosis water and pat dry. If collecting a whole stem sample, place whole stems into the sample container. Repeat cutting, cleaning, and rinsing stems until a sufficient sample mass is collected; approximately 200 grams wet-weight.

The outer bark of sumac and willow stems will also be collected. Bark will be removed after the stems are cleaned of leaves and twigs and have been rinsed. The first step in debarking stems is to soak them in water. To do this, fill a non-metallic bowl or other watertight container, such as a resealable plastic bag with deionized or reverse osmosis water. Place clean and rinsed stems in the water and soak them for at least 1 hour before stripping the bark. Peel the outer bark from the stems using gloved hands and a ceramic or stainless-steel knife. Place the bark directly into the sampling containers (one 8 oz wide-mouth glass jar for in-vitro bioaccessibility and one jar for total metals analysis) and discard the debarked stems. Repeat as necessary to obtain the required sample mass for each sample (ideal 50 grams wet-weight; minimum 20 grams wet-weight).

Note that bark stripping can also be conducted by the ESAT Region 8 laboratory. If processed in the laboratory, whole sampled plants will be placed in labeled brown paper bags in the field and carefully transported to the laboratory. Bagged plants should be stored in a cardboard box or similar breathable over pack container in a cool, dark place while in the field. They will be processed as soon as possible after being delivered to the ESAT Region 8 laboratory. Processing should occur within 1 week of collection. The sample label information on the brown paper bags will be transferred to the actual sample containers upon processing. The sample collection date and time will represent when the plants were harvested in the field.

B.2.2.6 Piñon nuts

Piñon pine nuts can be harvested in a variety of ways. The method described herein will be used to increase chances that a sufficient mass of piñon pine nuts can be collected under clean conditions. Harvesting pine nuts involves removing ripening cones from the tree and letting them dry out over the course of a couple weeks to a month. After drying, nuts can be extracted and processed into samples. Since cone collection occurs far in advance to processing, more cones should be collected than needed to ensure that enough mass is obtained.

At the beginning of September, piñon pine cones will be close to opening and are ready to harvest. First, put on a new pair of nitrile gloves. Next, remove the cone from the tree. Use the ladder to pick cones from higher branches. Look for green cones that have not opened and are covered with sticky pitch. Do not let the cones contact the ground to prevent soil and other unwanted debris from sticking to the cones. Once collected, place cones in a shallow pan in a dry, dark place. Do not stack cones more than two deep or they may get moldy before drying out. After three to four weeks, the cones will dry out and open. Use gloved hands or non-metallic tools to pick, shake, or beat nuts from the cone. Next, separate empty nuts by placing all nuts into a bowl of deionized or reverse osmosis water. Retain all of the nuts that sink and discard all nuts that float; floating nuts are most likely empty husks. Pat dry retained nuts with a paper towel. Next, nuts must be removed from the shells. Remove shells by pinching and rolling the husk with your fingers. Non-metallic tools can also be used to remove husks. Effort should be made to get husks off in one piece so that shelled nuts are clean. Discard husks and place shelled nuts into the sample container. Repeat as necessary to obtain the required sample mass for each sample (ideal 50 grams wet-weight; minimum 20 grams wet-weight).

B.2.2.7 Cambium (inner bark)

Cambium is a thin layer of pliable tissue, between the rough, outer bark and woody parts of the pine tree. Getting to and removing the cambium from the bark can be difficult, but is easier in the spring when the tree is producing a lot of sap.

First put on a new pair of nitrile gloves and obtain a newly decontaminated pry bar or similar tool. Use the pry bar to remove strips of outer bark from the tree. Cutting perpendicular slots at the top and bottom of the removal area might make removing strips of bark easier. The cambium is a distinct and separate wood colored layer that will come off the tree with the stripped outer bark. Working one strip of pealed bark at a time, peel the thin cambium layer from the outer bark. Depending on the size of the bark strip, the cambium layer should come off fairly easy and in one piece. It might be necessary to use a clean, decontaminated knife to start at a corner of the cambium before pealing from the outer bark. Once peeled, cambium should be rinsed off with deionized or reverse osmosis water and patted dry before placing in the sample container. Remove another strip of bark from the tree and repeat the steps listed above until a sufficient mass of sample is collected (ideal 50 grams wet-weight; minimum 20 grams wet-weight).

B.2.2.8 Resin

Resin will be collected by searching the trunk and branches for resin that is naturally present. This is a noninvasive, low effort way to collect resin. First, put on a new pair of nitrile gloves and obtain a new, labeled glass sampling jar. Search for resin oozing from damaged parts of the tree such as trunk scars and broken branches. Pick off of tree or collect resin directly into the sampling jar. Continue collecting resin until required sample mass is achieved (ideal 50 grams wet-weight; minimum 20 grams wet-weight).

B.2.3 Soil Sampling

Surficial soil samples will be collected from each SUIT-plant collection area. All soil samples will be collected in accordance with *Standard Operating Procedures for Soil Sampling* SOP FLD-05.00 (**Appendix A**) and as described in this SAP/QAPP.

A random 15 multi-point composite sampling method will be used to collect soil samples. Subsamples will target the same individual plants that were subject to tissue sampling. Sampling should also target fine-grained soil that does not contain rocks, soil invertebrates, or plant matter (roots, leaves, or sticks). All soil subsamples should be collected at a depth of 0 to 6 inches using dedicated, disposable Teflon scoops. Deeper sample may be collected for plant types with deep root systems. All subsamples will be combined in the sampling container or a decontaminated non-metallic or similar disposable mixing bowl, thoroughly homogenized, and sieved using a #10 mesh sieve. All soil samples collected by EPA will be sieved at the ESAT Region 8 laboratory before the samples are analyzed. All soil samples collected by SUIT will be sieved in a clean processing area. A minimum of 200 grams of soil will be collected in an 8 oz wide-mouth glass jar for each sample. The minimum sample weight should be enough to obtain the required sample mass for chemical analysis after considering sieving loss and analytical QA/QC sampling. If samples have limited fine-grained soil components, then an additional 8 oz glass jar should be collected. This sample label and COC entry should have the same sample information as the accompanying field sample.

All non-disposable sample collection and sample processing equipment will be decontaminated before collecting each plant collection area using the decontamination procedures described in Section B.2.9 of this SAP/QAPP.

Soil sample collection information will be written on the sample label, in the field notebook, and on the COC form. Each sample jar will be wrapped with clear packaging tape to cover the lid and label. The taped jar will then be placed into a secondary container, such as a resealable plastic or bubble baggie for protection. Samples will be placed in a hard-sided cooler containing ice packs or wet ice to maintain them at 4 degrees Celsius (˚C) plus or minus 2˚C.

B.2.4 Sample Preservation and Storage

Sample preservation procedures will be followed so that samples are maintained in good condition from the time of collection to analysis. Preservation methods described herein are consistent with the *Sample Preservation*. SOP FLD-03.00 (**Appendix A**).

In the field, soil and plant tissue samples will be stored in coolers on ice at approximately 4˚C. Ice packs or bagged ice should be used so that liquid does not contact sample containers. Samples should also be kept out of direct sunlight. After transport from the field, plant tissue and soil samples should be placed in a standard refrigerator as long as the refrigerator temperature maintains them at 4˚C plus or minus 2˚C. When received by the ESAT Region 8 laboratory, plant tissue samples will be stored at 4˚C prior to analysis.

B.2.5 Equipment Decontamination

All sample collection and preparation equipment will be decontaminated prior to and after collecting each plant sample. The same soil sampling collection and processing equipment can be used to collect all soil samples in a single plant collection area. New or decontaminated soil sampling equipment will be used at each plant collection area. Disposable plastic scoops do not require decontamination. Decontamination is not required between subsamples destined for the same composite sample. Decontamination will follow requirements and procedures described in the *Sample Equipment Decontamination* SOP FLD-02.00 (**Appendix A**) and this SAP/QAPP section.

A three step decontamination procedure will be used for this project. The nitric acid rinse step that is specified in the attached SOP will not be conducted because nitric acid is highly corrosive and a powerful oxidizing agent that can easily cause chemical burns to skin and eyes.

First put on a new pair of nitrile gloves. The first decontamination step involves using a phosphate-free detergent such as Alconox or Liquinox to thoroughly clean all residues off sampling equipment. A set of dedicated detergent-specific scrub brushes with different sizes and shapes should be used. The next step is a tap water rinse. Rinsing will continue until all traces of detergent are removed. The item should be thoroughly inspected for any signs of remaining contamination after being rinsed. If residues are found, repeat detergent and tap water rinse steps until it is visibly clean of residue. The final decontamination step is a deionized or reverse osmosis water rinse. After this final rinse, let the item air-dry before use. If not immediately being used, decontaminated items should be placed in a new, clean plastic bag.

Decontamination wastewater should be captured and collected in 5 gallon buckets when in the field. Wastewater should not contain any hazardous substances and be disposed of by pouring contents out down a municipal wastewater drain. Accumulated solids and sediments should be wiped out and disposed of in a municipal trash can.

The equipment list provides materials and equipment that will be needed to decontaminate sampling equipment (**Attachment 3**).

B.2.5.1 Equipment Rinsate Blank Sampling

Equipment rinsate blank samples should be collected to assess adequacy of sampling and sample processing equipment decontamination and evaluate the potential of sample contamination during collection.

Equipment rinsate blank samples should be collected after decontaminating field and laboratory sample collection and processing equipment. At least one rinsate blank sample should be collected each day of field sampling. At least one rinsate blank sample should also be collected when samples are sieved so that sample processing equipment decontamination procedures can be assessed. Multiple rinsate blank samples should be collected on a single day when extensive sample collection or sieving equipment decontamination is conducted or new sampling equipment is decontaminated and used.

Rinsate blanks will be processed in the field or laboratory by pouring metals-free distilled, deionized, or reverse osmosis filtered water over newly decontaminated sampling or sample processing equipment and into the sample container; one certified clean 250 milliliters high density polyethylene bottle per sample. All rinsate blank samples will be analyzed for TAL metals using EPA (1994a,b) Method 200.7 and 200.8 and mercury using EPA (2007) Method 245.1 Revision 3.0. Both TAL metals and mercury analyses will be conducted using the single 250 milliliter sample bottle. All samples were preserved with nitric acid in accordance with SOP FLD-03.00 *Sample Preservation* (EPA, 2012; **Appendix A**) once back from the field and in the laboratory.

All rinsate blank samples will be handled the same way as plant tissue and soil samples (Section B.3). This includes writing the date, time, and sampler's initials on the sample label and COC tracking form in accordance with *SOP FLD-11.00 Sample Custody and Labeling* (EPA, 2012; **Appendix A**). The label will be affixed to the sample container and should covered with clear tape to prevent loss of the label or information. All samples will be placed in a cooler with ice for transport to the EPA Region 8 ESAT laboratory and stored at 4˚C until analysis. Rinsate blank samples have the same maximum hold times as plant and soil sample which is 180 days for TAL metals and 28 days for mercury (**Table A.7-3;** Section B.3.1).

B.2.6 Deviations and Corrective Actions

Deviations from this SAP/QAPP will be documented using a task-dedicated field notebook. Deviations resulting in major modifications to this SAP/QAPP will be noted and incorporated into all related SAP/QAPP addenda, and applied, as necessary, to subsequent sampling events. Data obtained from these investigations will be used in accordance with the provisions outlined in the DQOs. A copy of the notebook will be provided to ESAT TOCOR Nicole Marotta (marotta.nicole@epa.gov). Original notebooks will be retained by the SUIT Field Team Manager, Curtis Hartenstine or the ESAT Field Task Lead, Steven Auer.

The SUIT Field Team Manager, Curtis Hartenstine or his designee will be responsible for directing Corrective Actions (CAs) if problems are encountered in the field which would impact the way this SAP/QAPP is implemented. Any problems encountered and CAs taken or deviations from this SAP/QAPP will be documented in the task-dedicated field notebook. When deviations occur and are recorded, corresponding field notebook entries will be scanned and emailed to ESAT TOCOR Nicole Marotta (marotta.nicole@epa.gov), ESAT Field Team Manager Steve Auer (auer.steven@epa.gov), and EPA RPM Jamie Miller (miller.jamie@epa.gov). The RPM or her designee will review the field notebook entries and follow up with the field manager as needed. The SUIT Field Team Manager will immediately notify the RPM or her designee to report and discuss CAs for any major deviations. Major deviations include any problems or deviations that jeopardize usability of field or laboratory data in supporting task objectives.

B.3 Sampling Handling and Custody

This section provides information on sample and data handling procedures, including maximum sample hold times, labeling samples, and instructions on how to prepare samples under COC for transport to the ESAT Region 8 laboratory for analysis.

B.3.1 Maximum Sample Hold Times

Sample hold times refer to the maximum length of time from collection to chemical analysis. If hold times are exceeded, samples may be compromised and may not be suitable for chemical analysis. Maximum holdings times differ depending on the target analytes (**Table A.7-3**). The maximum hold time is 180 days for total recoverable metals, except for mercury, which has a maximum hold time of 28 days. Samples should be sent to the ESAT Region 8 laboratory immediately after sampling to avoid the risk of exceeding maximum hold times.

B.3.2 Sample Identification and Labeling

Sample containers should always be labeled with a permanent marker with the sample identification (ID), date and time of collection, analysis to be performed, and sampler's name or initials prior to or upon sample collection. Sample labeling is required to chronicle all sample handling for collection or creation through analysis and/or disposal. Sample ID and labeling will follow procedures described in the *Sample Custody and Labeling* SOP FLD-11.00 (**Appendix A**) and this SAP/QAPP section.

Sample ID designation will consist of a series of letters and numbers to indicate the plant type, plant part, and unique ID. The plant type designations area listed in **Table A.6-1** and the plant part designations are listed in **Table B.3-1**. The unique ID number will follow the plant type and part designations for all plant samples collected for this sampling effort. The unique ID will be a sequential 3-digit number starting at -001 for the first sample collected and continue thereafter until the last sample. No samples will have the same unique ID designation. If more than one field team is simultaneously deployed, each team should be provided a block of unique IDs so that sample IDs are not duplicated. All duplicate plant samples will have the same sample ID (including the unique ID) as the corresponding assessment sample followed by a "-D".

The combination of the plant type, plant part, and unique ID will form the complete sample ID for all plant samples. Each designation will be separated by a dash. For example, the sample ID for a sumac stem sample that was the fifth sample collected would be SMC-STM-005. If the next sample was the bark of a willow stem, the corresponding sample ID would be WIL-BRK-006. A corresponding bark of a willow stem field duplicate sample ID would be WIL-BRK-006-D.

Soil samples will be labeled using the SOIL- sample ID designation followed by a sequential unique ID. The sequential unique ID will start with -01 and be independent from the plant samples. All duplicate soil samples will have the same sample ID (including the unique ID) as the corresponding assessment sample followed by a "-D". The COC form will clearly indicate which soil sample is associated with each or group of plant tissue samples. See Section B.3.3 for more details on how to complete COC forms.

Rinsate blank sample IDs will reference this project, sample type, and sequential sample count using the following designations:

SUIT Plants-Equipment Blank–##

The sequential sample count will start at -01 and continue sequentially until the last equipment blank sample is collected.

B.3.3 Sample Custody, Shipping and Receiving

All samples will be collected, stored and shipped or transported to the ESAT Region 8 laboratory following COC protocols described in the *Sample Custody and Labeling* SOP FLD-11.00 (**Appendix A**) and this SAP/QAPP section. The same COC procedures will also be used to document, store and ship sumac and willow bark samples that will be analyzed for bioaccessibility. The ESAT Region 8 laboratory will ship bioaccessibility samples to Dr. Drexler.

Sample COC is required to ensure that sample integrity is not compromised from time of collection and analysis to destruction. COC procedures also provide documentation of sample handling from time of collection to destruction. Sample custody begins with the physical collection of a sample. The individual sample collector or Field Team Manager supervising the sample collector will be the first person (Sample Custodian) that has custody of a sample. The Sample Custodian is responsible for ensuring that the custody of each sample is not jeopardized from collection to the time that it is relinquished to the analytical laboratory. The Sample Custodian is also responsible for documenting sample collection information on the sample label and COC form. When samples are sent to the ESAT Region 8 laboratory, the Sample Custodian is responsible for preparing samples for shipment and relinquishing samples using COC forms. The laboratory person that receives the samples and respective COC forms will reconcile sample shipments to COC forms and sign the COC forms. This procedure effectively transfers custody of the samples from the Sample Custodian to the analytical laboratory. If additional transfers are needed, the original sample COC forms will be used to document custody transfers.

The same original COC form will accompany all respective samples. However, when custody transfers require splitting samples recorded on a single COC, a new COC form is required to clearly documents custody actions.

Sample labels and COC forms will be completed as soon after sample collection as possible. The *Sample Custody and Labeling* SOP FLD-11.00 (**Appendix A**) provides direction on how to label samples and a blank copy of the project-specific COC form is provided in **Attachment 2**.

COC forms will be completely filled out by the Sample Custodian or Field Team Manager. First, the Sample Custodian or Field Team Manager contact information will be filled out in the upper left-hand corner of the COC form. The first couple of entries in the sample information entry lines contain the three elements of the plant sample ID. Sample date and time refer to the exact time when the sample was collected. Time entries should be in 24-hour time format. The COC form also contains an entry to identify whether the sampling location was potentially impacted from BPMD contamination (impacted) or is up gradient from BPMD contamination sources (reference). This is the lowest level of location information needed to use analytical data in the future BPMD human health risk assessment and is critical to this project. The "Soil sample ID" entry will be used to relinquish custody of the collocated soil sample and identify which soil samples correspond to which plant samples. Note that this entry will likely be repeated for every plant sample collected within a single plant collection area. The "Notes" entries should be used to document plant species when known or to document any important sample-specific issues or characteristics. Sample Custodian contact information is required and will be filled out accordingly. Samples are relinquished and transferred using the "Relinquished By" and "Received By" entries.

Samples should be shipped to the ESAT Region 8 laboratory as soon after collection as possible. Samples should be shipped in a hard-sided cooler. Samples should be carefully packaged with ice packs so that they are maintained at or below 4˚C. Samples packaged in brown paper bags should also be shipped in a cooler, but only with ice packs and not with wet ice. Samples should be double bagged with some airspace so that they a cushioned from one another and ice packs. A tap water filled plastic bottle, labeled "Temperature Blank" should be placed in each cooler. The laboratory will check this bottle to ensure that sample temperature was maintained during shipment. The temperature blank sample does not need to be documented on respective COC forms. Prior to sealing each shipping cooler, the original COC form will be signed and photocopied. The Sample Custodian will retain the photocopied COC form and seal the original COC form in a Ziploc bag in the shipment cooler. Note that each shipment cooler will only contain samples that are on respective COC forms. As such, COC forms cannot be split between coolers. Once the original signed COC form is placed in the cooler, the cooler will be sealed with packaging tape so that samples are not jeopardized during shipment.

Coolers will be shipped to the EPA Region 8 laboratory using a commercial carrier service. It is important that samples are not shipped on a Friday as samples will only be received Monday through Friday from 8:00am to 4:00pm.

Upon receipt, the ESAT Region 8 laboratory staff will inspect the coolers to make sure that the proper temperature was maintained, sample containers are intact and sealed, and samples in the coolers match the information provided on the COC forms. Any evidence that the cooler was opened will documented by the sample receiving group and the EPA analytical TOCOR will be notified. Once all of the samples are accounted for, the sample receiving group or designated laboratory staff member will sign and date the "Received By" entry on each COC form. This staff member will also contact the Sample Custodian via email to let them know which samples were received and in what condition. The Sample Custodian should reconcile what was received with their records and implement any changes that are needed to improve sample condition during shipment.

Once at the ESAT Region 8 laboratory, all samples will be stored in an access-controlled sample cooler. Sample receiving staff will then maintain custody until data management enters the information into Scribe and the Laboratory Information Management System (LIMS). An analytical chemist will log the samples in LIMS upon receipt and will enter all analytical data into the Scribe database for permanent storage and archiving.

The Region 8 laboratory shipping address is:

ESAT Region 8 Laboratory
Attention: ESAT/ SUIT Plant samples
16194 West 45th Drive
Golden, CO 80403
(303) 312-7773 or (303) 312-7702

Willow bark bioaccessibility analysis samples will be sent to Dr. Drexler using the following shipping and contact information:

Attn: Dr. John Drexler
UCB 399 Geological Sciences
University of Colorado Boulder
Boulder, CO 80305
Phone: (303)492-5251
Email: drexlerj@colorado.edu

B.4 Analytical Methods

This section describes the ESAT Region 8 laboratory methods regarding chemical analysis of plant tissue and soil samples. These methods are provided in this section and three SOPs available in **Appendix A** of this SAP/QAPP:

- *Determination of Metals by Inductively Coupled Argon Plasma-Mass Spectroscopy (ICP-MS)* – SOP 16-MET-01.01

- *Analysis of Trace Metals Using the Perkin Elmer Optima 4300DV ICP-OE* – SOP 16-MET-02.02
- *Mercury Analysis of Soils, Sludge, and Biota by Thermal Decomposition Using the NIC MA3000 Direct Mercury Analyzer* SOP 16-MET-04.01

Plant tissue and soil samples will be analyzed for TAL total recoverable metals and mercury. **Table A.7-3** includes the laboratory analytical instrumentation and methods to be used for sample analysis. Soil and tissue total recoverable metals analysis will follow EPA Method 200.7 *Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry*, Revision 4.4 (EPA, 1994a) or Method 200.8 *Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma-Mass Spectrometry*, Revision 5.4 (EPA, 1994b). Soil and tissue sample metals digestions will be in accordance with EPA Method 200.2 Sample Preparation Procedure for Spectrochemical Determination of Total Recoverable Elements (EPA, 1994c).

EPA Method 7473 *Mercury in Solids and Solutions by Thermal Decomposition, Amalgamation, and Atomic Absorption Spectrophotometry* (EPA, 2007) will be used to analyze tissue and soil samples for total mercury. This method uses a direct mercury analyzer in which samples are placed directly into a NIC MA3000 analyzer. An aliquot of each sample will be removed from the bulk sample in the laboratory and placed into the mercury analyzer so that separate total recoverable metals and mercury samples do not need to be collected in the field. The direct mercury analyzer will be the primary method for tissue and soil mercury analysis. Enough sample mass should be easily attained for soil and most tissue types. However, EPA Method 245.1 *Determination of Mercury in Water by Cold Vapor Atomic Absorption (CVAA) Spectrometry* (EPA, 1994a; **Appendix A**) will be used for mercury analysis when original sample mass is too small to split between total recoverable metals and mercury analyses. This situation may occur with cambium or resin samples where sample masses are expected to be small. This method splits the sample for total recoverable metals and total mercury analyses after digestion. The mercury digestion split is then analyzed using Perkin-Elmer Flow Injection Mercury System 100 mercury analyzer by CVAA. As such, Method 245.6 digestion is in accordance with EPA Method 200.2 (EPA, 1994c).

Analytical results for each plant tissue sample will be reported on both a wet- and dry-weight basis. Alternatively, percent moisture or solids results will be reported for each sample so that results can be normalized to wet- or dry-weight values. This information is needed to model human health exposure.

A subset of samples will be subject to an *in situ* bioaccessibility analysis. This analysis will be conducted by Dr. John Drexler at the University of Colorado at Boulder Laboratory for Environmental and Geological Studies Department. The analysis methods will be provided in the final SAR. More information on bioaccessibility analyses are provided in Section B.4.1 of this SAP/QAPP (next subsection).

Sample disposal of potentially hazardous waste will follow the protocol defined in the *Laboratory Waste Management* SOP 16-LAB-01.01 (**Appendix A**).

The ESAT laboratory Analytical Task Lead will report any sample analysis problems, failures, and deviations from this SAP/QAPP to the RPM or their designee. The Analytical TOCOR and RPM will be responsible for directing CAs if problems are encountered during sample analyses that may impact the implementation of this SAP/QAPP. Any problems encountered and CAs taken or deviations from this SAP/QAPP will be documented in the project data package narrative.

B.4.1 Bioaccessibility Analysis

A subset of sumac and willow shoot bark samples will be subjected to bioaccessibility analysis. This analysis involves a less aggressive sample digestion procedure than the standard EPA Method 200.7, 200.8, and 245.1 analyses. Note that bioaccessibility samples will only be analyzed for lead and arsenic.

Bioaccessibility analyses will be conducted by Dr. John Drexler of the University of Colorado at Boulder Laboratory for Environmental and Geological Studies Department. This *in vitro* analysis is designed to mimic metals exposure and uptake from using one's mouth and teeth to remove bark from sumac and willow shoots. The analysis methods will be provided in the final SAR.

Bioaccessibility samples will be sent from the ESAT Region 8 laboratory sample custodian to Dr. Drexler for analysis. Dr. Drexler will digest these samples using bioaccessibility protocols and likely analyze them for lead and arsenic in-house.

B.5 Quality Control

A series of QC sampling and analysis procedures will be implemented during the course of this project to ensure that data are of known quality and repeatability. These include the use of field duplicates and analytical laboratory QA/QC samples.

Field duplicate plant tissue and soil samples will be collected at a rate of one duplicate per ten samples collected. If possible, duplicate sample selection should also consider plant types, so that at least one duplicate sample is collected for each of the SUIT culturally important plant types.

The ESAT Region 8 laboratory will be responsible for ensuring that PQLs will be at or below those reported in **Table A.7-3**. Note that PQLs were selected so that analyses are sensitive enough to detect and measure metals at concentrations above their respective human health toxicity evaluation thresholds.

Tables A.7-4 and A.7-5 provide acceptable laboratory QC criteria, procedures, and calculations for applicable QC statistics used by the ESAT Region 8 laboratory. These criteria and procedures will be used to assess effectiveness of QC actions. The sample selection for laboratory QC will be determined by the laboratory staff. Where a specific

QC criteria table is not provided, the method's QC requirements will be met or exceeded by ESAT's and EPA's analytical processes.

QA actions and results will be documented in the narrative section of the analytical data report associated with this project. Sample results will also be flagged with data qualifiers as described in Section D.3 of this SAP/QAPP. If samples are flagged as unusable they will be reanalyzed; provided that enough sample is remaining.

B.6 Instrument and Equipment Testing, Inspection, and Maintenance

Measurement instruments and equipment that will be used during this project are associated with ESAT Region 8 laboratory analytical instrumentation. The laboratory manager will be responsible for ensuring that all required analytical instrumentation and equipment testing, inspections, and maintenance procedures are up-to-date and working as designed.

ESAT Region 8 laboratory analytical instrumentation will be subject to routine calibration, routine maintenance, and scheduled services. Maintenance/servicing schedules and applicable testing criteria will be followed in accordance with equipment manufacturer's specifications are included in the applicable user's manuals. Equipment and instrument calibration requirements and frequencies are detailed in the applicable user's manuals.

Any equipment deficiencies and maintenance requirements will be identified and mitigated (i.e., parts replaced, alternate equipment deployed, etc.). After mitigation, equipment will be re-inspected and the effectiveness of any repairs will be verified. All repair or maintenance activities will be documented in the designated equipment or instrument log book.

B.7 Instrument and Equipment Calibration and Frequency

As indicated in Section B.6, some analytical instrumentation equipment requires periodic calibration to verify function. The laboratory manager will be responsible for ensuring that all required analytical instrumentation and equipment is calibrated prior to analyzing plant tissue samples. Calibration requirements, procedures, testing criteria, and deficiency resolution procedures are discussed in the applicable SOPs and user's manuals. User's manuals for laboratory analytical instrumentation are on-file at the ESAT Region 8 laboratory. Any variations or inability to calibrate a piece of equipment or instrument will be noted in the relevant logbook, and appropriate mitigation procedures will be followed or replacement equipment obtained. Recalibration of any instrument that requires mitigation of a deficiency will be performed prior to use or deployment.

B.8 Inspection and Acceptance of Supplies and Consumables

All consumable field sampling and sample shipping supplies for this wild plant sampling event will be purchased by the SUIT. **Attachment 3** provides a list of supplies that should be assembled in advance of field data and sample collection.

Supplies and consumables associated with chemical analysis of plant samples will be supplied by EPA and obtained from approved vendors. Analysis supplies and consumables will be ordered, inspected upon receipt, accepted, tracked, and inventoried by the ESAT Region 8 laboratory. Acceptance of supplies and consumables will be based on the requirements of the end user.

B.9 Use of Existing Data (Non-Direct Measurements)

No non-direct measurements were used to prepare for project implementation.

B.10 Data Management

This section provides information on how data will be managed from inception to final use and storage. Data will be generated during field-based sample collection, plant tissue sample documentation and shipping, and chemical analysis of plant tissue samples.

All field measurements and observations will be recorded in project-specific field datasheets (**Attachment 1**) by field personnel at the time they are performed. All field datasheet and COC form entries will be filled out by SUIT staff. "N/A" will be recorded in entries that cannot be completed. The personnel doing the recording will initial and date all measurements, observations, and any other notations made. Corrections will be performed by drawing a single line through the error, accompanied by the date and the initials of the person performing the correction, followed by the proper entry. COC forms will be filled out during the time of sample collection. Blank project-specific COC forms are provided in **Attachment 2** of this SAP/QAPP. A project-specific field notebook will also be used and maintained to document deviations to this SAP/QAPP, problems encountered in the field, and any CAs that are implemented.

All field datasheets, COC form copies, and the project-specific field notebook will be in possession and maintained under custody by the SUIT Field Team Manager. Any electronic or hard-copy versions of any of the field data sources will also be in custody of the SUIT field team manager.

Specific management processes will be followed for data likely to be collected during field activities: field datasheet and notebook entries, COC forms, and analytical data. Field datasheets and notebooks will not be managed by EPA.

COC forms – when possible, COC forms will be generated prior to field activities and filled out when samples are collected following the protocol outlined in the *Sample Custody and Labeling* SOP FLD-11.00 (**Appendix A**). Otherwise, blank COC forms will be used to collect sample information during field activities (**Attachment 2**). Information entered on the forms during investigation activities will be entered into Scribe after returning to the ESAT Region 8 laboratory as a part of the Scribe upload process (see below). ESAT personnel will verify 100% of all the data entered into Scribe against the COC forms completed in the field. Hard copies of these forms will be stored at the Region 8 laboratory, Suite A127 until relinquished to EPA in accordance with ESAT Region 8 contract requirements.

Analytical Data – an analytical chemist will log all the samples into LIMS upon receipt at the Region 8 laboratory. All analytical results will be uploaded into the LIMS in accordance with the *Sample Receipt, Custody, Storage and LIMS Entry of Samples* SOP 16-LAB-05.04 (**Appendix A**). Peer review of the data package, at a 100% frequency of reported versus raw data, will be performed by the analytical laboratory before a final report is released. The final report will be in a standard CLP format, including all laboratory and instrument QC results. The laboratory Electronic Data Deliverable (EDD) will immediately be uploaded into a Scribe project for permanent electronic storage and archiving after the final report is generated. Hard copies of data reports (including bench sheets) will be stored at the Region 8 laboratory, Suite A127 until relinquished to EPA in accordance with ESAT Region 8 contract requirements.

Scribe project generation – as indicated above, data generated as a part of field investigation activities will be uploaded into a Scribe project (or update to a Scribe project) and subsequently published to Scribe.net in accordance with the *Data Management for Field Operations and Analytical Support* SOP 16-DAT-01.00 (**Appendix A**). ESAT personnel will verify 100% of each Scribe project against data collected in the field (COC forms) prior to publishing the project on Scribe.net. Verified Scribe projects will be published within one week of delivery of the analytical EDD when possible. The EPA Analytical TOCOR, Don Goodrich (goodrich.donald@epa.gov) will be immediately notified and an alternate publication date will be established. In the event that conditions preclude publication within that time period, the ESAT Field Task TOCOR, Nicole Marotta (marotta.nicole@epa.gov) will be notified and a new publication date will be established.

C. ASSESSMENT AND OVERSIGHT

This section describes assessment and oversight associated with these events, including field sampling assessments, laboratory assessments, field CAs, and reports to management.

C.1 Assessment and Response Actions

C.1.1 Field Sampling Assessments

Assessment and oversight of field sampling activities and implementation of the SAP/QAPP will include the following:

- Oversight of field sampling activities
- Oversight of sample handling and COC procedures

The SUIT Field Task Lead, Curtis Hartenstine or his designee will provide the above oversight roles. The SUIT Field Task Lead will be proficient in and understand all of the sampling and sample handling requirements and suggestions provided in this SAP/QAPP. The Field Task Lead will address minor problems prior to beginning work or anytime when in the field. The Field Task Lead also has the responsibility to stop work and communicate any issues with the RPM to resolve any issues associated with sample collection and handling.

C.1.2 Laboratory Assessments

System assessments of the designated laboratory may be performed by ESAT's QAO, Bill Fear or his designee.

Routine assessments will be conducted at least once a year, in accordance with ESAT's QMP. However, the frequency of the laboratory system assessments will also be based on the level of use and performance of individual designated laboratories. A member of the ESAT team will perform the assessment in accordance with the assessment checklist and the *Field Procedures - Analytical Support and Laboratory Selection* SOP 02-06-08 (**Appendix A)**. The checklist requires examining the laboratory documentation on sample receiving, sample log-in, sample storage, COC procedures, sample preparation and analysis, instrument operating records, etc. Routine assessments will also be performed before a laboratory is added to the approved laboratory list. Should one-time specialty analysis be requested, the need for on-site assessments will be evaluated and discussed with EPA before such assessment is conducted.

Performance assessments will require preparing blind QC samples and submitting them along with project samples to the laboratory for analysis. The analytical results of the QC sample analyses are evaluated by the QAO to ensure that the laboratory maintains acceptable QC performance. Performance assessments may be requested by ESAT or EPA. Performance Evaluation (PE) samples will be prepared by and obtained from vendors. The QAO will designate if a PE sample shall be submitted. PE samples should be submitted if a laboratory has not recently passed an outside PE sample or as requested by EPA.

Response Actions

CAs may be required at two phases corresponding to the two activities of data generation: 1) field activities (data gathering phase), and 2) laboratory activities (data analysis phase). CA required as a result of the data analysis phase is initiated by the ESAT QAO when analytical data are found to be outside the limits of acceptability, as specified in the laboratory SOPs.

C.1.3 Field Corrective Actions

It is the responsibility of SUIT Field Task Lead to provide assessment and oversight of field sampling activates that follows this SAP/QAPP. If issues are identified in the field, the Field Task Lead will contact and describe them with the EPA RPM. The Field Task Lead and the RPM will discuss the need to implement CAs to address sample collection and handling issues. The CAs will depend on the nature or severity of the problem and the level where the problem is detected, and may include, but shall not be limited to:

- Modifications to sampling procedures
- Additional training of field personnel
- Reassignment of staff personnel
- Re-sampling

C.2 Reports to Management

The results of all laboratory assessments will first be submitted to the ESAT Team Manager. The ESAT Team Manager will review the analytical results package(s) for completeness. When deemed complete, the ESAT analytical task lead will deliver the completed results package(s) to the ESAT TOCOR who will distribute information to the RPMs and the EPA human health risk assessor. EPA will negotiate all other transactions with external stakeholders, including the SUIT Field Task Lead.

D. DATA VALIDATION AND USABILITY

D.1 Data Review, Verification, and Validation

Laboratory data validation and verification will begin at the sample log-in stage where a sample log-in technician or chemist will compare received samples against COC forms and will document sample condition (damage, temperature, etc.). Validation and verification of data may be performed by QA/QC personnel following the *EPA Contract Laboratory Program National Functional Guidance for Inorganic Superfund Data Review* report (EPA, 2017c) to determine if the DQOs were met. Sample data deemed outside the expected range will be investigated, communicated to the analytical chemistry staff, flagged (if needed), and potentially re-sampled to verify or discredit the data. Data that are proven incorrect may be flagged, further reviewed, or invalidated. The cause of incorrect data will be investigated and appropriate response actions will be taken, including communication of any issues to the user in the data report.

Uncertainty of validated data will be evaluated by a RPM to determine if the DQOs were met. If the DQOs were not met, they will be reviewed to determine whether they are achievable, and if not, DQOs may be revised if necessary. Additionally, the data may be further evaluated to determine its impact to the project. Data usability and limitations will be evaluated by a RPM.

Abbreviated verification will be completed on 10% of the analytical results for data that are electronically uploaded directly from the analytical instrumentation into the ESAT LIMS. This verification will be performed to ensure that data were produced in accordance with procedures outlined in this SAP/QAPP. The following elements will be reviewed for compliance as part of the abbreviated data validation:

- Holding Times
- Calibration
- Blanks
- Spikes
- Duplicates
- LCSs
- Reporting Limits
- Analyte Quantification

D.2 Verification and Validation Methods

If conducted, either the acting ESAT Region 8 laboratory QAO or by a designated ESAT QAO outside of the Region 8 ESAT office will validate 10% the analytical results. The validation will include reviewing 10% of the samples for 100% of the analyses performed and reported. The following elements will be reviewed for compliance as part of the abbreviated data validation:

- Holding Times
- Calibration
- Blanks
- Spikes
- Duplicates
- LCSs
- MS/MSDs
- Post-digest Spike
- Internal Control Standard
- Dilution Sample
- Reporting Limits
- Analyte Identification
- Analyte Quantification
- Comparison of hard-copy results to the electronic data deliverable

Data validation will conform to the *EPA Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review* report (EPA, 2017c) and will use standard data qualifiers as described below.

D.3 Reconciliation with User Requirements

The following definitions provide brief explanations of the national qualifiers assigned to results in the data review process. If the ESAT Region 8 laboratory choose to use additional qualifiers, a complete explanation of those qualifiers should accompany the data review.

U	The analyte was not detected above the level of the reported sample quantitation limit.
J	The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.
J+	The result is an estimated quantity, but the results may be biased high.
J-	The result is an estimated quantity, but the results may be biased low.
R	The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria. The analyte may or may not be present in the sample.
UJ	The analyte was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.

D.4 Reconciliation with DQOs

Information obtained from the field investigation will be evaluated through the Data Quality Assessment (DQA) process to determine if the data obtained are of adequate quality and quantity to support its intended use. The DQA process consists of five steps, as summarized below (EPA, 2006):

1) *Review the project's objectives and sampling design*: Review the objectives defined during the systematic planning to assure that they are still applicable. Ensure objectives have been deployed before evaluating the data for the project's objectives. Review the sampling design and data collection documentation for consistency with the project objectives observing any potential discrepancies.

2) *Conduct a preliminary data review*: Review QA reports (when possible) for the validation of data, calculate basic statistics, and generate data graphs. Use this information to learn about the structures of the data and identify patterns, relationships, or potential anomalies.

3) *Select the statistical method*: Select the appropriate procedures to summarize and analyze the data based on the review of the performance and acceptance criteria associated with the project objectives, the sampling design, and the preliminary data review. Identify the key underlying assumptions associated with the statistical tests.

4) *Verify the assumptions of the statistical method*: Evaluate whether the underlying assumptions hold, or whether departures are acceptable, given the actual data and other information about the study.

5) *Draw conclusions from the data*: Perform the calculations necessary to draw reasonable conclusions from the data. If the design is to be used again, evaluate the performance of the sampling design.

Uncertainty of validated data will be evaluated by the RPM to determine if the DQOs were met. If the DQOs were not met, they will be reviewed to determine if they are achievable and, if not, may be revised if necessary, and the data may be further evaluated to determine the impact to the project. Data usability and limitations will be evaluated by the RPM.

E. References

Brett, J.A. 2003. *Ethnographic Assessment and Documentation of Rocky Mountain Nation Park*. Project Report. Department of Anthropology University of Colorado at Denver.

Environmental Services Assistance Team. 2015. *Final Revision 2 Quality Management Plan*

McBeth, S. 2010. *Ethnographic Overview Draft #2: Colorado National Monument*. National Park Service. February 26.

Storosh, M. 2013. *Mine Site Remediation by Good Samaritans in the Animas Watershed, Colorado*. Fort Lewis College.

U.S. Environmental Protection Agency. 1994a. *Determination of Mercury in Water by Cold Vapor Atomic Absorption Spectrometry*. Method 245.1. Revision 3.0, May.

U.S. Environmental Protection Agency. 1994b. *Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry*. Method 200.7 Revision 4.4. May.

U.S Environmental Protection Agency. 1994c. *Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma-Mass Spectrometry*. Method 200.8 Revision 5.4.

U.S Environmental Protection Agency. 1994d. *Sample Preparation Procedure for Spectrochemical Determination of Total Recoverable Elements*. Method 200.2 Revision 2.8.

U.S. Environmental Protection Agency. 1997. *Ecological Risk Assessment Guidance for Superfund: Process for Designing and Conducting Ecological Risk Assessments*. Interim Final. EPA 540-R-97-006.

U.S. Environmental Protection Agency. 2001. *Requirements for Quality Assurance Project Plans*. EPA QA/R-5. March.

U.S. Environmental Protection Agency. 2002a. *Guidance on Choosing a Sampling Design for Environmental Data Collection*. EPA QA/G-5S. December.

U.S. Environmental Protection Agency. 2002b. *Guidance for Quality Assurance Project Plans*. EPA QA/G-5. December.

U.S. Environmental Protection Agency. 2006. *Guidance on Systematic Planning Using the Data Quality Objectives Process*. EPA QA/G-4. February.

U.S. Environmental Protection Agency. 2007. *Mercury in Solids and Solutions by Thermal Decomposition, Amalgamation, and Atomic Absorption Spectrophotometry*. Method 7473. Revision 0. February.

U.S. Environmental Protection Agency. 2016. Federal Register. EPA-HQ-SFUND-1994-0002. Vol. 81. No. 67. Thursday, April 7. Proposed Rules pp. 20277-20283

U.S. Environmental Protection Agency. 2017a. *Draft Terrestrial Screening-level Ecological Risk Assessment, Upper Animas Mining District, San Juan County, CO.* April. Prepared for: USEPA Region 8, 1595 Wynkoop Street, Denver, CO 80202.

U.S. Environmental Protection Agency. 2017b. *Final Sampling and Analysis Plan/Quality Assurance Project Plan 2017-2018 Combined Risk Assessment Sampling Events Bonita Peak Mining District, San Juan County, CO.* August. Prepared for: USEPA Region 8, 1595 Wynkoop Street, Denver, CO 80202.

U.S. Environmental Protection Agency. 2017c. National Functional Guidelines for Inorganic Superfund Methods Data Review. EPA-540-R-2017-001. January. Available at https://www.epa.gov/sites/production/files/2017-01/documents/national_functional_guidelines_for_inorganic_superfund_methods_data_review_01302017.pdf

U.S. Environmental Protection Agency. 2017d. Regional Screening Level (RSL) Resident Soil Table (TR=1E-06, HQ=1). June. Available at: https://semspub.epa.gov/work/03/2245085.pdf

Standard Operating Procedures Provided in Appendix A:

Standard Operating Procedures for Soil Sampling SOP FLD-05.00

Analysis of Trace Metals Using the Perkin Elmer Optima 4300DV ICP-OE. SOP 16-MET-02.02

Mercury Analysis of Soils, Sludge, and Biota by Thermal Decomposition Using the NIC MA3000 Direct Mercury Analyzer SOP 16-MET-04.01

Data Management for Field Operations and Analytical Support. SOP 16-DAT-01.00

Determination of Metals by Inductively Coupled Argon Plasma-Mass Spectroscopy (ICP-MS). SOP 16-MET-01.01

Field Procedures - Analytical Support and Laboratory Selection. SOP 02-06-08

General Field Sampling Protocols. SOP FLD-12.00

Laboratory Waste Management. SOP 16-LAB 01.01

Sample Custody and Labeling. SOP FLD-11.00

Sampling Equipment Decontamination. SOP FLD-02.00

Sample Preservation. SOP FLD-03.00

Sample Receipt, Custody, Storage and LIMS Entry of Samples. SOP 16-LAB-05.04

Tables

Table A.6-1 Plant Species List and Sample Types

SUIT Plants	Plant Type ID	Common Name	Genus/Species	Sample Type	Season Harvested
Camas	**CAM**	Common camas	*Camassia quamash*	Roots/bulb only	Fall
		Mountain death camas	*Anticlea elegans*		
		Desert death camas	*Toxicoscordion paniculatum*		
Wild onion	**ONI**	Nodding onion	*Allium cernuum*	Entire plant	Spring
		Geyer's onion	*Allium geyeri*		
		Taper-tip onion	*Allium acuminatum*		
		Large-petaled onion	*Allium macropetalum*		
		Nevada onion	*Allium nevadense*		
		Textile onion	*Allium textile*		
Osha	**OCH**	Loveroot	*Ligusticum porteri*	Roots/bulb only	Fall
		Cow parsnip	*Heracleum sphondylium*		
Wild carrots	**CRR**	Wild Carrot	*Daucus carota*	Roots/bulb only	Spring/summer
		Yampa	*Perideridia* sp		
Balsam/Arrow root	**ARW**	Arrowleaf balsamroot	*Balsamorhiza sagittata*	Entire plant	Spring to fall
Cattails	**CTL**	Cattail	*Typha latifolia*	Entire plant	Summer
Chokecherry	**CCH**	Chokecherry	*Prunus virginiana*	Fruit only	Spring/fall
Sumac	**SMC**	Aromatic sumac	*Rhus aromatica*	Stems, outer bark, leaves, and fruits	Spring
		Smooth sumac	*Rhus glabra*		
Willow	**WIL**	Willow	*Salix* spp.	Stems and outer bark	Spring
Primrose	**PRM**	Yellow evening primrose	*Oenothera flava*	Roots	Spring
		Bridges evening primrose	*Oenothera longissima*		
Wild olive	**OLV**	New Mexico forestiera	*Forestiera neomexicana*	Fruit only	Late summer/fall
		Desert olive	*Forestiera pubescens*		
Currant	**CRT**	Wax currant	*Ribes cereum*	Leaves and fruit	Late summer/fall
		Whiskey currant	*Ribes inerme*		
		Golden currant	*Ribes aureum*		
		Trailing currant	*Ribes laxiflorum*		
		Mountain currant	*Ribes montigenum*		
		Wolf's currant	*Ribes wolfii*		
		Prickly currant	*Ribes lacustre*		
Sego lily	**LLY**	Gunnison's sego lily	*Calochortus gunnisonii*	Roots/bulb only	Any
		Nuttall's sego lily	*Calochortus nuttallii*		
Kinnikinnick	**KIK**	Kinnikinnick	*Arctostaphylos uva-ursi*	Leaves and fruit	Late summer/fall
		Greenleaf manzanita	*Arctostaphylos patula*		
		Pointleaf manzanita	*Arctostaphylos patula*		
		Red osier dogwood	*Cornus alba*		
		Wild licorice	*Glycyrrhiza lepidota*		
		Tobacco	*Nicotiana attenuata*		
Buffaloberry	**BUF**	Canada buffaloberry	*Shepherdia canadensis*	Fruit only	Late summer/fall
		Silver buffaloberry	*Shepherdia argentea*		
		Round leaf buffaloberry	*Shepherdia rotundifolia*		
Barberry/Mahonia	**BAR**	Fremont barberry	*Mahonia fremontii*	Fruit only	Summer/fall
		Creeping mahonia/Oregon grape	*Mahonia repens*		
Bitterbrush	**BIT**	Cliff rose	*Purshia stansburiana*	Leaves	Spring/summer
		Bitterbrush/Buckbrush	*Purshia tridentata*		
		Mountain spray	*Holodiscus dumosus*		
Dandelion	**DAN**	Common dandelion	*Taraxacum officinale*	Entire plant	Spring to fall
		Alpine dandelion	*Taraxacum ceratophorum*		
		Woolbearing dandelion	*Taraxacum eriophorum*		
		Dandelion	*Taraxacum ovinum*		
		Dandelion	*Taraxacum scopulorum*		
Wild grape	**GRP**	Virginia creeper	*Parthenocissus quinquefolia*	Young shoots and fruit	Spring/summer
		Canyon grape	*Vitis arizonica*		
Wild strawberry	**STB**	Little strawberry	*Fragaria vesca*	Fruit only	Spring/summer
		Virginia strawberry	*Fragaria virginiana*		
Rubus berries	**RUB**	Raspberry	*Rubus idaeus*	Leaves and fruit	Spring/summer
		Thimbleberry	*Rubus parviflorus*		
Serviceberry/Roses	**RSE**	Alder-leaf serviceberry	*Amelanchier alnifolia*	Rose hips/pomes	Late summer/fall
		Utah serviceberry	*Amelanchier utahensis*		
		Woods rose	*Rosa woodsii*		
		Squaw apple	*Peraphyllum ramosissimum*		
Pussytoes	**PST**	Flat-top pussytoes	*Antennaria corymbosa*	Stems and leaves	Spring/summer
		White-margined pussytoes	*Antennaria marginata*		
		Rocky Mountain pussytoes	*Antennaria media*		
		Small-leaf pussytoes	*Antennaria parvifolia*		
		Red pussytoes	*Antennaria rosea*		
Rabbitbrush	**RBT**	Gray rabbitbrush	*Ericameria nauseosa*	Stems and leaves	Spring/summer
Spiderflower	**SPD**	Rocky Mountain bee plant	*Peritoma serrulata*	Seed pods, leaves and flowers	Spring/summer
Fleabane/Daisies	**DSY**	Spreading daisy	*Erigeron divergens*	Stems and leaves	Spring/summer
		Rayless fleabane	*Erigeron aphanactis*		
		Silvery daisy	*Erigeron argentatus*		
		Hairy daisy	*Erigeron concinnus*		
		Glacial daisy	*Erigeron glacialis*		
		Tracy's daisy	*Erigeron tracyi*		
		Utah daisy	*Erigeron utahensis*		
Beardtongues	**BRT**	Scarlet bugler	*Penstemon barbatus*	Stems and leaves	Summer
		Eaton's penstemon	*Penstemon eatonii*		
Grasses	**GRS**	Wheatgrass	*Agropyron cristatum*	Stems and leaves	Summer
		Blue gamma grass	*Bouteloua gracilis*		
		Arizona fescue	*Festuca arizonica*		
Buckwheat	**BCW**	Nodding buckwheat	*Eriogonum cernuum*	Stems and leaves	Spring
		Sulfur buckwheat	*Eriogonum umbellatum*		
		Curly dock	*Rumex crispus*		

Table A.6-1 Plant Species List and Sample Types

SUIT Plants	Plant Type ID	Common Name	Genus/Species	Sample Type	Season Harvested
Asters	**ASR**	Alpine aster	*Aster alpinus*	Stems and leaves	Spring/summer
		Waxy aster	*Herrickia glauca var. glauca*		
		Dwarf golden aster	*Heterotheca pumila*		
		Hairy golden aster	*Heterotheca villosa*		
		Zion golden aster	*Heterotheca zionensis*		
		White aster	*Symphyotrichum ericoides*		
		Leafy aster	*Symphyotrichum foliaceum*		
		Purple aster	*Symphyotrichum puniceum*		
		Spatula aster	*Symphyotrichum spathulatum*		
Groundsel/Packera	**GRL**	New Mexico groundsel	*Packera neomexicana*	Stems and leaves	Spring/summer
		Many-lobed packera	*Packera multilobata*		
		Streambank packera	*Packera pseudaurea*		
		Groundsel	*Senecio vulgaris*		
Goldenrods	**GLD**	Simple goldenrod	*Solidago simplex*	Stems and leaves	Spring/summer
		Missouri goldenrod	*Solidago missouriensis*		
		Rocky Mountain goldenrod	*Solidago multiradiata*		
		Dwarf goldenrod	*Solidago nana*		
		Velvet goldenrod	*Solidago velutina*		
Spearmint	**SMT**	Field mint	*Mentha arvensis*	Entire plant	Spring
		Aromatic little monarda	*Monardella odoratissima*		
Peppermint	**PMT**	Peppermint	*Mentha piperita*	Entire plant	Spring
Apache plume	**PLM**	Apache plume	*Fallugia paradoxa*	Flower tops and roots	Spring/summer
Deerbrush	**DER**	Deerbrush	*Ceanothus integerrimus*	Stems and flowers	Spring/summer
Scarlet globe mallow	**MAL**	Scarlet globe mallow	*Sphaeralcea coccinea*	Leaves	Fall
Mountain mahogany	**MAH**	Mountain mahogany	*Cercocarpus montanus*	Roots and leaves	Spring/summer
Buckthorn	**BCT**	Buckthorn	*Frangula obovata*	Inner bark	Spring/summer
New Mexico locust	**LOC**	New Mexico locust	*Robinia neomexicana*	Flowers	Spring/summer
Twinberry	**TWN**	Twinberry	*Lonicera involucrata*	Fruit only	Spring/summer
Showy milkweed	**MLK**	Showy milkweed	*Asclepias speciosa*	Stems	Spring/summer
Beebalm	**BEE**	Beebalm	*Monarda fistulosa*	Stems and leaves	Spring/summer
Western virgins bower	**WVB**	Western virgins bower	*Clematis ligusticifolia*	Stems and leaves	Spring/summer
Sagebrush	**SAG**	Fringed sagewort	*Artemisia frigida*	Stems and leaves	Spring
		Silvery sagewort	*Artemisia ludoviciana*		
		Bigelow's sagebrush	*Artemisia bigelovii*		
		Northern sagewort	*Artemisia borealis*		
		Carruth's sagewort	*Artemisia carruthii*		
		Wild tarragon	*Artemisia dracunculus*		
		Sand sagebrush	*Artemisia filifolia*		
		Ragweed sagebrush	*Artemisia franserioides*		
		Black sagebrush	*Artemisia nova*		
		Alpine sagewort	*Artemisia scopulorum*		
		Big sagebrush	*Artemisia tridentata*		
Yarrow	**YRO**	Yarrow	*Achillea millefolium*	Stems and leaves	Spring
		Common yarrow	*Achillea lanulosa*		
Mormon tea	**MOR**	Cutler's mormon tea	*Ephedra cutleri*	Stems and leaves	Spring
		Green mormon tea	*Ephedra viridis*		
		Torrey's mormon tea	*Ephedra torreyana*		
Saltbush/Winterfat	**SLT**	Four wing saltbush	*Atriplex canescens*	Roots and flowers	Spring/summer
		Winterfat	*Krascheninnikovia ceratoides*		
Cholla	**CLA**	Cholla	*Cylindropuntia fulgida*	Fruit only	When present
		Cane cholla	*Cylindropuntia imbricata*		
		Whipple's cholla	*Cylindropuntia whipplei*		
Yuccas	**YCA**	Banana yucca	*Yucca baccata*	Seeds and flowers	Summer/fall
		Small soapweed	*Yucca glauca*		
Junipers	**JPR**	Common juniper	*Juniperus communis*	Tender needles and berries	Late summer/fall
		Alligator juniper	*Juniperus deppeana*		
		One seed juniper	*Juniperus monosperma*		
		Utah juniper	*Juniperus osteosperma*		
		Rocky Mountain juniper	*Juniperus scopulorum*		
		Red cedar	*Juniperus virginiana*		
Pines	**PNS**	Colorado spruce	*Picea pungens*	Needles	Spring/summer
		Lodgepole pine	*Pinus contorta*		
		Douglas fir	*Pseudotsuga menziesii*		
Cottonwood	**CTW**	Narrowleaf cottonwood	*Populus angustifolia*	Buds only	Spring
Aspen	**APN**	Aspen	*Populus spp.*	Bark and leaves	Summer
Maple	**MPL**	Rocky Mountain maple	*Acer glabrum*	Stems and bark	Spring/summer
Pinion nuts	**PIN**	Pinyon pine	*Pinus edulis*	Seeds only	Late fall
Bristle cone	**BRC**	Rocky Mountain bristlecone pin	*Pinus aristata*	Seeds only	Late fall
Ponderosa	**PON**	Ponderosa pine	*Pinus ponderosa*	Cambium, seeds, cones, and resin	Spring
Acorns	**OAK**	Gambel oak	*Quercus gambelii*	Acorns only	Fall

Table A.7-1 Data Quality Objectives Planning Team

Name	Organization	Area of Technical Expertise
Rebecca Thomas	EPA Region 8	Remedial Project Manager
Jamie Miller	EPA Region 8	Remedial Project Manager
Robert Parker	EPA Region 8	Remedial Project Manager
Deborah McKean	EPA Region 8	Toxicologist
Joyel Dhieux	EPA Region 8	On-Scene Coordinator
Don Goodrich	EPA Region 8	Chemist
Sherry Skipper	USFWS	Biologist
Lisa Richardson	BLM	Physical Scientist
Michael Carney	ESAT	Toxicologist
Scott Walker	ESAT	Chemist
Steve Auer	ESAT	Biologist

Table A.7-2 Stakeholders

Organization	Represented By
SUIT	Tom Johnson
SUIT	Curtis Hartenstine
SUIT	Cassandra Naranjo
USFS	Ben Martinez
BLM	Brent Lewis
ARSG	Peter Butler
USFWS	Sherry Skipper

Table A.7-3 Total Recoverable Metals Target Analyte List

Target Analytes	EPA Digestion Method [1]	EPA Analysis Method [1]	Instrument	Fraction Evaluated	Min Sample Weight[2]	Preservative	Max Hold Time	Laboratory PQL (mg/Kg)	EPA RSL[3] (mg/Kg)
Aluminum (Al)	200.2	200.7	ICP-OE	TR	50 grams[b] 4 oz for soil	Ice	180 days	5.0	77,000
Beryllium (Be)	200.2	200.7	ICP-OE					0.5	160
Calcium (Ca)	200.2	200.7	ICP-OE					25	NA
Chromium (Cr)	200.2	200.7	ICP-OE					0.5	120,000[a]
Copper (Cu)	200.2	200.8	ICP-OE					0.2	3,100
Iron (Fe)	200.2	200.7	ICP-OE					25	55,000
Magnesium (Mg)	200.2	200.7	ICP-OE					25	NA
Manganese (Mn)	200.2	200.7	ICP-OE					0.5	1,800
Potassium (K)	200.2	200.7	ICP-OE					100	NA
Zinc (Zn)	200.2	200.7	ICP-OE					2	23,000
Antimony (Sb)	200.2	200.8	ICP-MS					0.1	31
Arsenic (As)	200.2	200.8	ICP-MS					0.2	0.77
Barium (Ba)	200.2	200.8	ICP-MS					0.5	15,000
Cadmium (Cd)	200.2	200.8	ICP-MS					0.02	71
Cobalt (Co)	200.2	200.8	ICP-MS					0.5	23
Lead (Pb)	200.2	200.8	ICP-MS					0.02	400
Molybdenum (Mo)	200.2	200.8	ICP-MS					2	390
Nickel (Ni)	200.2	200.8	ICP-MS					0.1	820[c]
Selenium (Se)	200.2	200.8	ICP-MS					0.2	390
Silver (Ag)	200.2	200.8	ICP-MS					0.1	390
Thallium (Tl)	200.2	200.8	ICP-MS					0.1	NA
Vanadium (V)	200.2	200.8	ICP-MS					5	390
Mercury (Hg)	7473	415.3	CVAA				28 days	0.02	11

TR = Total recoverable metals from bulk tissues and soils
PQL - Practical Quantitation Limit
RSL = Regional Screening Level
ICP-OE = Inductively coupled plasma-optical emission
ICP-MS = Inductively coupled plasma mass spectrometry
CVAA = Cold vapor atomic absorption
NA = Not Available

[1] EPA's Method for the Determination of Metals in Environmental Samples, Supplement I, May 1994 (Series 200 Methods)

[2] Sample containers should be plastic or glass and capable of holding the required sample mass/volume

[3] Lowest available EPA (2017d) RSL for residential soils

[a] RSL for chromium (III), insoluble salts

[b] 50 grams is the minimum amound of tissue that is required; 200 gams of tissue is recommended for most plant and sample types; see Section B.2 for more details

[c] RSL for nickel as refinery dust

Table A.7-4 ESAT Region 8 Laboratory Metals Quality Control Criteria

QC Check / Symbol	Explanation	Run Frequency	Acceptance Criteria	Corrective Action
Initial Calibration Verification (ICV)	Certified standard or standard from a different lot/source than calibration standards	Beginning of run to verify calibration	90-110% recovery (%R) of "true value"	Terminate analysis, restandardize
Continuing Calibration Verification (CCV)	Approximate mid-range standard made from working standards stock	Every 10 unknowns and at end of run	90-110%R "True" value	Re-analyze immediately (once). Then: Restandardize and rerun all samples following last "acceptable" CCV. If recovery >110% and <120% and all associated samples (same analyte) show non-detected, no action required.
Spectral/Mass Interference Check for ICP-OE & ICP-MS (ICSA / ICSAB)	Analyze spectral interferents at high concentrations alone (ICSA) and with other target analytes (ICSAB) to evaluate the effect on analyte recovery	Once per analytical run, prior to sample analyses	ICSAB: ± 20%R 'true value' ICSA: ± 20%R 'true value' or < ±PQL whichever is greater	Evaluate the sample analyte levels. Rerun ICSA/AB or use an alternate wavelength. If interferent levels in the samples don't approach ICSA interferent levels, no action is required. If necessary, recalculate IECs & rerun associated samples
Calibration Blanks, Initial & Continuing (ICB & CCB)	Blank with same reagents as working standards; i.e. zero point on curve	Beginning, end, and after each ICV/CCV during analytical run	≤ ±PQL	Re-analyze immediately once. If still unacceptable, terminate analysis & restandardize. Rerun all samples analyzed after last "acceptable" blank. Evaluate interferent level(s) vs samples, use prof judgement for addit'l required sample reruns.
Preparation Blank (PB)	Digested or prepared blank processed identical to samples. Aliquot of clean water prepared using same reagents/volumes as unknown samples.	Once per preparation batch/per matrix, or at 5% frequency, whichever is greatest	≤ ±PQL	PB > PQL: Redigest all samples >MDL and <10x PB value PB < -PQL: Re-calibrate and re-analyze all associated samples
Matrix Spike & Matrix Spike Duplicate (MS & MSD)	Unknown sample (NOT a field blank) fortified at approximately 10-100x MDL for each target analyte. High concentration samples (spike <25% sample target analyte concentration), no calculation is required	1 per 20 unknowns per matrix, whichever is greatest (One PB Spike per PB)	Spike recovered at: 80-120% (ICP& MS) - waters 65-135% (all) - solids	Compose 1 post-digest spike (PS) and retest, note in the narrative. (Analyze original sample with PS) Evaluate duplicate reproducibility. Compare results to LFB/PBS for similar trends. If no similar trends observed, assume a matrix effect. Qualify corresponding analyte data as estimated 'J' for similar matrix samples in set.
Lab Fortified Blank (LFB or PBS)	Spike of reagent blank at same level as MS (analyze/prep identical to samples)	Recommend: once/run	85-115%R of expected (for target analytes)	Used for comparison to Matrix Spike. If MS/MSD in-control no corrective action necessary.
Lab Control Sample (LCS)	For solid & liquid digested samples. A known of similar matrix prepared the same as unknown samples.	1 per prep batch or one per matrix, whichever is greater.	Aq: 80-120%R of "true" Solids: 70-130%R of "true" or published limits	Recalibrate & reanalyze. If still unacceptable, check for corresponding high or low results in pre-digest spikes, if similar, redigest all associated samples
Serial Dilution (L)	Sample analyzed at 5x the reported analysis. (for matrix effect evaluation) Applies to analytes >50x MDL (in the original analyzed solution)	1 per 20 unknown	Diluted value 90-110% of original analysis.	Concentrations compared/reported from the analyzed solution only. Check IECs and re-analyze. May re-analyze both sample and 'L' at a higher dilution. Use professional judgement, and discuss outliers in the narrative.
Detection Limit Standard (CRI/CRA)	Low level standard ≈3-5x MDL concentration. Applies to all target analytes except Al, Ca, Fe, Mg, Na, & K	Once per analytical batch prior to unknowns	50-150%R for Sb, Pb, and Tl. 70-130%R for other target analytes*.	1. Rerun 2. If all associated samples ≥CCV for outlier analyte, no action required 3. Correct instrument's sens. problem or else need to redetermine and raise reporting limits *[Al, Ca, Fe, Mg, Na, & K are monitored without corrective actions]
ICP-MS Internal Standard (IS)	IS standard solution added to all samples, blanks, and standards.	All samples and standards corrected for IS response.	60% - 125%R of IS associated with target analyte(s)	[IS recovery determined versus calibration blank response.] Dilute sample by 2, re-analyze. Continue to dilute until IS %R acceptable.

Table A.7-5 QA/QC Calculation Algorithms

Statistical QC Parameter Evaluated	Acronym	Analyses Applied to	Calculation Algorithm
Percent Recovery	%R	Spike recovery determinations	$\%R = ((C_s - S_a) \div (S_a)) \times 100$
Percent Recovery	%R	ICV/CCV, ICSAB, LCS	$\%R = (A_T \div T) \times 100$
Relative Percent Difference	RPD	Variance between duplicates	$RPD = ((C - C_D) / ((C + C_D) \div 2)) \times 100$
Percent Difference	%D	Serial dilution variance	$\%D = ((C - C_L) / C) \times 100$

Notes:

C = Sample extract

C_s = Sample extract, spiked

S_a = Spike amount added

T = True (possibly certified) amount in standard solution

C_D = Duplicate sample concentration

C_L = Sample extract concentration, dilution factor corrected.

AT = Analyzed concentration for the known standard.

Table B.3-1 Sample Identification Plant Part Designations

Plant Part	Sample ID Designation
Roots/bulb only	RTB
Entire plant	ALL
Fruit/Berries only	FRT
Stems with bark	STM
Outter bark only	BRK
Leaves[1]	LEV
Grape shoots	GST
Pomes and seed pods	POM
Seeds	SEE
Flowers	FLW
Cambium (inner bark)	CBM
Needles	NED
Cottonwood buds	BUD
Piñon nuts	NUT
Cones	CNE
Resin	RSN
Acorns	ACN

[1] LEV will be used for samples that contain leaves, flowers, and buds for a single plant type; see Section B.2.2.3 for more details

Figures

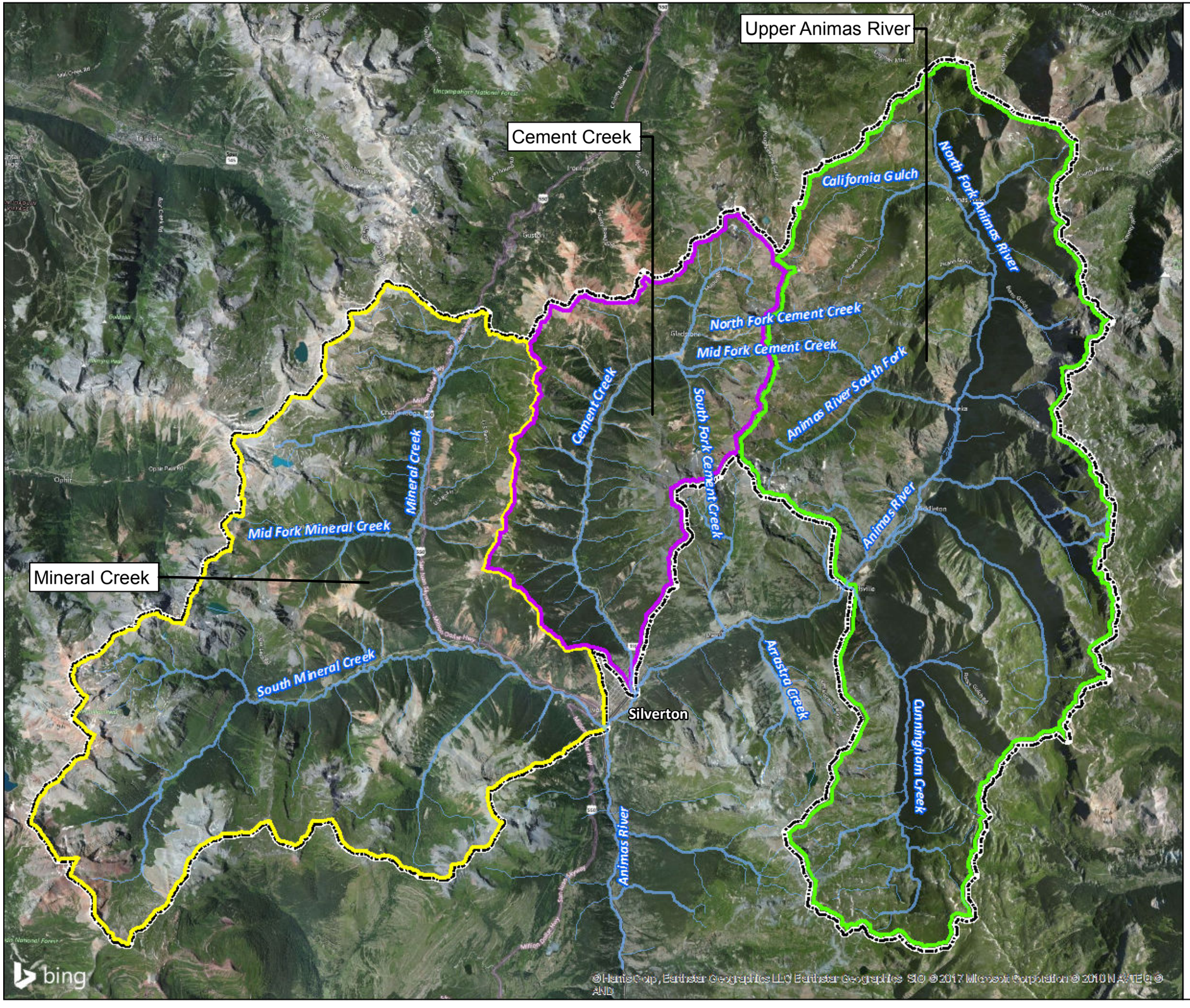


Figure A.6-1
Bonita Peak Mining District
Overview Map of Assessment Area

- Minor Streams
- Major Streams
- Assessment Area
- Upper Animas River
- Cement Creek
- Mineral Creek

Map Date: February 15, 2017

Data Sources:
Streams: CDOW (2004)
Assessment reas & Sub Watershed: USDA (2013)
Imagery: Microsoft Bing Hybrid Web Service (2017)

Map Projection: UTM Zone 13 N, NAD83, Meters

N
W E
S

0 1 2
Miles

Area of Interest

Appendix A
Standard Operating Procedures

FIELD PROCEDURES –
ANALYTICAL SUPPORT AND
LABORATORY SELECTION

SOP Number: 02-06-08
Effective Date: 2/3/2017

Technical Approval: _____ Date: 2/3/2017

QA Management Approval: _____ Date: 2/3/2017

SOP Description

This Standard Operating Procedure (SOP) describes the process to be followed by TechLaw Holdings and Subsidiary Companies (TechLaw Holdings) staff when acquiring analytical support. All requests for analytical services are to be arranged through the Laboratory Assistance Team (LAT) Coordinator, or alternatively through a LAT member (see Attachment A for a list of approved LAT members). This SOP is to be followed by the TechLaw Holdings Project Manager (PM), or designee, when completing and submitting the Analytical Support Request Form (ASRF) (see Attachment B), and after the sampling event is completed by submitting copies of the chain-of-custody (COC) forms to the LAT and forwarding invoices to the project files. A LAT member checklist is provided in Attachment D, and a TechLaw Holdings Project Manager Checklist is provided in Attachment E.

This SOP is also to be followed by the LAT Coordinator and assigned LAT members when processing the ASRF, selecting a laboratory, and reviewing data packages and invoices.

General Procedures

Related SOPs

This SOP is to be used in conjunction with other applicable SOPs found in the following SOP categories:

Category No.	Category Title
01	General Procedures
02	Field Procedures
03	Field Documentation Procedures
04	Packaging and Shipping Procedures
05	Field Equipment Operation and Maintenance Procedures

06	Groundwater Sampling/Monitoring and Analysis Procedures
07	Soil/Sediment Sampling and Analysis Procedures
08	Surface Water Sampling and Analysis Procedures
09	Health and Safety Procedures
10	Regulatory Compliance Procedures
11	Quality Assurance Procedures
12	Incineration/BIF Sampling and Analysis Procedures
13	Waste Sampling and Analysis Procedures
14	Asbestos Handling
15	Region 5 ESAT-Specific SOPs
16	Region 8 ESAT-Specific SOPs
17	Region 1 ESAT-Specific SOPs

Procedures for Submitting and Processing the Form

An analytical support request must be submitted to the assigned LAT member at least five business days prior to the sampling event to avoid additional charges for rush shipping of any necessary supplies. The request should be submitted as a completed ASRF (see Attachment B). Note that either this form or an email containing the same information will be considered an ASRF. If supplies are not required from the laboratory, the ASRF may be submitted three business days prior to the sampling event. If shorter turnaround time is required, every effort will be made by the LAT to process the request.

The assigned LAT member will ensure that all necessary information is included in the ASRF and make the necessary arrangements with the laboratory (e.g., request delivery of glassware or other sample collection media and equipment). The assigned LAT member will procure sample containers on an as-needed basis, as indicated on the ASRF.

Upon completion of the ASRF by the assigned LAT member, a copy is uploaded to the project folder under LAT on TechLaw Holdings' SharePoint Site. All samples must be sent to a TechLaw Holdings-approved laboratory. The laboratory is selected on the ability to perform the requested analysis, availability of laboratory space, and analysis cost. The LAT member must send the winning bid to the TechLaw Holdings PM for approval prior to shipment of samples to the laboratory (approval can be received formally in writing or via e-mail). In cases of emergency responses or expedited requests for analytical services (e.g., within 24 hours), TechLaw Holdings PM approval will be obtained as soon as possible. This documented approval will be uploaded to the project folder under LAT on SharePoint. A Work Authorization

(Attachment C) is submitted to the selected laboratory by the assigned LAT member prior to sampling activities. The Work Authorization is also e-mailed to TechLaw Holdings contracting staff for generation of a purchase order (PO). A copy of the finalized Work Authorization is placed in the project folder under LAT on SharePoint.

In cases where the sampling work is in the initial stages of being bid, an ASRF is not required; however, the LAT member must make sure that all information required from the request for proposal (RFP) is obtained by the PM prior to providing costs (i.e., requirements for certifications, such as State, DoD, etc., as well as EDD requirements).

Procedures for Completing the Form

The ASRF (see Attachment B) must contain complete sampling and analytical information. This information shall include the following:

- Project number (billing code) and project manager name
- Site name and location
- Date(s) of sampling event
- Required turnaround time
- Type of data package (i.e., Level II, III or IV, requirements for summary forms)
- Special considerations, if any
- For the table: sample matrix, number of field samples, parameter (i.e., the appropriate analytical method numbers), required detection limits, and the numbers of quality control (QC) samples (i.e., field duplicates, trip blanks, field blanks, and matrix spike/matrix spike duplicates [MS/MSD]).

Procedures for Changes

If changes occur in the number of samples or type of sampling methods during the field activities, the project manager will notify the LAT member by email.

Procedures After Sampling Event Completion

COC Forms

Within one week of shipment of samples (within 24 hours ideally), the laboratory must send copies of all COC forms to the assigned LAT member. Scanned copies are acceptable. The LAT member should do a cursory review of the COC forms to ensure the information contained therein is consistent with the ASRF.

Data Package Review and Delivery

The laboratory sends all data packages directly to the assigned LAT member who performs a preliminary review to ensure that the laboratory has submitted the requested information. This review shall be performed within approximately 1 day of receipt. Each data package is evaluated by the assigned LAT member for the following criteria ONLY:

- Laboratory reports address methods specified on COCs
- Requested results present for all samples
- Appropriate level data package provided
- Data received within requested turnaround time

Upon completion of this review, the original data package is forwarded to the PM unless the PM specifically requests otherwise.

Invoices

The laboratory sends all invoices to the assigned LAT member. Upon receipt of the invoice, it is reviewed for agreement with the Work Authorization and project sampling documentation. Upon confirmation (within approximately 24 hours of receipt), the LAT member will forward the invoice to TechLaw Holdings Accounts Payable and send a copy to the TechLaw Holdings PM.

Communication with the Laboratories

A member of the LAT, preferably the assigned LAT member, is to participate in all communications with laboratories. This is to ensure that all procedures required under the laboratory agreements and the TechLaw Holdings Quality Management Plan (QMP) are met. The LAT member will notify the laboratory of any unusual situations, including the expected presence of high concentrations of contaminants at the site.

Obtaining New Laboratories

All samples must be sent to a TechLaw Holdings-approved laboratory. Names of approved laboratories may be obtained from the LAT Coordinator. Only senior members of the LAT, with the concurrence of the TechLaw Holdings Quality Assurance Director (QAD), may approve a new laboratory; this approval is conditional upon examination of laboratory-specific documentation. Necessary documentation consists of a laboratory quality assurance (QA) plan (must include a description and number of instruments, staff resumes and a summary of QA/QC procedures), current accreditation certification if routine analyses are being conducted (including updates when they become available), SOPs, method detection limits (MDLs), quantitation limits, proficiency examination (PE) sample results, and pricing.

An onsite laboratory audit may also be performed by the TechLaw Holdings LAT. A laboratory audit will be performed at the discretion of the LAT Coordinator and TechLaw Holdings QAD. The laboratory audit checklist is included as Attachment F to this SOP. Note that the laboratory audit checklist is based on International Organization for Standardization (ISO) and International Electrotechnical Commission (IEC) 17025, second edition, dated May 15, 2005.

After a laboratory has been approved by the LAT, a Laboratory Agreement shall be arranged by the TechLaw Holdings Contracts Administrator working in conjunction with the senior LAT member responsible for assessing the laboratory's qualifications. Only after these procedures have been completed may a new laboratory be used for analytical services.

The laboratory list is reviewed and updated biennially. If a problem is encountered with an approved laboratory, its approval status will be reviewed at that time (i.e., more frequently). LAT laboratories are contacted to obtain any updated laboratory documentation and pricing during the biennial review.

Health and Safety

Not applicable

QA/QC

None at this time

Comments/Notes

Under no circumstances is it acceptable to provide the laboratory with the name, location or other identifying information for the site (this includes listing facility information on the COC). Facility initials, TechLaw Holdings project number, or other identifier should be used that will not reveal facility information to the laboratory, but will be evident to TechLaw Holdings employees involved with the project. If the laboratory becomes aware of the site name, the LAT member should inform the TechLaw Holdings Conflict of Interest (COI) Officer immediately. The COI Officer will ensure that the laboratory does not have a COI and will post documentation of this confirmation to the LAT project files on SharePoint.

The time required to arrange analytical services and process data packages and invoices will be charged to the appropriate project.

Attachments

Attachment A – Approved LAT Members

Attachment B – Analytical Support Request Form

Attachment C – Laboratory Work Authorization (generic)

Attachment D – LAT Member Checklist

Attachment E – TechLaw Holdings Project Manager Checklist

Attachment F – Laboratory Audit Checklist

References

International Organization for Standardization and International Electrotechnical Commission 17025, second edition, dated May 15, 2005, reference number ISO/IEC 17025:2005(E)

TechLaw Holdings and Subsidiary Companies, Corporate Quality Management Plan, current version.

TechLaw Holdings and Subsidiary Companies, Health and Safety Program, current version.

ATTACHMENT A [Revised 2/3/2017]
SOP Number: 02-06-08

TechLaw Holdings Approved LAT Members

LAT Coordinator: Jim Burden
536 South Clark Street, Suite 734
Chicago, IL 60605
(312) 353-2964
(312) 877-0048 (mobile)

LAT Members:

Ms. Nikki Thomsen (Backup LAT Coordinator) 600 12th Street., Suite 110 Golden, CO 80401 (303) 552-5809 (303) 746-7647 (mobile)	Ms. Rachel Ireland 7 Technology Drive, Unit 202 North Chelmsford, MA 01863 (978) 275-9749 (617) 283-1332 (mobile)
Mr. Eric Middleditch 600 12th Street., Suite 110 Golden, CO 80401 (303) 586-4822 (720) 364-7899 (mobile)	Mr. Bruce Fritz (TLI Solutions only) 112 N. Rubey Drive, Suite 210 Golden, CO 80401 (303) 416-6295 (720) 292-0659 (mobile)
Mr. Gene Nance (START Region 3 only) 5455 County Road 2 Chesapeake, OH 45619 (740) 867-0968 (304) 830-1442 (mobile)	Mr. Scott Walker (ESAT Region 8 only) 16194 W. 45th Dr. Golden, CO 80403 (303) 312-7726 (303) 453-9018 (mobile)
Mr. Doug Kent (ESAT Region 8 only) 16194 W. 45th Dr. Golden, CO 80403 (303) 312-7725 (303) 489-0793 (mobile)	Mr. Nathan DelHierro (ESAT Region 8 only) 16194 W. 45th Dr. Golden, CO 80403 (303) 312-7790 (720) 695-6304 (mobile)

ATTACHMENT A [Revised 2/3/2017]
SOP Number: 02-06-08

If none of the above LAT Members are available, and an urgent laboratory need/data issue arises, please contact:

Mr. Bill Fear
600 12th Street, Suite 110
Golden, CO 80401
(303) 586-4572
(303) 204-4540 (mobile)

Mr. David Dobb
7411 Beach Drive East
Port Orchard, WA 98366
(360) 871-8750
(360) 337-0625 (mobile)

TechLaw Holdings COI Officer: Ms. Judy Manley
14500 Avion Parkway, Suite 300
Chantilly, VA 20151
(703) 818-3233
(703) 209-5187 (mobile)

ATTACHMENT B [Revised 5/31/2013]
SOP Number: 02-06-08

ANALYTICAL SUPPORT REQUEST FORM

Project Number (Bill Code): _____

TechLaw Holdings Project Manager: _____

Site Name and Location: _____

Site Code (identifier)*: _____

***Site Code should not reveal the site name or location and will be the only identifier used in communication with the laboratory (including COCs) to prevent COI issues**

Example: Site Name and Location: Buckeye Products in Adrian, Michigan
Site Code: BAM (used the first letter of facility name, city, and state)

Date(s) of Sampling Event: _____

Glassware: Date Needed: _____ Location: _____

Turnaround Time (circle one): Standard (21 days) / Rush-_____ days (extra charge)

Data Package: Level IV (full "CLP-like") / Other (Level II or III)_____

Electronic Date Deliverable? Yes / No Format (i.e., Excel): _____

Special Considerations:

- **Are any special certifications required?**
- **Are there minimum volume or filter requirements?**
- **Is there a specific QAPP requirement?**
- **Are high concentrations expected?**
- **Are verbal or preliminary results required?**

PLEASE ATTACH A TABLE OF THE APPLICABLE SCREENING LEVELS FOR ALL COMPOUNDS TO BE ANALYZED. The LAT staff will use this information to verify that the laboratory reporting limits will meet the specified screening levels.

Matrix [1]	Number of field samples	Parameter (method #'s)[2]	Required Reporting Limits	Number of field duplicates	Number of trip blanks	Number of field blanks	Number MS/MSD

1 Be specific (i.e., surface H2O, liquid fuel, slag; not just solid/water).

2 Be specific; if split sampling, attach applicable MDLs, action levels, methodologies, etc., from facility work plan. For metals, please specify which compound list should be utilized: RCRA 8, priority pollutants, Target Analyte List

Note: Target Analyte List compounds will be utilized, unless otherwise specified by the Project Manager.

TechLaw Holdings Project Manager Signature: _____ Date: _____

LAT Signature:_____ Date: _____

ATTACHMENT C [Revised 4/22/2016]
SOP Number: 02-06-08

LABORATORY ANALYTICAL WORK AUTHORIZATION

Date:

To: Contact
Laboratory
Address
Address
Phone
Email

From: [LAT Member], LAT Representative
Address
Address
Phone
Email

Re: EPA Prime Contract XXXXX
TechLaw Holdings Laboratory Agreement
Task Order Authorization Number: XXXX
Site Code (identifier): XXX
Project Code: [Insert billing code]

This document authorizes work on the subject Contract as outlined in the attached Scope of Work and Pricing quotation (Attachment A). The expenditure limits on the Contract are XXXXX for work performed through (insert date). If it is anticipated that these funding limitations will be exceeded in performance of this work, you must notify us in a timely manner. Failure to notify and negotiate additional funding will result in forfeiture of costs incurred in excess of the funding limitations. Invoices should be sent to the Laboratory Assistance Team (LAT) representative noted above.

Please acknowledge your acceptance of work by signing in the space provided on this Laboratory Analytical Work Authorization Form, and return the signed form to [TechLaw Holdings Contracting Staff (insert email address)] and [LAT Member (insert email address)]. By acceptance of this Work Authorization, the Laboratory confirms that: no known personal or organizational conflict of interest exists; best efforts will be employed to conduct the work specified to the satisfaction of TechLaw Holdings representatives; all terms and conditions of the Agreement

identified and the Scope of Work and Pricing document will be met in performance of the work specified herein.

Authorized Signatures:

TechLaw Holdings	**Laboratory Name**
Name:	Name:
Title:	Title:
Date:	Date:

ATTACHMENT D [Revised 2/3/2017]
SOP Number: 02-06-08

LAT Member Checklist

1. If a TechLaw Holdings Project Manager (PM) contacts you regarding laboratory procurement, please ask them to fill out the Analytical Support Request Form (ASRF) in Attachment B of SOP 02-06-XX (current version) or send an email providing all information contained in this form, if they have not already done so. Upload completed form to the project file on SharePoint.
    a. Be sure to ask if there are any reporting limit requirements, and verify with the laboratory that they can achieve such requirements
2. Contact three TechLaw Holdings Approved Laboratories to obtain price quotes and to ensure they have capacity to analyze the samples within the requested turnaround time.
    a. If a specialized analysis is required and is not performed by a TechLaw Holdings Approved Laboratory, another laboratory may be used upon consultation with the LAT Coordinator.
3. Select the laboratory based on lowest pricing and ability to perform the requested analyses.
4. Send the winning laboratory bid to the TechLaw Holdings PM and request approval.
5. Upload TechLaw Holdings PM approval documentation to SharePoint.
6. Fill out the Work Authorization Form in Appendix C of SOP 02-06-XX (current version), convert it to a PDF file, and attach the analytical quote to end of the PDF. Submit the form to the selected laboratory via e-mail (copy TechLaw Holdings Contracting Staff). TechLaw Holdings Contracting Staff will email the LAT member the purchase order (PO) associated with the analytical request.
7. Check the appropriate laboratory folder under LAT on SharePoint to ensure we have the SOP for the methods requested for the project. If these are not available already on SharePoint, request a copy of the SOP from the lab and upload to the laboratory folder on SharePoint.
8. Create a folder for your project under LAT > Project Files.
9. Update the Project Tracking spreadsheet under LAT > Project Tracking and ensure justification for laboratory selection is included in the Comments column.
10. Upon submission to the laboratory, upload a copy of the Work Authorization Form (unsigned by lab) to LAT > Project Files > Project Name.
11. Once a signed copy of the Work Authorization Form is received from laboratory, upload to LAT > Project Files > Project Name.
12. Confirm with laboratory when/where to send bottleware.
13. Inform the laboratory when to expect samples (it is also a good idea to remind them one day before they will receive samples).
14. Ensure the laboratory submits a copy of the COC to you to confirm that samples submitted are consistent with the ASRF and for verification of invoicing/sample data.
15. Upon receipt of the analytical data, review for the following:
    a. Laboratory reports address methods specified on COCs

b. Requested results present for all samples submitted to the lab
c. Appropriate level data package provided
d. Data received within the requested turnaround time
e. Scan through the laboratory case narrative for any major issues that would result in rejection of data.

16. If the items above are acceptable, approve invoice by signing, dating, and adding the proper PO number (including line item number) and forward to TechLaw Holdings Accounts Payable at ap@techlawholdings.com (copy the TechLaw Holdings PM).
17. Ask the TechLaw Holdings PM where the data should be sent, and send it out as soon as possible. If electronic data is available, you may upload a copy to the project folder under LAT > Project Files.

ATTACHMENT E [Revised 5/31/2013]
SOP Number: 02-06-08

TechLaw Holdings Project Manager Checklist for LAT

1. Fill out the Analytical Support Request Form (ASRF) in Attachment B of SOP 02-06-07 and send an email to the LAT providing all information contained in this form.
2. Notify LAT member of any changes in schedule/requirements as soon as possible.
3. Inform LAT member where to send data upon receipt.

LAT members will:

- Procure laboratories
- Order bottleware, preservatives, and laboratory-grade water for blanks (if requested to do so)
- Handle all communication with the laboratory
- Add another laboratory to the TechLaw Holdings Approved Lab List only if required by the project (i.e., 24-hour turnaround for an emergency response) and current approved laboratories cannot fulfill the project requirements. Note: Additional time will be required to obtain the necessary information and add the laboratory to the approved list.

LAT members do NOT:

- Verify reporting limits if no requirements have been provided by the TechLaw Holdings Project Manager
- Perform data validation (unless qualified and asked to do so)
- Coordinate a data validator (unless asked to do so and authorized hours are provided)

ATTACHMENT F [Revised 5/31/2013]
SOP Number: 02-06-08

LABORATORY AUDIT CHECKLIST

Laboratory On-Site Visits

LABORATORY AUDIT CHECKLIST
Section A - Overview

There are several purposes for making on-site visits to analytical laboratories. The most common purposes are:

1. Prior to award of a contract or delivery of samples, a client visits the laboratory to verify that the laboratory has the capability to perform the needed work. The areas for which capability must be judged are:

   - Physical facility – adequate work space, adequate and appropriate air handling, adequate storage space.
   - Equipment – all equipment (instrumentation, reagents, glassware, etc.) needed to do the job at the needed frequency.
   - Personnel – trained, experienced personnel who meet the clients' requirements.
   - Standard Operating Procedures (SOPs) – written procedures must be in place (and updated when changes to "modus operandi" are made) for all operations of the laboratory so that consistency and continuity are maintained where appropriate.
   - Quality Assurance (QA) Program – includes all aspects of EPA's "Good Automated Laboratory Practices" (GALP) Guidance.
   - Appropriate evidentiary procedures, chain-of-custody (COC) documentation, and security systems must be in place.

2. Post contract award or after sample delivery by a client (at intervals determined appropriate by the client), the laboratory can be visited to verify that the capabilities evaluated in Number one (1) still exist, or improvements cited as needed or deficiencies cited as requiring correction in Number one (1) have occurred.

3. Problem resolution visits – When problems are noted by the client (e.g., performance evaluation samples not analyzed acceptably, lateness, non-compliance with contract requirements, etc.), laboratories can be visited to isolate problem areas and identify where corrective action must be taken by laboratory management.

4. Unannounced visits – To verify that the laboratory follows procedures and maintains systems per the client's requirements, even when the client's visit is not expected.

5. Unannounced visits – When there is reason to believe a laboratory may be involved in improper practices (e.g., data falsification/alteration), a client may want a "surprise" visit. This visit should focus an audit on the area perceived to be vulnerable.

6. Routine – Even when a laboratory is performing well, a client presence to show interest (and maybe a "pat on the back") is important every other year if possible.

As we look at the cited purposes, we should take the opportunity to identify what we really need to do during an on-site visit to meet our needs. We should minimize the universe of possible targets for evaluation and focus on what is important.

We should recognize that the capability to meet our needs (produce our required product) may come in many forms, and a stereotyped approach on our part is unreasonable and unnecessary. If we accept this premise, we go a long way towards minimizing our efforts in auditing the laboratory and opening the door to innovation and creativity on the part of our laboratories that may save time and money and may produce a better product.

Since the first two types (and sometimes Number 6) of on-site visits are the most common and involve looking at the same things (which are primarily amenable to a checklist approach), the first effort at a new design of an on-site visit will consist of an appropriate checklist.

It is important to note that a checklist only meets part of our needs: one-on-one conversations with laboratory personnel who will perform our work should occur to make sure they understand our requirements, follow their SOPs, are properly trained, etc.

Proposed checklists for Evidentiary and Technical on-site visits (audits) are in the following sections of this Laboratory Audit Checklist. They are designed so one auditor can perform the full gamut of evaluation in one swing through a laboratory. The auditor should become familiar with the checklists prior to an audit so that all necessary checks can be made in one location in a laboratory at one time.

These checklists are not contract specific. They can be used for non-CLP and CLP because they do not demand conformance, only observance of what is in place. The auditor is responsible for determining if the laboratory appears to meet the requirements of the client (which in the case of CLP labs, is the SOW).

The auditor should only evaluate the laboratory according to the items that the client considers relevant to meet needs (for CLP contracts, this is all items). Judgment should be used when determining what items are relevant to meet needs.

LABORATORY AUDIT CHECKLIST
Section B - General

This section must be completed for every on-site laboratory audit

Laboratory Name: _____

Address: _____

_____

_____

_____

Date of on-site visit: _____

General Information

1. Audit Staff
   - Name: _____ _____
   - Phone: _____ _____
   - E-mail: _____ _____
2. Management Staff
   - Name: _____ _____
   - Title: _____ _____
   - Phone: _____ _____
   - E-mail: _____ _____
3. Quality Assurance Officer
   - Name: _____
   - Phone: _____
   - E-mail: _____
4. State the reason for the on-site visit (refer to Items 1-6 in Section A - Overview):

   _____

   _____

   _____
5. Type of Evaluation

   Q Evidentiary Q Organics Q Inorganics Q Asbestos

   Note: If you are performing an Evidentiary audit, do not complete the Organics, Inorganics or Asbestos checklists. If you are performing an Organics, Inorganics, and/or Asbestos audit, do not complete the Evidentiary checklist.

LABORATORY AUDIT CHECKLIST

Section C - Evidentiary Procedures Evaluation Checklist

Intructions: Complete this section for Evidentiary audits only. Do not complete this section if you are performing an Organics, Inorganics and/or Asbestos audit.

Place an "X" beside each checklist item that represents a nonconformity. Place a "C" beside each item on which you are commenting for other reasons. Record the item number and written nonconformity explanation and/or comment on the comment sheet(s) at the end of this section. Write "N/A" beside any item that is not applicable to the current audit. Write "OK" beside all other items you observed or verified as compliant.

Personnel Interviewed:

Name	Title/Department

I. Sample Receipt

_____ 1. The laboratory has a designated sample custodian and alternate for each shift.

Names:

Sample Custodians	Alternates

_____ 2. SOPs for sample receipt are in place and readily available.

_____ 3. SOPs for sample receipt are followed by laboratory personnel.

_____ 4. The sample receipt area is secured against non-authorized personnel.

5. The sample custodian verifies the following:

_____ a. Condition of shipping cooler

_____ b. Presence or absence of custody seals

_____ c. Condition of custody seals, when present

_____ d. Custody seal numbers, when present

_____ e. Presence or absence of COC records

_____ f. Presence or absence of airbill stickers

_____ g. Airbill or airbill sticker number

_____ h. Presence or absence of sample tags

_____ i. Sample tag numbers

_____ j. Condition of sample containers

_____ k. Discrepancies in any information recorded on COC records, client requests, airbills, sample containers, etc.

_____ l. Documentation of hand deliveries

_____ m. Problems encountered

Obtain examples of all forms used during sample receipt.

II. Sample Identification

_____ 6. The laboratory has a unique sample identification system (i.e., vs. using client sample ID numbers).

_____ a. The number is assigned upon receipt. If no, when?

_____ _____

_____ b. If yes, the numbers are cross referenced to client numbers in a log.

_____ 7. The system clearly applies to samples, extracts, digestates, etc.

_____ 8. SOPs are readily available for sample identification.

_____ 9. SOPs for sample identification are followed by laboratory personnel.

Obtain example of laboratory's sample ID system (e.g., example sample number with cross reference).

III. Sample Storage and Tracking

_____ 10. Sample (extracts, etc.) storage areas are secured and access to samples (extracts, etc.) is available only to authorized personnel.

_____ 11. Samples (extracts, etc.) are logged in/out of storage area(s) when accessed.

_____ 12. Samples (extracts, etc.) are tracked throughout analytical process (e.g., a traveler sheet).

_____ 13. SOPs for storage and tracking of samples (extracts, etc.) are readily available.

_____ 14. SOPs for storage and tracking are followed by laboratory personnel.

Obtain examples of all forms/documents used for storage and tracking records.

IV. Document Review

15. Evaluate documents with the following in mind:

_____ a. Activities (e.g., GC/MS-VOA, ICP-metals) are identified on all analysis documents.

_____ b. Titles are on all documents.

_____ c. Columns are labeled with headers.

_____ d. Reviewers' signatures are identified when applicable.

_____ e. The laboratory's name is on all documents.

_____ f. All entries are fully dated (day, month, year).

_____ g. Entries are signed by the person responsible for performing and recording activities.

_____ h. All logbook and other document entries are in ink.

_____ i. Error correction protocol is followed (single line through area to be corrected and corrector's initials - no "white out").

_____ j. Pages in bound and unbound logbooks are sequentially numbered.

_____ k. Logbook entries are in chronological order.

_____ l. Inserted information taped into logbooks is signed and dated when activity is performed.

_____ m. Unused portions of documents are lined out.

_____ 16. Documents provide a complete record of activities observed by the evaluator.

_____ 17. Instrument run logs are maintained to enable a reconstruction of the run sequence on an instrument.

_____ 18. Records of failed runs are maintained.

_____ 19. Disposal/depletion of samples (extracts, etc.) is documented.

_____ 20. For data transferred electronically within the laboratory, a hardcopy is printed and retained in a client/case file.

21. If data is transferred electronically, the following information recorded:

_____ a. Person responsible for electronic data transfer.

_____ b. Date of electronic transfer.

_____ c. Person to whom data was electronically transferred.

_____ d. Status of electronically transferred data (e.g., draft, final, etc.).

_____ e. Numerical identifier assigned to electronic data transfer.

_____ 22. SOPs are readily available for electronic data transfer.

_____ 23. SOPs are followed by laboratory personnel for electronic data transfer.

V. Confidential Information

24. If the laboratory receives confidential information/documents, there is a system set up to maintain that confidentiality, including for data generated on associated samples.

VI. Case (Client's Designated Group of Samples) File Organization and Assembly

Names:

Document Control Officer _____ Alternate _____

_____ 25. Case documents are maintained in a secure area.

_____ 26. Shipments of deliverables to clients are documented.

_____ 27. The recipient is identified.

_____ 28. Deliverables are sealed with custody seals.

_____ 29. Custody seals are signed and dated.

_____ 30. The document control officer assembles and cross checks information to assure that data on each case file is consistent and complete.

VII. Security of the Facility

_____ 31. Visitors are required to sign in.

_____ 32. Visitors are required to display distinct badges/ID.

_____ 33. All doors to outside lock except to the reception area.

_____ 34. Access to laboratory and data reduction/report preparation areas is limited to authorized personnel.

COMMENTS AND NONCONFORMITIES

Section C - Evidentiary Procedures Evaluation Checklist

Instructions: Use this sheet to document comments or nonconformities. For each, identify the appropriate item number from the checklist. Identify comments with a "C" and nonconformities with an "X." If additional space is needed, make copies of this page (or use additional blank sheets).

Item No.	***C or X***	***Comments and/or Nonconformities***

COMMENTS AND NONCONFORMITIES (Cont.)

Section C - Evidentiary Procedures Evaluation Checklist

Item No.	***C or X***	***Comments and/or Nonconformities***

LABORATORY AUDIT CHECKLIST

Section D - Organics Technical Procedures Evaluation Checklist

Instructions: Place an "X" beside each checklist item that represents a nonconformity. Place a "C" beside each item on which you are commenting for other reasons. Record the item number and written nonconformity explanation and/or comment on the comment sheet(s) at the end of this section. Write "N/A" beside any item that is not applicable to the current audit. Write "OK" beside all other items you observed or verified as compliant.

Personnel Interviewed:

Name	Title/Department

I. Sample Receipt

_____ 1. The laboratory has a designated sample custodian and alternate for each shift.

Names:

Sample Custodians	Alternates

_____ 2. SOPs for sample receipt are in place and readily available.

_____ 3. SOPs for sample receipt are followed by laboratory personnel.

_____ 4. The sample receipt area is secured against non-authorized personnel.

5. The sample custodian verifies the following:

_____ a. Condition of shipping cooler

_____ b. Presence or absence of custody seals

_____ c. Condition of custody seals, when present

_____ d. Custody seal numbers, when present

_____ e. Presence or absence of COC records

_____ f. Presence or absence of airbill stickers

_____ g. Airbill or airbill sticker number

_____ h. Presence or absence of sample tags

_____ i. Sample tag numbers

_____ j. Condition of sample containers

k. Discrepancies in any information recorded on COC records, client requests, airbills, sample containers, etc.

l. Documentation of hand deliveries

m. Problems encountered

Obtain examples of all forms used during sample receipt.

II. Sample Identification

6. The laboratory has a unique sample identification system (i.e., vs. using client sample ID numbers).

a. The number is assigned upon receipt. If no, when? _____

b. If yes, the numbers are cross referenced to client numbers in a log.

7. The system clearly applies to samples, extracts, digestates, etc.

8. SOPs are readily available for sample identification.

9. SOPs for sample identification are followed by laboratory personnel.

Obtain example of laboratory's sample ID system (e.g., example sample number with cross reference).

III. Sample Storage and Tracking

10. Sample (extracts, etc.) storage areas are secured and access to samples (extracts, etc.) is available only to authorized personnel.

11. Samples (extracts, etc.) are logged in/out of storage area(s) when accessed.

12. Samples (extracts, etc.) are tracked throughout analytical process (e.g., a traveler sheet).

13. SOPs for storage and tracking of samples (extracts, etc.) are readily available.

14. SOPs for storage and tracking are followed by laboratory personnel.

Obtain examples of all forms/documents used for storage and tracking records.

IV. Sample Receipt and Storage Area

15. Sample shipping coolers are opened in a contamination-free area (e.g., fume hood or vented area).

16. Adequate facilities are provided for the cold storage of samples and unused samples for 60 days after data submission.

a. The temperature of the cold storage is recorded daily in a logbook.

b. Temperature excursions are noted and appropriate actions are taken when required.

17. Volatile samples are stored separately from semi-volatile samples and extracts.

_____ 18. Sample extracts are properly stored (2-6◦C, separate) and easy to locate by reference to a logbook.

V. Sample Preparation Area

19. The laboratory is maintained in a clean and organized manner appropriate for trace level analyses (contamination free).

_____ 20. The laboratory appears to have adequate work space (6 linear feet of unencumbered bench top per analyst).

_____ 21. The laboratory benches are made of suitable chemically resistant materials.

_____ 22. Sufficient functional hoods are available.

_____ 23. Documented organic free water (for organics standards, blanks, dilutions) is available.

24. Analytical balances are located away from drafts and areas subject to rapid temperature changes.

_____ a. Balances are checked routinely (e.g., before each weighing session) with the appropriate range of weights and the results are recorded in a permanent logbook.

_____ b. Routine weights are checked against Class S (or equivalent) weights at least once a month and the results are recorded in a permanent logbook.

_____ c. Balances have been calibrated within one year by a certified technician.

_____ d. Data generated from balances are electronically transferred or manually entered into LIMS.

_____ 25. Sample preparation SOPs are readily available.

_____ 26. Sample preparation SOPs are followed by laboratory personnel.

_____ 27. Glassware preparation/cleaning SOPs are readily available.

_____ 28. Glassware preparation/cleaning SOPs are followed by laboratory personnel.

_____ 29. All required sample preparation equipment is available.

_____ a. Sonicator Make _____
Model _____
Backup (Y/N) _____

_____ b. GPC Make _____
Model _____
Backup (Y/N)

_____ c. GPC UV Detector
Make _____
Model _____

_____ d. GPC logs indicate that corrective actions are taken when there is a problem with calibration.

_____ e. The number of continuous liquid/liquid extractors: _____

30. Analysts record bench data in a neat and accurate manner.
31. Analysts record the lot number of solvents, spiking solutions, etc. on bench sheets.
32. There is evidence of a secondary review of all documents and logbooks by someone other than the person generating the documents.
33. Review the following procedures for oven drying:
    a. Temperatures in the drying ovens are verified against NIST traceable thermometers.
    b. Ovens have temperature logbooks.
    c. "In" and "Out" drying times are recorded.

VI. Standards Preparation and Storage

34. SOPs for standards preparation are readily available.
35. SOPs for standards preparation are followed by laboratory personnel.
36. Reagent grade or higher purity chemicals are used to prepare standards.
37. Standards are properly labeled with concentrations, date of preparation, expiration date, and/or traceable reference code number.
38. Spiking/calibration standards preparation and tracking logbooks for:
    a. VOCs
    b. SVOCs
39. Logbook numbers and series of stock solutions and reagents are recorded.
40. For commercially prepared standard mixes, appropriate documentation is available (Manufacturer's Certificate of Analysis).

VII. Analytical Instrumentation and Analyses-Specific Items

List the following for each instrument:

Type	ID Number	Manufacturer/Model	Software

41. The manufacturer's operating manuals are readily available.
42. The laboratory has service contracts for each instrument.
43. The laboratory has extensive replacement parts available.
44. A permanent service record is maintained for each instrument.
45. The laboratory uses a recent mass spectral library.
46. Magnetic tape storage of GC/MS electronic data:
    a. Raw data, including quantitative output files and libraries, are archived on magnetic tape.
    b. A log of raw data contents of tapes is maintained.
47. For VOA analyses:
    a. Equipment is available for heated purge and trap for low level soil analysis.
    b. VOA holding blanks results are available.
48. The instrument operator can show from the run log that corrective actions were taken for:
    a. Re-analysis when internal standard areas are out of control.
    b. Dilutions when calibration range is exceeded.
    c. Blanks when previous sample showed saturation.
49. SOPs for analyses and logbook completion are readily available.
50. SOPs for analyses and logbook completion are followed by laboratory personnel.
51. There is evidence of a secondary review of all documents and logbooks by someone other than the person generating the documents.

VIII. Data Handling and Review (GALP)

52. Data calculations are spot checked by a second person.
53. Records indicate appropriate corrective action when QC criteria are not met.
54. Supervisory personnel review the data and QC results prior to submission.
55. SOPs for data handling/review are readily available.
56. SOPs for data handling/review are followed by laboratory personnel.
57. Deliverables are checked for completeness and accuracy (hardcopy and electronic), including resubmittals.
58. The monthly data entry error rate is determined and recorded.
59. When changes to deliverables are required, the changes are properly documented (rationale, review, initials, etc.).
60. User manuals and operations/systems manuals are available.

_____ 61. A written software test and acceptance plan is available for installation of system changes.

IX. Quality Assurance Internal Inspections

_____ 62. There is an internal QA inspection procedure.

_____ 63. The QA Officer reports to senior management.

_____ 64. Corrective actions are documented.

_____ 65. Internal audits are performed.

List the types of internal audits

_____ _____

_____ _____

_____ _____

66. The following QA records are kept:

_____ a. PE sample results
_____ b. Records of recoveries (extractions, etc.)
_____ c. Training/experience records of personnel
_____ d. Method sensitivities
_____ e. Control charts for QC purposes
_____ f. Other _____

_____ 67. A Quality Assurance Manual (or equivalent) is available.

68. The Quality Manual addresses the following:

_____ a. Organization and philosophy
_____ b. Facilities and equipment
_____ c. Document control
_____ d. Analytical methodology
_____ e. Data generation
_____ f. QA
_____ g. QC
_____ h. Corporate ethics policy

X. Standard Operating Procedures

69. In addition to the previously mentioned SOPs, the following SOPs are also available:

_____ a. Calibration (balance)
_____ b. Calibration (instruments)
_____ c. Maintenance activities (for each system)
_____ d. Data reduction procedures

e. Documentation policy/procedures

f. Data validation/self-inspection procedures

XI. Organization and Personnel Summary

70. Personnel assigned to this project have the appropriate educational background to accomplish the objectives of the program (see Key Personnel list below).

71. The laboratory is adequately staffed to meet project commitments in a timely manner.

72. All key personnel were present and available for the audit.

XII. Laboratory Capacity

73. The laboratory has sufficient analytical instrumentation to analyze the needed number of samples.

74. The laboratory has sufficient technical and administrative personnel to deliver the number of needed analyses.

75. The laboratory has an adequate sample and data tracking system to handle the needed number of analyses.

XIII. Key Personnel List (not previously identified)

Organics Supervisor

Name: _____

Generally requires Bachelor's degree in chemistry/science/engineering + 3 years organics experience, including 1 year supervisory experience.

Sample Preparation Laboratory Supervisor

Name: _____

Generally requires Bachelor's degree in chemistry/science/engineering + 3 years organics experience, including 1 year supervisory experience. Three additional years experience may substitute for education requirement.

GC/MS Operator

Name: _____

Generally requires Bachelor's degree in chemistry/science/engineering + 1 year GC/MS/DS experience or 3 years GC/MS/DS experience and GC/MS interpretation. Three additional years experience may substitute for education requirement.

GC/EC Operator

Name: _____

Generally requires Bachelor's degree in chemistry/science/engineering + 1 year GC/EC experience or 3 years GC/EC experience and GC/EC interpretation. Three additional years experience may substitute for education requirement.

Extraction Concentration Expert

Name: _____

Generally requires high school diploma and college level course in general chemistry + 1 year experience in extraction/concentration.

Backup Chemist (Technical Staff Redundancy)

Name: _____

Generally requires Bachelor's degree in chemistry/science/engineering + 1 year lab experience in GC/MS operation, MS interpretation, extraction, and pesticide analysis.

COMMENTS AND NONCONFORMITIES

Section D - Organics Technical Procedures Evaluation Checklist

Instructions: Use this sheet to document comments or nonconformities. For each, identify the appropriate item number from the checklist. Identify comments with a "C" and nonconformities with an "X." If additional space is needed, make copies of this page (or use additional blank sheets).

Item No.	***C or X***	***Comments and/or Nonconformities***

COMMENTS AND NONCONFORMITIES (Cont.)

Section D - Organics Technical Procedures Evaluation Checklist

Item No.	***C or X***	***Comments and/or Nonconformities***

LABORATORY AUDIT CHECKLIST

Section E - Inorganics Technical Procedures Evaluation Checklist

Instructions: Place an "X" beside each checklist item that represents a nonconformity. Place a "C" beside each item on which you are commenting for other reasons. Record the item number and written nonconformity explanation and/or comment on the comment sheet(s) at the end of this section. Write "N/A" beside any item that is not applicable to the current audit. Write "OK" beside all other items you observed or verified as compliant.

Personnel Interviewed:

Name	Title/Department

I. Sample Receipt

_____ 1. The laboratory has a designated sample custodian and alternate for each shift.

Names:

Sample Custodians	Alternates

_____ 2. SOPs for sample receipt are in place and readily available.

_____ 3. SOPs for sample receipt are followed by laboratory personnel.

_____ 4. The sample receipt area is secured against non-authorized personnel.

5. The sample custodian verifies the following:

_____ a. Condition of shipping cooler

_____ b. Presence or absence of custody seals

_____ c. Condition of custody seals, when present

_____ d. Custody seal numbers, when present

_____ e. Presence or absence of COC records

_____ f. Presence or absence of airbill stickers

_____ g. Airbill or airbill sticker number

_____ h. Presence or absence of sample tags

_____ i. Sample tag numbers

_____ j. Condition of sample containers

k. Discrepancies in any information recorded on COC records, client requests, airbills, sample containers, etc.

l. Documentation of hand deliveries

m. Problems encountered

Obtain examples of all forms used during sample receipt.

II. Sample Identification

6. The laboratory has a unique sample identification system (i.e., vs. using client sample ID numbers).

a. The number is assigned upon receipt. If no, when? _____

b. If yes, the numbers are cross referenced to client numbers in a log.

7. The system clearly applies to samples, extracts, digestates, etc.

8. SOPs are readily available for sample identification.

9. SOPs for sample identification are followed by laboratory personnel.

Obtain example of laboratory's sample ID system (e.g., example sample number with cross reference).

III. Sample Storage and Tracking

10. Sample (extracts, etc.) storage areas are secured and access to samples (extracts, etc.) is available only to authorized personnel.

11. Samples (extracts, etc.) are logged in/out of storage area(s) when accessed.

12. Samples (extracts, etc.) are tracked throughout analytical process (e.g., a traveler sheet).

13. SOPs for storage and tracking of samples (extracts, etc.) are readily available.

14. SOPs for storage and tracking are followed by laboratory personnel.

Obtain examples of all forms/documents used for storage and tracking records.

IV. Sample Receipt and Storage Area

15. Sample shipping coolers are opened in a contamination-free area (e.g., fume hood or vented area).

16. Adequate facilities are provided for the cold storage of samples and unused samples for 60 days after data submission.

a. The temperature of the cold storage is recorded daily in a logbook.

b. Temperature excursions are noted and appropriate actions are taken when required.

17. The pH of the samples is recorded and available for data review.

V. Sample Preparation Area

18. The laboratory is maintained in a clean and organized manner appropriate for trace level analyses (contamination free).

19. The laboratory appears to have adequate work space (6 linear feet of unencumbered bench top per analyst).

20. The laboratory benches are made of suitable chemically resistant materials.

21. Sufficient functional hoods are available.

22. Documented organic free water (for organics standards, blanks, dilutions) is available.

23. Analytical balances are located away from drafts and areas subject to rapid temperature changes.

    a. Balances are checked routinely (e.g., before each weighing session) with the appropriate range of weights and the results are recorded in a permanent logbook.
    b. Routine weights are checked against Class S (or equivalent) weights at least once a month and the results are recorded in a permanent logbook.
    c. Balances have been calibrated within one year by a certified technician.
    d. Data generated from balances are electronically transferred or manually entered into LIMS.

24. Sample preparation SOPs are readily available.

25. Sample preparation SOPs are followed by laboratory personnel.

26. Glassware preparation/cleaning SOPs are readily available.

27. Glassware preparation/cleaning SOPs are followed by laboratory personnel.

28. If microwave digestion is used, adequate microwave ovens (programmable power setting up to 600 watts) are available.

29. Analysts record bench data in a neat and accurate manner.

30. Analysts record the lot number of solvents, spiking solutions, etc. on bench sheets.

31. There is evidence of a secondary review of all documents and logbooks by someone other than the person generating the documents.

32 Review the following procedures for oven drying:

    a. Temperatures in the drying ovens are verified against NIST traceable thermometers.
    b. Ovens have temperature logbooks.
    c. "In" and "Out" drying times are recorded.

VI. Standards Preparation and Storage

_____ 33. SOPs for standards preparation are readily available.

_____ 34. SOPs for standards preparation are followed by laboratory personnel.

_____ 35. Reagent grade or higher purity chemicals are used to prepare standards.

_____ 36. Standards are properly labeled with concentrations, date of preparation, expiration date, and/or traceable reference code number.

37. Spiking/calibration standards preparation and tracking logbooks for:

_____ a. Inorganics

_____ b. Pesticides

_____ 38. Logbook numbers and series of stock solutions and reagents are recorded.

_____ 39. For commercially prepared standard mixes, appropriate documentation is available (Manufacturer's Certificate of Analysis).

_____ 40. If the laboratory uses automatic pipets for preparing standards, they are routinely calibrated.

VII. Analytical Instrumentation and Analyses-Specific Items

List the following for each instrument:

Type	ID Number	Manufacturer/Model	Software

_____ 41. The manufacturer's operating manuals are readily available.

_____ 42. The laboratory has service contracts for each instrument.

_____ 43. SOPs for analyses and logbook completion are readily available.

_____ 44. SOPs for analyses and logbook completion are followed by laboratory personnel.

_____ 45. Stock standards are current.

_____ 46. Calibration standards are made from a ready-made stock standard.
Manufacturer: _____

47. Calibration standards are prepared at least monthly.

48. If any instrument has been modified, list which one and how:

49. Explain how calibration intensity and gains are kept:

50. A mass flow controller is used.

51. Interference correction is done automatically and interelement correction factors are determined on at least an annual basis.

    Re-analyses are performed when internal standards are out of control.

52. A permanent service record is maintained for each instrument.

53. There is evidence of a secondary review of all documents and logbooks by someone other than the person generating the documents.

54. For AA Spectrometer, element-specific SOPs which list instrument conditions, background corrections, and required instrument sensitivity are readily available and followed.

55. An autosampler is used.

56. List EPA or instrument manufacturer's matrix modifiers used:

    As
    Pb
    Se
    Tl

57. For Mercury Analyzer - Cold Vapor AAs, an absorbance record is kept to monitor sensitivity.

58. For Cyanide Distillation Apparatus, there is a stock cyanide standard from a commercial source. If no, the standard is made from KCN salt. The standard is titrated.

59. For Cyanide Distillation Apparatus, the titrimetric manual or semi-automated colorimetric method is used. Method: _____

60. For Cyanide Distillation Apparatus, the pH of the samples is recorded and available for review.

61. For Cyanide Distillation Apparatus, samples are checked for the presence of sulfide and chlorine.

VIII. Data Handling and Review (GALP)

_____ 62. Data calculations are spot checked by a second person.

_____ 63. Records indicate appropriate corrective action when QC criteria are not met.

_____ 64. Supervisory personnel review the data and QC results prior to submission.

_____ 65. SOPs for data handling/review are readily available.

_____ 66. SOPs for data handling/review are followed by laboratory personnel.

_____ 67. Deliverables are checked for completeness and accuracy (hardcopy and electronic), including resubmittals.

_____ 68. The monthly data entry error rate is determined and recorded.

_____ 69. When changes to deliverables are required, the changes are properly documented (rationale, review, initials, etc.).

_____ 70. User manuals and operations/systems manuals are available.

_____ 71. A written software test and acceptance plan is available for installation of system changes.

IX. Quality Assurance Internal Inspections

_____ 72. There is an internal QA inspection procedure.

_____ 73. The QA Officer reports to senior management.

_____ 74. Corrective actions are documented.

_____ 75. Internal audits are performed.

List the types of internal audits

_____ _____

_____ _____

_____ _____

76. The following QA records are kept:

_____ a. PE sample results

_____ b. Records of recoveries (extractions, etc.)

_____ c. Training/experience records of personnel

_____ d. Method sensitivities

_____ e. Control charts for QC purposes

_____ f. Other _____

_____ 77. A Quality Assurance Manual (or equivalent) is available.

78. The Quality Manual addresses the following:

_____ a. Organization and philosophy
_____ b. Facilities and equipment
_____ c. Document control
_____ d. Analytical methodology
_____ e. Data generation
_____ f. QA
_____ g. QC
_____ h. Corporate ethics policy

X. Standard Operating Procedures

79. In addition to the previously mentioned SOPs, the following SOPs are also available:

_____ a. Calibration (balance)
_____ b. Calibration (instruments)
_____ c. Maintenance activities (for each system)
_____ d. Data reduction procedures
_____ e. Documentation policy/procedures
_____ f. Data validation/self-inspection procedures

XI. Organization and Personnel Summary

_____ 80. Personnel assigned to this project have the appropriate educational background to accomplish the objectives of the program (see Key Personnel list below).

_____ 81. The laboratory is adequately staffed to meet project commitments in a timely manner.

_____ 82. All key personnel were present and available for the audit.

XII. Laboratory Capacity

83. The laboratory has sufficient analytical instrumentation to analyze the needed number of samples.

_____

_____ 84. The laboratory has sufficient technical and administrative personnel to deliver the number of needed analyses.

_____ 85. The laboratory has an adequate sample and data tracking system to handle the needed number of analyses.

XIII. Key Personnel List (not previously identified)

Inorganics Supervisor

Name: _____

Generally requires a BS or BA in science and 1 year related experience, including 1 year supervisory experience.

ICP/ICP-MS Operator

Name: _____

Generally requires a BS or BA in science and 1 year ICP experience. Three additional years experience may substitute for education requirement.

Lachat Operator

Name: _____

Generally requires a BS or BA in science and 1 year Lachat experience. Three additional years experience may substitute for education requirement.

AA/Mercury Operator

Name: _____

Generally requires a BS or BA in science and 1 year experience for each of the following AA techniques: flame, graphite furnace, and cold vapor. Three additional years experience may substitute for education requirement.

Inorganic Sample Preparation Specialist

Name: _____

Generally requires high school diploma and college level course in general chemistry + 1 year experience in extraction/concentration. An additional 6 months experience with microwave digestion is required if microwave technique is used.

Wet Chemistry Analyst

Name: _____

Generally requires a BS or BA in science and 1 year experience. Three additional years experience may substitute for education requirement.

COMMENTS AND NONCONFORMITIES

Section E - Inorganics Technical Procedures Evaluation Checklist

Instructions: Use this sheet to document comments or nonconformities. For each, identify the appropriate item number from the checklist. Identify comments with a "C" and nonconformities with an "X." If additional space is needed, make copies of this page (or use additional blank sheets).

Item No.	***C or X***	***Comments and/or Nonconformities***

COMMENTS AND NONCONFORMITIES (Cont.)

Section E - Inorganics Technical Procedures Evaluation Checklist

Item No.	***C or X***	***Comments and/or Nonconformities***

LABORATORY AUDIT CHECKLIST

Section F - Asbestos Technical Procedures Evaluation Checklist

Instructions: Place an "X" beside each checklist item that represents a nonconformity. Place a "C" beside each item on which you are commenting for other reasons. Record the item number and written nonconformity explanation and/or comment on the comment sheet(s) at the end of this section. Write "N/A" beside any item that is not applicable to the current audit. Write "OK" beside all other items you observed or verified as compliant.

Personnel Interviewed:

Name	Title/Department

I. Sample Receipt

_____ 1. The laboratory has a designated sample custodian and alternate for each shift.

Names:

Sample Custodians	Alternates

_____ 2. SOPs for sample receipt are in place and readily available.

_____ 3. SOPs for sample receipt are followed by laboratory personnel.

_____ 4. The sample receipt area is secured against non-authorized personnel.

5. The sample custodian verifies the following:

_____ a. Condition of shipping package

_____ b. Presence or absence of custody seals

_____ c. Condition of custody seals, when present

_____ d. Custody seal numbers, when present

_____ e. Presence or absence of COC records

_____ f. Presence or absence of airbill stickers

_____ g. Airbill or airbill sticker number

_____ h. Presence or absence of sample tags/labels

_____ i. Sample tag/labels numbers

_____ j. Condition of sample containers

k. Discrepancies in any information recorded on COC records, client requests, airbills, sample containers, etc.

l. Documentation of hand deliveries

m. Problems encountered

Obtain examples of all forms used during sample receipt.

II. Sample Identification

6. The laboratory has a unique sample identification system (i.e., vs. using client sample ID numbers).

a. The number is assigned upon receipt. If no, when? _____

b. If yes, the numbers are cross referenced to client numbers in a log.

7. The system applies to all asbestos samples, including QC, PE and round robin samples.

8. SOPs are readily available for sample identification.

9. SOPs for sample identification are followed by laboratory personnel.

Obtain example of laboratory's sample ID system (e.g., example sample number with cross reference).

III. Sample Storage and Tracking

10. Sample storage areas are secured and access to samples is available only to authorized personnel.

11. Samples are logged in/out of storage area(s) when accessed.

12. Samples are tracked throughout analytical process (e.g., a traveler sheet).

13. SOPs for storage and tracking of samples are readily available.

14. SOPs for storage and tracking are followed by laboratory personnel.

Obtain examples of all forms/documents used for storage and tracking records.

IV. Sample Receipt and Storage Area

15. Sample shipping packages are opened in a contamination-free area (e.g., fume hood or vented area).

16. Adequate facilities are provided for the storage of samples and unused samples for 6 months after data submission.

V. Sample Preparation Area

17. The laboratory is maintained in a clean and organized manner appropriate for trace level analyses (contamination free).

18. The laboratory appears to have adequate work space (6 linear feet of unencumbered bench top per analyst).
19. The laboratory benches are made of suitable chemically resistant materials.
20. Sufficient functional HEPA-filtered hoods are available.
21. Analytical balances are located away from drafts and areas subject to rapid temperature changes.
    a. Balances are checked routinely (e.g., before each weighing session) with the appropriate range of weights and the results are recorded in a permanent logbook.
    b. Routine weights are checked against Class S (or equivalent) weights at least once a month and the results are recorded in a permanent logbook.
    c. Balances have been calibrated within one year by a certified technician.
    d. Data generated from balances are electronically transferred or manually entered into LIMS.
22. Sample preparation SOPs are readily available.
23. Sample preparation SOPs are followed by laboratory personnel.
24. SOPs for sample preparation area decontamination are readily available.
25. SOPs for sample preparation area decontamination are followed by laboratory personnel.
26. Analysts record bench data in a neat and accurate manner.
27. Analysts record ID numbers of equipment and materials used (e.g., microscope, RI liquids, etc.) on bench sheets.
28. There is evidence of a secondary review of all documents and logbooks by someone other than the person generating the documents.
29. Air flow through the HEPA-filtered hoods is checked on a routine basis and results are documented in a logbook.
30. RI liquids are calibrated on a routine basis and results are documented in a logbook.
31. The HEPA-filtered hoods used for sample preparation are checked for contamination each time samples are prepared and results are documented in a logbook,
32. Corrective action procedures are in place if contamination is detected.

VI. Analytical Instrumentation and Analyses-Specific Items

List the following for all instrumentation/equipment used:

Type	ID Number	Manufacturer/Model	Software

33. The manufacturer's operating manuals are readily available.
34. The laboratory has service contracts for each instrument.
35. The laboratory has extensive replacement parts available.
36. A permanent service record is maintained for each instrument.
37. SOPs for analyses and logbook completion are readily available.
38. SOPs for analyses and logbook completion are followed by laboratory personnel.
39. There is evidence of a secondary review of all documents and logbooks by someone other than the person generating the documents.

VII. Data Handling and Review (GALP)

40. Data calculations are spot checked by a second person.
41. Records indicate appropriate corrective action when QC criteria are not met.
42. Supervisory personnel review the data and QC results prior to submission.
43. SOPs for data handling/review are readily available.
44. SOPs for data handling/review are followed by laboratory personnel.
45. Deliverables are checked for completeness and accuracy (hardcopy and electronic), including resubmittals.
46. The monthly data entry error rate is determined and recorded.
47. When changes to deliverables are required, the changes are properly documented (rationale, review, initials, etc.).
48. User manuals and operations/systems manuals are available.
49. A written software test and acceptance plan is available for installation of system changes.

VIII. Quality Assurance Internal Inspections

50. There is an internal QA inspection procedure.
51. The QA Officer reports to senior management.

_____ 52. Corrective actions are documented.

_____ 53. Internal audits are performed.

List the types of internal audits

_____ _____

_____ _____

_____ _____

54. The following QA records are kept:

_____ a. PE sample results
_____ b. Round robin/interlab results
_____ c. Reference standards results
_____ d. Training/experience records of personnel
_____ e. Method sensitivities
_____ f. Control charts for QC purposes
_____ g. Other _____

_____ 55. A Quality Assurance Manual (or equivalent) is available.

56. The Quality Manual addresses the following:

_____ a. Organization and philosophy
_____ b. Facilities and equipment
_____ c. Document control
_____ d. Analytical methodology
_____ e. Data generation
_____ f. QA
_____ g. QC
_____ h. Corporate ethics policy

IX. Standard Operating Procedures

57. In addition to the previously mentioned SOPs, the following SOPs are also available:

_____ a. Calibration (balance)
_____ b. Calibration (instruments)
_____ c. Maintenance activities (for each system)
_____ d. Data reduction procedures
_____ e. Documentation policy/procedures
_____ f. Data validation/self-inspection procedures

X. Organization and Personnel Summary

58. Personnel assigned to this project have the appropriate educational background to accomplish the objectives of the program (see Key Personnel list below). _____

59. The laboratory is adequately staffed to meet project commitments in a timely manner. _____

60. All key personnel were present and available for the audit. _____

XI. Laboratory Capacity

61. The laboratory has sufficient analytical instrumentation to analyze the needed number of samples. _____

62. The laboratory has sufficient technical and administrative personnel to deliver the number of needed analyses. _____

63. The laboratory has an adequate sample and data tracking system to handle the needed number of analyses. _____

XII. Key Personnel List (not previously identified)

TEM Supervisor

Name: _____

Generally requires a BA or BS in science + 3 years TEM analysis experience. Requires knowledge of statistics and extensive familiarity with the silicate minerals, as well as the following methods: ISO 10312, AHERA, EPA 100.2, and ASTM D5755.

PLM Supervisor

Name: _____

Generally requires a BA or BS in science + 3 years PLM analysis experience. Requires knowledge of statistics and extensive training in optical mineralogy.

PCM Supervisor

Name: _____

Generally requires a BA or BS in science + 3 years PCM analysis experience.

TEM/PLM/PCM Analyst

Name: _____

Generally requires a BA or BS in science; however, three additional years laboratory experience may substitute for education requirement.

COMMENTS AND NONCONFORMITIES

Section F - Asbestos Technical Procedures Evaluation Checklist

Instructions: Use this sheet to document comments or nonconformities. For each, identify the appropriate item number from the checklist. Identify comments with a "C" and nonconformities with an "X." If additional space is needed, make copies of this page (or use additional blank sheets).

Item No.	***C or X***	***Comments and/or Nonconformities***

COMMENTS AND NONCONFORMITIES (Cont.)

Section F - Asbestos Technical Procedures Evaluation Checklist

Item No.	***C or X***	***Comments and/or Nonconformities***

RECORD OF CHANGES/REVIEW		
Rev. Number	**Revision/ Review Date**	**Document Name (If other than entire document, list sections)**
02-06-07	4/22/2016	Revised to apply to TechLaw Holdings and Subsidiary Companies.
02-06-08	2/3/2017	Minor changes to LAT Members and Appendix D

16-DAT-01.00
Data Management for Field Operations and Analytical Support

Technical Approval: Mark McDaniel

PRINT NAME

SIGNATURE **Date:** 3/12/14

QA Management Approval: Nikki MacDonald

PRINT NAME

SIGNATURE **Date:** 03/12/14

Table of Contents

1.0 SOP Description

The purpose of this standard operating procedure (SOP) is to provide a consistent format for all Region 8 Environmental Services Assistance Team (ESAT) data management personnel who perform uploads to Scribe and management of associated databases and reports.

This SOP is applicable to all ESAT personnel who prepare, process, review, and load analytical data into the Scribe database for the Field Operations Group and the Analytical Support and Data Validation Group.

2.0 Abbreviations and Acronyms

EDD	Electronic Data Deliverable
ERT	Environmental Response Team
ESAT	Environmental Services and Assistance Team
LIMS	Laboratory Information Management System
SOP	Standard Operating Procedure
TDF	Technical Direction Form
TO	Task Order
USEPA	United States Environmental Protection Agency

3.0 Health and Safety

All office-related safety precautions must be followed. Consideration is given to ergonomics for staff members using a keyboard and sitting in front of a computer terminal for extended periods of time and all other work conditions where ergonomics may be an issue.

4.0 Equipment and Supplies

Standard office supplies are required for this SOP, such as a personal computer and central filing system. Specific equipment and supplies are listed below:

- Internet connection and access to the ESAT network drive
- Access to Scribe and the Scribe databases associated with Technical Direction Forms (TDFs) issued by the client
- Login capabilities to the Scribe.NET website
- Microsoft Office software applications
- External hard drive containing the appropriate databases for upload

5.0 General Procedures

ESAT personnel are responsible for acquiring, compiling, reviewing, and loading analytical data into the appropriate Scribe database associated with a specific TDF. Electronic Data Deliverables (EDDs) are posted to the network drive from the Laboratory Information Management System (LIMS) on or before the EDD due date.

5.1 Review, Obtain, and Prepare EDDs to be uploaded to Scribe

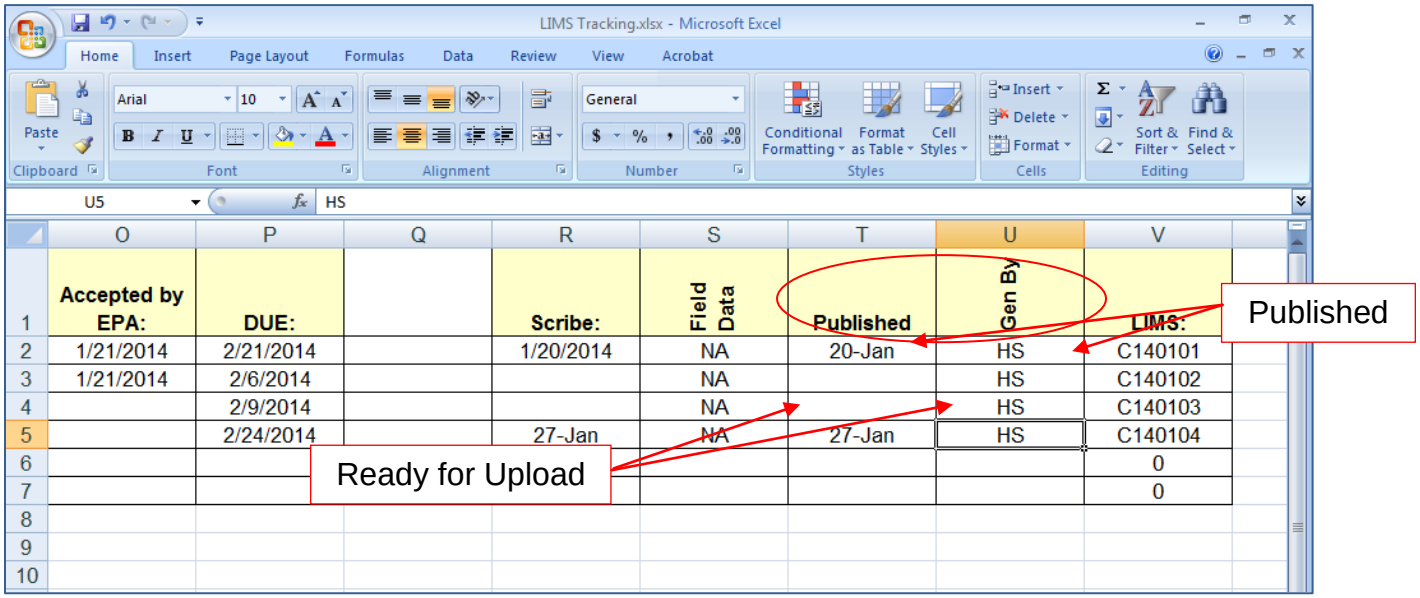
The analysts and/or Data Package Coordinator posts EDDs to the appropriate Task Order (TO) and project folder on the network drive upon completion. Prior to uploading or publishing a project to Scribe, review the LIMS Tracking spreadsheet to ensure that the

Data Package Coordinator has completed assembly and finalization of the current Sample Event(s) (Figure 5.2). EDDs that are complete and ready for upload are listed in the LIMS Tracking spreadsheet (located on the network drive), and will be signed off in the “Gen By” (generated by) column (Figure 5.1). EDDs that have been published will contain a date in the “Published” column (Figure 5.1).

Note: TO Numbers and TDFs are not permanent, and are subject to change based on the contract year, as well as the type and number of sample events.

- Review the "Gen By" and "Published" columns. If the Data Package Coordinator's initials are listed in the "Gen by" column, but a date is not included in the "Published" column, that sampling event is ready for uploading to the database.

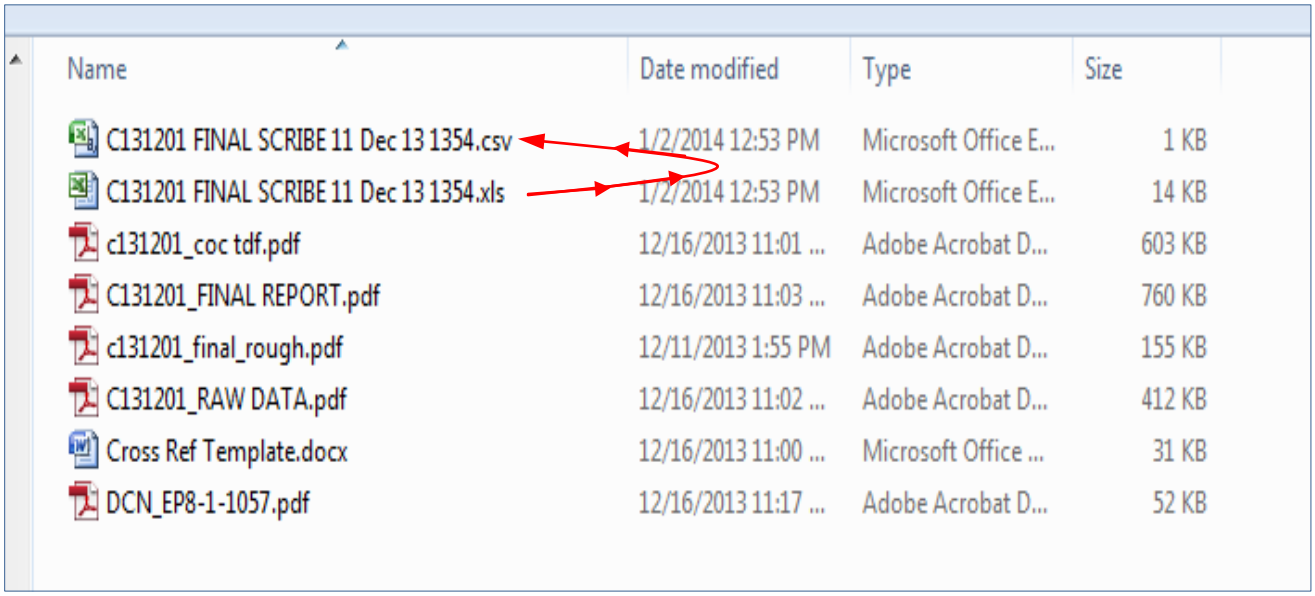
Figure 5.1 LIMS Tracking Spreadsheet Example



	O	P	Q	R	S	T	U	V
1	Accepted by EPA:	DUE:		Scribe:	Field Data	Published	Gen By	LIMS:
2	1/21/2014	2/21/2014		1/20/2014	NA	20-Jan	HS	C140101
3	1/21/2014	2/6/2014			NA		HS	C140102
4		2/9/2014			NA		HS	C140103
5		2/24/2014		27-Jan	NA	27-Jan	HS	C140104
6								0
7								0
8								
9								
10								

- Navigate to the TO and Projects to be uploaded
- Select the .xlsx EDD files, and within the same folder, convert them to .csv format for uploading to Scribe
- Repeat with all other EDDs noted from the LIMS Tracking spreadsheet

Figure 5.2 Example of Sample Event Folder from the Network Drive

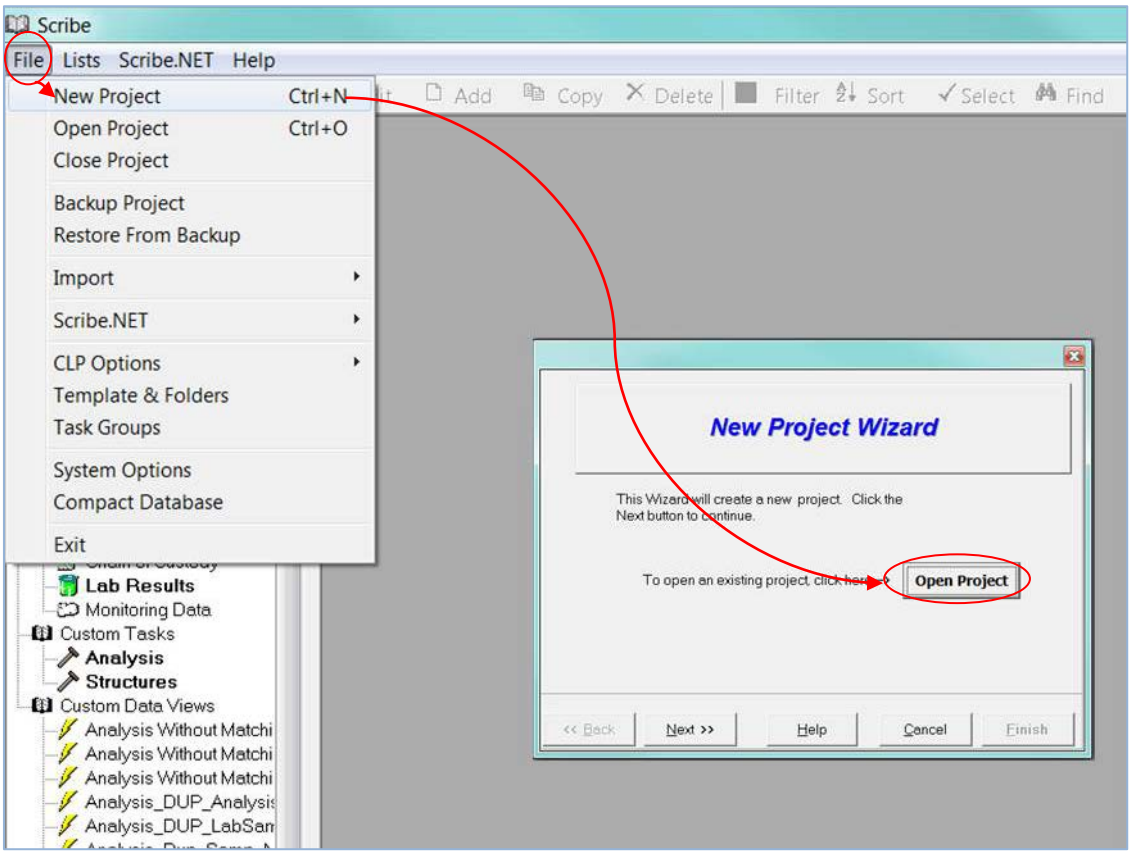


5.2 Scribe Data Load

Each TO project/TDF has its own database. Several separate sample events may occur under each project and will be managed according to the associated TO and project.

- Access the external hard drive containing the appropriate TO databases
- Open the Scribe Database Program
- Select "File" from the menu then "New Project"
- Select "Open Project" in the "New Project Wizard" dialogue box (this will open a Windows Explorer window from which to choose the appropriate folder pathway and database file)

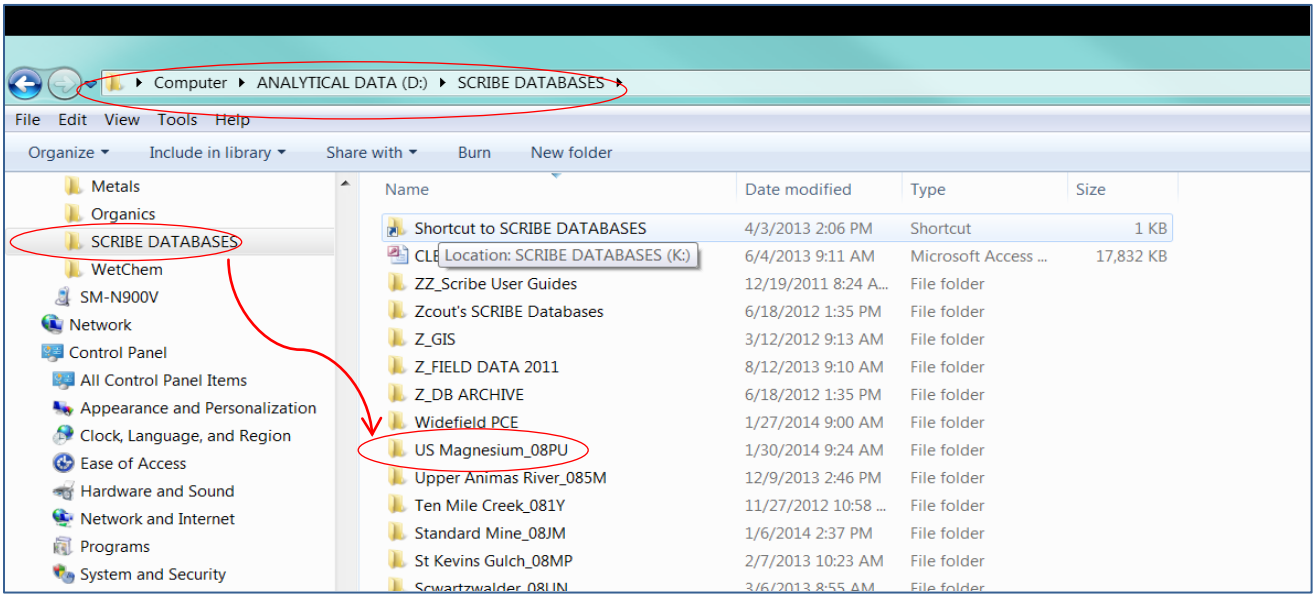
Figure 5.3 Scribe Database "Open Project"



- Navigate to the current TO and Project to be uploaded

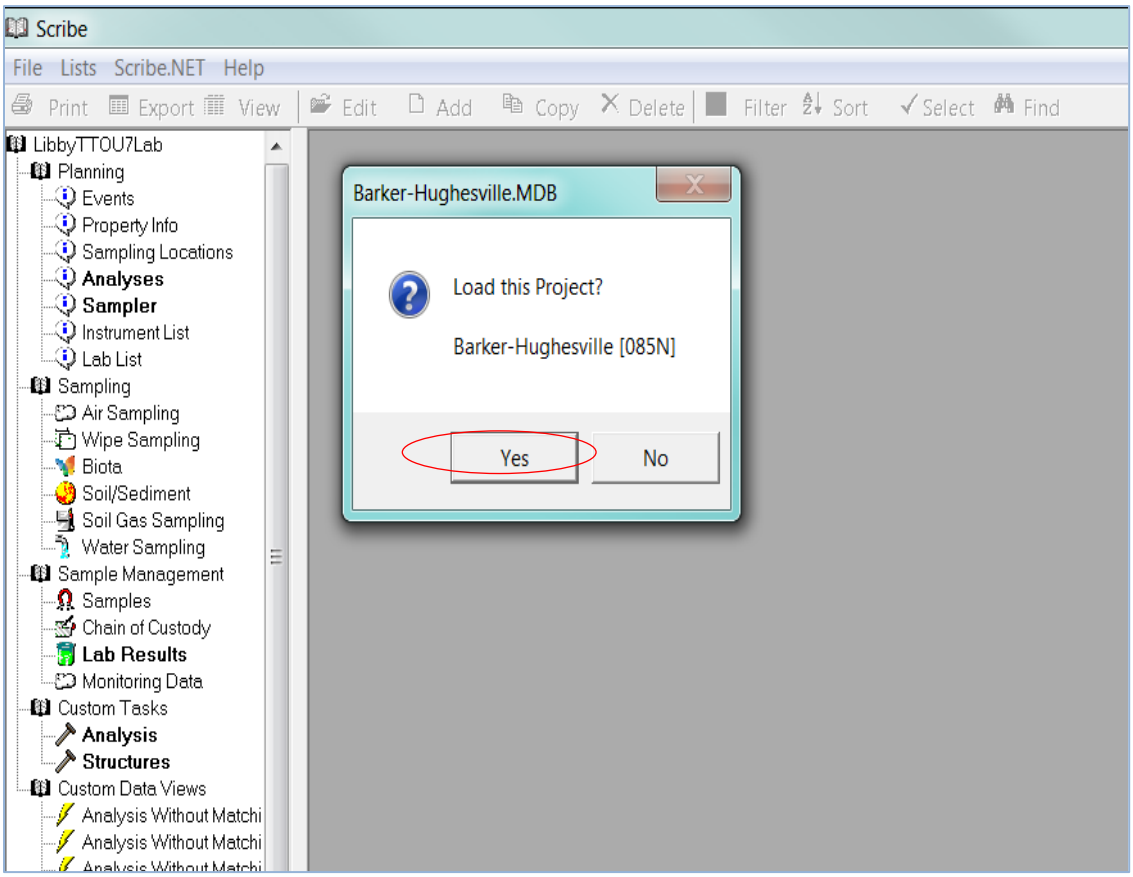
  Note: Databases for the current TO are stored on an external hard drive as shown in Figure 5.4, but the external storage location, pathways, and TO numbers are subject to change.

Figure 5.4 Scribe Database Project Pathway



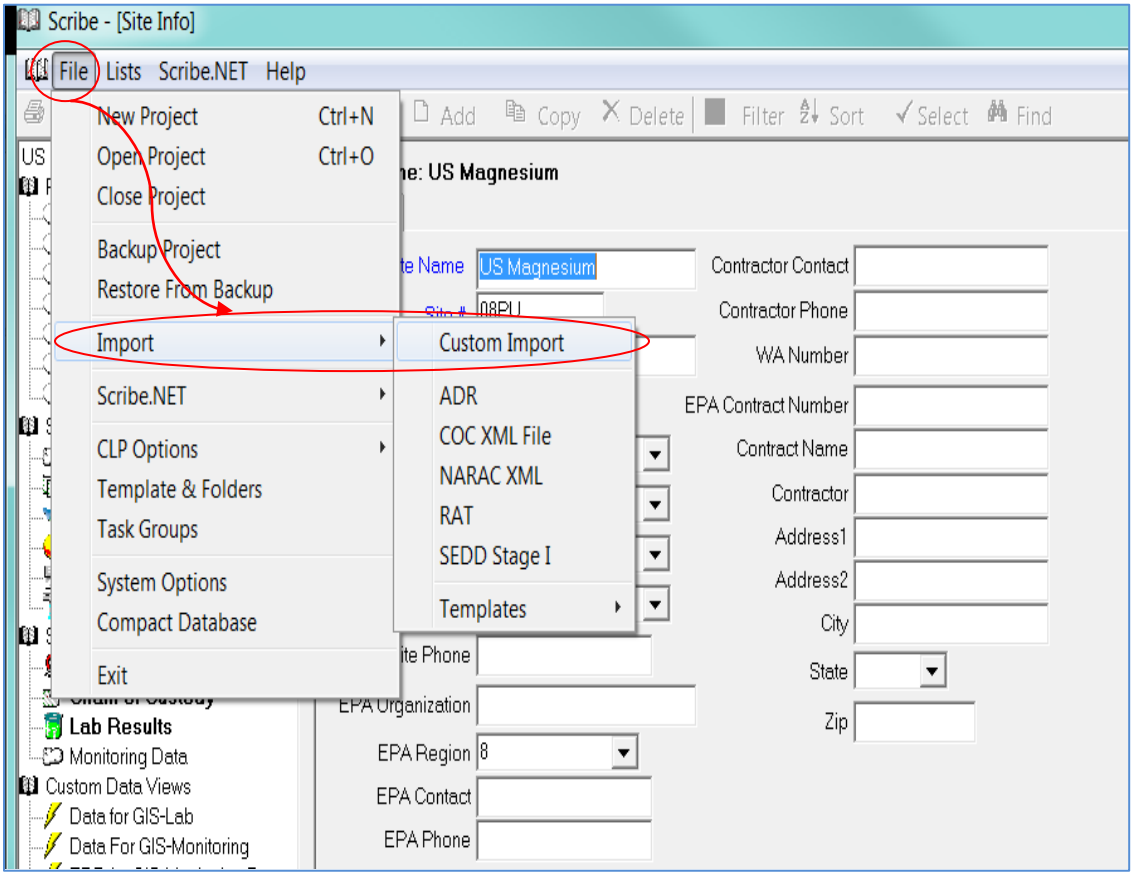
- Select "Yes" in the "Load this Project" dialogue box

Figure 5.5 Scribe Database Load Project



- Select "File -> Import -> Custom Import"
- Select "No" in the resulting "Backup Now?" dialogue box

Figure 5.6 Scribe "File/Import/Custom Import"

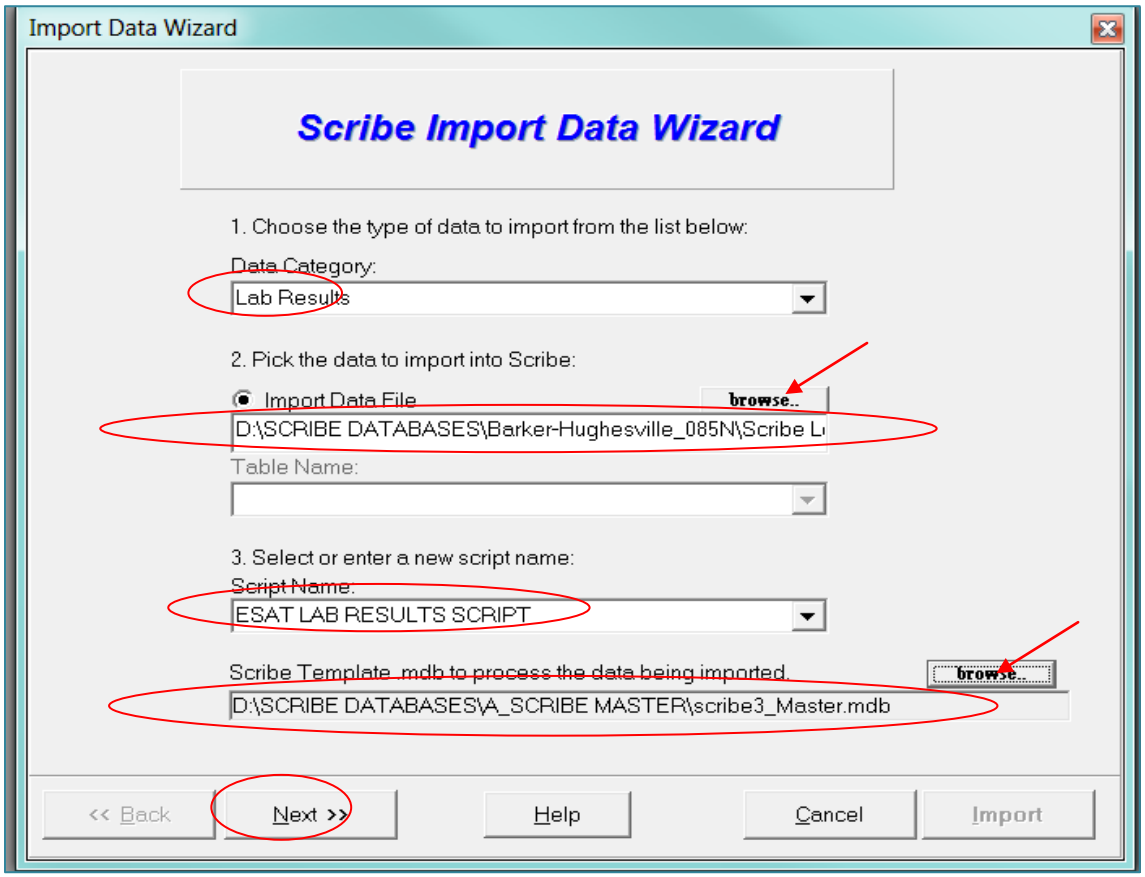


- Select "Lab Results" from the drop down arrow list in the Data Category field
- Select the correct EDD using the "browse" button above the Import Data File field

  Note: EDDs will be obtained from the folders located on the network drive pathways discussed in Section 5.1.

- Select "ESAT Lab Results Script" for the "Script Name" field
- Select the Master Scribe template using the "browse" button above the Scribe Template field; this template is located on the external drive in Scribe Databases/Master
- Select "Next"

Figure 5.7 Scribe "Import Data Wizard"

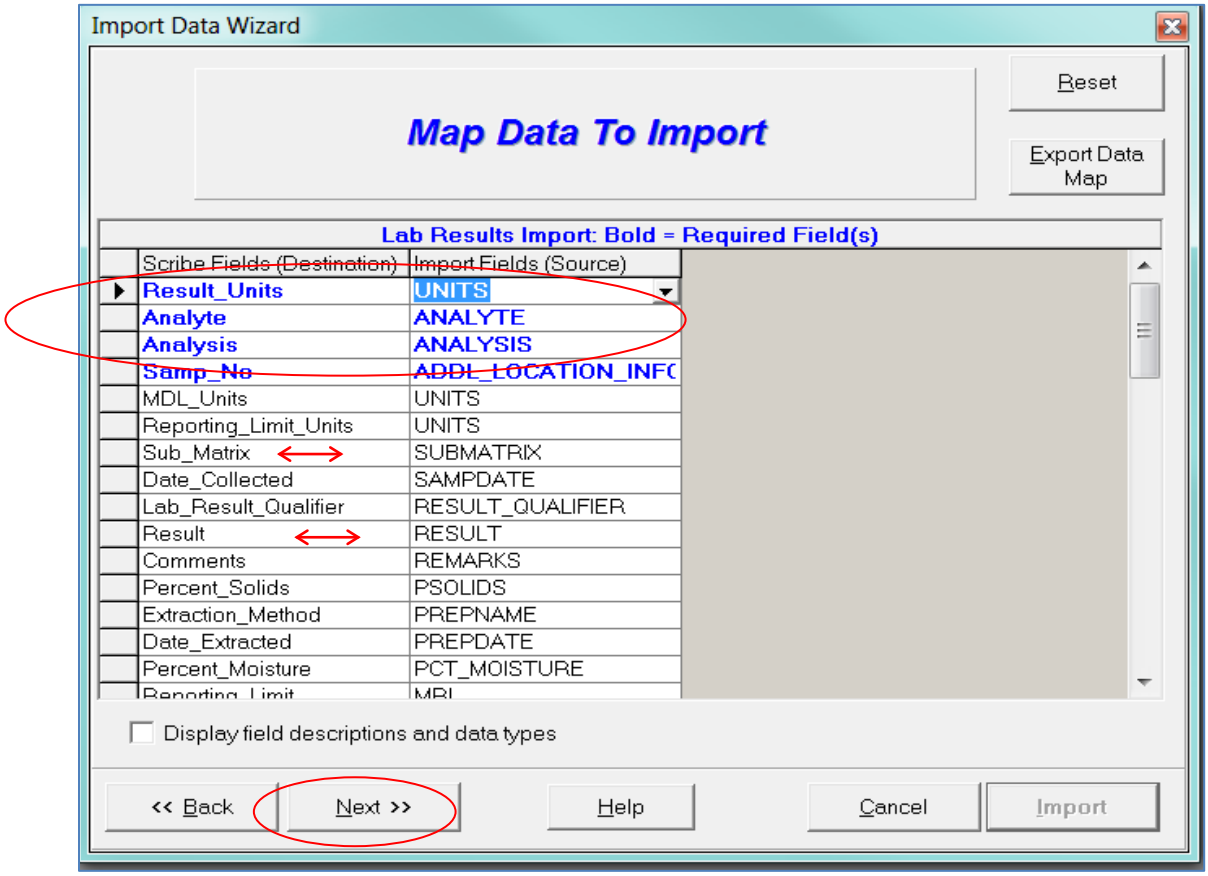


- Review the field mapping dialogue box to ensure that all fields match up with Scribe requirements

  Note: The fields in blue bolded text are required fields. The remaining fields (listed below the blue bolded font) may not match exactly in name. Ensure that they match in type and meaning regardless of the slight differences in names and case. In addition, not all "Import Fields" fields will be present for "Scribe Fields".

- Select "Next"

Figure 5.8 Scribe "Map Data to Import"

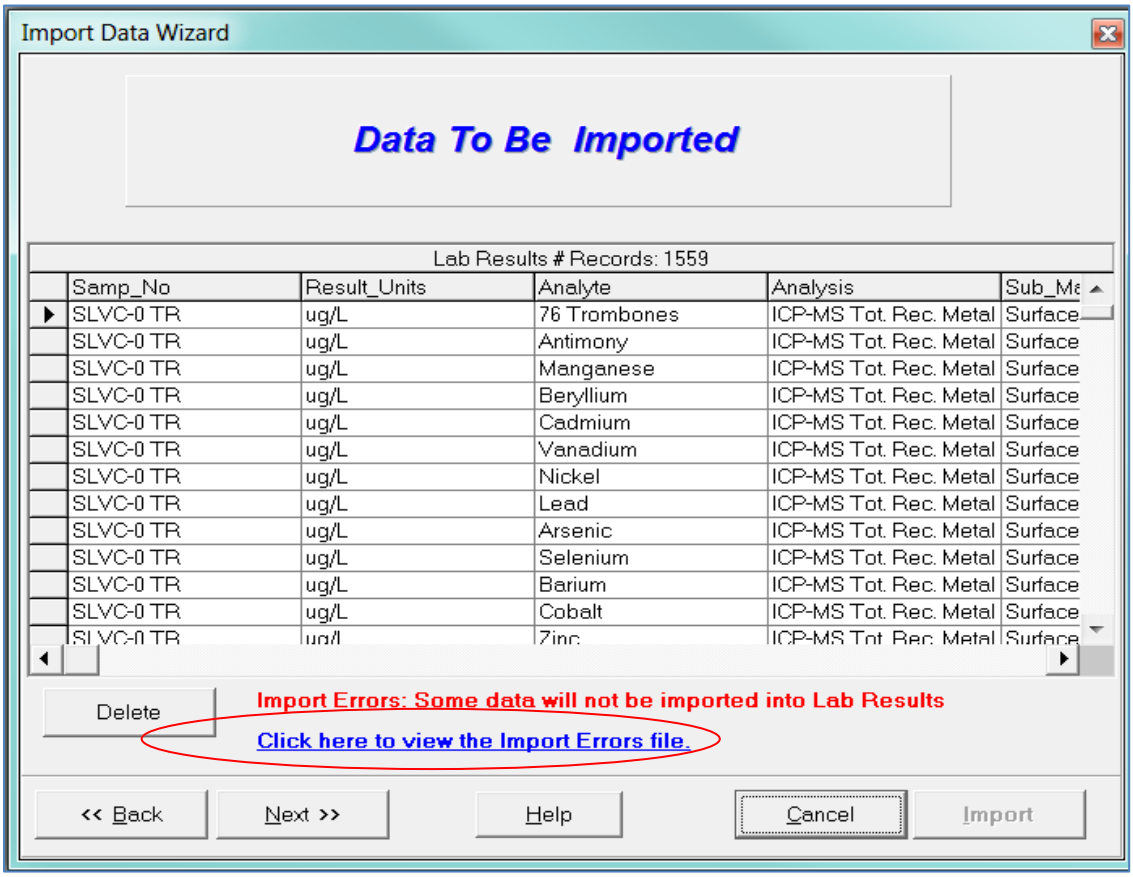


- Review the total in the "Lab Results # Records:" field
- Ensure that the total number of records in the "Lab Results # Records" field matches the total in the EDD

  Note: The Excel formatted EDD can be opened and reviewed to verify that the total number of samples to be imported matches the number of samples contained in the EDD.

- Select the blue bolded "Import Errors file" link to obtain a list of errors if the import is unsuccessful (list will open in Excel format)

Figure 5.9 Scribe "Data to be Imported" Import Errors

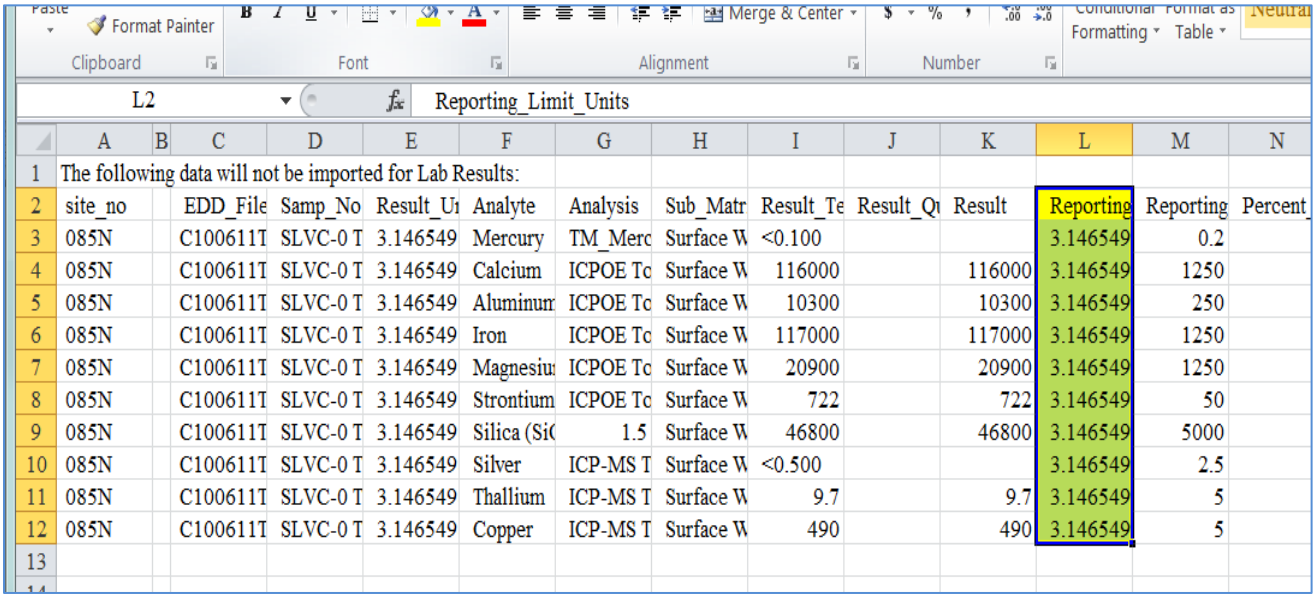


- Review the error report, open the appropriate EDD, and correct the errors. As shown in the example report below, the "Reporting Units" field has a numerical value, which is incorrect. Because this is a text field, the necessary correction would be to insert the appropriate unit text, which for this EDD would be ug/L.

  Note: The example below contains one of the common types of errors that may occur. That is, one that is correctable within the EDD itself and by the Scribe uploader. However, this is not the only possible error type. If a more complex error occurs, one that cannot be corrected within the EDD, an ESAT analyst may need to be contacted for assistance and the error may need to be corrected at an earlier point in the process.

  Errors within the Scribe program itself are essentially non-existent for the Analytical Support Group and Field Operations databases. If an upload error occurs, it is generally caused by incorrect selections of either the database script or the Scribe Master template. In those cases, return to the import screen and ensure that all scripts and templates selected are correct. If the database still shows errors that are not correctable in the EDD, the error(s) may be Scribe-related. If an error occurs that cannot be corrected within the EDD, or by correcting possible upload procedure errors, contact the Environmental Response Team (ERT) Software Support department for assistance. ERT can be contacted by email: ertsupport@epa.gov or by phone: 800-999-6990.

Figure 5.10 Scribe Example of Import Errors File



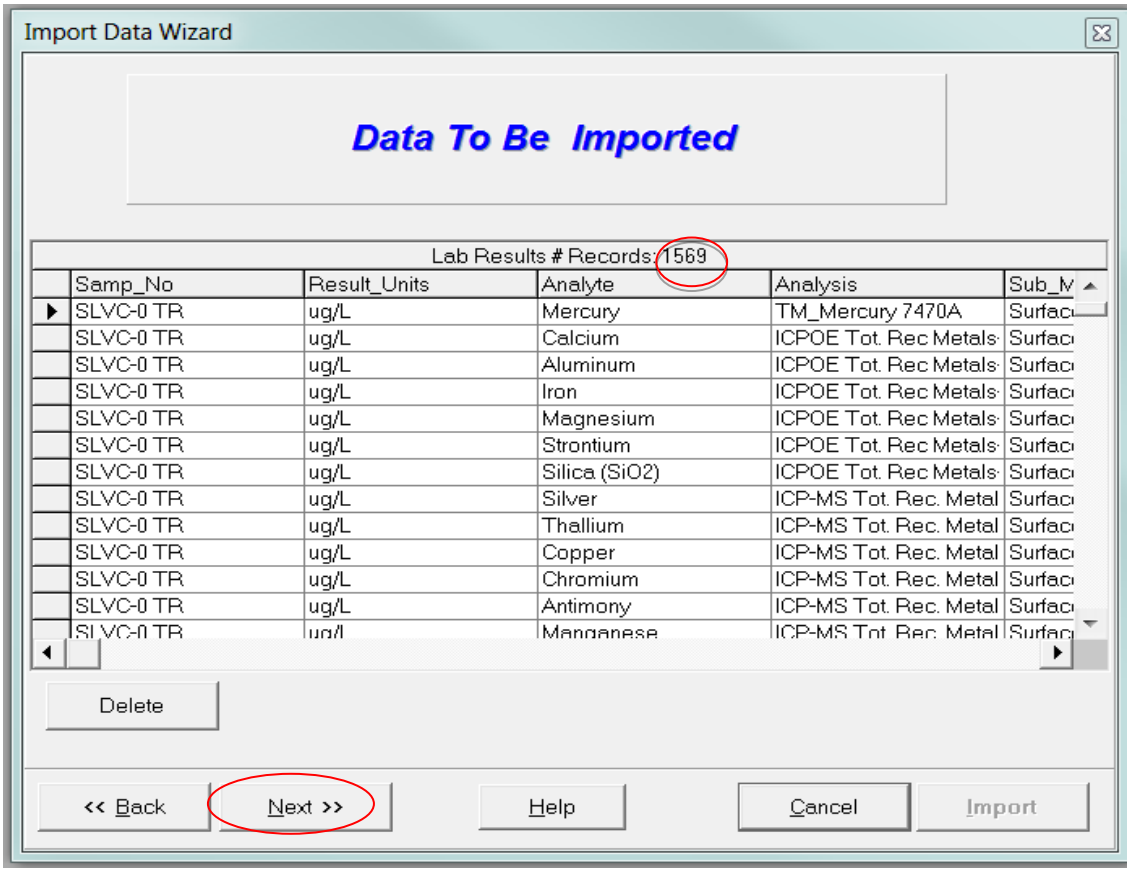
Format Painter | Clipboard | Font | Merge & Center | Alignment | Number | Formatting | Table

L2 | Reporting_Limit_Units

	A	B	C	D	E	F	G	H	I	J	K	L	M	N
1	The following data will not be imported for Lab Results:													
2	site_no		EDD_File	Samp_No	Result_Ui	Analyte	Analysis	Sub_Matr	Result_Te	Result_Qu	Result	Reporting	Reporting	Percent_
3	085N		C100611T	SLVC-0 T	3.146549	Mercury	TM_Merc	Surface W	<0.100			3.146549	0.2	
4	085N		C100611T	SLVC-0 T	3.146549	Calcium	ICPOE To	Surface W	116000		116000	3.146549	1250	
5	085N		C100611T	SLVC-0 T	3.146549	Aluminum	ICPOE To	Surface W	10300		10300	3.146549	250	
6	085N		C100611T	SLVC-0 T	3.146549	Iron	ICPOE To	Surface W	117000		117000	3.146549	1250	
7	085N		C100611T	SLVC-0 T	3.146549	Magnesiu	ICPOE To	Surface W	20900		20900	3.146549	1250	
8	085N		C100611T	SLVC-0 T	3.146549	Strontium	ICPOE To	Surface W	722		722	3.146549	50	
9	085N		C100611T	SLVC-0 T	3.146549	Silica (Si(	1.5	Surface W	46800		46800	3.146549	5000	
10	085N		C100611T	SLVC-0 T	3.146549	Silver	ICP-MS T	Surface W	<0.500			3.146549	2.5	
11	085N		C100611T	SLVC-0 T	3.146549	Thallium	ICP-MS T	Surface W	9.7		9.7	3.146549	5	
12	085N		C100611T	SLVC-0 T	3.146549	Copper	ICP-MS T	Surface W	490		490	3.146549	5	
13														

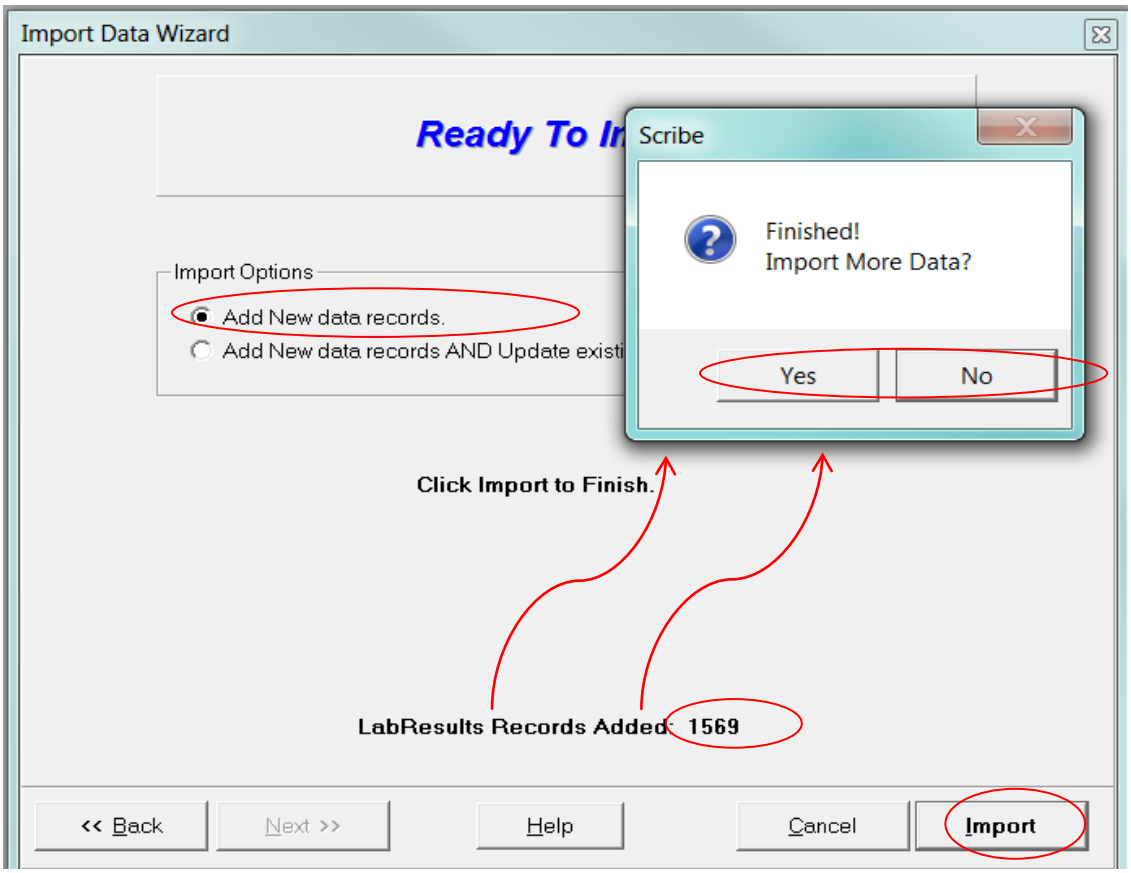
- Select "Next" once the errors have been corrected and continue the import process

Figure 5.11 Scribe Corrected "Data to be Imported"



- Select the "Add New Records" box
- Select "Import"
- Move the "Finished!" dialogue box away from the center portion of the "Import Data Wizard" dialogue box so that the "LabResults Records Added" total can be viewed
- Ensure that the "LabResults Records Added" is the same as the total in the "Data to be Imported" dialogue box (Figure 5.11)
- Select "Yes" if more EDDs for the same TO and Project will be loaded
- Select "No" if complete

Figure 5.12 Scribe "Import More Data?"



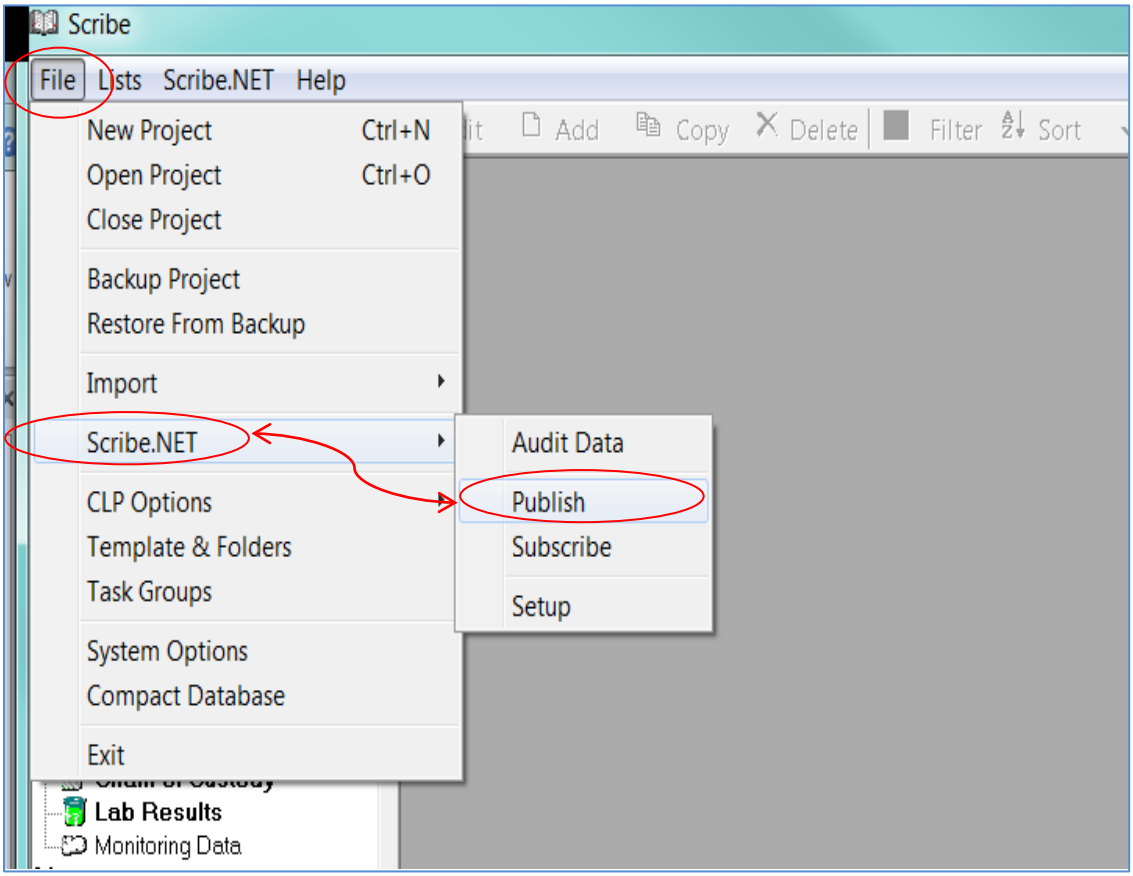
- Continue with all completed EDDs (those noted as ready to upload in the LIMS Tracking spreadsheet)

5.3 Publish Databases in Scribe

Once all EDD uploads for a specific project are loaded to Scribe, the project can be published to Scribe.NET.

- Navigate to the LIMS Tracking spreadsheet on the network drive as described in Section 5.1 and shown in Figure 5.1
- Open the LIMS Tracking spreadsheet
- Follow the steps for opening a specific database as shown in Section 5.2, Figure 5.3, Figure 5.4, and Figure 5.5
- Select "File ->Scribe.net->Publish"

Figure 5.13 Scribe "File/Scribe.NET/Publish"

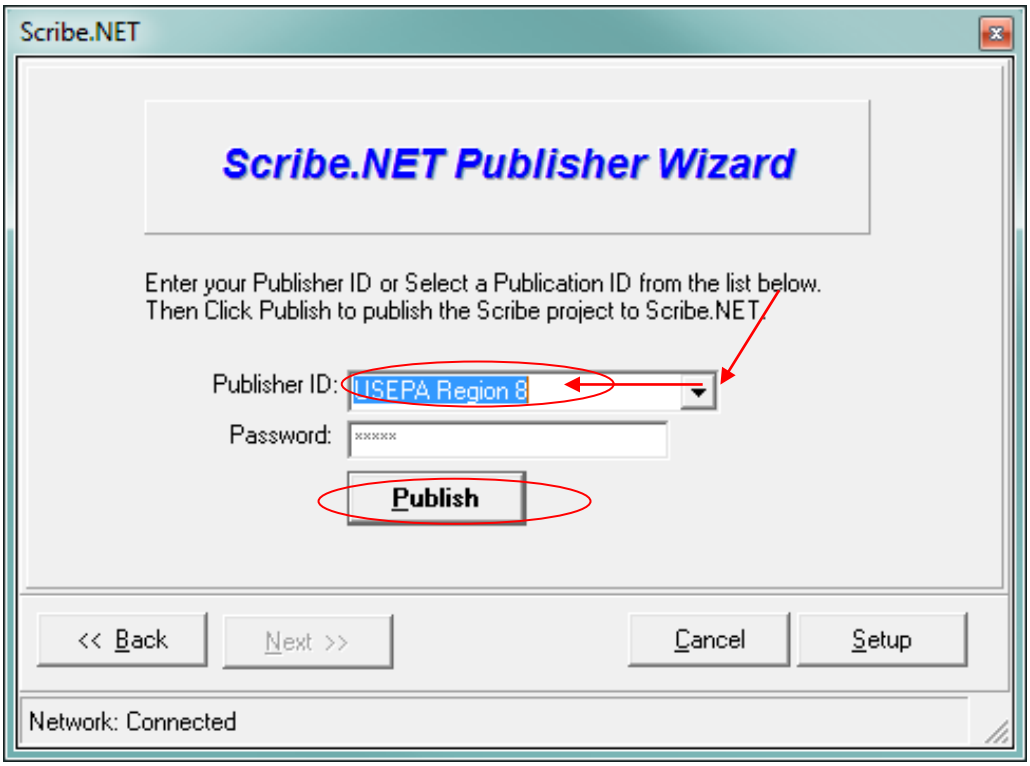


- Select "Next" on the Scribe.NET Publisher Wizard dialogue box

Figure 5.14 "Scribe.NET Publisher Wizard"

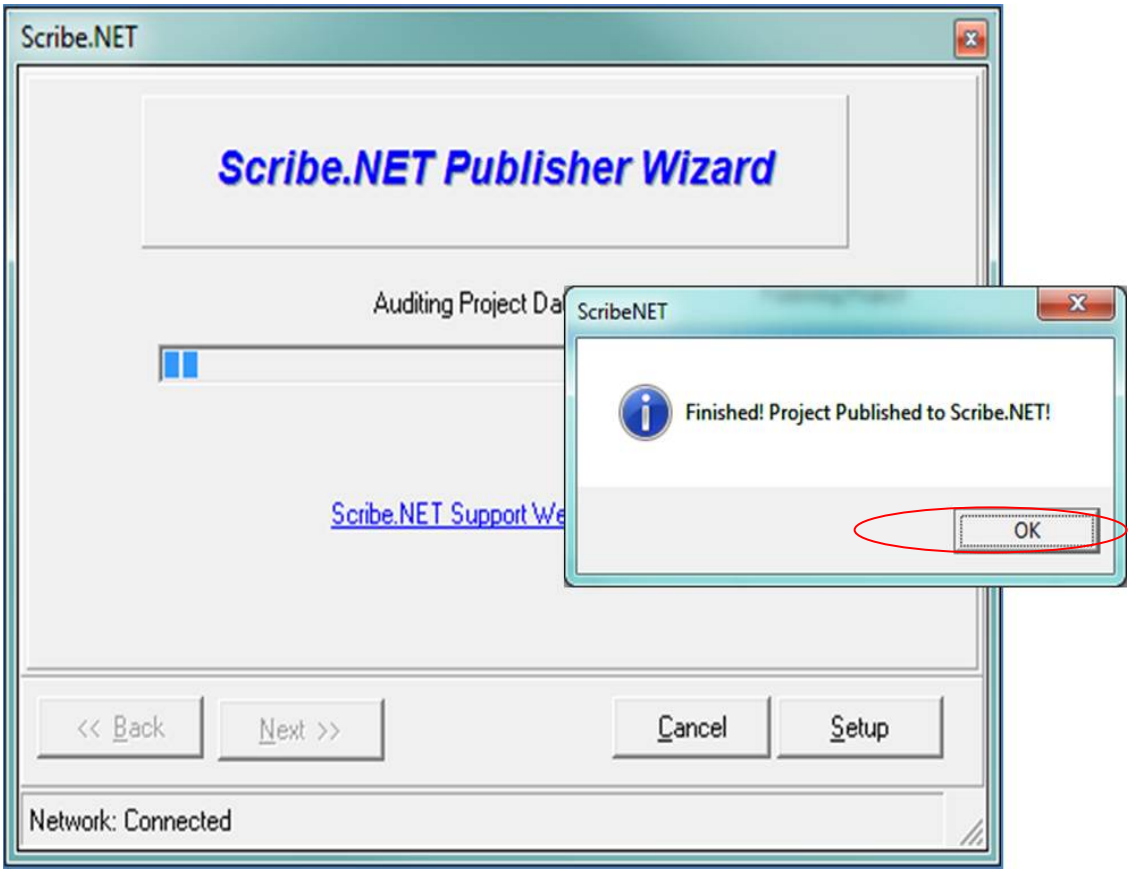
- Select "USEPA Region 8" from the dropdown list on the resulting dialogue box; leave the password field as is
- Select "Publish"

Figure 5.15 Scribe Publish Dropdown



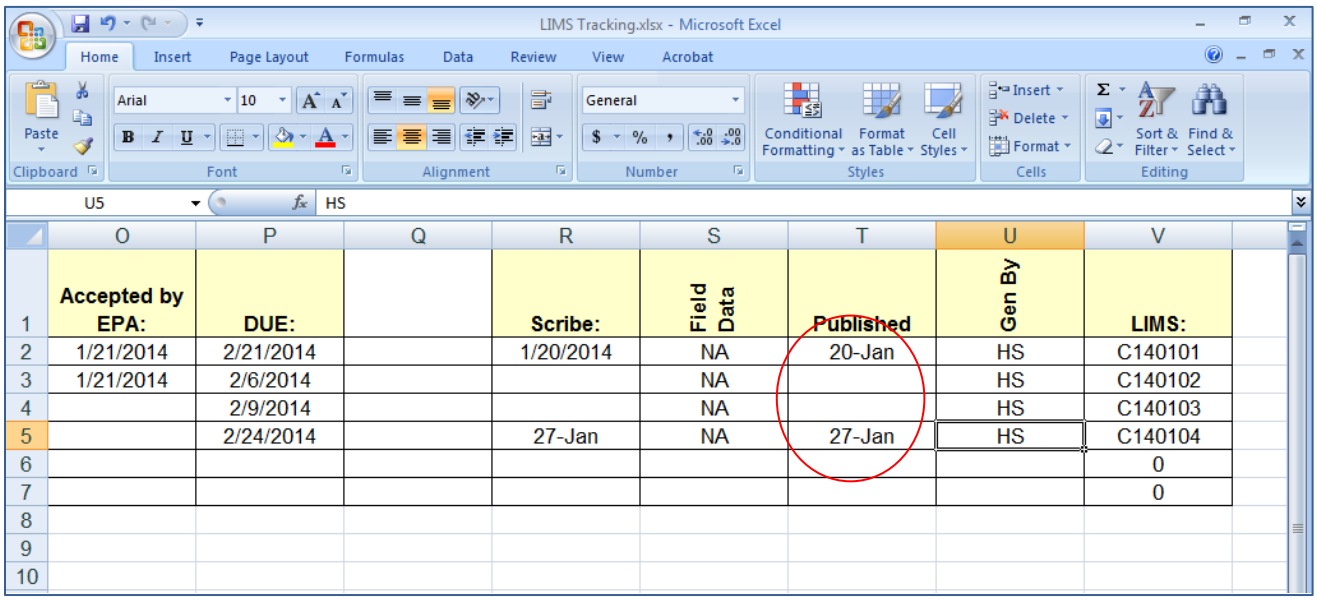
- Select "OK" in the "Finished! Project Published to Scribe.NET!" dialogue box

- **Figure 5.16 Scribe.NET "Project Published"**



- Move completed and published project folders to the correct TO subfolder titled "Final Folder" (located on the network drive, as described in Section 5.1)
- Open the LIMS Tracking spreadsheet and record the Publish date (date format defaults to spreadsheet formatting) for each Sample Event completed
- Continue with remaining TO Sample Events

Figure 5.17 LIMS Tracking Spreadsheet Input Published Date



	O	P	Q	R	S	T	U	V
1	Accepted by EPA:	DUE:		Scribe:	Field Data	Published	Gen By	LIMS:
2	1/21/2014	2/21/2014		1/20/2014	NA	20-Jan	HS	C140101
3	1/21/2014	2/6/2014			NA		HS	C140102
4		2/9/2014			NA		HS	C140103
5		2/24/2014		27-Jan	NA	27-Jan	HS	C140104
6								0
7								0
8								
9								
10								

6.0 Data Records and Management

The Data Package Coordinator will combine documents contained within each specific TO and Project folder and create and assemble the final Data Package for submittal to the client. Both the Excel and .csv versions of the EDD, as well as the data package and other associated documents, will be located in the appropriate TO Project file and Sample Event folder as shown in Figure 5.3.

7.0 Quality Control and Assurance

This SOP meets all the requirements of the ESAT Quality Management Plan.

8.0 References

ESAT Region 8, SOP,16-QAQ-03.00, Document Control, effective November 11, 2013.

ESAT Region 8, Quality Management Plan, version 7, effective June 2013.

TechLaw, Inc., Health and Safety Program Plan, effective November 2013.

TechLaw Inc., Corporate Quality Management Plan, effective November 2013.

United States Environmental Protection Agency, Environmental Response Team Software Support, accessed online at: http://www.ertsupport.org/scribe_home.htm. February 7, 2014.

United States Environmental Protection Agency Guidance for Preparing Standard Operation Procedures, EPA QA/G-6, April 2007.

Document Change History			
Revision No.	**Status[1] (I, R, C)**	**Effective Date**	**Changes Made**
0	I	03/12/14	Initial Document

[1] Status: I = Initial, R= Revision, or C = Cancelled

16-LAB-01.01

Laboratory Waste Management

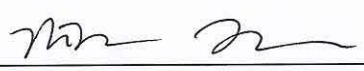
Technical Approval: Scott C Walker

PRINT NAME

SIGNATURE **Date:** 3/29/2016

QA Management Approval: Nikki Thomsen

PRINT NAME



SIGNATURE **Date:** 03/28/16

Table of Contents

1.0 SOP Description

This standard operating procedure (SOP) describes the procedures for safely collecting, storing, analyzing, and disposing of laboratory waste. This SOP is applicable to the following aqueous waste streams: acidic instrument waste, acidic reagents (except standards), sample digestates, and samples preserved for analysis with acid. This SOP is also applicable to solid wastes such as soils, vegetation and biota samples.

Aqueous samples, digestates, reagents, and some instrument wastes contain small amounts of mineral acids. The presence of these acids causes the pH of the waste to be below 2, and hence, be defined as hazardous. In addition, these wastes may contain metal concentrations which exceed discharge standards.

Solid wastes may contain metal concentrations which exceed disposal standards. This waste must be properly labeled, contained, and stored in accordance with all state and federal regulations. This SOP includes the initiation of satellite waste containers, documentation accompanying the waste, and procedures for placing the waste in the designated waste storage area.

2.0 Acronyms and Definitions

CHP	Chemical Hygiene Plan
EPA	United States Environmental Protection Agency
ESAT	Environmental Services Assistance Team
GHS	Globally Harmonized System for Classification and Labeling of Chemicals
HASP	Health and Safety Plan
HSO	Health and Safety Officer
HWTS	Hazardous Waste Tracking System
LIMS	Laboratory Information Management System
PPE	Personal Protective Equipment
SHEMP	Safety, Health and Environmental Management Program
Satellite Waste Container	A container used to collect waste during generation (see 40 CFR 261.31)
Secondary Containment	A second level of containment ensuring no release if the initial containment fails
SOP	Standard Operating Procedure
WCO	Waste Control Officer

3.0 Health and Safety

Follow general laboratory health and safety policies and regulations in the current versions of the Environmental Services Assistance Team (ESAT) Health and Safety Plan (HASP) and the United States Environmental Protection Agency (EPA) Region 8 Chemical Hygiene Plan (CHP) when handling wastes. The use of laboratory equipment and chemicals exposes the analyst to several potential hazards. Good laboratory practices should be followed at all times.

Solutions classified as aqueous corrosive wastes normally contain percentage levels of mineral acids and can contain certain inorganic elements known to be hazardous. Avoid contact with water or wastewater samples. Personal protective equipment (PPE), such as gloves, protective eye wear and laboratory coats, should be worn at all times when handling samples, reagents, or when in the vicinity of others handling these items.

Satellite waste containers must always be tightly capped when not in use. Satellite waste containers can weigh in excess of 50 pounds and should be lifted carefully.

When in doubt as to the proper procedure to follow, contact the EPA Health and Safety Officer (HSO) for guidance. Personnel should minimize exposure to potential health hazards through the use of engineering and administrative controls, work practice procedures, and proper PPE.

4.0 Personnel Qualifications

Personnel responsibilities for hazardous waste management at the EPA Region 8 laboratory are described in the Introduction/Executive Summary of the *Safety, Health and Environmental Management Program (SHEMP) Manual* (Section 2.2 Hazardous Waste Management).

Federal and state regulations require that employees who handle hazardous waste be provided with initial and annual training. Initial orientation and on-the-job training are provided to new ESAT employees within their first month of employment, and refresher training is provided on an annual basis thereafter. This training is designed to keep employees familiar with waste handling procedures in place at the Region 8 laboratory, along with applicable regulations. Training completion will be enforced by the supervisor and documented for each individual by the HSO.

5.0 Equipment and Supplies

The EPA Waste Control Officer (WCO) will ensure that a supply of appropriate waste containers and labels are available for use. The WCO is responsible for maintaining control of all documentation of waste disposal.

Waste containers must be able to be tightly capped, and both the container and the secondary containment must be chemically resistant to corrosive materials.

6.0 General Procedures

6.1 Satellite Waste Container Preparation

6.1.1 Labeling the Waste Container

The waste container will be properly labeled as appropriate. A waste container ID is assigned to the container by the person who initiates the container's designation. The container ID must be written on the container with a permanent marker in a location that is easily seen.

The format for the container ID is "YYMMDD-XXXX- #" where "XXXX" is the laboratory room number and "#" is the sequential number of the container generated on that particular day.

Affix an appropriate red and white hazardous label for containers used for suspected or known hazardous materials on the waste container. Affix the proper Globally Harmonized System for Classification and Labeling of Chemicals (GHS) label for hazard communication warning of the presence of corrosives (pH<2) when the contents to be added to the container are known to be acidic.

6.1.2 Waste Container Inventory Log

The waste container inventory log must be initiated and placed next to the

container. The hazardous waste container inventory log is either attached or located in the near vicinity of the waste container, and lists the accumulated waste maintained by the generator(s).

The waste inventory log must include the container ID number and its date, description and amount of waste added, date of the addition, and the name of the person making each addition.

Note: The waste inventory log serves two important purposes. It guards against addition of incompatible chemicals to the container mix, and it allows packers to determine the correct classification of the waste for transport and disposal.

6.2 Waste Collection and Analysis

6.2.1 Waste Collection

Waste must be collected as near as possible to the point of generation and have secondary containment.

Containers must be kept closed except when waste is being added. When a container of waste is approximately 85% full, the waste inventory sheet must be signed, dated and entered into the inventory system by the WCO.

Waste containers that are full or otherwise ready for disposal will be transported to the F wing where the wastes are segregated by waste categories.

Each unit will be labeled with the start of accumulation date and, as appropriate, a “hazardous waste” or “non-hazardous waste” label.

6.2.2 Waste Analysis

<u>Aqueous Waste</u>

Aqueous waste containers must be sub-sampled. The waste inventory sheet must be filled out and transported to the waste storage area within 24 hours and must be clearly labeled as “Awaiting Analysis.”

Analysis of each aqueous waste container must be completed within 10 days. The results are then entered into the waste inventory sheet.

<u>Solid Waste</u>

Solid wastes designated for disposal are collected in an approved container.

In lieu of analyzing the solids, the data collected during the analysis of the samples is used to indicate the level, if any, of metals concentration in the solid waste. In general, segregating the solids into Laboratory Information Management System (LIMS) Work Order-specific groups will make calculating the metals concentration less complicated.

A copy of the completed solid waste inventory sheet, along with the raw data, is provided to the WCO, and the original is attached to the waste container.

6.3 Waste Minimization

Metals laden waste volumes are minimized by the use of a dedicated waste receptacle in which no other laboratory waste is placed.

Waste concentrations are minimized by judicious use of metals standard solutions and materials. In addition, ESAT chemists work with field personnel to reduce the amount of excess sample collected.

7.0 Data and Records Management

Waste is tracked through the use of container labels, waste container logs, an in-house tracking system, physical inventories, hazardous waste shipping manifests, and certificates of disposal.

Data from waste container sheets for wastes generated in the laboratory is entered into the Hazardous Waste Tracking System (HWTS) by the WCO at the time of transport to the F-wing.

8.0 References

EPA Region 8, Chemical Hygiene Plan, current version

EPA Region 8, Health and Safety Plan, current version

EPA Region 8, Waste Management SOP, current version

Document Change History			
Revision No.	Status[1] (I, R, C)	Effective Date	Changes Made
0	I	01/17/07	Not applicable
1	R	03/29/16	Entire document reviewed and updated

[1] Status: I = Initial, R = Revision, or C = Cancelled

16-LAB-05.04

Sample Receipt, Custody, Storage, and LIMS Data Entry

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SIGNATURE **Date:** 10/14/15

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Table of Contents

1.0 SOP Description

The purpose of this standard operating procedure (SOP) is to establish a safe, traceable, and consistent laboratory process for receiving, tracking, and storage of Environmental Services Assistant Team (ESAT) samples at the United States Environmental Protection Agency (EPA) Region 8 Laboratory. These may include surface waters, ground waters, soils, sediments, biological materials, and proficiency testing (PT) samples.

This SOP specifies the requirements for project definition, sample receipt, control, and record keeping by ESAT. The following objectives are defined in detail within this document:

1.1 EPA Project Definition – Prior to accepting client samples at the laboratory, an agreement between the EPA Task Order Project Officer (TOPO) and ESAT must be set forth in a Technical Direction Form (TDF), which details the required analytical methods, target analytes, approximate quantity of samples, receipt date, analytical quality control (QC) procedures, and data deliverables.

1.2 ESAT Project Definition – Following receipt of the TDF, ESAT personnel will create a project in the Laboratory Information Management System (LIMS) that defines the requirements detailed in the TDF.

Note: ESAT does not receive a TDF for PT samples; however, ESAT will still create a project in LIMS that defines the requirements of the PT provider and the National Environmental Laboratory Accreditation Conference (NELAC).

1.3 Sample Integrity Inspection

- Samples listed on the chain of custody (COC) are compared to the actual samples received in order to identify any discrepancies
- Samples and shipping coolers are inspected for leakage or breakage
- Temperature of the samples is recorded
- Sample preservation is verified
- Any breach of the sample integrity will be noted and become a part of the project record
- A copy of the *Sample Receipt Form* – TLF-51.XX (current version) will be completed, which documents all of the parameters taken and anomalies, if any.

1.4 COC Verification

- The COC establishes a traceable, legal record of the possession of the samples from sampling through analysis
- Laboratory personnel compares the sample identification as listed on the COC to the identification on the samples
- Identify any sample requiring analyses with short holding times and notify laboratory personnel of the sample arrival
- Note any and all discrepancies on the *Sample Receipt Form*, which become part of the project record
- Maintain sample custody by storing the samples in a locked cooler
- Track movement of the samples in and out of the cooler in a logbook

1.5 LIMS Sample Login – After completing sample receipt procedures, the samples are logged into the LIMS by utilizing the ESAT project definition and either an electronic XML

file or by hand entering sample information and any noted discrepancies from the *Sample Receipt Form*.

2.0 Acronyms

^{o}C	Degrees Celsius
CHP	Chemical Hygiene Plan
COC	Chain of Custody
EDD	Electronic Data Deliverable
EPA	United States Environmental Protection Agency
ESAT	Environmental Services Assistance Team
HSO	Health and Safety Officer
ID	Identification
IR	Infrared
LIMS	Laboratory Information Management System
NELAC	National Environmental Laboratory Accreditation Conference
PPE	Personal Protective Equipment
PT	Proficiency Testing
QA	Quality Assurance
QAO	Quality Assurance Officer
QC	Quality Control
SOP	Standard Operating Procedure
TDF	Technical Direction Form
TO	Task Order
TOPO	Task Order Project Officer

3.0 Health and Safety

3.1 The person receiving the samples ensures that the sample login area is clean and free of any potential contaminants prior to working in the area.

3.2 Proper personal protective equipment (PPE) is required for sample receipt, including a minimum of gloves, eye protection, and lab coat.

3.3 Leaking containers can pose a health risk due to the possible presence of acids and other toxic components making inhalation of toxic vapors a potential hazard.

- All coolers should be opened in a room with adequate ventilation
- If broken sample containers are present, additional PPE and engineering controls (e.g., chemical fume hood) may be required. If the use of spill cleanup material is necessary, the proper method of cleanup and disposal must be followed. Refer to the EPA Region 8 HSP-001, *Chemical Hygiene Plan* (CHP), current version.
- Assistance from the ESAT or EPA Health and Safety Officer (HSO) for proper handling and disposal procedures may be required

3.4 Sample receipt personnel must be familiar with the location of additional safety equipment.

- Spill and neutralizer equipment are available in the sample receipt area
- The eye wash station and safety shower in the sample receiving area should be verified as unobstructed prior to unpacking the samples

4.0 Equipment and Supplies

- Calibrated and certified thermometers – Thermometers used for measuring sample storage cooler temperatures are calibrated annually under the supervision of the EPA laboratory Quality Assurance Officer (QAO) using a certified thermometer. An infrared (IR) temperature indicator is maintained and used by ESAT personnel for recording the temperature of the samples upon arrival.
- Wide-range pH paper (non-bleeding)
- Waste container (properly labeled according to the CHP)
- Promium ELEMENT LIMS for sample tracking and reporting
- Laboratory chemical fume hood for opening sample coolers
- Refrigerated and secured sample storage cooler

5.0 Personnel Qualifications and Responsibilities

5.1 ESAT Personnel

- The receiving and checking of incoming samples must be performed by an ESAT team member trained in the proper performance of this SOP
- The sample receiver must be familiar with interpreting COC documentation, performing pH determinations, and maintaining custody of samples
- Personnel receiving samples should have a baseline physical examination performed prior to receiving samples
- Some lifting of 30-50 pound coolers/containers may be required

5.2 EPA Personnel

- EPA personnel will periodically move coolers containing ESAT samples into the ESAT sample storage cooler
- EPA personnel will notify ESAT team members of the arrival of the samples

6.0 Cooler Receipt and Acceptance

6.1 Sample Integrity Inspection

6.1.1 Generally, samples are received through the main entrance of the laboratory via FedEx or delivery from the sampling contractor. Note the method of delivery on the *Sample Receipt Form*. This is indicated in the project later when the samples are logged into the LIMS.

6.1.2 Retrieve a sample cart, and move the coolers to the sample receipt area (E-115).

6.1.3 Examine the shipping coolers for any damage or leaks, and note their presence for inclusion into the project folder.

6.1.4 Open the cooler(s) while the cooler is located under the exhaust hood in the sample receipt area.

6.1.5 Remove the COC from the cooler.

6.1.6 On each page of the COC, sign the “Received” section and record the date and time of receipt.

Note: Whenever possible, the sampler or customer should be present during the transition of the samples into ESAT custody, including opening of the coolers and cross-checking of information.

6.1.7 Unpack the cooler, and use the COC to organize the samples on the work table in the sample receipt area.
- If any issues with sample integrity are observed (e.g., damage to the sample container, contamination, etc.), the analyst should note on the *Sample Receipt Form* and in the case narrative of the data package so that data users are aware that the sample may have been compromised.
- Any correspondence with and direction received from the TOPO regarding a compromised sample should be received in writing via email, and that email should be included in the data package.

6.1.8 Temporarily place the ice or baggies filled with ice in the deep sink under the exhaust hood.

6.1.9 Using the IR thermometer, measure the temperature of the first unpacked sample. This temperature is recorded on the *Sample Receipt Form* and in LIMS.

6.1.10 Inspect each sample container for damage or leaking, and note any circumstance for inclusion in the project folder.

6.1.11 Verify the preservation of any samples that are indicated on the COC as having been preserved to a specific pH. Note the lot number(s) of the pH strips on the *Sample Receipt Form.*

6.1.12 Place a drop or two of sample on the pH indicator strip using a disposable pipet and compare strip to color scale that is provided on pH strip package to obtain sample pH.

6.1.13 If the sample is properly preserved, no further action is required.

6.1.14 Recap the sample container and proceed with the login procedure (see Section 7.0).

6.1.15 Improperly preserved samples must be preserved before placing into the storage cooler. Carefully note on the *Sample Receipt Form* which samples were not properly preserved.

6.2 COC Verification

6.2.1 The samples should be accompanied by a COC, sample identification (ID) tags, and custody seals.
- All information required on the forms and tags must be properly completed and legible.
- The sample ID tag information must be verified against the corresponding sample information provided on the COC.

6.2.2 In the case of COC discrepancies, the sample ID tag will be assumed as the true information, and the discrepancies must be clearly noted on the *Sample Receipt Form* and on the COC with the login personnel's initials and date.
- All COC discrepancies should be discussed in the *Sample Receipt Form* of the data package.
- If a COC discrepancy requires contact with the TOPO, this should also be discussed in the case narrative of the data package. If COC discrepancies are resolved verbally with the TOPO, an email should be sent to confirm the reconciliation of discrepancies, and a copy of the email should be included in the data package with the COC.

6.2.3 If the documentation is incomplete, the ESAT Contract Project Manager and TOPO must be notified of the discrepancy. The TOPO will decide if the process will continue.

6.2.4 After each sample is unpacked from the shipping container and the sampling information is verified, it is segregated into various storage trays by analytical method.

6.2.5 The trays are labeled with a tag in a plastic shield with the following information:

project name, LIMS number, TDF number, due date, and requested analysis.

6.2.6 The labeled trays are then placed in walk-in cooler "A" and secured by locking the cooler with the provided padlock.

6.2.7 The trays are removed by the analyst prior to analysis. The analyst records the removal of the samples from the cooler in the logbook in the sample receipt area.

6.2.8 Empty the plastic bags filled with ice that were placed in the sink, and put the empty bags into the provided waste container in the sample receipt area.

7.0 Project Creation and Sample Entry in LIMS

7.1 Project Creation in LIMS

- Open the LIMS software
- In the Project Management dropdown menu, select "Projects"
- Highlight a similar project. Be sure to check that it has the required test codes.
- Select the "Copy" option
- Double click the "Superfund" client option
- Rename the project in the dialog window
- From the new project screen select "Edit"
- Put the TDF number in both the "Project Number" and "PO number" fields
- Select the "Project Manager" from the drop down menu
- Check that the default Electronic Data Deliverable (EDD) is "StdESATExel_rev1.exe"
- Enter the appropriate project name in the comments field
- If the test codes for the new project need to be changed, double click on "Test Codes" and select the correct test codes for the project from the drop down menu
- Save the project

7.2 Work Order Creation in LIMS

- From the "Sample Control" menu, select "Work Order"
- Select "Import" and select the file location of the XML/Scribe file from the drop down menu
- Click the "Import" button
- From the "Analysis" tab, match the appropriate test codes
- From the "Matrices" tab, match the sample preservatives
- From the "Container" tab, select "Default"
- Click "Done" and the new work order screen will appear

7.3 Work Order Information Editing

- Select the work order from the dropdown menu and click "Edit"
- Select the project from the drop down menu in the top right corner
- The Project number and the PO number should match the TDF for the project
- In the "Submitted By" window, select the appropriate sampler from the drop down menu
- In the "SDG Identifier" window, type in the TDF number
- In the "Shipped By" window, select either "Walk-in" or" FedEx" from the drop down menu. If shipping was by Fed Ex, enter the tracking number in that window.
- Select the turn around time to calculate the appropriate due date for the project
- Check the appropriate "Condition" boxes for the samples received
- Ensure the Analysis Test Codes are accurate and add/delete as needed

- Save the work order

7.4 Editing Samples in the Work Order

- Click on the “Samples” tab and “Edit”
- Verify that the sample name, container, location, and comment (EPA Tag #) are correct
- In the “Report Matrix” drop down window, select the one listed on the COC
- In the “Sample Type” drop down window, select “Field Sample”
- In the “Sampled By” drop down window, select the one listed on the COC
- In the “Work Analysis” windows, the test codes may or may not be applied. If more tests are needed, click the “Work Analysis” tab to see all of the available test codes.
- Repeat sample entry/editing for all samples in the project
- Save the work order, and click the printer icon to print the sample information

7.5 Project Folder Creation

7.5.1 Master Project Folder
- Label a new folder with the work order number, project name, TDF number, and due date
- Place the original COC, TDF, and the shipping label in this folder
- Place the LIMS printout of the samples entered in this folder
- Place any E-mail or other documents pertaining to the project in this folder
- All analytical data will be placed in this folder until final report generation

7.5.2 Analytical Folder
- On the LIMS computer, go to ”Explore”
- Go to the “X” drive and click on “Metals_Data_Files”
- Select the appropriate year
- Go to “File”, “New”, “Folder”
- Name the new folder using the following format: Work Order_TDF Project Name (e.g., C606006_SC010 CalGulch June Monthly)
- Repeat the file creation sequence in “WetChem_Data_Files” if the project requires this type of analysis

7.5.3 Reporting Folder
- On an ESAT computer (not the LIMS computer), navigate to the appropriate Task Order (TO) folder on the network drive
- Click on “Analytical Reports” and then “Final Reports”
- Go to “File”, “New”, “Folder”
- Name the folder using the same convention as the Analytical Folder (Section 7.5.2)

8.0 Data and Records Management

- The sample checkout logbook is maintained by ESAT quality assurance (QA)/ QC personnel
- Completed logbooks are archived and new ones provided when necessary
- EPA QA/QC personnel verify thermometer calibration and log cooler temperatures daily
- COC records, LIMS reports, and all other correspondence become part of the ESAT retained records data file
- All custody records and entries in the sample checkout logbook will be recorded in blue or black indelible ink
- When an entry error occurs, the author will draw a single line through the error, initial and

date, and record the correct information. If the space is too small for further legible entries, either the next line will be used or the correction must be footnoted to ensure legibility of the correct entry.

- Internal audits will be conducted periodically by the ESAT QAO or designee to verify the procedures outlined in this SOP are being performed
- Refrigerated cooler temperatures are checked and recorded daily according to EPA Region 8 SOP EQOP-805, *Monitoring Refrigerator and Cooler Temperatures*, current version.

9.0 Waste Minimization

- The analyzed samples are separated for consolidation and disposal. Refer to ESAT SOP 16-LAB-01.XX, *Collection, Analysis and Disposal of Laboratory Waste*, current version
- Plastic sample tag holders are reused, as are the washable trays, coolers, and carts. Sample containers are not reused due to high possibility of cross contamination.
- In order to minimize contamination of large volumes of liquids, compatible samples marked for disposal will be consolidated without further dilution
- Field coolers and some packing materials (e.g., foam, bubble wrap) can be cleaned, dried, and reused

10.0 References

EPA Region 8 Laboratory HSP-001, *Chemical Hygiene Plan*, current revision

EPA Region 8 Laboratory SOP EQOP-805, *Monitoring Refrigerator and Cooler Temperatures,* current revision

EPA Region 8 Laboratory SOP GENLP-808, *Sample Receipt and Custody*, current revision

ESAT Region 8 SOP 16-LAB-01.XX, *Collection, Analysis and Disposal of Laboratory Waste*, current revision

ESAT Region 8 Health and Safety Plan, current revision

Document Change History			
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[1] Status: I = Initial, R= Revision, or C = Cancelled

16-MET-01.01
Determination of Metals by Inductively Coupled Argon Plasma-Mass Spectroscopy (ICP-MS)

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SIGNATURE **Date:** 9/23/2013

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Table of Contents

1.0 SOP Description

This standard operating procedure (SOP) describes the daily operation, tuning, optimization, and analytical procedures according to United States Environmental Protection Agency (EPA) Method 6020 and EPA Method 200.8 for the elements listed as analytes in Appendix I using the ELAN® 6000 Inductively Coupled Plasma-Mass Spectrometer (ICP-MS). Aqueous sample, digestates, leachates, etc. are nebulized into a spray chamber where a stream of argon carries the sample aerosol through a quartz torch and injects it into a R.F. plasma. There, the sample is decomposed and desolvated. The ions produced are entrained in the plasma gas and by means of a water-cooled differentially pumped interface, introduced into a high-vacuum chamber that houses a quadruple mass spectrometer. The ions are sorted according to their mass-to-charge ratio and measured with a detector. The quantity and identity of the unknown analytes are determined from a mass spectral comparison with known standards. This method is applicable to the measurement of like aqueous matrices. The method further defines general instrument operational protocols.

This method is applicable to the following sample matrices analyzed by the Region 8 Environmental Services Assistance Team (ESAT): ground waters, surface waters, industrial wastes, sludge's, and soil samples. These operational guidelines are to be used by an experienced operator to prepare the instrument for optimum performance.

Routine operation and maintenance procedures for the ELAN® 6000 ICP-MS may be found in the ELAN® 6000 Hardware Manual provided by the instrument manufacturer. Detailed instructions on operating of the ELAN® 6000 ICP-MS operating software may be found in the ELAN® 6000 Software Manual. Detailed information regarding requirements by EPA SW-846 Method 6020 may be found in Revision 0 (September 1994) of Method 6020. Detailed information regarding requirements by EPA SW-846 Method 200.8 may be found in EPA *Methods for the Determination of Metals in Environmental Samples*, Supplement I, May 1994.

2.0 Acronyms

amu	Atomic Mass Unit
ASTM	American Society for Testing and Materials
BS	Blank Spike
CCB	Continuing Calibration Blank (QC sample)
CCV	Continuing Calibration Verification (QC sample)
CDOC	Continuing Demonstration of Capability
CHP	Chemical Hygiene Plan
CPS	Counts Per Second
ICP-MS	Inductively Coupled Plasma-Mass Spectrometer
CRDL	Contract Reporting Detection Limit
ESAT	Environmental Services Assistance Team
EPA	United States Environmental Protection Agency
HCl	Hydrochloric Acid
HNO_3	Nitric Acid
ICB	Initial Calibration Blank (QC sample)
ICSA(B)	Interference Check Solution A/AB
ICV	Initial Calibration Verification (QC sample)
IDL	Instrument Detection Limit
IDOC	Initial Demonstration of Capability
IS	Internal Standard
L	Liter

LCS	Laboratory Control Sample (QC sample)
LIMS	Laboratory Information Management System
MB	Method Blank
MDL	Method Detection Limit
mg	Milligram
ml	Milliliter
mm	Millimeter
MS	Matrix Spike
PQL	Practical Quantitation Limit
PS	Post-Digestion Spike
PT	Proficiency Testing
QA	Quality Assurance
QC	Quality Control
RPD	Relative Percent Difference
RPM	Revolutions Per Minute
RSD	Relative Standard Deviation
SCV	Secondary Calibration Verification
SHEMP	Safety, Health and Environment Management Program
SOP	Standard Operating Procedure
SRM	Standard Reference Material
TDF	Technical Direction Form
ug/L	Microgram Per Liter
µL	Microliter

3.0 Health and Safety

3.1 All pertinent procedures outlined in the EPA Region 8 Chemical Hygiene Plan (CHP) will be followed in performance of ICP-MS analyses. The use of laboratory equipment and chemicals exposes the analyst to several potential hazards. Good laboratory technique and safety practices must be followed at all times.

3.2 Solutions prepared for ICP-MS analysis normally contain percentage levels of mineral acids and can contain certain inorganic elements known to be hazardous. Gloves, protective eye wear and laboratory coats must be worn at all times when handling samples or reagents or when in the vicinity of others handling these items.

3.3 Liquid argon represents a potential cryogenic hazard, and safe handling procedures should be used when handling liquid argon tanks at all times.

3.4 The ELAN® 6000 is fully interlocked to protect the user from dangers such as high voltages, radio frequency generators, and intense ultra-violet light. At no time should the operator attempt to disable these interlocks or operate the ELAN® 6000 if any safety interlock is known to be disabled or malfunctioning.

3.5 The torch and interface remain hot for some time after the plasma has been extinguished. Do not attempt to perform adjustments or maintenance until all equipment is sufficiently cool.

3.6 Spilled samples, reagents, and water must be cleaned up from instrument and autosampler surfaces immediately. In the case of acid spills, the acid must be neutralized with acid spill kits available in the laboratory.

4.0 Cautions

This SOP provides guidelines for ICP-MS analyses. Following this SOP will result in data of known quality. However, situations may arise in which changes are required. Undocumented non-compliance with this SOP may lead to challenges to the validity of data produced. Any deviation from the SOP must be addressed in the technical narrative prepared as part of the data report.

5.0 Interferences

5.1 Isobaric Interferences

- This type of interference occurs when an isotope of one element is at the same nominal mass as an isotope of another element (e.g., Mo 98 and Ru 98).
- Corrections for isobaric interferences may be made by measuring the intensity due to the interfering element at another isotope and using its natural abundance ratios to correct for its presence at the analytical mass of interest.
- Most commonly used corrections for isobaric interferences are already present as default interference equations in the ELAN® 6000 software.
- Care should be taken that the isotope measured for correction purposes does not suffer from overlap with other isotopes that may be present in the sample.

5.2 Molecular Interferences

- This interference is caused by molecular species formed in the plasma with plasma or matrix ions (examples of common molecular interferences include ArCl, ClO, Nitrogen dimmer, oxygen dimmer and oxide species.
- Predictions about the type of molecular interferences may be made using knowledge about the sample matrix.
- Molecular interferences can often be corrected for in the same manner as isobaric interferences, i.e., measuring the intensity present at another isotope and using isotope ratios to calculate the amount of the interfering species. For example, corrections for interferences of $Ar^{40}Cl^{35}$ on As at mass 75 may be made by measuring the intensity of ArCl present at mass 77 ($Ar^{40}Cl^{37}$) and converting to the apparent intensity of ArCl at mass 75 by using the isotopic ratio of Cl^{37} to Cl^{35}.
- A list of the corrections used is given in the listing of the isotopes monitored in Appendix I to this SOP.

6.0 Personnel Qualifications

The analysis may be performed by ESAT team members with experience in laboratory analytical methods and certified laboratory health and safety training. Technical staff may include lab technicians with demonstrated ability, prior experience, or personnel with technical degrees. Certified health and safety training will include successful completion of the 24 hour EPA Region 8 Safety, Health and Environmental Management Program (SHEMP) laboratory safety training course.

7.0 Equipment and Supplies

7.1 The Perkin-Elmer ELAN® 6000 ICP-MS system includes ELAN® 6000 instrument, computer system, ELAN® 6000 software, printer, and auto sampler and chiller.

7.2 Peristaltic pump tubing:

- Black/Black - 0.75 mm i.d. (for sample introduction)
- Yellow/Orange - 0.51 mm i.d. (for internal standard introduction)
- Purple/White - 2.79 mm i.d. (for drain)
- Red/Red - 1.14 mm i.d. (for rinse station)

7.3 T-connector, polypropylene, 1/16 inch i.d. (Cole Parmer Part Number: H-06365-77) for hooking up on-line addition of internal standards.

7.4 Calibrated mechanical pipettes and tips:

- 10-100 µL
- 100 - 1000 µL
- 5000 - 10000 µL

7.5 14 mL polypropylene auto sampler test tubes (Falcon 2018, VWR 60819-670 or equivalent).

7.6 50-mL plastic SCP digestion tubes with caps (SCP Part # 010-500-061 or equivalent).

8.0 Reagents and Solutions

8.1 All reagents may contain impurities that could affect the integrity of analytical results. Due to the high sensitivity of ICP-MS, high-purity reagents, water, and acids must be used whenever possible.

8.2 All acids used for this method must be of high-purity grade.

- Nitric Acid (HNO_3) is preferred for ICP-MS in order to minimize polyatomic interferences.
- It should be noted that hydrochloric acid (HCl) is required to maintain stability in solutions containing antimony and silver.
- When HCl is used, corrections for poly-atomic ion interferences must be used.

8.3 HNO_3, concentrated, suitable for trace metals analysis.

8.4 HCl, concentrated, suitable for trace metals analysis.

8.5 Reagent water equivalent to American Society for Testing and Materials (ASTM) Type I water (ASTM D1193).

8.6 Single element stock solutions of the analytes of interest can be purchased from various vendors. Suggested suppliers include High Purity, Inorganic Ventures, and Spex.

8.7 Calibration Blank, 1% HNO_3 – In a 2 L volumetric flask, add approximately 1 L of reagent water and 20 mL of concentrated HNO_3. Bring to volume with reagent water, stopper and mix.

8.8 ESAT Tuning Solution Stock: 10 mg/L Be, Mg, Co, Rh, In, Ba, Ce, Pb, U and Zn

Note: Method 6020 does not meet the tuning requirements of the ELAN® 6000

manufacturer. The tuning solution defined in this section of the SOP allows all manufacturer tuning parameters to be met while also meeting the stated requirements of the tuning solution in Section 5.8 of Method 6020, Revision 0.

8.8.1 Obtain a clean, dry 50 mL volumetric flask.
8.8.2 Add approximately 25 mL of reagent water and 1 mL of concentrated HNO_3 into the flask and swirl to mix.
8.8.3 Pipet 500 µL of each 1000 mg/L single element stock solution (Section 8.9) into the flask.
8.8.4 Dilute to 50 mL with reagent water, stopper and swirl to mix.
8.8.5 Transfer the solution to a labeled 60 mL Nalgene® bottle.

8.9 ESAT Tuning Solution, Working: 10 µg/L Be, Mg, Co, Rh, In, Ba, Ce, Pb,U and Zn

8.9.1 Obtain a clean, dry 1000 mL volumetric flask.
8.9.2 Add approximately 500 mL of reagent water and 10 mL of concentrated HNO_3 to the flask.
8.9.3 Pipet 1 mL of the Tuning Stock solution (Section 8.8) into the flask and dilute to volume with reagent water and mix well.
8.9.4 Transfer the solution to the labeled container near the ICP-MS auto sampler.

8.10 ESAT Internal Standard (IS) Stock Solution: 10 mg/L Li^6, Sc, Y, In, Rh, Tb, Ho, Bi; 50 mg/L Ge, Au

8.10.1 Obtain a clean, dry 50 mL volumetric flask.
8.10.2 Add approximately 25 mL of reagent water and 1 ml of concentrated HNO_3 to the flask and swirl to mix.
8.10.3 Pipet 500 µL of each of the following 1000 mg/L stock solutions into the flask: Rh, Ho, Li^6, Sc, Y, In, Tb and Bi.
8.10.4 Pipet 2.5 mL of the 1000 mg/L Ge and Au standards into the flask.
8.10.5 Bring to volume with reagent water, stopper and mix.
8.10.6 Transfer the solution to a labeled 60 mL Nalgene® bottle.

Note: The IS is added to the samples and standards via online addition of the IS solution by using an additional channel on the peristaltic pump with yellow/orange 2-stop tubing.

8.11 ESAT IS Working Solution

8.11.1 Obtain a clean, dry 500 mL volumetric flask.
8.11.2 Add approximately 400 mL of reagent water and 5 mL of concentrated HNO_3 to the flask. Swirl to mix.
8.11.3 Pipet 3.0 mL of the IS stock solution (9.10) into the flask.
8.11.4 Bring to volume with reagent water, stopper and mix.
8.11.5 Transfer the solution to the labeled container near the ICP-MS autosampler.

8.12 ESAT Calibration Standards - Stock Solutions

8.12.1 Calibration Standard #1, Stock: 10 mg/L of As, Sb, Be, Cd, Cr, Co, Cu, Pb, Mn, Mo, Ni, Se, Tl, Th, U, V and Zn; 50 mg/L of Al
8.12.1.1 Obtain a clean, dry 50 mL volumetric flask.
8.12.1.2 Add approximately 10 mL of reagent water and 1 mL of concentrated

HNO_3 to the flask. Swirl to mix.

8.12.1.3 Pipet 2500 uL of 1000 mg/L Al and 500 µL of each 1,000 mg/L standard of the analytes listed above (9.12.1) into the flask.

8.12.1.4 Bring to volume with reagent water, stopper and mix.

8.12.1.5 Enter the prep information and date into the Secondary Standard Preparation and Tracking Logbook.

8.12.1.6 Transfer the solution to a labeled 60 mL Nalgene® bottle.

8.12.2 Calibration Standard #2, Stock: 10 mg/L of Ag and Ba

8.12.2.1 Obtain a clean, dry 50 mL volumetric flask.

8.12.2.2 Add approximately 10 mL of reagent water and 1 mL of concentrated HNO_3 to the flask. Swirl to mix.

8.12.2.3 Pipet 500 µL of each 1,000 mg/L Ag and Ba standard into the flask.

8.12.2.4 Bring to volume with reagent water, stopper and mix.

8.12.2.5 Enter the prep information and date into the Secondary Standard Preparation and Tracking Logbook.

8.12.2.6 Transfer the solution to a labeled 60 mL Nalgene® bottle.

8.12.3 Calibration Standard #3, Stock: 100 mg/L of Na, Ca, Mg, K, and Fe

8.12.3.1 Obtain a clean, dry 50 mL volumetric flask.

8.12.3.2 Add approximately 10 mL of reagent water and 1 mL of concentrated HNO_3 to the flask. Swirl to mix.

8.12.3.3 Pipet 500 µL of each 10,000 mg/L standard above into the flask.

8.12.3.4 Bring to volume with reagent water, stopper and mix.

8.12.3.5 Enter the prep information and date into the Secondary Standard Preparation and Tracking Logbook.

8.12.3.6 Transfer the solution to a labeled 60 mL Nalgene® bottle.

8.13 ESAT Calibration Standards - Working Solutions

8.13.1 Calibration Std. #1, Working 10 µg/L: As, Sb, Be, Cd, Cr, Co, Cu, Pb, Mn, Mo, Ni, Se, Tl, Th, U, V and Zn; 50 ug/L: Al; 100 µg/L: Na, Ca, Mg, K and Fe

8.13.1.1 Obtain a clean, dry 50 mL volumetric flask.

8.13.1.2 Add approximately 40 mL of reagent water and 1 mL of concentrated HNO_3 to the flask. Swirl to mix.

8.13.1.3 Pipet 50 µL of each calibration stock standard above (9.12.1, 9.12.2 and 9.12.3) into the flask.

8.13.1.4 Bring to volume with reagent water, stopper and mix.

8.13.1.5 Record the prep date in the Secondary Standard Preparation and Tracking Logbook.

8.13.2 Calibration Std. #2, Working 20 µg/L: As, Sb, Be, Cd, Cr, Co, Cu, Pb, Mn, Mo, Ni, Se, Tl, Th, U, V and Zn; 100 ug/L Al; 1 mg/L: Na, Ca, Mg, K and Fe

8.13.2.1 Obtain a clean, dry 50 mL volumetric flask.

8.13.2.2 Add approximately 40 mL of reagent water and 1 mL of concentrated HNO_3 to the flask. Swirl to mix.

8.13.2.3 Pipet 100 µL of each calibration stock standard above (9.12.1, 9.12.2) and 500 µL of calibration stock #3 (9.12.3) into the flask.

8.13.2.4 Bring to volume with reagent water, stopper and mix.

8.13.2.5 Record the prep date in the Secondary Standard Preparation and Tracking Logbook.

8.13.3 Calibration Std. #3, Working 100 µg/L: As, Sb, Be, Cd, Cr, Co, Cu, Pb, Mn, Mo,

Ni, Se, Tl, Th, U, V and Zn; 500 ug/L Al; 10 mg/L: Na, Ca, Mg, K and Fe

8.13.3.1 Obtain a clean, dry 50 mL volumetric flask.

8.13.3.2 Add approximately 40 mL of reagent water and 1 mL of concentrated HNO_3 to the flask. Swirl to mix.

8.13.3.3 Pipet 500 µL of each calibration stock standard above (9.12.1, 9.12.2) and 5000 µL of calibration stock #3 (9.12.3) into the flask.

8.13.3.4 Bring to volume with reagent water, stopper and mix.

8.13.3.5 Record the prep date in the Secondary Standard Preparation and Tracking Logbook.

8.14 ESAT Secondary Calibration Verification (SCV) Standard

The SCV stock MUST BE from a source independent of that used for calibration. The ESAT current SCV preparation is described below.

8.14.1 Obtain a clean, dry 50 mL volumetric flask.

8.14.2 Add approximately 40 mL of reagent water and 1 mL of concentrated HNO_3 to the flask. Swirl to mix.

8.14.3 Pipet 250 µL of Inorganic Ventures QCP-QCS-3 standard into the flask.

8.14.4 Pipet 250 µL of each 1,000 mg/L Na, Ca, Mg, K and Fe standards into the flask.

8.14.5 Bring to volume with reagent water, stopper and mix.

8.14.6 Record the prep date in the Secondary Standard preparation and Tracking Logbook. This solution simultaneously expires with the elemental stocks.

8.15 ESAT Initial Calibration Verification (ICV)/Continuing Calibration Verification (CCV) Standard

8.15.1 Obtain a clean, dry 50 mL volumetric flask.

8.15.2 Add approximately 40 mL of reagent water and 1 mL of concentrated HNO_3 to the flask. Swirl to mix.

8.15.3 Pipette 250 µL each of ICP-MS Cal Stock 1 and ICP-MS Cal Stock 2 into the flask.

8.15.4 Pipette 2500 µL of ICP-MS Cal Stock 3 into the flask.

8.15.5 Bring to volume with reagent water, stopper and mix.

8.15.6 Record the prep date in the Secondary Standard preparation and Tracking Logbook. This solution simultaneously expires with the elemental stocks.

8.16 ESAT Dual Detector Cross-Calibration Solution

This solution contains 250 mg/L each: Al, Sb, As, Ba, Be, Cd, Cr, Co, Cu, Pb, Mn, Mo, Ni, Se, Tl, Th, U, V, Zn, Sc, Y, In, Rh, Tb, Ho, and Bi: 1250 mg/L Ge and Au and 3 mg/L Na, Ca, Mg, K, Fe

8.16.1 Pipette 2.5 mL of Calibration Stock 1, 2.5 mL of Calibration Stock 2, 250 µL of Calibration Stock 3, and 2.5 mL of the Internal Standard stock solution into a 100 mL volumetric flask containing 50 mL of reagent water and 1 mL of concentrated HNO_3.

8.16.2 Dilute to volume with reagent water and mix well.

8.16.3 If fewer than five points are collected during calibration for a specific element, add 100 - 500 µL of a 1000 mg/L standard and re-calibrate.

Note: You should also add comparable concentrations of any interfering elements or

species, such as chloride, whose intensities may be used in interference corrections to ensure correct equations if the measured intensities are in the analog range of the detector. For example, if the EPA SW-846 3050 digestion is to be measured at a 1:10 dilution, resulting in an hydrochloric acid concentration of 0.5%, the detector cross calibration solution should be made in 0.5% hydrochloric acid.

8.17 ESAT Interference Check Solution Stock A (ICSA)

8.17.1 This solution contains 1000 mg/L each of Al, Ca, Fe, Mg, Na, P, K, and S; 2000 mg/L of C; 10,000 mg/L Cl, and 20 mg/L each of Mo and Ti.
8.17.2 ESAT currently purchases this stock from Inorganic Ventures with the catalog number: 6020ICS-0A.

8.18 ESAT Interference Check Solution Stock AB (ICSAB)

This solution contains 10 mg/L each of As, Cd, Cr, Co, Cu, Mn, Ni, Ag, and Zn (ESAT currently purchases this stock from Inorganic Ventures with the catalog number: 6020ICS-0B).

8.19 ESAT ICSA Working Solution

8.19.1 This solution contains 10 mg/L each of Al, Ca, Fe, Mg, Na, P, K, and S; 20 mg/L of C; 100 mg/L Cl, and 0.2 mg/L each of Mo and Ti.
8.19.2 Pipette 0.5 mL of the ICSA stock into a 50 mL volumetric flask and dilute to 50 mL with 1% HNO_3.
8.19.3 Transfer the solution to the labeled centrifuge tube in the ICP-MS auto sampler and record the preparation in the Secondary Standard Preparation and Tracking logbook. This solution simultaneously expires with the elemental stocks.

8.20 ESAT ICSAB Working Solution

8.20.1 This solution contains 10 mg/L each of Al, Ca, Fe, Mg, Na, P, K, and S; 20 mg/L of C; 100 mg/L Cl, 0.2 mg/L each of Mo and Ti, and 0.020 mg/L each of As, Cd, Cr, Co, Cu, Mn, Ni, Ag, and Zn.
8.20.2 Pipette 0.5 mL of the ICSA stock into a 50 mL volumetric flask containing approximately 40 mL of reagent water and 1 mL of concentrated HNO_3.
8.20.3 Pipette 0.5 mL of the ICSAB stock into a 50 mL volumetric flask containing approximately 40 mL of reagent water and 1 mL of concentrated HNO_3.
8.20.4 Bring to volume with reagent water.
8.20.5 Transfer the solution to the labeled centrifuge tube in the ICP-MS autosampler and record the preparation in the Secondary Standard Preparation and Tracking logbook. This solution simultaneously expires with the elemental stocks.

8.21 ESAT Contract Reporting Detection Limit (CRDL) Stock Solution

8.21.1 Obtain a clean, dry 500 mL volumetric flask and add approximately 100 mL of reagent water and 10 mL of concentrated HNO_3.
8.21.2 Stopper and swirl to mix.
8.21.3 Pipet the following volumes of 1000 mg/L concentration of each of the analytes listed below into the flask.
8.21.4 Record the preparation in the Secondary Standard Preparation logbook.

8.21.5 This solution simultaneously expires with the elemental stocks.

Element	Volume µL	Final Conc. µg/L
Beryllium	100	200
Manganese	100	200
Cobalt	100	200
Nickel	100	200
Arsenic	250	500
Molybdenum	100	200
Silver	250	500
Cadmium	100	200
Thorium	500	1000
Thallium	250	500
Uranium	100	200
Lead	100	200
Chromium	500	1000
Selenium	250	500
Antimony	250	500
Barium	250	500
Copper	500	1000
Vanadium	1000	2000
Zinc	2500	5000
Aluminum	10000	20000

8.22 ESAT CRDL Daily Solution

8.22.1 Obtain a clean, dry 200mL volumetric flask and add approximately 100mL of reagent water and 4mL of concentrated HNO_3.
8.22.2 Stopper and swirl to mix.
8.22.3 Pipet 20mL of the CRDL Stock Solution into the flask and bring to volume with reagent water.
8.22.4 Transfer the solution to the labeled centrifuge tube in the ICP-MS auto sampler. This solution simultaneously expires with the elemental stocks.

9.0 Sample Handling

Routine field preserved water samples and sealed soil samples require no special handling to ensure their integrity. Acid preservation of water samples and cold storage of soil samples in glass containers are sufficient to preserve integrity for the holding times indicated for this method. Proper acidification will aid in minimizing losses. Loss of analyte from soil samples may be attributed to or accelerated by changes in soil oxidation states and/or microbial action.

10.0 Sample Preparation

10.1 Total and Total Recoverable Metals

10.1.1 Acid digestion prior to filtration and analysis is required for groundwater, aqueous samples, industrial wastes, soils, sludges, sediments, and for other solid wastes for which total recoverable (acid leachable) elements are required.
10.1.2 Routine sample preparation methods may be found in EPA SW-846 or the appropriate ESAT SOP.

10.2 Dissolved Metals

Samples for dissolved metals are generally received filtered and preserved and require no preparation prior to analysis.

11.0 Instrument Analysis

11.1 Instrument Readiness

11.1.1 Open the ELAN® 6000 software and click on the “Instrument” tab.
11.1.2 Click on the “Front Panel” tab.
11.1.3 Ensure the “Ready” section is illuminated indicating that the vacuum is operational.
11.1.4 Ensure that no portion of the instrument graphic is highlighted in red.
11.1.5 Check the temperature of the instrument chiller.
11.1.6 Verify flow through the instrument ventilation.

11.2 Instrument Preparation

11.2.1 Open the instrument cover and slide the instrument module away.
11.2.2 Observe the condition of the skimmer cone, torch and load coil.
11.2.3 Return the module to its closed position and close the cover.
11.2.4 Replace all peristaltic pump tubing prior to igniting plasma.
11.2.5 With the ELAN® 6000 software open, click on the “Devices” window.
11.2.6 Click on the “Connect” button for the pump.
11.2.7 Place the sample and IS sippers into the reagent water container.
11.2.8 Type “24” into the RPM window for the pump.
11.2.9 Click the forward arrow to start the pump.
11.2.10 Observe flow through each of the pump tubes and instrument drain.

11.3 Instrument Warm-Up

11.3.1 Click on the “Front Panel” tab.
11.3.2 Click on the “Plasma Start” button.
11.3.3 Allow the instrument to run and aspirate 1% HNO_3 for 30-60 minutes.

11.4 ESAT Daily Instrument Optimization

11.4.1 Lens Voltage (Static)
11.4.1.1 Move the sippers from the reagent water to the tuning solution.
11.4.1.2 Open the ESAT Optimize workspace.
11.4.1.3 Ensure the method ESAT Optimize is included in the workspace.
11.4.1.4 Click the Auto Optimize tab and select Lens Voltage.
11.4.1.5 Click the “Get Analyte List” button and ensure Rh appears in the window.
11.4.1.6 Click the Optimize button.
11.4.1.7 Save the file after optimization is complete.

11.4.2 Nebulizer Flow
11.4.2.1 Click the Optimize tab and select Nebulizer Gas Flow
11.4.2.2 Click on “Realtime” and then “Analytes” and select CeO and Ce.

11.4.2.3 In the “Optimization Criteria” group, choose “Formula”.
11.4.2.4 Fill in the drop down menus with “CeO/Ce < 0.03”.
11.4.2.5 Click the Optimize button.
11.4.2.6 If the instrument is operating well, the flow should be 0.75 to 0.79.
11.4.2.7 Save the file after optimization is complete.

11.4.3 Tuning the Mass Spec
11.4.3.1 Open the “ESAT Tuning” workspace in the software.
11.4.3.2 Ensure He, Be, Mg, Co, Ce, Zn, Cd, In, Tl, Pb and U are the analytes in the window.
11.4.3.3 Click the “Tune Mass Spec” button.
11.4.3.4 Save the file after tuning is complete.

Note: The measured mass must be within 0.1atomic mass unit (amu) of the exact mass and the resolution must be <0.9 amu full width at 10% peak height to meet tuning criteria.

11.4.4 Auto Lens Calibration
11.4.4.1 Open the “ESAT Auto Lens Calibration” workspace.
11.4.4.2 Click on the Auto Lens tab.
11.4.4.3 Ensure Be, Co and In are listed in the analyte window.
11.4.4.4 Click the Calibrate button and save the file following calibration.

11.4.5 Daily Performance Check
11.4.5.1 Open the “ESAT Daily Performance” workspace.
11.4.5.2 Click the “Method” button and ensure that “ESAT daily.mth” is loaded.
11.4.5.3 Click on “Sample” button on the top menu.
11.4.5.4 Click on the “Manual” tab.
11.4.5.5 Click the cursor into the “Sample” window and type the date and initials.
11.4.5.6 Click “Analyze Sample”.
11.4.5.7 Evaluate the criteria and ensure they meet specifications.
11.4.5.7.1 Check that the relative standard deviations (RSDs) for the five replicates for all Be, Mg, Co, In, Rh, and Pb are all less than 5%.
11.4.5.7.2 Rh sensitivity should be >150,000 cps.
11.4.5.7.3 The background at mass 220 should be < 30 cps and the % double charged and % oxide levels should be < 3%.

11.5 Instrument Calibration

11.5.1 Open the “ESAT 2006 Work” workspace.
11.5.1.1 A previous sample information file will load with the workspace.
11.5.1.2 Click on the SAMPLE icon on the top menu.
11.5.1.3 The previous Sample Info table will be on the screen.
11.5.1.4 Click File/Open and select the appropriate Sample Info template for either waters or soils and the number of samples in the analytical batch.
11.5.1.5 Click File/Save As and select to create a New Folder.
11.5.1.6 Name the folder with the Laboratory Information Management System (LIMS) Work Order number.
11.5.1.7 Rename the sample info file using the format: “Batch_Prep_Date”.

11.5.1.8 Click on the “Method” icon on the top menu.
11.5.1.9 Click on the Report tab.
11.5.1.10 Rename the file output file in the Report File name dialog box.
11.5.1.11 Save the file to retain the new output file name by clicking File / Save.
11.5.1.12 Click on the Sample icon on the top of the page.
11.5.1.13 Enter samples to be analyzed on the Sample Info page.
11.5.1.14 Save the new sample info by clicking File/Save.
11.5.1.15 Save the workspace to retain all changes.

11.5.2 Prepare calibration standards as in Section 8.13.
11.5.2.1 Load the calibration blank and calibration standards into the autosampler positions specified in the autosampler page of the analytical method.
11.5.2.2 Load the Quality Control (QC) samples defined in the QC checking part of the method into the autosampler according to the positions entered in the QC Autosampler page of the method.

Note: If automatic QC checking is used, the method must be the same for all samples.

11.5.2.3 The Calibration action for the first sample for which concentration results are desired must be “Analyze Blank, Standards, and Sample”.
11.5.2.4 Enter peristaltic-pump control speeds for all samples.

Note: Typical pump speeds are 24 rpm for analysis and 48 rpm, (Maximum rate), for sample flush and rinse.

11.5.3 Prior to loading the autosampler, print the Sample Information table.
11.5.3.1 Arrow down to the last entry on the Sample Info table.
11.5.3.2 Click, Hold and Drag to the top of the table highlighting all entries.
11.5.3.3 Click File / Print Preview.
11.5.3.4 Click on the “Report Option” button and select ESAT Batch Report.
11.5.3.5 Click on the “Print” button.
11.5.3.6 Highlight the sample entries as before.
11.5.3.7 Click the “Build Run List” button.
11.5.3.8 Click “OK” to clear any previous QC.
11.5.3.9 Click on “Printable View”.
11.5.3.10 Click the printer icon to print the autosampler sequence.
11.5.3.11 Close the window.
11.5.3.12 Click the “Analyze Batch” button to start the sequence.

11.6 Sample Analysis

11.6.1 Load the samples into the autosampler positions specified in the Sample file.
11.6.2 Dilute samples as needed and load into their autosampler position.

11.7 Sample Sequence

A typical analytical sequence will begin with the following samples:

Blank
Standard 1 (10ug/L)
Standard 2 (20ug/L)

Standard 3 (100ug/L)
ICV
SCV
Initial calibration blank (ICB)
CRDL
ICSA
ICSAB
Laboratory or digestion blank
Blank spike (BS) or laboratory control sample (LCS)
Sample
Sample duplicate
Sample serial dilution
Sample matrix spike (MS)
Sample post-digestion spike (PS; digested samples, optional)
Sample
CCV
Continuing calibration blank (CCB)

12.0 Troubleshooting

12.1 QC Analytes

If poor recoveries are obtained on only particular analytes in a QC Standard and it is verified that the sample was properly prepared, it is possible that the problem is related to the associated internal standard.

12.1.1 Examine the list of elements grouped with an internal standard.
12.1.2 If the results for elements associated with a particular internal standard are not satisfactory, but the results for other elements not grouped with that internal standard are acceptable, there could be a problem with the internal standard used for that grouping.
12.1.3 Try using a different internal standard and reprocessing the data.
12.1.4 Look at the monitored intensities of the internal standards. If the internal standard used for the elements with unacceptable results is not within the allowable range or the percent recovery for this internal standard significantly different that the others, use a different internal standard and reprocess the data.

12.2 RSD for Standards or Samples

12.2.1 Check that the sampler and skimmer cones are in good condition and the orifices of both cones are round and of the proper size.
12.2.2 Check that the nebulizer is operating properly by checking the aerosol with the plasma off and the spray chamber removed.
12.2.2.1 Turn on the nebulizer gas and the peristaltic pump; there should be a visible aerosol leaving the spray chamber.
12.2.2.2 If there is not, clean or replace the nebulizer gem tips.
12.2.3 Check that the peristaltic pump tubing is in good condition and not worn.
12.2.3.1 When the auto sampler probe is removed and reinserted in the wash solution an air bubble will be visible in the tubing.
12.2.3.2 Watch the progress of this bubble and adjust the tension on the pump tubing so the flow is smooth without any pulsations.

12.3 Tuning Criteria

If the tuning criteria are not met, refer to the optimization protocols in section 11.4 of this SOP.

13.0 Data Analysis, Calculations and LIMS Transfer

13.1 All calculations necessary to convert raw data (ion counts/second) are performed by the ELAN® 6000 software.

13.1.1 The calculated quantities are selected by choosing the desired options in the Report Options screen.
13.1.2 The default report option for the ELAN® 6000 6020 Method is EPA6020QCReport.rop.
If the user desires, this format can be edited and saved under a new name.
13.1.3 All calculations performed in the ELAN® 6000 software are based on the ratio of the analyte intensity (cps) to the internal standard intensity (cps). In all calculations where internal standards are used the ratio of the analyte intensity to internal standard intensity is taken before any other calculation is performed.

Note: Method 6020 requires the use of internal standards.

13.2 QC Sample Results

13.2.1 These may be checked using the QC Checking features in the ELAN® 6000 software.
13.2.2 All values entered in the default ELAN® 6000 Method 6020 should be checked and edited to match the true values used by the laboratory.
13.2.3 The modified method can then be saved under a different name.

13.3 Data Transfer

13.3.1 Minimize the ELAN® 6000 software.
13.3.2 Using Microsoft Explorer open the ELAN® 6000 Report Output folder.
13.3.3 Open the appropriate data file using Microsoft Notepad.
13.3.4 For all sample dilutions including the serial insert an @ before the dilution.
13.3.5 Save the Report Output File.
13.3.6 Open the TEXT File folder and save the file with a .txt extension on the hard drive and on the X: drive under Metals_Data_Files.

14.0 Computer Hardware and Software

The computer interface with the current system is a Pentium class personal computer utilizing Windows operating system. Instrument control of the ICP-MS is facilitated through Perkin-Elmer ELAN® 6000 software. ESAT has created methods within the software for specific EPA protocols which include window layouts, QC sample acceptance criteria, and instrument operating conditions.

15.0 Data and Records Management

All instrument printouts, worksheets, and associated documentation must be filed in the final report folder. Copies may be required in submitted data reports. Any maintenance performed

should be recorded in the Perkin-Elmer ELAN® 6000 ICP-MS Instrument Logbook.

16.0 Waste Minimization

Metals laden waste volumes are minimized by the use of a dedicated waste receptacle in which no other laboratory waste is placed. Waste concentrations are minimized by judicious use of metals standard solutions and materials. General waste disposal regulations and policies are included in the CHP and must be strictly followed.

17.0 Quality Control and Quality Assurance

A summary of QC acceptability limits and associated corrective action is provided in Appendix II, QC Criteria-ICP/MS. The Instrument Detection Limit (IDL) and Method Detection Limit (MDL) procedures are also discussed in their associated SOPs. Consult the current version of the applicable SOPs for further details.

17.1 Initial Demonstration of Capability (IDOC)/Continuing Demonstration of Capability (CDOC)

17.1.1 Analyst certification for this method requires that an IDOC is performed by preparing a set of 4 digested water and soil LCSs and analyzing them.
17.1.2 The LCS results must be with the current established QC acceptance limits.
17.1.3 The analyst will perform a CDOC annually by either repeating the IDOC procedure or by the analysis of a proficiency testing (PT) sample.
17.1.4 Specific details of the IDOC/CDOC requirements are described in the ESAT IDOC SOP.

17.2 IDLs

17.2.1 IDLs should be determined according to the procedure outlined in Section 8.2 of Method 6020.
17.2.2 This procedure states that the IDLs be estimated by calculating the average of the standard deviations of the three runs on three nonconsecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day.
17.2.3 Each measurement must be performed as though it were a separate sample (i.e., with rinsing in between).
17.2.4 IDLs can be used to determine or troubleshoot instrumental problems and as a routine check on instrumental performance.

17.3 MDLs

17.3.1 The reference procedure for determination of MDLs is in Chapter 1, Section 5 of SW-846. Alternatively, specific protocols for MDL determination may be specified by the EPA by issuing a Technical Direction Form (TDF).
17.3.2 In order to determine the MDL in a matrix, the analytes should be spiked into the matrix of interest (e.g., a Method 3050 or Method 3051 sample preparation blank, ground water matrix, or other sample preparation procedure matrix) at a level that is three to five times the estimated MDL.
17.3.3 The spiked matrix is then carried through the entire sample preparation procedure.
17.3.4 The MDL is calculated by multiplying the standard deviation obtained from a

minimum of three analyses of the matrix spike by the one-sided 99% confidence level t-statistic. The table of t-statistics can be found in Chapter 1 of SW-846.

17.3.5 The procedure used in this SOP is to analyze the spiked matrix seven times and then multiplying the standard deviation of the seven measurements by 3.14 (the t- statistic for 7 samples).

17.3.6 MDLs should be re-determined whenever the following occurs:

17.3.6.1 Annually

17.3.6.2 Any change to the sample preparation procedure

17.3.6.3 Any significant change to the instrument (new detector or different sample introduction system used)

17.4 Linear Range Study

17.4.1 Calibrate the instrument, as described in Section 11.0 of this SOP.

17.4.1.1 Run a series of increasing concentration standards close to the upper linear range of the instrument.

17.4.1.2 The linear range is defined as the highest concentration where the measured value is within 10% of the actual prepared value of the standard.

17.4.2 The Upper Linear Dynamic Range should be re-determined whenever one of the following occurs:

17.4.2.1 A new detector is installed.

17.4.2.2 A new PA tube is installed in the RF generator.

17.4.2.3 A different sample introduction system (change in nebulizer or spray chamber type) is installed.

17.4.2.4 Whenever a significant change in the instruments performance is observed.

17.5 ICV Analysis

17.5.1 The ICV analysis must be performed immediately after calibration to verify calibration standards.

17.5.2 The ICV results must be within ±10% of the stated value. If the ICV is not within limits, the analysis should be terminated and the source of the problem identified before continuing.

17.6 SCV and CCV Analysis

17.6.1 The SCV is required to be run before samples are analyzed. The CCV is required to be run after every 10 samples and at the end of the analytical sequence.

17.6.2 The limits are ±10% of the true value. The action to be taken upon failure of the limits is to stop the analysis and correct the problem.

17.6.3 Samples run between CCVs not within limits must be rerun.

17.7 ICB and CCB Analysis

17.7.1 The solution used is the calibration blank.

17.7.2 Analyze the ICB immediately following the ICV and the CCB immediately after each CCV analysis. The limits are ± the Practical Quantitation Limit (PQL).

17.7.3 The action upon failure is to find and correct the cause of the out of control situation.

17.7.4 Samples run between CCBs not within limits must be evaluated before continuing. For results between the PQL and 2x the PQL, raise the MRL to the applicable level of detection in the blank. Rerun bracketed samples if the blank level exceeds 2x the PQL.
17.7.5 If failure occurs consistently, the MDL must be re-evaluated.

17.8 CRDL

17.8.1 After instrument calibration verification the sensitivity of the instrument is verified by analyzing the CRDL solution. Verify that the acceptance criteria (±30%) have been met for all required elements.
17.8.2 The acceptance criteria for Pb, Tl and Sb are ±50%.
17.8.3 Al, Ca, Fe, Mg and K are monitored but do not have control criteria.
17.8.4 Failure to meet these criteria requires identification and resolution of the problem, including recalibration, if necessary.

17.9 Interference Check Solutions ICSA and ICSAB

17.9.1 Required at the beginning of analytical run or every 12 hours, whichever is more frequent.
17.9.2 Limits for these solutions are ±20% of the true value. The analytes not contained in the ICSA should not be present at greater than ± the PQL.

17.10 Internal Standards

17.10.1 Intensities must be monitored in all solutions.
17.10.2 Intensities of internal standards in all subsequent analyses of CCV and CCB solutions must be within 60-125% of the levels in the original calibration blank.
17.10.3 Failure action is to terminate the analysis, correct the problem, re-calibrate, and reanalyze all affected samples.
17.10.4 Intensities of internal standards in samples must be within 60-125% of that in the original calibration blank.
17.10.5 Failure action is to dilute the sample and reanalyze with appropriate amounts of internal standards. This procedure is followed until the internal standard intensities fall within the prescribed window.

17.11 Serial Dilution Test

17.11.1 If the analyte concentration is within the linear range of the instrument and is a factor of at least 10 times the PQL, the analysis of a five-fold dilution of the sample must agree within 10% of the original determination.
17.11.2 One dilution test must be performed for each twenty samples or less of each matrix in a sample batch.

17.12 Post Digestion Spike (optional)

17.12.1 An aqueous analyte spike added to a portion of a prepared sample or its dilution should be recovered to within 85-115% of the known value of the spike or within laboratory derived acceptance criteria.
17.12.2 The spike value should be based upon the indigenous level of the analyte in the sample. If the spike is not recovered within the acceptance limits, the

sample must be diluted and reanalyzed to compensate for the matrix effect.

17.12.3 The use of the method of standard additions may be used to compensate for matrix effects.

17.13 Method BS/Standard Reference Material (SRM)

17.13.1 A BS is prepared and analyzed for aqueous dissolved metals analysis and an SRM is prepared and analyzed for aqueous and solid total recoverable metals analysis.

17.13.2 A BS should be analyzed using the same sample preparations, analytical methods, and quality assurance (QA)/QC procedures used for test samples.

17.13.3 One BS or SMR should be prepared and analyzed for each sample batch of 20 samples or less.

17.13.4 The recovery limits are 85-115%. For solid samples the limits vary by element and certification.

17.14 Dissolved Sample Analysis

17.14.1 A batch of dissolved samples requires the analysis one Blank, BS, Sample Duplicate, MS.

17.14.2 These are prepared at the time of analysis. Spiked samples are prepared by adding 100 uL each of QCP-QCS-3 and the Salt Spike to either 10mL of sample or 10mL of 1% HNO_3.

17.15 Duplicate Sample Analysis

17.15.1 Analyze one duplicate sample for every matrix in a batch of twenty samples or less.

17.15.2 The Relative Percent Difference (RPD) between the duplicate determinations shall be 20% for waters and 35% RPD for soils. This criteria should not be exceeded for analyte values greater than five times the PQL.

17.15.3 If the control limit is exceeded, the reason for the out of control situation should be corrected and any samples analyzed during the out of control condition reanalyzed.

17.16 MS

17.16.1 The laboratory must analyze at least one with each batch of 10 or fewer samples of the sample matrix, if sample volume permits.

17.16.2 MS recovery data are used to evaluate effects of the sample matrix on the recovery of sample analytes.

17.16.3 When MS recovery values exceed control limits a possible matrix effect must be noted in the case narrative.

17.16.4 Recovery limits for matrix spikes are 70-130%. Matrix spike recoveries are not applicable for sample concentrations that exceed four times the spike added concentration.

17.16.5 For digested samples, a post-digestion spike (PS), optional, may also be performed to confirm sample matrix effects.

17.16.6 Recovery limits for PS samples are 85-115%, however, there is no corrective action for failed PS samples as they are used for verification purposes only.

17.17 Method Blank (MB)

17.17.1 One MB must be prepared with each set of samples.

17.17.2 The MB for dissolved metals analysis is prepared by adding calibration blank to an autosampler tube and analyzing as the MB.

17.17.3 Corrective action is taken for detectable MB levels greater than ± PQL as follows:

17.17.3.1 If blank level is greater than PQL but less than 2 times PQL, raise the MRL to applicable level. Verify client reporting requirements before raising levels.

17.17.3.2 If Blank level is greater than 2 times the PQL, all samples associated with the batch should be re-prepared including digestion, if applicable.

18.0 References

Application Note: RCRA SW-846 Method 6020 for the ICP-MS Analysis of Soils and Sediments, 1996, Perkin-Elmer Corporation.

ELAN® 6000 Hardware Manual, 1995, Perkin-Elmer Corporation.

ELAN® 6000 ICP-MS Software Manual, 1995, Perkin-Elmer Corporation.

ELAN® 6000 Method 6020 Quick Start Guide, 1996, Perkin-Elmer Corporation.

United States Environmental Protection Agency, SW-846 Method 6020, Revision 0, “Inductively Coupled Plasma-Mass Spectrometry,” September 1994, EPA Publication SW-846, Second Update.

United States Environmental Protection Agency, “Methods for the Determination of Metals in Environmental Samples - Supplement 1,” EPA-600/R-94-111, May 1994, Available at NTIS, PB 94-184942, Method 200.8.

Document Change History			
Revision No.	**Status[1] (I, R, C)**	**Effective Date**	**Changes Made**
0	I	03/25/08	Not applicable
1	R	09/23/13	Entire document reviewed and updated; reformatted to conform with TechLaw SOPs 01-01-04 and 11-07-00

[1] Status: I = Initial, R= Revision, or C = Cancelled

Appendix I
Table of Isotopes Monitored and Equations

Analyte	Symbol	Isotopes Monitored	Correction Equations
Aluminum	Al	27	
Antimony	Sb	121,123	Sb 123 = Sb 123 - 0.127189 * Te 125
Arsenic	As	75	As 75 = As 75 - 3.127 * [ArCl 77 - (0.815*Se 82)]
Barium	Ba	135,137	
Beryllium	Be	9	
Cadmium	Cd	106,108,111,114	Cd 111 = Cd 111 - 1.073 * [MoO 108 - (0.712*Pd 106)] Cd 114 = Cd 114 - 0.026826 * Sn 118
Chromium	Cr	52,53	
Cobalt	Co	59	
Copper	Cu	63,65	
Lead	Pb	206,207,208	Pb 208 = Pb 208 + 1* Pb 206 + 1* Pb 207
Manganese	Mn	55	
Nickel	Ni	60,62	
Silver	Ag	107,109	
Thallium	Tl	203,205	
Zinc	Zn	66,67,68	
Internal Standards			
Lithium	Li	6	
Scandium	Sc	45	
Yttrium	Y	89	
Rhodium	Rh	103	
Indium	In	115	
Terbium	Tb	159	
Bismuth	Bi	209	
Germanium	Ge	72	
(Information	Only)		
Molybdenum	Mo	95,97,98	Mo 98= Mo 98 - 0.110588 * Ru 101
Selenium	Se	77,82	Se 82 = Se 82 - 1.008696 * Kr 83
Thorium	Th	232	
Uranium	U	238	

Vanadium	**V**	**51**	**V 51 = V 51 - 3.127*[ClO 53 - (0.113*Cr 52)]**
Calcium	**Ca**	**44**	
Magnesium	**Mg**	**24**	
Sodium	**Na**	**23**	
Potassium	**K**	**39**	
Iron	**Fe**	**54**	**Fe 54 = Fe 54 - 0.028226 * Cr 52**

Appendix II
QC Criteria – ICP-MS

QC Sample	Limits	Action
ICV	**90-110%**	**Recalibrate the instrument and reanalyze**
CCV/SCV	**90-110%**	**Recalibrate the instrument and reanalyze all samples since last acceptable CCV**
ICSA	**80-120% for Al, Mo** **Other analytes present < PQL** **Analytes > PQL**	**Recalibrate the instrument and reanalyze** **Evaluate data (consult Analytical Lead) and comment in case narrative**
ICSAB	**80-120% for all analytes in the spike**	**Recalibrate the instrument and reanalyze**
CRDL	**70-130%** **50-150% for Pb, Sb,Tl**	**Recalibrate the instrument and reanalyze** **Monitored but no corrective action for Al,Ca,Fe,Mg and K**
MB, ICB, CCB	**Results < 2x PQL**	**Reanalyze all samples with levels greater than 2 times PQL for ICB, CCB. If blank >PQL but <2xPQL, raise MRL to applicable level.** **For MB, reprep samples for concentrations >2x PQL. Results are valid if analyte concentration exceeds 5 times level detected in the blank.**
BS/SRM	**85-115% Aqueous** **Soil SRM varies per manufacturer certification**	**Reprepare BS for aqueous and analyze, reprepare/digest LCS and samples associated w/ failed SRM.**
Matrix Spike **Post Spike (Optional)**	**70-130%** **85-115%**	**Note in the Case Narrative that potential matrix interferences exist. Results are valid if native analyte concentration exceeds 4 times the spike concentration.** **Consult Analytical Lead for data acceptability and qualification** **No corrective action for failed post spike - verification purposes only**
Sample Duplicate RPD	**20% Aqueous** **35% Soils**	**Applicable for original results >5X PQL.** **Consult Analytical Lead for acceptability**
Serial Dilution	**90-110%**	**Applicable for original results >10X PQL**

		Consult Analytical Lead for acceptability.
Internal Standards	**60-125%**	**Dilute sample and reanalyze**

16-MET-02.02
ANALYSIS OF TRACE METALS USING THE PERKIN ELMER OPTIMA 4300DV® ICP-OE

Technical Approval: Scott C. Walker
PRINT NAME

SIGNATURE Date: 9/23/2013

QA Management Approval: Nikki MacDonald
PRINT NAME

SIGNATURE Date: 09/23/13

Table of Contents

1.0 SOP Description

This standard operating procedure (SOP) describes the daily operation, optimization, calibration, routine maintenance and analysis procedures for the analysis of samples according to SW-846 Method 6010 and United States Environmental Protection Agency (EPA) Method 200.7 using the Perkin Elmer Optima 4300DV® (referred to as Optima) Inductively Coupled Plasma-Optical Emission (ICP-OE).

An aqueous sample is nebulized into a spray chamber where a stream of argon carries the sample aerosol through a quartz torch and injects it into radio frequency plasma. Once in the plasma, the sample is decomposed and desolvated. The individual atoms produced emit discrete spectral lines characteristic to each element. The spectral line intensities for the elements of interest are measured and compared to the intensities resulting from similar analysis of standard solutions containing known amounts of the elements of interest. The quantity and identity of the unknown analytes are determined from a designated spectral line comparison. This method is applicable to the measurement of like aqueous matrices. The method further defines general instrument operation protocols.

This method is applicable to the following sample matrices analyzed by the Region 8 Environmental Services Assistance Team (ESAT): ground waters, surface waters, industrial wastes, sludges, and soil samples. These operational guidelines are to be used by an experienced operator to prepare the instrument for optimum performance.

2.0 Acronyms

ASTM	American Society for Testing and Materials
BS	Blank Spike
CCB	Continuing Calibration Blank (QC sample)
CCV	Continuing Calibration Verification (QC sample)
CDOC	Continuing Demonstration of Capability
CHP	Chemical Hygiene Plan
CRDL	Contract Reporting Detection Limit
EPA	United States Environmental Protection Agency
ESAT	Environmental Services Assistance Team
HCl	Hydrochloric Acid
HNO_3	Nitric Acid
ICB	Initial Calibration Blank (QC sample)
ICP-OE	Inductively Coupled Plasma-Optical Emission
ICSA(B)	Interference Check Sample (QC sample)
ICV	Initial Calibration Verification (QC sample)
IEC	Inter-Element Correction
IDL	Instrument Detection Limit
IDOC	Initial Demonstration of Capabilities
IS	Internal Standard
LCS	Laboratory Control Sample (QC sample)
LIMS	Laboratory Information Management System
MB	Method Blank
MDL	Method Detection limit
MS	Matrix Spike
PC	Personal Computer
PS	Post-digestion Spike

PT	Proficiency Testing
PQL	Practical Detection Limit
QA	Quality Assurance
QC	Quality Control
SCV	Secondary Calibration Verification
SOP	Standard Operating Procedure
SRM	Standard Reference Material
TDF	Technical Directive Form

3.0 Health and Safety

3.1 All pertinent procedures outlined in the EPA Region 8 Chemical Hygiene Plan (CHP) will be followed in performance of ICP-OE analyses. The use of laboratory equipment and chemicals exposes the analyst to several potential hazards. Good laboratory technique and safety practices must be followed at all times.

3.2 Safety glasses and a lab coat must be worn at all times when handling samples, reagents, or when in the vicinity of others handling these items.

3.3 Liquid argon represents a potential cryogenic hazard and safe handling procedures must be used at all times when handling liquid argon tanks.

3.4 The Optima is fully interlocked to protect the user from dangers such as high voltages, radio frequency generators, and intense ultraviolet light. At no time should the operator attempt to disable these interlocks or operate the Optima if any safety interlock is known to be disabled or malfunctioning.

3.5 Spilled samples, reagents, and water must be cleaned up from instrument and autosampler surfaces immediately. In the case of acid spills, the acid must be neutralized with acid spill kits available in the laboratory.

3.6 Solutions prepared for ICP analysis are normally percentage level in mineral acid concentration and can contain inorganic elements known to be hazardous to health. Normal laboratory requirements for protection against these hazards must be followed. At a minimum, this includes wearing protective eyewear, protective gloves, and laboratory coats while handling the solutions.

4.0 Cautions

This Standard Operating Procedure (SOP) provides guidelines for ICP-OE analyses. Following it will result in data of known quality, and is acceptable for most environmental analyses. However, situations may arise in which changes are required. Undocumented non-compliance with the protocols and procedures included in this SOP may lead to challenges to the validity of data produced. Any deviation from the SOP shall be addressed in the technical narrative prepared as part of the data report.

5.0 Interferences

Interferences in the determination of elemental concentrations by ICP-OE may occur for a variety of reasons. Spectral peak overlap between an interferent and analyte and background shifts may result in false readings. Unusual physical properties of some sample solution matrices may

adversely affect uptake rates and proper nebulization and presentation of the solutions to the plasma torch. Evaluations of this effect are normally incorporated in the quality assurance/quality control (QA/QC) protocols of the specific method incorporated for sample analysis.

6.0 Personnel Qualifications

The use of this SOP is limited to the spectroscopist's knowledge of the production and evaluation of spectroscopic data. Additionally, the ICP-OE operator will be familiar with routine chemical laboratory procedures and well-versed in the discipline of chemistry. Generally, this requires a degree in chemistry and at least one year of experience operating an ICP-OE instrument under an experienced spectroscopist's supervision.

7.0 Equipment and Supplies

7.1 Perkin Elmer Optima 4300DV® High Resolution ICP-OE system, which includes Optima instrument, computer system, WinLab32™ software, printer, and autosampler.

7.2 Peristaltic pump tubing:

- Black/Black – 0.76 mm i.d. (for sample introduction)
- Yellow/Orange – 0.51 mm i.d. (for internal standard introduction)
- Red/Red – 1.14 mm i.d. (for rinse station and drain)

7.3 T-connector, polypropylene, 1/16 inch i.d. (Cole Parmer Part Number: H-06365-77) for hooking up on-line addition of internal standards.

7.4 Calibrated mechanical pipettes:

- 10 - 100 µL
- 100 - 1000 µL
- 1000 - 5000 µL
- 5000 µL - 10,000 µL

7.5 Metal-free plastic pipette tips (for the pipettes specified in Section 7.4)

7.6 Argon Gas – Argon from gas pressure cylinders is not recommended by the manufacturer; however, the use of argon from Dewar (cryogenic) containers is suggested.

8.0 Reagents and Standards

8.1 Nitric acid (HNO_3), concentrated, suitable for trace metals analyses.

8.2 Hydrochloric acid (HCl), concentrated, suitable for trace metals analyses.

8.3 Reagent water equivalent to American Society for Testing and Materials (ASTM) Type I water (ASTM D1193).

8.4 Single element stock solutions of high purity grade. Suggested suppliers include: High Purity, Inorganic Ventures, and Spex.

8.5 Initial Calibration Verification (ICV) Standard

8.5.1 The ICV stock MUST BE from a source independent of that used for calibration. The concentrations of the analytes in the ICV are prepared near the mid-point of the calibration range and a concentration not used for calibration. Commercially prepared ICV stock solutions suitable for this method are available from several vendors. Suggested suppliers include: High Purity, Inorganic Ventures, and Spex.

8.5.2 ESAT ICV Preparation

8.5.2.1 Obtain a clean, dry 100 mL volumetric flask with stopper.
8.5.2.2 Add approximately 50 mL of E-Pure water and 2 mL of concentrated HNO_3 to the flask.
8.5.2.3 Stopper and swirl to mix.
8.5.2.4 Pipet 1.0 mL each of Inorganic Ventures multi-element standards QCP-QCS-1 and QCP-QCS-2 into the flask.
8.5.2.5 Bring to volume with E-Pure water.
8.5.2.6 Stopper and swirl to mix.
8.5.2.7 Record the preparation in the appropriate standard logbook.

Element	Conc. µg/L
Ag	250
Al	1000
As	2000
B	1000
Ba	1000
Be	1000
Ca	1000
Cd	1000
Co	1000
Cr	1000
Cu	1000
Fe	1000
K	5000
Mg	1000
Mn	1000
Mo	1000
Na	1000
Ni	1000
Pb	2000
Sb	2000
Se	1000
SiO_2	5000
Sr	1000
Ti	1000
Tl	5000
V	1000
Zn	1000

8.6 Interference Check Solutions ICSA and ICSAB

8.6.1 The ICSA contains interfering elements that will provide an adequate test for inter-element correction (IEC) factors applied in the analytical method. The ICSAB contains the interfering elements, as well as elements with known concentrations.

8.6.2 ESAT ICSA Preparation

8.6.2.1 Obtain a clean, dry 100 mL volumetric flask.
8.6.2.2 Add approximately 50 mL of E-Pure water to the flask.
8.6.2.3 Add 2 mL of concentrated HNO_3 to the flask.
8.6.2.4 Stopper and swirl to mix.
8.6.2.5 Pipet 2.0 mL of Inorganic Ventures standard 2007ICS-4 into the flask.
8.6.2.6 Stopper and swirl to mix.

8.6.3 ESAT ICSAB Preparation

8.6.3.1 Obtain a clean, dry 100 mL volumetric flask.
8.6.3.2 Add approximately 50 mL of E-Pure water to the flask.
8.6.3.3 Add 2 mL of concentrated HNO_3 to the flask.
8.6.3.4 Stopper and swirl to mix.
8.6.3.5 Pipet 2.0 mL of Inorganic Ventures standard 2007ICS-4 (or current standard) into the flask.
8.6.3.6 Pipet 0.1 mL each of Inorganic Ventures standards 2007ICS-1, 2007ICS-2, 2007ICS-3 (or current standards) and 1,000 mg/L Sr into the flask.
8.6.3.7 Stopper and swirl to mix.

Element	ICSA Conc. µg/L	ICSAB Conc. µg/L
Ag	0	300
Al	60,000	60,000
As	0	1000
B	0	500
Ba	0	300
Be	0	100
Ca	300,000	300,000
Cd	0	300
Co	0	300
Cr	0	300
Cu	0	300
Fe	250,000	250,000
K	0	20,000
Mg	150,000	150,000
Mn	0	200
Mo	0	300
Na	50,000	50,000
Ni	0	300
Pb	0	1000
Sb	0	1000
Se	0	500
SiO_2	0	490
Sr	0	1000
Ti	0	1000
Tl	0	1000
V	0	300
Zn	0	300

8.7 ESAT ICP Calibration Solution

8.7.1 The calibration solution is prepared from single element standards.
8.7.2 To a 200 mL volumetric flask, add approximately 50 mL of E-pure water and 4 mL of concentrated HNO_3.
8.7.3 Pipet the volumes of the individual standards into the flask as indicated in the table below. Bring to volume with E-pure water.
8.7.4 Record the preparation of this standard in the appropriate logbook.

Element	Vol. (µL)	Std. Conc. mg/L	Calib. Conc. mg/L
Ag	100	1000	0.5
Al	500	10,000	25
As	1000	1000	5
B	2000	1000	10
Ba	200	1000	1
Be	200	1000	1
Ca	500	10,000	25
Cd	200	1000	1
Co	200	1000	1
Cr	1000	1000	5
Cu	400	1000	2
Fe	500	10,000	25
K	1000	10,000	50
Mg	500	10,000	25
Mn	400	1000	2
Mo	200	1000	1
Na	500	10,000	25
Ni	1000	1000	5
Pb	1000	1000	5
Sb	1000	1000	5
Se	1000	1000	5
SiO_2	4000	1000	20
Sr	200	1000	1
Ti	200	1000	1
Tl	1000	1000	5
V	400	100	2
Zn	1000	1000	5

8.8 ESAT Continuing Calibration Verification (CCV) Standard

8.8.1 The CCV is prepared by diluting the calibration standard by a factor of 2X with the 1% HNO_3 calibration blank.
8.8.2 Prepare this solution as needed for daily analysis.

8.9 ESAT Contract Reporting Detection Limit (CRDL) Standard

8.9.1 The CRDL is prepared daily by diluting the CRDL stock solution with calibration blank.
8.9.2 Prepare the CRDL stock by adding approximately 25 mL of E-pure water and 2 mL

of concentrated HNO_3 to a 100 mL volumetric flask.

8.9.3 Pipet the volumes of the individual standards into the flask as indicated in the table below. Bring to volume with E-pure water.

CRDL Stock Solution

Element	Vol. (µL)	Std. Conc. mg/L	CRDL Stock µg/L
Ag	100	1000	1000
Al	100	10,000	10,000
As	500	1000	5000
B	2500	1000	25,000
Ba	100	1000	1000
Be	50	1000	500
Ca	250	10,000	25,000
Cd	100	1000	1000
Co	100	1000	1000
Cr	100	1000	1000
Cu	100	1000	1000
Fe	1000	1000	10,000
K	1000	10,000	100,000
Mg	1000	10,000	100,000
Mn	100	1000	1000
Mo	100	1000	1000
Na	1000	10,000	100,000
Ni	100	1000	1000
Pb	300	1000	3000
Sb	500	1000	5000
Se	1000	1000	10,000
SiO_2	2500	1000	25,000
Sr	100	1000	1000
Ti	200	1000	5000
Tl	500	1000	5000
V	500	1000	5000
Zn	500	1000	5000

8.9.4 Prepare the daily CRDL standard by diluting the CRDL stock 100X with calibration blank. The standard procedure is to dilute 100 µL of the stock to 10 mL.

8.9.5 This solution is prepared daily.

8.10 ICP Internal Standard (IS) Solution, 6.0 mg/L Scandium

8.10.1 Prepare the IS solution by adding approximately 250 mL of E-pure water and 5 mL of concentrated HNO_3 to a 500 mL volumetric flask.

8.10.2 Pipet 3.0 mL of a 1,000 mg/L Sc standard into the flask.

8.10.3 Bring to final volume with E-pure water.

8.11 Calibration Blank, 1% HNO_3

8.11.1 Prepare 100 mL of 1% HNO_3 by adding approximately 50 mL of E-pure water to a 100 mL volumetric flask and adding 1.0 mL of concentrated HNO_3 to the flask.

Swirl to mix.

8.11.2 Bring to volume with E-pure water.

9.0 General Procedures

9.1 Instrument Preparation

9.1.1 Pump Tubing

9.1.1.1 Prior to igniting the plasma, the rinse, sample and internal standard pump tubing are replaced with new tubing or as needed.

9.1.1.2 Set the tubing tensions by manually starting the pump and observing the flow of E-Pure water through the individual lines.

9.1.1.3 After setting the tensions, use the software to stop the pump.

9.1.2 Instrument Warm-Up

9.1.2.1 The instrument is warmed-up for at least 30 minutes prior to calibration by igniting the plasma and aspirating rinse solution through the sample probe and internal standard probe.

9.1.2.2 Open the WinLab software by double-clicking on the icon.

Note: There is an off-line version of the software. Do not initiate that version of the software for analysis.

9.1.2.3 The auto sampler rinse pump will start. Fasten the pump tubing across the notched mounts and lift up on the tension handle to start the flow of rinse solution to the sample probe position.

9.1.2.4 Place the internal standard probe into the internal standard solution.

9.1.2.5 Click on FILE, choose OPEN, choose WORKSPACE and select ESAT_2006.1.3. This will load the analytical method for analysis.

9.1.2.6 Click on the PLASMA icon to open the dialog window.

9.1.2.7 Click on the PUMP icon in the dialog window. The peristaltic pump on the instrument will start.

9.1.2.8 Lift each probe in and out of their solution briefly and then watch the flow of solution through each and adjust the tension on the instrument pump as needed to ensure smooth, non-pulsing flow.

9.1.2.9 Observe the waste flow from the red-red tubing attached to the bottom of the spray chamber and ensure that a smooth, segmented flow exists.

9.1.2.10 Ignite the plasma by clicking the ON switch in the dialog box.

9.1.2.11 After the plasma ignites, the pump should automatically start. If not, initiate the pump by clicking the icon.

9.1.2.12 Close the plasma dialog box and allow the instrument to warm-up.

9.1.3 Hg Alignment of Instrument

9.1.3.1 Using the software controls, align the instrument using the Hg alignment procedure while aspirating rinse.

9.1.3.2 From the top menu, select TOOLS, choose SPECTROMETER CONTROL.

9.1.3.3 The Alignment dialog box will open.

9.1.3.4 Click the Hg Align box and then click OK.

9.1.3.5 After the alignment is complete, click the APPLY button.

9.1.4 Manganese Optimization of Radial and Axial Viewing Windows

9.1.4.1 Move the sample probe from the E-Pure water container to the 10 mg/L Mn solution.

9.1.4.2 Click the RADIAL button in the dialog box.
9.1.4.3 Click the ALIGN VIEW button.
9.1.4.4 After the Mn solution reaches the instrument, click OK to begin the radial view alignment.
9.1.4.5 When complete, close the spectral view window and click the APPLY button.
9.1.4.6 Move the sample probe from the 10 mg/L Mn solution to the 1 mg/L Mn solution container.
9.1.4.7 Using the software controls, perform the axial viewing optimization.
9.1.4.8 After the axial alignment is complete, move the sample probe to the rinse station of the autosampler.
9.1.4.9 Rinse the instrument for about 5 minutes prior to calibration.

9.2 Instrument Calibration and Analysis

9.2.1 Open either the Soil or Water sample information table template or a previously used sample information file.
9.2.2 Enter the sample information from the sample batch sheets into the sample information table.
9.2.3 Save the sample information table in the appropriate directory.
9.2.4 Using the “Auto Run” function of the software, designate the appropriate sample information file and the name and location of the associated data file that will be generated during the sample analysis.
9.2.5 Build the run list by clicking on the “Build List” button on the “Auto Run” tab and print this list by clicking the “Print List” button.
9.2.6 Load the calibration blank, calibration standard and QC samples as defined on the run list into the auto-sampler rack according to the positions specified on the run list.
9.2.7 Click the “Analyze All” button to initiate calibration. The analysis can be halted if any part of the QC samples does not meet requirements.

9.3 Troubleshooting

QA/QC sample compliance issues may require troubleshooting of some limited hardware and/or software components of the ICP-OE system.

9.3.1 Analyst troubleshooting will also include investigations of sample preparation processes and sample introduction problems.
9.3.2 Improper system operation troubleshooting is limited to sample introduction hardware components of the ICP-OE.
9.3.3 No troubleshooting of the power supply system is to be performed by ESAT, due to the high voltage hazards associated with that module.
9.3.4 Software troubleshooting may be performed by instrument operators. Problems with sample solutions, QC or calibration solutions will be addressed by the instrument operator.
9.3.5 Any hardware, software, or solution chemistry problems should be cited in the instrument log book along with the measures taken to address the failure so that future occurrences can be more easily resolved.

9.4 Computer Hardware and Software

The computer interface with the current system is a Pentium class personal computer (PC) utilizing Windows operating system. Instrument control of the Optima is facilitated through WinLab32™ software. ESAT has created methods within the software for specific EPA protocols which include window layouts, QC sample acceptance criteria, and instrument operating conditions.

9.5 Waste Minimization

Metals-laden waste volumes are minimized by the use of a dedicated waste receptacle in which no other laboratory waste is placed. Waste concentrations are minimized by judicious use of metals standard solutions and materials. General waste disposal regulations and policies are included in the CHP and must be strictly followed.

10.0 Data and Records Management

All instrument printouts, worksheets, and associated documentation must be filed in the final report folder. Copies of the documentation may be required in submitted data reports. Any maintenance performed should be recorded in the Perkin Elmer Optima 4300 ICP-OE Instrument Logbook.

All calculations necessary to convert raw data (emission counts/second) are performed by the WinLab32™ software. The calculated quantities are selected by choosing the desired options in the Method Preferences screen. Quality Control sample results may be checked using the QC Checking features in the WinLab32™ software.

10.1 Following completion of an analytical sequence, the electronic data file is copied from the instrument PC to the data server for Laboratory Information Management System (LIMS) upload.
10.2 Minimize the WinLab software and open the Data Manager software by double-clicking the icon.
10.3 The most recent data file will be at the top of the file list. Single-click on this file to highlight.
10.4 Click on the EXPORT icon at the top of the menu.
10.5 Click on the USE EXISTING DESIGN button and then click BROWSE.
10.6 Select the element.xpt file and click OPEN.
10.7 Click the NEXT button.
10.8 Click the ENABLE/DISABLE button. The analytical sequence associated with the data file will be displayed. Select the standards and samples that you want to export.

Note: If a CCV was rerun, both the initial and re-run will appear in the sequence. Select the correct one for the upload.

10.9 Click NEXT and then NEXT again.
10.10 Click on the button to uniquely name the data file.
10.11 Type in the name of the data file.
10.12 Click on the button next to C:\pe\Administrator\Reports.
10.13 Click on the button "…" to open the directory dialog box.
10.14 Select the drive the data file is to be transferred to.
10.15 Move the drop-down arrow until the Metals_Data_Files directory appears and double-click the directory.
10.16 Double-click the appropriate directory for the current year.

10.17 Select the appropriate project name from the directory list.
10.18 Click OK, click NEXT and click FINISH.
10.19 Click the EXPORT DATA button.
10.20 Click the FINISH button.

11.0 Quality Assurance and Quality Control

A summary of QC acceptability limits and associated corrective action is provided in Appendix I, QC criteria – ICP. The Instrument Detection Limit (IDL) and Method Detection Limit (MDL) procedures are also discussed in their associated SOP. Consult the applicable SOP for further details.

11.1 Initial Demonstration of Capability (IDOC)/Continuing Demonstration of Capability (CDOC)

11.1.1 The analyst will prepare a set of 4 digested water and soil Laboratory Control Standards (LCSs) and analyze them.
11.1.2 The LCS results must be with the current established quality control acceptance limits.
11.1.3 The analyst will perform a CDOC annually by either repeating the IDOC procedure or by analyzing a proficiency testing (PT) sample.
11.1.4 Specific details of the IDOC/CDOC requirements are described in ESAT SOP QAQ-02.xx (current version).

11.2 MDLs

11.2.1 The reference procedure for determination of MDLs is in Chapter 1, Section 5 of SW-846. Alternatively, specific protocols for MDL determination may be specified by the EPA by issuing a Technical Direction Form (TDF).
11.2.2 In order to determine the MDL in a matrix, the analytes should be spiked into the matrix of interest (e.g., a Method 3050 or Method 3051 sample preparation blank, ground water matrix, or other sample preparation procedure matrix) at a level that is three to five times the estimated MDL.
11.2.3 The spiked matrix is then carried through the entire sample preparation procedure.
11.2.4 The MDL is calculated by multiplying the standard deviation obtained from a minimum of three analyses of the matrix spike by the one-sided 99% confidence level t-statistic. The table of t-statistics can be found in Chapter 1 of SW-846. The procedure used in this SOP is to analyze the spiked matrix seven times and then multiplying the standard deviation of the seven measurements by 3.14 (the t-statistic for 7 samples).
11.2.5 MDLs should be re-determined whenever the following occurs:
- Annually
- Any change to the sample preparation procedure.
- Any significant change to the instrument (new detector or different sample introduction system used).

11.3 Linear Calibration Ranges

11.3.1 Calibrate the instrument, as described in Section 9.2.
11.3.2 Run a series of increasing concentration standards close to the upper linear range of the instrument. In order to use an established definition of linear range, the definition from EPA Method 200.7 will be used. In this reference, the linear range

is defined as the highest concentration where the measured value is within 10% of the actual prepared value of the standard.

11.3.3 The Upper Linear Dynamic Range should be re-determined every year or whenever one of the following occurs:

- A new detector is installed.
- The detector voltages are changed.
- A new PA tube is installed in the RF generator.
- A different sample introduction system (change in type of nebulizer or spray chamber type) is installed.
- Whenever a significant change in the instruments performance is observed.

11.4 ICV

11.4.1 Analyze immediately after calibration to verify calibration.
11.4.2 Required at the beginning of each analytical run.
11.4.3 The limits must be within ±5% of the stated value.
11.4.4 If the ICV is not within limits, the analysis should be terminated and the source of the problem identified before continuing.

11.5 Secondary Calibration Verification (SCV)/ CCV

11.5.1 The SCV is required before the analysis of any samples and the CCV is required to be run after every 10 samples.
11.5.2 The limits are ±10% of the true value.
11.5.3 The action to be taken upon failure of the limits is to stop the analysis and correct the problem. Samples run between CCVs not within limits must be rerun.

11.6 Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB)

11.6.1 The solution used is the calibration blank.
11.6.2 Analyze the CCB immediately after each ICV and CCV analysis.
11.6.3 The limits are ± Practical Quantitation Limit (PQL) for each element.
11.6.4 The action upon failure is to find and correct the cause of the out of control situation.
11.6.4.1 If blank level is greater than PQL but less than 2 times PQL, raise the MRL to applicable level. Verify client reporting requirements before raising levels.
11.6.4.2 All samples affected must be reanalyzed.
11.6.4.3 If failure occurs consistently, the MDL must be re-evaluated.

11.7 ICSA and ICSAB

11.7.1 Required at the beginning of each analytical run or every 12 hours, whichever is more frequent.
11.7.2 The ICSA limits are ± 20% of the true value for Al, Ca, Fe, Mg and Na.
11.7.3 The analytes not present in the ICSA should not be present as either a false positive or negative value greater than their respective PQL.
11.7.4 The ICSAB limits are ± 20% of the true value for all spiked analytes.

11.8 Method Blank (MB)

11.8.1 One MB must be prepared with each set of digested samples.
11.8.2 The MB for dissolved metals analysis is prepared by adding calibration blank to an autosampler tube and analyzing as the PB.
11.8.3 Corrective action is taken for detectable MB levels greater than ± PQL as follows:
11.8.3.1 If blank level is greater than PQL but less than 2 times PQL, raise the MRL to applicable level. Verify client reporting requirements before raising levels.
11.8.3.2 If Blank level is greater than 2 times the PQL, all samples associated with the batch should be re-prepared including digestion if applicable.

11.9 Method Blank Spike (BS)/Standard Reference Material (SRM)

11.9.1 Recommended one for each dissolved batch of 20 samples or less.
11.9.2 The recovery limits are 85-115%.
11.9.3 For solid samples the recovery limits for SRM vary by element and certification.
11.9.4 The dissolved BS is prepared by pipetting 0.1 mL each of the Salt Spike and QCP-QCS-3 into 10 mL of calibration blank.

11.10 Salt Spike Stock Solution

11.10.1 Add 10 mL of E-Pure reagent water and 1 mL of concentrated HNO_3 to a clean, dry 50 mL volumetric flask.
11.10.2 The Salt Spike is prepared by pipetting the following analytes as detailed in the table below in to the 50 mL volumetric flask.

Element	Vol. (mL)	Std. Conc. mg/L	Salt Spike mg/L
Al	5.0	10,000	1,000
Ca	5.0	10,000	1,000
Fe	5.0	10,000	1,000
K	5.0	10,000	1,000
Mg	5.0	10,000	1,000
Na	5.0	10,000	1,000
Sr	2.5	1,000	50

11.10.3 Bring to volume with E-Pure reagent water.
11.10.4 Typically, the Salt Spike is used to spike samples for dissolved metals analysis.

11.11 CRDL

11.11.1 One CRDL is required in each analytical run.
11.11.2 The limits are 70-130% recovery for most analytes (Sb, Pb, and Tl are 50-150%). The major cations (Al, Ca, Fe, Mg, Na, and K) are monitored without corrective action.
11.11.3 The action to be taken upon failure of the limits is to stop the analysis and correct the problem.
11.11.4 If sample analysis was allowed to continue following an unsuccessful CRDL, those samples must be rerun and the data would not be reportable.

11.12 Matrix Spike (MS)

11.12.1 One MS is required for 10% of the samples or less.

11.12.2 The recovery limits are 70-130%
11.12.3 The MS is prepared by pipetting 0.5 mL of each Inorganic Ventures standard WW-LFS-1 and WW-LFS-2 into the digestion vial with an aliquot of the parent sample and digesting as a sample using the appropriate reagents.
11.12.4 A MS for dissolved analysis is prepared by spiking a separate 10 mL aliquot of the parent sample with 100 µL each of the Salt Spike and QCP-QCS-3.

11.13 Post-Digestion Spike (PS; Optional)

11.13.1 A post-digestion spike may be analyzed with each analytical digestion batch and is reported for analytes that do not meet MS acceptance criteria.
11.13.2 Prepare the PS by spiking 10 mL of the parent sample digest with 100 µL each of the Salt Spike and QCP-QCS-3.
11.13.3 The recovery limits are 85-115%
11.13.4 There is no corrective action for failed post-spike criteria and is used for verification purposes only.

11.14 Serial Dilution

11.14.1 One serial dilution must be performed for each twenty samples or less of each matrix in a sample batch.
11.14.2 If the analyte concentration is within the linear range of the instrument and is a factor of at least 10 times the PQL, the analysis of a five-fold dilution of the sample must agree within ±10% of the original determination.
11.14.3 If the recovery is not within these limits, an interference must be suspected.

11.15 Sample Duplicate

11.15.1 Analyze one duplicate sample for every matrix in a batch of twenty samples or less.
11.15.2 A control limit of 20% Relative Percent Difference (RPD) should not be exceeded for aqueous samples and 35% for solid samples. Evaluation of acceptance criteria is valid for native sample concentrations greater than 5 times its PQL.
11.15.3 If the control limit is exceeded, the reason for the out of control situation should be corrected, and any samples analyzed during the out of control condition re-analyzed.

11.16 Internal Standards

11.16.1 The Internal standard scandium is automatically added to all calibration standards, samples and quality control standards to monitor the performance of the instrument and for matrix interference effects.
11.16.2 The recovery limit is 80-120% of the true value for axial and radial views as compared to that initially recovered in the calibration blank.
11.16.3 If the control limit is exceeded in any of the quality control samples, the problem must be corrected and all bracketed samples reanalyzed. The most likely cause for the exceedance will be the sample introduction system or the internal standard needs to be re-prepared.
11.16.4 If the limit is exceeded in the samples, dilute the samples until an acceptable limit is achieved. The most likely cause for the exceedance is sample matrix interferences.

12.0 References

Environmental Services Assistance Team, Region 8, *Health and Safety Plan,* current version.

Perkin Elmer Optima 4300DV® User's Guide.

Perkin Elmer WinLab32™ Software Manual.

United States Environmental Agency, Region 8, *Chemical Hygiene Plan,* current version.

United States Environmental Agency SW-846 Method 6010, Revision 2, December 1996.

United States Environmental Agency Method 200.7, Revision 4.4, 1994.

Document Change History			
Revision No.	**Status[1] (I, R, C)**	**Effective Date**	**Changes Made**
0	I	03/27/07	Not applicable
1	R	03/27/08	Entire document reviewed and updated
2	R	09/23/13	Entire document reviewed and updated; reformatted to conform with TechLaw SOPs 01-01-04 and 11-07-00

[1] Status: I = Initial, R= Revision, or C = Cancelled

Appendix I
QC Criteria – ICP

QC Sample	Limits	Action
ICV	**95-105%**	**Recalibrate the instrument and reanalyze**
CCV/SCV	**90-110%**	**Recalibrate the instrument and reanalyze all samples since last acceptable CCV**
ICSA	**80-120% for Al, Mo** **Other analytes present < PQL** **Analytes > PQL**	**Recalibrate the instrument and reanalyze** **Evaluate data (consult Analytical Lead) and comment in case narrative**
ICSAB	**80-120% for all analytes in the spike**	**Recalibrate the instrument and reanalyze**
CRDL	**70-130%** **50-150% for Pb, Sb,Tl** **No criteria for Al,Ca,Fe,Mg,Na,K**	**Recalibrate the instrument and reanalyze**
MB, ICB, CCB	**Results < PQL**	**Reanalyze all samples with levels greater than 2 times PQL for ICB, CCB. If blank >PQL but <2xPQL, raise MRL to applicable level.** **For MB, reprepare samples for concentrations >2x PQL. Results are valid if analyte concentration exceeds 5 times level detected in the blank.**
BS/SRM	**85-115%** **Soil varies per manufacturer certification**	**Reprepare BS for dissolved aqueous and analyze, reprepare/digest TRC BS and associated samples.** **Reprepare and digest samples associated with failed soil SRM.**

Matrix Spike **Post Spike (optional)**	**70-1** **30%** **85-115%**	**Note in the Case Narrative that potential matrix interferences exist. Results are valid if native analyte concentration exceeds 4 times the spike concentration.** **Consult Analytical Lead for data acceptability and qualification**
Sample Duplicate RPD	**20% Aqueous** **35% Soils**	**Applicable for original results >5X PQL** **Consult Analytical Lead for acceptability**
Serial Dilution	**90-110%**	**Applicable for original results >10X PQL** **Consult Analytical Lead for acceptability**
Internal Standards	**80-120%**	**Dilute and reanalyze samples** **Address problem for QA samples (sample introduction or reprepare IS)**

16-MET-04.01

Mercury Analysis of Solids, Sludge, and Biota by Thermal Decomposition Using NIC MA3000 Direct Mercury Analyzer

Technical Approval: Scott C. Walker
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Table of Contents

1.0 SOP Description

This standard operating procedure (SOP) is designed for determination of mercury in solids. Integration of thermal decomposition sample preparation and atomic absorption detection reduces the total analysis time of most samples to less than five minutes. Total mercury in soils, sediments, bottom deposits, sludge-type, and biota material can be determined without sample chemical pretreatment using this method. Alternatively, this method can be used for the detection of total mercury from total decomposition sample preparation methods, such as United States Environmental Protection Agency (EPA) Method 3052, or for the detection of extracted or leached mercury compounds or species from methods such as SW-846 3000 series. This procedure is applicable to samples analyzed by the Region 8 Environmental Services Assistance Team (ESAT).

Controlled heating in an oxygenated decomposition furnace is used to liberate mercury from solid and aqueous samples in the instrument. The sample is dried and then thermally and chemically decomposed within the decomposition furnace. The decomposition products are carried by flowing oxygen to the catalytic section of the furnace. Here, oxidation is completed, and halogens and nitrogen/sulfur oxides are trapped. The remaining decomposition products are then carried to an amalgamator that selectively traps mercury. After the system is flushed with oxygen to remove any remaining gases or decomposition products, the amalgamator is rapidly heated, releasing mercury vapor. Flowing oxygen carries mercury vapor through absorbance cells positioned in the light path of a single wavelength atomic absorption spectrophotometer. Absorbance (peak height or peak area) is measured at 253.7 nm as a function of mercury concentration.

2.0 Acronyms

CCB	Continuing Calibration Blank
CCV	Continuing Calibration Verification
CHP	Chemical Hygiene Plan
°C	Degrees Celsius
DI	Deionized
EPA	United States Environmental Protection Agency
ESAT	Environmental Services Assistance Team
g	Grams
H_2SO_4	Sulfuric Acid
Hg	Mercury
$Hg(NO_3)_2$	Mercury (II) Nitrate
HNO_3	Nitric Acid
ICB	Initial Calibration Blank
ICV	Initial Calibration Verification
ID	Identification
LCS	Laboratory Control Sample
LIMS	Laboratory Information Management System
mg/L	Milligram per liter
mL	Milliliter
MS	Matrix Spike
MSD	Matrix Spike Duplicate
ng	Nanogram
PQL	Practical Quantitation Limit
QC	Quality Control
QA	Quality Assurance
RPD	Relative Percent Difference
SRM	Standard Reference Material

SOP	Standard Operating Procedure
TDF	Technical Direction Form
ug/L	Micrograms per liter
nm	Nanometer
uL	Microliter

3.0 Health and Safety

All pertinent procedures outlined in the EPA Region 8 Chemical Hygiene Plan (CHP) will be followed during the analysis of mercury samples with the following special precautions:

- Many mercury compounds are highly toxic if swallowed, inhaled, or absorbed though the skin. Extreme care must be exercised in the handling of concentrated mercury reagents. Concentrated mercury reagents should only be handled by analysts knowledgeable of their risks and with safe handling procedures. Safety glasses, laboratory coats and gloves must be worn while handling these materials.

- The generation and evolution of ground state mercury vapor must be performed in a well ventilated area. The instrument analytical gas stream outlet must be vented to a hood or attached to a scrubber to remove mercury vapors. Adequate ventilation must be provided to ensure that the analyst is not exposed to any residual vapors.

4.0 Cautions

This SOP provides guidelines for thermal decomposition analyses. Following it will result in data of known quality, and is acceptable for most environmental analyses. However, situations may arise that require changes to the procedure. Undocumented, non-compliance with this SOP may lead to challenges to the validity of data produced. Any deviation from the SOP must be addressed in the technical narrative prepared as part of the data report.

Care must be taken to ensure that only dry mercury-vapor/carrier gas is introduced into the sample cell. The gas/liquid separation membrane must be checked, or replaced if necessary, before each set of analyses. Significant loss of gas flow, with corresponding loss of response signal, will begin to occur in as few as 30 sample cycles.

5.0 Interferences

In areas where mercury contamination is an existing problem, the background signal may be significantly increased.

Memory effects between analyses may be encountered when analyzing a sample of high mercury concentration (> 2000 ng) prior to analyzing one of low concentration (< 25 ng). Typically, to minimize memory effects, analyze samples in batches of low and high concentrations, always analyzing those of low concentration first. If this batch processing cannot be accomplished, a blank analysis with an extended decomposition time may be required following the analysis of a highly concentrated sample in order to limit memory effects.

Co-absorbing gases, such as free chlorine and certain organics, should not interfere due to the release of decomposition products by the decomposition furnace, removal of some decomposition products by the decomposition catalyst, and the selective entrapment of mercury vapor on the amalgamator.

6.0 Personnel Qualifications

The use of this SOP is restricted to analysts experienced in the use of the Nippon MA3000 Direct Mercury Analyzer. Each analyst must demonstrate the ability to generate acceptable results with this test method using the reference material. Refer to the current revision of SOP 16-QAQ-02.xx, *Analytical Chemist Demonstration of Capabilities*, for specific requirements.

7.0 Equipment and Supplies

- Nitric acid, concentrated: Reagent grade of low mercury content
- Chemical fume hood
- L-cysteine crystals
- Stock 1000 mg/L Mercury Solution: Dissolve 0.1354 g of mercuric chloride in 75 mL of distilled water. Add 10 mL concentrated nitric acid and adjust the volume to 100.0 mL with distilled water. A commercially available certified calibration stock solution may be substituted for the stock mercury solution. This solution expires one year after preparation or expiration of the purchased stock solution.
- Second Source Stock 5000 mg/L Solution: A commercially available certified calibration stock solution is purchased annually. This solution expires one year after receipt and must be from a source different from the Stock Mercury Solution.

Note: All reagents used should be checked for low mercury background prior to use in mercury analysis. It is advised to use reagents listed as suitable for mercury analysis by the manufacturer.

- Safety glasses, laboratory coats and gloves
- NIC MA3000 Analyzer: This system includes an autosampler and spectrophotometer monitoring 253.7 nm, and a personal computer for data handling.

8.0 Standards and Reagents

8.1 Calibrations Standards

A minimum of five calibration standard aliquots and a calibration blank will be analyzed with each digestion/analysis batch, with a linear regression coefficient for the concentration versus absorbance plot equal to or greater than 0.995. A typical calibration curve is provided in the tables below.

Low concentration mode

Vol of 10 ug/L Std added to Sample boat (uL)	ng of std added to sample boat	typical peak height
25	0.25	0.02
50	0.50	0.04
100	1.0	0.08
200	2.0	0.16
400	4.0	0.32
500	5.0	0.40

High concentration mode

vol of 1000 ug/L std added to sample boat	ng of std added to sample boat	typical peak height
10	10	20
20	20	40
50	50	95
100	100	180
200	200	360
400	400	650

8.2 Reagents

- Reagent Water – Reagent water will be interference free. All references to water in this method refer to reagent water unless otherwise specified.

- L-cysteine Solution – Add 10 mg L-cysteine crystals to one liter of reagent water. This solution is stable for one month if kept away from light.

- Stock Mercury Solution (1000 mg/L) – In 100 mL volumetric flask, dissolve 0.1354 g of mercuric chloride in 75 mL water. Add 1.0 mL concentrated nitric acid, bring to volume with water. A commercially available calibration stock solution may be substituted for the stock mercury solution. Do not use $Hg(NO_3)_2$ as a stock standard.

- Stock Mercury Solution 2 (1000 ug/L) – From stock mercury solution (1000 mg/L), prepare a 1:1000 dilution by placing 100 uL of stock mercury solution in a 100 mL volumetric flask and diluting to volume with L-cysteine solution.

- Working Mercury Solution (10 ug/L) – From mercury stock solution 2, prepare a 1:1000 dilution by placing 1.0 mL of stock mercury solution in a 100 mL volumetric flask and diluting to volume with L-cysteine solution.

- Standard Reference Material (SRM) – In place of aqueous mercury standards, solid reference material with a certified value for mercury may be used for calibration.

9.0 Sample Handling

9.1 Preservation Integrity

- Soil samples are stored at 4°C before preparation and analysis.
- Loss of analyte from soil samples may be attributed to, or accelerated by, changes in soil oxidation states and/or microbial action. Cold storage of the samples will help prevent these losses.

9.2 Contamination can occur when samples are stored in a mercury contaminated laboratory area (e.g., where a thermometer was broken or samples of high concentration were stored).

9.3 Holding Time – Analysis of mercury solid samples must be preformed within 28 days of sampling.

10.0 Sample Preparation

10.1 Pour 25 mL of each sample, standard, and quality control (QC) sample into a labeled 50 mL digestion tube. Use 1.0 g for soil samples and add 25 mL of reagent water for volume.

10.2 Spike the laboratory control sample (LCS), matrix spike (MS) and matrix spike duplicate (MSD) samples with 25 µL of the 5000 µg/L Internal Calibration Verification (ICV) stock standard. This will yield a spike level of 5.0 ug/L Hg.

10.3 Add 1.25 mL concentrated H_2SO_4, 0.625 mL concentrated HNO_3, 3.75 mL of 5% potassium permanganate and 2 mL of 5% potassium persulfate to each tube. These additions must be performed in a hood.

10.4 Place watch covers over the tubes and place in a preheated digestion block for 2 hours.

10.5 Remove tubes from digestion block and allow cooling for 30 minutes.

10.6 Add 1.5 mL 12% Sodium Chloride-Hydroxylamine Sulfate.

10.7 Swirl until all traces of coloration are gone.

10.8 Dilute to 50 mL and cap. Samples should be analyzed as soon as possible within 24 hours of digestion.

11.0 Sample Analysis

11.1 Ensure that the Flow Injection Mercury System (FIMS)-cell and tubing are properly installed. Check tubing for wear and replace as necessary.

11.2 Verify proper operation of the fume ventilation system.

11.3 Turn on the carrier gas supply and adjust pressure to 52 pounds per square inch.

Note: To save carrier gas, the gas flow does not need to be turned on until approximately five minutes before analyses are performed.

11.4 Switch on the FIMS and AS-90 auto-sampler.

11.5 Turn on the computer and start the Windows operating system. Double click on the Winlab icon to initialize the software and establish communication with the FIMS.

11.6 Open the desired work space and analytical method from the software.

11.7 Prepare a sample queue in Winlab by selecting *Sample Information* from the top menu bar in the software.

- Specify sample identification (ID) number, sample location, and dilution information.
- Each unique sample set will receive a project specific name and be saved as a unique sample queue.
- The analyst should print the sample queue as a guide to accurately load the auto-sampler stage.

11.8 Place the inlets of the carrier pump tube, reductant pump tube, and sampling tube in deionized (DI) water.

11.9 Start the peristaltic pump from the FIAS control window in the Winlab software.

11.10 Adjust the tension on the pump windings to ensure smooth flow of reagents through the tubing.

- Check all tubing for leaks, signs of damage, and proper connections.
- Any damaged tubing or associated fitting should be replaced prior to analysis.

11.11 Specify a file pathway to save the analytical information in the Automated Analysis control window in the Winlab software for data processing and review and for subsequent data

transfer to the Laboratory Information Management System (LIMS). Additionally, specify the range of sample locations to be included in the analytical run.

11.12 Remove the caps on the digestion tubes containing the samples and standards.

11.13 Load standards, QC solutions and samples onto the auto-sampler stage.

11.14 Place the inlets of the carrier pump tube, reductant pump tube, and sampling tube in their respective solutions, and allow sufficient time for the reagents to reach the reaction cell of the FIMS.

11.15 Click on the Reset button in the Automated Analysis Control window, and then click on the Analyze All button.

- The FIMS will analyze the blank, standards, QC solutions, and samples in the order specified in the method and the sample queue.
- No control functions, method modifications, or sample queue modification will be accessible in the software until the analytical run is completed or aborted.

11.16 After completion of the analytical run, place the inlets of the carrier pump tube, reductant pump tube, and sampling tube into DI water and allow the system to flush for a minimum of 10 minutes.

11.17 After the system is flushed, remove the tube inlets and place them into a dry container to allow the system to be pumped dry before instrument shutdown.

11.18 Once the tubing is pumped dry, shut off the peristaltic pump from the FIAS control window and loosen the pump windings from their respective cassettes.

11.19 Shut down the instrument in the reverse order in which it was turned on.

12.0 Troubleshooting

Troubleshooting of problems must address both the preparation and instrument measurement phases of the procedure. Problems arising from the preparation of samples include:

12.1 Apparent Loss of Analyte – Commonly attributable to loss of oxidizing conditions in the digestion step (see method instructions for addition of extra oxidizers).

12.2 Apparent Loss of Signal or Erratic Results

- For known analyte samples, check for signs of incomplete reactions in both reduction steps.
- All permanganate color and/or dark manganese residue must be completely eliminated by the hydroxylamine reagent.
- Erratic results may occur if the stannous solution is beginning to change phase (become a heavier suspension over time).

12.3 Incomplete Digestion – Organic or humic components may not digest completely. This may be alleviated by using a smaller sample aliquot or digestion of the material by an alternate method, and analysis of the resultant aqueous phase.

12.4 Instrument Drift – Decreasing sensitivity or excessive downward drift of response to known samples may be due to a leak in the gas/liquid separation membrane. Check/remove excess moisture in the sample outlet connection and replace the membrane as required.

13.0 Data Acquisition, Calculations, and Data Reduction

13.1 Data Acquisition – Analytical response data is recorded by the Winlab software as peaks representing excitation line absorbance. The header for each trial contains the sequence number of the set, date, and time of initiation of each measurement.

13.2 Calculations

- The sample absorbance versus time plot is recorded as a peak height.
- Peak height values for unknown samples are quantified by comparison with peak heights from known mercury masses in the standard solutions.
- Sample values and QC standard values are calculated by the software and obtained by comparison with the linear regression curve derived from the measurement of the standards.
- Values obtained for the samples or soil digestates are in µg/L.

13.3 Data Reduction – Instrument raw data is electronically transferred to the LIMS. The LIMS combines the raw instrument data with the sample information in the digestion batch in LIMS to calculate the final reportable value.

14.0 Computer Hardware and Software

The computer interface with the current system is a Pentium class personal computer utilizing Windows operating system. Instrument control of the FIMS is facilitated through Perkin-Elmer Winlab software.

15.0 Data and Records Management

All instrument printouts, worksheets, and associated documentation must be filed in the final report folder. Copies of the documentation may be required in submitted data reports. Any maintenance performed must be recorded in the Perkin-Elmer FIMS Instrument Logbook (document controlled).

16.0 Waste Minimization

Mercury laden waste volumes are minimized by the use of a dedicated mercury waste receptacle in which no other laboratory waste is placed. Waste concentrations are minimized by judicious use of mercury standard solutions and materials. General waste disposal regulations and policies are included in the CHP and must be strictly followed.

17.0 QA/QC

QC requirements may be detailed in cited analytical methods or specified by individual Technical Directive Form (TDFs). The minimum QC sample requirements for a mercury digestion/analysis batch include:

17.1 Calibration Standards – A minimum of four calibration standard aliquots and a calibration blank will be digested and analyzed with each digestion/analysis batch. The linear regression coefficient for the curve should be 0.995 or greater.

17.2 ICV/LCS – The recovery of the ICV and LCS should be 90%-110%.

17.3 Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB) – A reagent blank digested and analyzed following each calibration verification standard and at the end of the run. The CCB should be less than the practical quantitation limit (PQL).

17.4 Continuing Calibration Verification (CCV) – A calibration-midpoint drift check solution analyzed every ten samples and at the end of the run. The recovery should be 80%-120%.

17.5 Sample Duplicate – One sample prepared, digested, and analyzed in duplicate. At least one sample duplicate per matrix type will be analyzed with each digestion/analysis batch. The relative percent difference (RPD) between duplicates greater than the PQL should be 35% or less for soil samples.

17.6 MS/MSD – A mercury spiked solution added to a representative sample prior to sample preparation steps. At least one MS and MSD sample per matrix type will be analyzed with each digestion/analysis batch. The calculated recovery of the spike is used to evaluate sample matrix effects. The recovery limits for sample MS/MSDs are 75-125%. The RPD between the MS and MSD should be 20% or less for aqueous samples and 35% or less for soil samples.

17.7 Corrective Actions

- Failure to comply with quality assurance/QC requirements in steps 17.1 through 17.6 may require re-starting the analysis.
- Failure of any subsequent CCV sample will require re-analysis of any samples bracketed by the failed CCV.
- ICV and CCV failures due to slightly high recoveries may be accepted if no samples associated with these QC samples contain reportable amounts of mercury.

18.0 References

Environmental Services Assistance Team Region 8 Health and Safety Plan (current revision)

FIMS (Flow Injection Mercury System): Setting Up and Performing Analyses. Part Number 0993-5203, Publication B3118.20, Release 1.1/Mar.94, Perkin-Elmer Corporation

Flow Injection Mercury/Hydride Analyses: Recommended Analytical Conditions and General Information. Part Number B050-1820 Publication B3505.10 Release 4.0, Perkin-Elmer Corporation

United States Environmental Protection Agency, Mercury in Solid or Semi-solid Waste (manual cold-vapor technique) Method 7471A, EPA SW-846, Test Methods for Evaluating Solid Waste, Revision 1, Sept. 1994

United States Environmental Protection Agency, Mercury in Liquid Waste (manual cold-vapor technique) Method 7470A, EPA SW-846, Test Methods for Evaluating Solid Waste, Revision 1, Sept. 1994

United States Environmental Protection Agency Region 8 Chemical Hygiene Plan (current revision)

Document Change History			
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Sampling Equipment Decontamination

APPROVED:

ESAT Region 8 QA Coordinator — Date: 06/06/12

ESAT Region 8 Team Manager — Date: 6/6/12

EPA Task Order Project Officer — Date: 7/10/12

ESAT Region 8 Task Lead — Date: 6/8/12

DCN: EP8-7-7061

TABLE OF CONTENTS

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide a description of the methods used for preventing, minimizing, or limiting cross-contamination of samples due to inappropriate or inadequate equipment decontamination and to provide general guidelines for developing decontamination procedures for sampling equipment to be used during hazardous waste operations as per 29 Code of Federal Regulations (CFR) 1910.120. This SOP does not address personnel decontamination.

2.0 SCOPE AND APPLICABILITY

These are standard procedures which may be varied or changed as required, dependent upon site conditions, equipment limitation, or limitations imposed by the procedure. In all instances, the ultimate procedures employed should be documented and included with the final report. Mention of trade names or commercial products does not constitute Techlaw, Inc. endorsement or recommendation for use.

3.0 SUMMARY OF METHOD

Removing or neutralizing contaminants from equipment minimizes the likelihood of sample cross contamination, reduces or eliminates transfer of contaminants to clean areas, and prevents the mixing of incompatible substances. Gross contamination can be removed by physical decontamination procedures. These abrasive and non-abrasive methods include the use of brushes, air and wet blasting, and high and low pressure water cleaning.

The first step, a soap and water wash, removes all visible particulate matter and residual oils and grease. This may be preceded by a steam or high pressure water wash to facilitate residuals removal. The second step involves a tap water rinse and a distilled/deionized water rinse to remove the detergent. An acid rinse provides a low pH media for trace metals removal and is included in the decontamination process if metal samples are to be collected. It is followed by another distilled/deionized water rinse. If sample analysis does not include metals, the acid rinse step can be omitted.

Next, a high purity solvent rinse is performed for trace organics removal if organics are a concern at the site. Typical solvents used for removal of organic contaminants include acetone, hexane, or water. Acetone is typically chosen because it is an excellent solvent, miscible in water, and not a target analyte on the Priority Pollutant List. If acetone is known to be a contaminant of concern at a given site or if Target Compound List analysis (which includes acetone) is to be performed, another solvent may be substituted. The solvent must be allowed to evaporate completely and then a final distilled/deionized water rinse is performed. This rinse removes any residual traces of the solvent.

The decontamination procedure described above may be summarized as follows:

1. Physical removal
2. Non-phosphate detergent wash
3. Tap water rinse
4. Distilled/deionized water rinse
5. 10% nitric acid rinse
6. Distilled/deionized water rinse
7. Solvent rinse (pesticide grade)
8. Air dry
9. Distilled/deionized water rinse

If a particular contaminant fraction is not present at the site, the nine step decontamination procedure specified above may be modified for site specificity. For example, the nitric acid rinse may be eliminated if metals are not of concern at a site. Similarly, the solvent rinse may be eliminated if organics are not of concern at a site. Modifications to the standard procedure should be documented in the site-specific work plan or subsequent report.

4.0 ACRONYMS AND DEFINITIONS

CFR	Code of Federal Regulations
CRC	Contamination Reduction Corridor
CRZ	Contamination Reduction Zone
DOT	Department of Transportation
EPA	United States Environmental Protection Agency
ESAT	Environmental Services Assistance Team
EZ	Exclusion Zone
HASP	Health and Safety Plan
OSHA	Occupation Safety and Health Administration
PPE	Personal Protective Equipment
QAPP	Quality Assurance Project Plan
SAP	Sampling and Analysis Plan
SOP	Standard Operating Procedure
SZ	Safe Zone

Code of Federal Regulations (CFR): The codification of the general and permanent rules published in the Federal Register by the executive departments and agencies of the Federal Government

Contamination Reduction Corridor (CRC): An area between the exclusion zone and the safe zone where equipment goes through the decontamination process. The decontamination line is usually set up in the CRC.

Contamination Reduction Zone (CRZ): An area between the exclusion zone and contamination reduction corridor where preliminary decontamination activities occur.

Department of Transportation (DOT): A government agency that oversees and regulates transportation functions.

Exclusion Zone (EZ): The area at a site where work is being performed.

Health and Safety Plan (HASP): A site-specific document that identifies safety hazards and proper safety procedures. This normally includes hospital route maps and material safety data sheets.

Occupational Safety and Health Administration (OSHA): A regulatory agency that governs health and safety standards in the United States.

Personal Protective Equipment (PPE): Refers to protective clothing, helmets, goggles, or other garment designed to protect the wearer's body from injury by blunt impacts, electrical hazards, heat, chemicals, and infection, for job-related occupational safety and health purposes.

Safe Zone (SZ): An area at a site where work is not being performed. Equipment and personnel in the SZ should be considered contaminant free.

Standard Operating Procedure (SOP): A set of written instructions that document a routine or repetitive activity followed by an organization (EPA, 2007).

5.0 HEALTH AND SAFETY

When working with potentially hazardous materials, follow OSHA, U.S. Environmental Protection Agency (EPA), corporate, and other applicable health and safety procedures.

Decontamination can pose hazards under certain circumstances. Hazardous substances may be incompatible with decontamination materials. For example, the decontamination solution may react with contaminants to produce heat, explosion, or toxic products. Also, vapors from decontamination solutions may pose a direct health hazard to workers by inhalation, contact, fire, or explosion.

The decontamination solutions must be determined to be acceptable before use. Decontamination materials may degrade protective clothing or equipment; some solvents can permeate protective clothing. If decontamination materials do pose a health hazard, measures should be taken to protect personnel or substitutions should be made to eliminate the hazard. The choice of respiratory protection based on contaminants of concern from the site may not be appropriate for solvents used in the decontamination process.

Safety considerations should be addressed when using abrasive and non-abrasive decontamination equipment. Maximum air pressure produced by abrasive equipment could cause physical injury. Displaced material requires control mechanisms. Material generated from decontamination activities requires proper handling, storage, and disposal. Proper PPE may be required for these activities. Material safety data sheets are required for all decontamination solvents or solutions as required by the Hazard Communication Standard (i.e., acetone, alcohol, and trisodiumphosphate).

6.0 CAUTIONS

Any personnel who participate in decontamination activities in the field must familiarize themselves with the site decontamination plan, which is part of the site HASP. Furthermore, decontamination activities should be done so with proper PPE. The HASP should provide guidelines for required PPE in the field.

7.0 INTERFERENCES

The use of distilled/deionized water commonly available from commercial vendors may be acceptable for decontamination of sampling equipment provided that it has been verified by laboratory analysis to be analyte free (specifically for the contaminants of concern).

The use of an untreated potable water supply is not an acceptable substitute for tap water. Tap water may be used from any municipal or industrial water treatment system. If acids or solvents are utilized in

decontamination they raise health and safety, and waste disposal concerns. Damage can be incurred by acid and solvent washing of complex and sophisticated sampling equipment.

8.0 PERSONNEL QUALIFICATIONS

All personnel who participate in field activities are required to obtain clearance in three mandatory health and safety programs: medical monitoring, respirator fit testing, and OSHA Hazardous Waste Operations and Emergency Response (HAZWOPER) 40-hour training. In addition, any personnel who will participate in decontamination activities must read, understand, and sign the site-specific HASP and associated Sampling and Analysis Plan and Quality Assurance Project Plan (SAP/QAPP).

9.0 EQUIPMENT AND SUPPLIES

Decontamination equipment, materials, and supplies are generally selected based on availability. Other considerations include the ease of decontaminating or disposing of the equipment. Most equipment and supplies can be easily procured. For example, soft-bristle scrub brushes or long-handled bottle brushes can be used to remove contaminants. Large galvanized wash tubs, stock tanks, or buckets can hold wash and rinse solutions. Children's wading pools can also be used. Large plastic garbage cans or other similar containers lined with plastic bags can help segregate contaminated equipment. Contaminated liquid can be stored temporarily in metal or plastic cans or drums. The following standard materials and equipment are recommended for decontamination activities:

9.1 Decontamination Solutions

Non-phosphate detergent selected solvents, (acetone, hexane, nitric acid, etc.) Virkon® disinfectant virucide, tap water distilled, or deionized water.

9.2 Decontamination Tools/Supplies

Long and short handled brushes, bottle brushes, drop cloth/plastic sheeting, paper towels, plastic or galvanized tubs or buckets, pressurized sprayers (H_2O) solvent sprayers, and aluminum foil.

9.3 Health and Safety Equipment

Safety glasses or splash shield, appropriate gloves, aprons or coveralls, respirator, and emergency eye wash.

9.4 Waste Disposal

Trash bags, trash containers, 55-gallon drums, metal/plastic buckets/containers for storage and disposal of decontamination solutions

10.0 STANDARDS AND REAGENTS

There are no reagents used in this procedure aside from the actual decontamination solutions. Table 1 lists solvent rinses which may be required for elimination of particular chemicals. In general, the following solvents are typically utilized for decontamination purposes: 10% nitric acid is typically used for

inorganic compounds such as metals. An acid rinse may not be required if inorganics are not a contaminant of concern. Acetone, hexane, and methanol are used for organic compound decontamination. A solvent may not be required if organics are not a chemical of concern. Virkon® is used for decontamination of non-disposable PPE (see section 11.3 Personal Protective Equipment Decontamination).

11.0 PROCEDURES

As part of the HASP, a decontamination plan should be developed and reviewed. The decontamination line should be set up before any personnel or equipment enters the areas of potential exposure. The equipment decontamination plan should include:

- The number, location, and layout of decontamination stations
- Decontamination equipment needed
- Appropriate decontamination methods
- Methods for disposal of contaminated clothing, equipment, and solutions

Procedures can be established to minimize the potential for contamination. This may include: (1) work practices that minimize contact with potential contaminants; (2) using remote sampling techniques; (3) covering monitoring and sampling equipment with plastic, aluminum foil, or other protective material; (4) watering down dusty areas; (5) avoiding laying down equipment in areas of obvious contamination; and (6) use of disposable sampling equipment.

11.1 Decontamination Methods

All samples and equipment leaving the contaminated area of a site must be decontaminated to remove any contamination that may have adhered to equipment. Various decontamination methods will remove contaminants by: (1) flushing or other physical action, or (2) chemical complexing to inactivate contaminants by neutralization, chemical reaction, disinfection, or sterilization.

Physical decontamination techniques can be grouped into two categories: abrasive methods and non-abrasive methods, as follows:

11.1.1 Abrasive Cleaning Methods

Abrasive cleaning methods work by rubbing and wearing away the top layer of the surface containing the contaminant. The mechanical abrasive cleaning methods are most commonly used at hazardous waste sites. The following abrasive methods are available:

<u>Mechanical</u>
Mechanical methods of decontamination include using metal or nylon brushes. The amount and type of contaminants removed will vary with the hardness of bristles, length of time brushed, degree of brush contact, degree of contamination, nature of the surface being cleaned, and degree of contaminant adherence to the surface.

<u>Air Blasting</u>
Air blasting equipment uses compressed air to force abrasive material through a nozzle at

high velocities. The distance between nozzle and surface cleaned, air pressure, time of application, and angle at which the abrasive strikes the surface will dictate cleaning efficiency. Disadvantages of this method are the inability to control the amount of material removed and the large amount of waste generated.

Wet Blasting
Wet blast cleaning involves use of a suspended fine abrasive. The abrasive/water mixture is delivered by compressed air to the contaminated area. By using a very fine abrasive, the amount of materials removed can be carefully controlled.

11.1.2 Non-Abrasive Cleaning Methods

Non-abrasive cleaning methods work by forcing the contaminant off a surface with pressure. In general, the equipment surface is not removed using non-abrasive methods.

Low-Pressure Water
This method consists of a container which is filled with water. The user pumps air out of the container to create a vacuum. A slender nozzle and hose allow the user to spray in hard-to-reach places.

High-Pressure Water
This method consists of a high-pressure pump, an operator controlled directional nozzle, and a high-pressure hose. Operating pressure usually ranges from 340 to 680 atmospheres (atm) and flow rates usually range from 20 to 140 liters per minute.

Ultra-High-Pressure Water
This system produces a water jet that is pressured from 1,000 to 4,000 atm. This ultra-high-pressure spray can remove tightly-adhered surface films. The water velocity ranges from 500 meters/second (m/s) (1,000 atm) to 900 m/s (4,000 atm). Additives can be used to enhance the cleaning action.

Rinsing
Contaminants are removed by rinsing through dilution, physical attraction, and solubilization.

Damp Cloth Removal
In some instances, due to sensitive, non-waterproof equipment or due to the unlikelihood of equipment being contaminated, it is not necessary to conduct an extensive decontamination procedure. For example, air sampling pumps hooked on a fence, placed on a drum, or wrapped in plastic bags are not likely to become heavily contaminated. A damp cloth should be used to wipe off contaminants which may have adhered to equipment through airborne contaminants or from surfaces upon which the equipment was set.

Disinfection/Sterilization
Disinfectants are a practical means of inactivating infectious agents. Unfortunately, standard sterilization methods are impractical for large equipment. This method of decontamination is typically performed off-site.

11.2 Field Sampling Equipment Decontamination Procedures

The decontamination line is setup so that the first station is used to clean the most contaminated item. It progresses to the last station where the least contaminated item is cleaned. The spread of contaminants is further reduced by separating each decontamination station by a minimum of three feet. Ideally, the contamination should decrease as the equipment progresses from one station to another farther along in the line.

A site is typically divided up into the following boundaries: Hot Zone or Exclusion Zone (EZ), the Contamination Reduction Zone (CRZ), and the Support or Safe Zone (SZ). The decontamination line should be setup in the Contamination Reduction Corridor (CRC) which is in the CRZ. Figure 1 shows a typical contaminant reduction zone layout. The CRC controls access into and out of the exclusion zone and confines decontamination activities to a limited area. The CRC boundaries should be conspicuously marked. The far end is the hotline, the boundary between the exclusion zone and the contamination reduction zone. The size of the decontamination corridor depends on the number of stations in the decontamination process, overall dimensions of the work zones, and amount of space available at the site. Whenever possible, it should be a straight line.

Anyone in the CRC should be wearing the level of protection designated for the decontamination crew. Another corridor may be required for the entry and exit of heavy equipment. Sampling and monitoring equipment and sampling supplies are all maintained outside of the CRC. Personnel don their equipment away from the CRC and enter the exclusion zone through a separate access control point at the hotline. One person (or more) dedicated to decontaminating equipment is recommended.

11.2.1 Decontamination Setup

Starting with the most contaminated station, the decontamination setup should be as follows:

Station 1: Segregate Equipment Drop
Place plastic sheeting on the ground (Figure 2). Size will depend on amount of equipment to be decontaminated. Provide containers lined with plastic if equipment is to be segregated. Segregation may be required if sensitive equipment or mildly contaminated equipment is used at the same time as equipment which is likely to be heavily contaminated.

Station 2: Physical Removal with A High-Pressure Washer (Optional)
Fill a low-pressure sprayer with distilled/deionized water. Provide a 5-gallon bucket or basin to contain the rinsate. A high-pressure wash may be required to remove compounds which are difficult to remove by washing with brushes. High pressure washers require water and electricity.

A decontamination pad may be required for the high-pressure wash area. An example of a wash pad may consist of an approximately 1 ½ foot deep basin lined with plastic sheeting and sloped to a sump at one corner. A layer of sand can be placed over the plastic and the basin is filled with gravel or shell. The sump is also lined with visqueen

and a barrel is placed over the hole to prevent collapse. A sump pump is used to remove the water from the sump for transfer into a drum.

Station 3: Physical Removal with Brushes and a Wash Station
Prior to setting up station 3, place plastic sheeting on the ground to cover the areas under station 3 through station 10.

Fill a wash basin or large bucket with non-phosphate detergent soap and tap water. Several bottle and bristle brushes to physically remove contamination should be dedicated to this station. Approximately 10-50 gallons of water may be required initially depending on the amount of equipment to decontaminate and the amount of gross contamination.

Station 4: Low-Pressure Sprayers
Fill a low-pressure sprayer with distilled/deionized water. Provide a 5-gallon bucket or basin to contain the rinsate.

Station 5: Nitric Acid Sprayers
Fill a spray/squeeze bottle with 10% nitric acid. This procedure is useful only for inorganic contaminants. Provide a 5-gallon bucket or basin to collect acid during the rinsing process.

Station 6: Organic Solvent Sprayers
Fill a spray/squeeze bottle with an organic solvent. After each rinse, the equipment should be rinsed with distilled/deionized water and dried. Amount of solvent will depend on the amount of equipment to decontaminate. Provide a bucket or basin to contain the rinsate.

11.2.2 Decontamination Procedures

Station 1: Segregate Equipment Drop
Deposit equipment used on-site (i.e., tools, sampling devices and containers, monitoring instruments radios, clipboards, etc.) on the plastic drop cloth/sheet or in different containers with plastic liners. Each will be contaminated to a different degree. Segregation at the drop reduces the probability of cross contamination. Loose leaf sampling data sheets or maps can be placed in plastic zip lock bags if contamination is evident.

Station 2: Physical Removal with a High-Pressure Washer (Optional)
Use high pressure wash on grossly contaminated equipment. Do not use high- pressure wash on sensitive or non-waterproof equipment.

Station 3: Physical Removal with Brushes and a Wash Basin
Scrub equipment with soap and water using bottle and bristle brushes. Only sensitive equipment (i.e., radios, air monitoring and sampling equipment) which is waterproof should be washed. Equipment which is not waterproof should have plastic bags removed and wiped down with a damp cloth. Acids and organic rinses may also ruin sensitive equipment. Consult the manufacturers for recommended decontamination solutions.

Station 4: Low-Pressure Rinse
Rinse sampling equipment with distilled/deionized water with a low-pressure sprayer.

Station 5: Nitric Acid Sprayers (required only if metals are a contaminant of concern)
Using a spray bottle, rinse sampling equipment with nitric acid. Begin spraying (inside and outside) at one end of the equipment allowing the acid to drip to the other end into a 5-gallon bucket. A rinsate blank may be required at this station. Refer to Section 9.

Station 6: Organic Solvent Sprayers
Rinse sampling equipment with a solvent. Begin spraying (inside and outside) at one end of the equipment allowing the solvent to drip to the other end into a 5-gallon bucket. Allow the solvent to evaporate from the equipment before going to the next station. A QC rinsate sample may be required at this station.

11.2.3 Post Decontamination Procedures

1. Collect high-pressure pad and heavy equipment decontamination area liquid and waste and store in appropriate drum or container. A sump pump can aid in the collection process. Refer to the Department of Transportation (DOT) requirements for appropriate containers based on the contaminant of concern.

2. Collect high-pressure pad and heavy equipment decontamination area solid waste and store in appropriate drum or container. Refer to the DOT requirements for appropriate containers based on the contaminant of concern.

3. Empty soap and water liquid wastes from basins and buckets and store in appropriate drum or container. Refer to the DOT requirements for appropriate containers based on the contaminant of concern.

4. Empty acid rinse waste and place in appropriate container or neutralize with a base and place in appropriate drum. pH paper or an equivalent pH test is required for neutralization. Consult DOT requirements for appropriate drum for acid rinse waste.

5. Empty solvent rinse sprayer and solvent waste into an appropriate container. Consult DOT requirements for appropriate drum for solvent rinse waste.

6. Using low-pressure sprayers, rinse basins, and brushes. Place liquid generated from this process into the wash water rinse container.

7. Empty low-pressure sprayer water onto the ground.

8. Place all solid waste materials generated from the decontamination area (i.e., gloves and plastic sheeting, etc.) in an approved DOT drum. Refer to the DOT requirements for appropriate containers based on the contaminant of concern.

9. Write appropriate labels for waste and make arrangements for disposal. Consult DOT regulations for the appropriate label for each drum generated

from the decontamination process.

11.3 Personal Protective Equipment Decontamination

Non-disposable PPE such as waders, wading boots, or life vests will require special decontamination (if exposed) between sites in order to mitigate cross-contamination and/or transfer of invasive species. The EPA approved disinfectant is Virkon®, a compound that is used widely for various cleaning purposes. The manufacturer recommends for equipment disinfection to add 10 grams of Virkon® to a liter of water and pressure wash or brush until the surface appears clean. If soaking the PPE is preferred, place in 1:100 diluted Virkon® until solution appears soiled or for a period of 4-5 days.

12.0 DATA RECORDS AND MANAGEMENT

Results of quality control samples will be evaluated for contamination. This information will be utilized to qualify the environmental sample results in accordance with the project's data quality objectives.

13.0 QUALITY CONTROL AND ASSURANCE

A rinsate blank is one specific type of quality control sample associated with the field decontamination process. This sample will provide information on the effectiveness of the decontamination process employed in the field. Rinsate blanks are samples obtained by running analyte-free water over decontaminated sampling equipment to test for residual contamination. The blank water is collected in sample containers for handling, shipment, and analysis. These samples are treated identical to samples collected that day. A rinsate blank is used to assess cross-contamination brought about by improper decontamination procedures. Where dedicated sampling equipment is not utilized, collect one rinsate blank per day per type of sampling device samples to meet QA2 and QA3 objectives.

If sampling equipment requires the use of plastic tubing it should be disposed of as contaminated and replaced with clean tubing before additional sampling occurs.

14.0 REFERENCES

EPA Guidance for Preparing Standard Operating Procedures, EPA QA/G-6, April 2007.

Field Sampling Procedures Manual, New Jersey Department of Environmental Protection, February, 1988.

A Compendium of Superfund Field Operations Methods, EPA 540/p-87/001. Engineering Support Branch Standard Operating Procedures and Quality Assurance Manual, EPA Region IV, April 1, 1986.

Guidelines for the Selection of Chemical Protective Clothing, Volume 1, Third Edition, American Conference of Governmental Industrial Hygienists, Inc., February, 1987.

Occupational Safety and Health Guidance Manual for Hazardous Waste Site Activities, NIOSH/OSHA/USCG/EPA, October, 1985.

EPA, Environmental Response Team, Standard Operating Procedures - Sampling Equipment Decontamination: U.S. Environmental Protection Agency, 1994, SOP #2006.

15.0 ATTACHMENTS

Table 1 - Soluble Contaminants and Recommended Solvent Rinse

SOLVENT(1)	EXAMPLES OF SOLVENTS	SOLUBLE CONTAMINANTS
Water	Deionized water, tap water	Low-chain hydrocarbons Inorganic compounds Salts Some organic acids and other polar compounds
Dilute Acids	Nitric acid, acetic acid, boric acid	Basic (caustic) compounds (e.g., amines and hydrazines)
Dilute Bases	Sodium bicarbonate (e.g., soap detergent)	Acidic compounds phenol thiols some nitro and sulfonic compounds
Organic Solvents (2)	Alcohols ethers ketones aromatics straight chain alkalines (e.g., hexane) common petroleum products (e.g., fuel, oil, kerosene)	Nonpolar compounds (e.g., some organic compounds)
Organic Solvent(2)	Hexane	PCBs

(1) Material Safety Data Sheets are required for all decontamination solvents or solutions as required by the Hazard Communication Standard.

(2) WARNING: Some organic solvents can permeate and/or degrade the protective clothing.

Figure 1 - Contamination Reduction Zone Layout

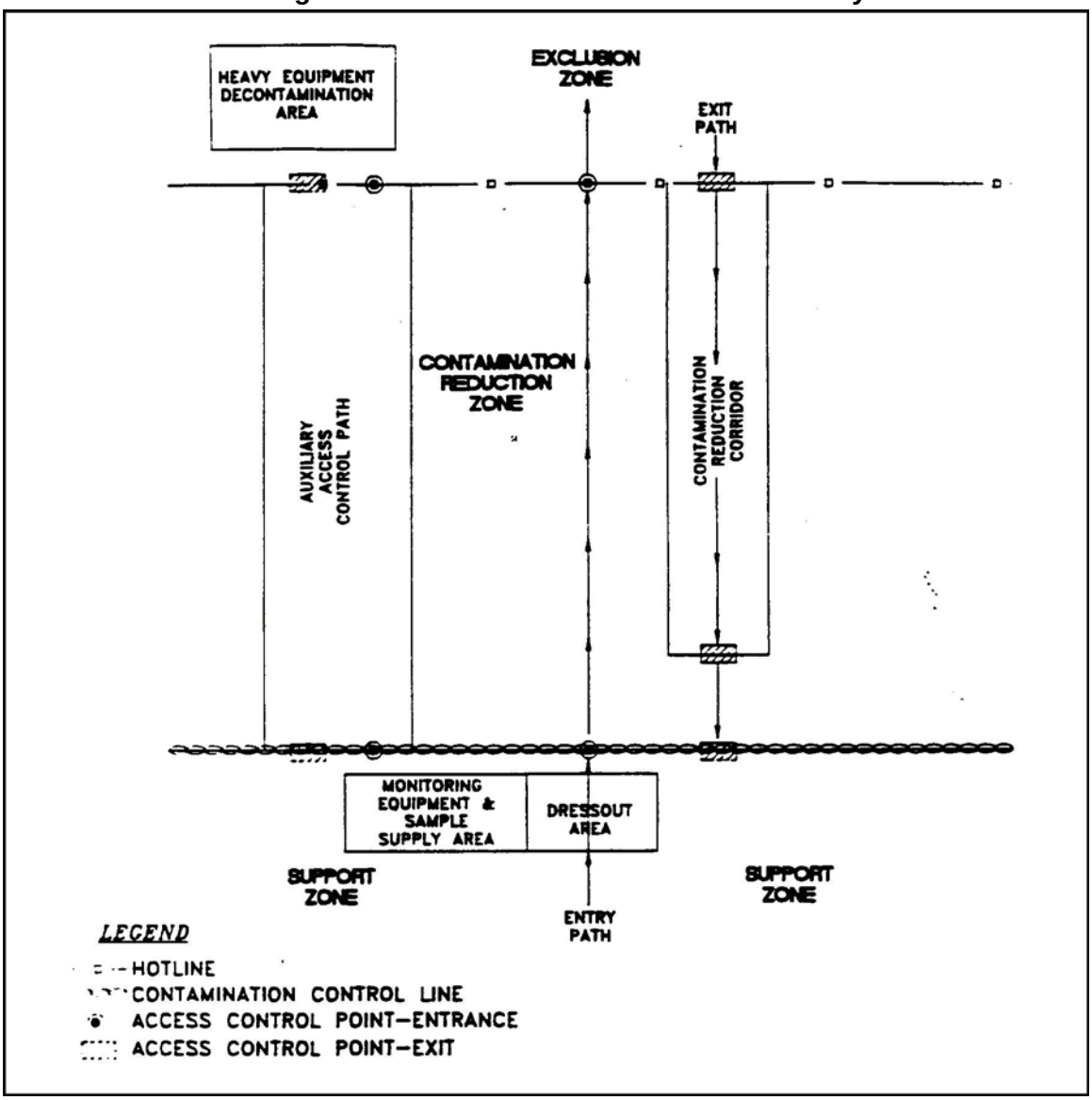
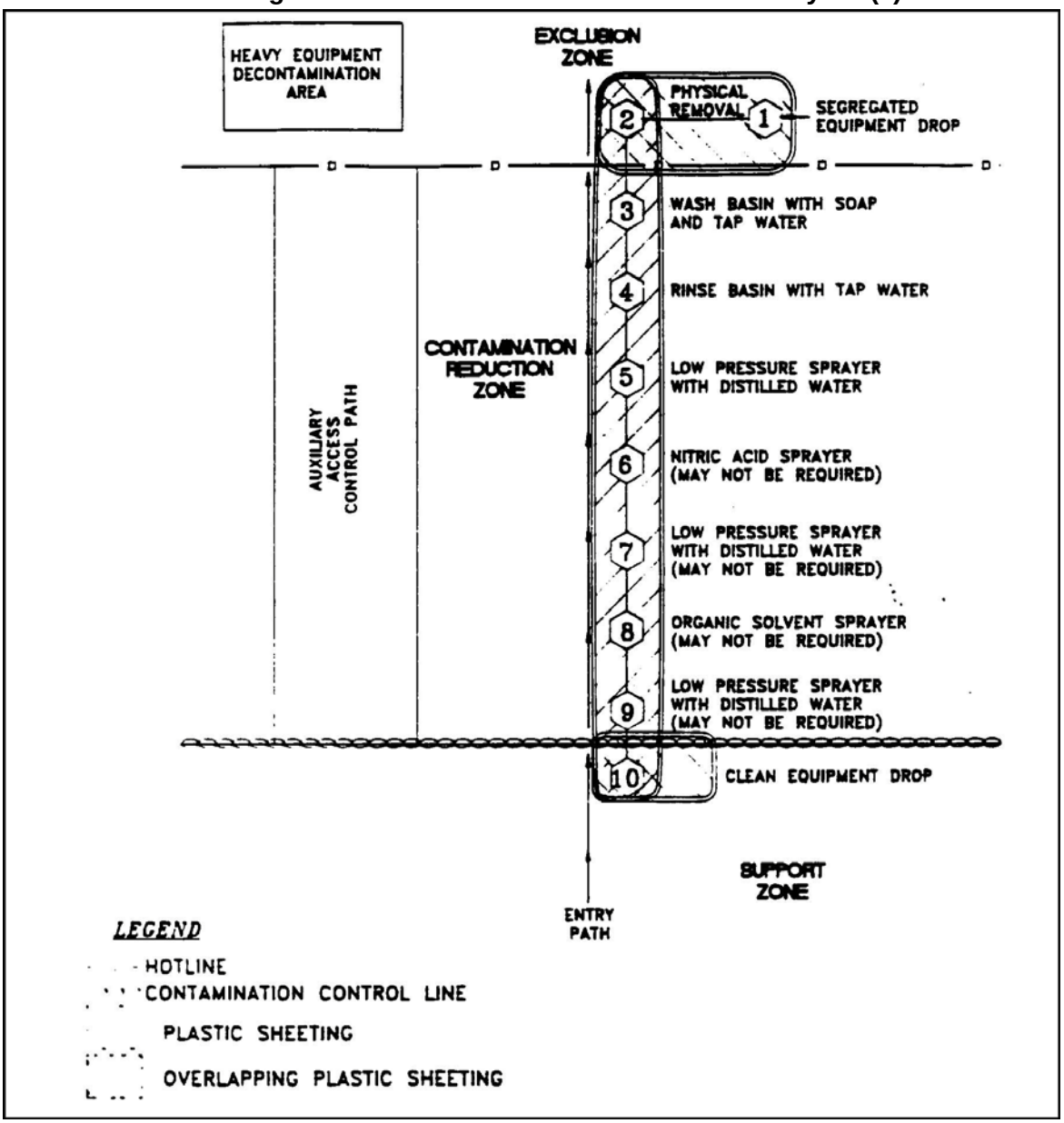


Figure 2 - Contamination Reduction Zone Layout (2)



Sample Preservation

APPROVED:

ESAT Region 8 QA Coordinator — Date: 06/06/12

ESAT Region 8 Team Manager — Date: 6/6/12

EPA Task Order Project Officer — Date: 7/10/12

ESAT Region 8 Task Lead — Date: 6/8/12

DCN: EP8-7-7061

TABLE OF CONTENTS

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide a standard approach for Environmental Protection Agency (EPA) and Environmental Services Assistance Team (ESAT) Region 8 personnel to preserve samples during field activities.

2.0 SCOPE AND APPLICATION

This SOP is specifically intended for application by EPA and ESAT personnel who conduct sample preservation in field work activities.

3.0 SUMMARY OF METHOD

For purposes of this SOP, proper sample preservation techniques and methods are reviewed. This SOP is based on industry standard instructions.

4.0 ACRONYMS AND DEFINITIONS

ESAT	Environmental Services Assistance Team
DOC	Dissolved Organic Carbon
HASP	Health and Safety Plan
HAZWOPER	Hazardous Waste Operations and Emergency Response
MSDS	Material Safety Data Sheet
PPE	Personal Protective Equipment
QAPP	Quality Assurance Project Plan
QC	Quality Control
SAP	Sampling Analysis Plan
SOP	Standard Operating Procedure
EPA	United States Environmental Protection Agency
VOA	Volatile Organic Analytes

Ampule: A small sealed vial which is used to preserve a sample usually nitric, phosphoric, hydrochloric, or sulfuric acid.

Health and Safety Plan (HASP): A site-specific document that outlines potential site hazards and hazard mitigation practices.

Material Safety Data Sheets (MSDS): A form with data regarding the properties of a particular substance.

Personal Protective Equipment (PPE): Refers to protective clothing, helmets, goggles, or other garment designed to protect the wearer's body from injury by blunt impacts, electrical hazards, heat, chemicals, and infection, for job-related occupational safety and health purposes.

Quality Assurance Project Plan (QAPP): A site-specific document that specifies quality assurance activities and data quality objectives.

Sampling and Analysis Plan (SAP): A site-specific document that specifies events to take place in the field.

Standard Operating Procedure (SOP): A set of written instructions that document a routine or repetitive activity followed by an organization (EPA, 2007).

5.0 HEALTH AND SAFETY

The procedures outlined in this SOP have general health and safety issues associated with it. This includes the use of proper PPE when conducting sample preservation. The most important health and safety items while preserving samples are latex gloves to prevent skin contact and safety eyewear to protect from splash hazards. Always refer to the applicable HASP and MSDS any time field work or preservation activities are conducted.

6.0 EQUIPMENT

- Acid Ampules
- PPE (gloves, eye protection, cover-alls)
- First-Aid kit
- Disposal equipment (ampoule waste container, garbage bags)
- Sample Filter equipment

7.0 SAMPLE PRESERVATION

Complete and unequivocal preservation of samples with total stability of every constituent maintained, regardless of the nature of the sample, can never be achieved. At best, preservation techniques can only minimize the chemical and biological changes that inevitably continue after the sample is removed from the parent source. Proper preservation methods, such as pH control, chemical addition, filtration, and refrigeration are intended to retard biological and chemical effects, reduce volatility of constituents, and limit absorption effects.

Because of the potential vulnerability samples may have to a number of influences, it is best that sample analyses occur as soon as possible after collection. However, since the majority of sampling events do not have on-site mobile laboratories, and travel time from the field to the laboratory may be multiple days, it is critical that effective sample preservation techniques are employed to ensure sample integrity.

7.1 Chemical Influences on Samples

Chemical changes to samples may result when physical conditions alter the chemical structure of the constituents. Many of the chemical processes that occur once a sample is taken will ultimately depend on the type and amount of sample taken, the medium to which the sample is housed, and the storage and transportation environment for that particular sample. Examples of chemical effects to samples might include metal cations precipitating as hydroxides or forming complexes with other constituents; cations or anions changing valence states under certain reducing or oxidizing conditions; or other constituents dissolving or volatilizing with the passage of time. Metal cations may also adsorb onto surfaces (glass, plastic, quartz, etc.), such as, iron and lead.

7.2 Biological Influences on Samples

Biological changes may occur when a sample changes the valence of an element or a radical to a different valence. Soluble constituents may be converted to organically bound materials in cell structures, or cell lysis may result in release of cellular material into solution. Nitrogen and phosphorus cycles are examples of biological influence on sample composition.

7.3 Sample Types to Preserve

Sample preservation mainly pertains to water samples (surface water, groundwater, pore water), but other sample types that might require preservation include sediment, macroinvertabrates, or waste rock. For all non-water based samples, the basic practice of storing and transporting in coolers with ice will suffice for a preservation method. However, be sure to consult with the analyzing laboratory with regard to any specific preservation requirements (for both water and non-water samples) before going into the field.

8.0 PRESERVATION PROCEDURES AND TECHNIQUES

Samples collected in the field are generally preserved by chilling or chemical treatment. It is important that the sample crew be knowledgeable of the following before and during the field event:

1. The required sample-designation code for each sample.

2. The sample requirements for filtration, chilling and chemical treatment.

3. The holding time restrictions required by the analyzing laboratory.

8.1 Water Sample Preservation

There are a number of things to consider when preparing for a water sampling event. Before field deployment, be sure to have all the equipment and supplies necessary to collect, preserve, store, and transport your samples in the proper way.

8.2 Meet Sample Volume Requirements

Collecting sufficient sample volume is critical. There must be sufficient physical sample volume for the analysis of all required parameters and completion of all QC determinations. The type of analytical procedure(s) to be performed will often dictate the sample volume to collect. It is extremely important that samplers refer to their specific SAP and QAPP to identify and collect the correct sample volume during each sampling event. Once the sample volume requirement is understood, the appropriate container size can be chosen to accommodate the sample.

8.3 Proper Preservation for Water Sample

Whether the preservation method is chilling or chemical treatment, the preservation specifics will vary based on the analysis. The variability involved in sample preservation can best be understood in the Preservation Requirement Tables (section 8.5.1 – 8.5.4). In these tables, you

will see how different analyses (physical, metals, organics, inorganics or non-metallics), dictate a different set of preservation requirements.

8.4 Preservation Methods

Each preservation method has specific standard procedures that need to be followed when preserving a sample.

8.4.1 Chemical Treatment

Chemicals used for sample preservation will depend on the target analyte (see section 8.5.1 – 8.5.4). For purposes of EPA Region 8 sampling, nitric acid (HNO_3), sulfuric acid (H_2SO_4) hydrochloric acid (HCl), and phosphoric acid (H_3PO_4) are most commonly used. It is important to wear appropriate PPE when involved in any part of the sample preservation process, especially in the chemical treatment process. An MSDS should be available for all preservatives to be used on site.

Preservation chemicals may be in the form of bulk liquid or ampoules.

Common chemical preservation in EPA Region 8 water sampling:

Measurement	**Chemical Treatment**
Total and Dissolved Metals	Nitric Acid (HNO_3)
Semi-Volatile Organics	Hydrochloric Acid (HCl)
Dissolved Organic Carbon (DOC)	Phosphoric Acid (H_3PO_4)
Nutrients	Sulfuric Acid (H_2SO_4)

8.4.2 Using Preservation Ampules

Preservation chemicals, such as nitric acid (HNO_3), hydrochloric acid (HCl), and phosphoric acid (H_3PO_4), can come in the form of ampoules. Ampoules are small plastic or glass containers that hold an exact amount of a chemical in a liquid form. They are designed to be used once per sample.

When preparing to treat a sample with a chemical from an ampoule, first break the tip of the ampoule with two hands (while wearing proper PPE), and pour the liquid into the sample bottle. When all liquid has been removed from the ampoule, place the broken components of the glass ampoule into a specifically designated and labeled acid neutralization container that has a secure screw top. It is acceptable for one acid waste container to be used to neutralize all acids.

8.4.3 Using Bulk Concentrated Acid

Preservation acid can also come in the form of bulk concentrated acid, typically at a concentration level of 70%. When using bulk concentrated acid, a disposable pipet may be used to extract the necessary quantity from the parent vessel to then be released in the water sample. The pipet may be used multiple times if the same chemical is being transferred. Never cross-contaminate pipets with different chemicals or different samples.

8.4.4 Chilling

Chilling samples is almost always a part of the sample preservation process. Once a sample is collected (and potentially treated with chemicals) it is to be immediately packed in ice or placed in a refrigerator and maintained at a temperature of 4 degrees Celsius or less, without freezing, until analyzed. To avoid problems that can result from sample expansion, allow sufficient headspace in the sample bottle before chilling it (An exception to this method includes Volatile Organic Analytes (VOA). In the case of VOAs, do not leave head space in the sample bottle). If using glass bottles, use foam sleeves to protect them. Another method that can be used to avoid the potential of melting ice water seeping into sample bottles is the use of plastic bags to contain the samples. This method doubles as a way to group subsamples from the same sample location.

8.5 Preservation Requirement Tables

Preservation requirements for physical, metals, organics, and inorganic sample types can be understood in the following tables.

8.5.1 Physical Preservation Requirements

Measurement	Volume (ml)	Container	Preservative	Holding Time
Color	50	Plastic or Glass	Cool, 4 Degrees Celsius	48 Hours
Conductance	100	Plastic or Glass	Cool, 4 Degrees Celsius	28 Days
Hardness	100	Plastic or Glass	HNO_3 – pH below 2	6 Months
Odor	200	Glass	Cool, 4 Degrees Celsius	24 Hours
pH	25	Plastic or Glass	None required	Analyze Immediately
Temperature	1000	Plastic or Glass	None required	Analyze Immediately
Turbidity	100	Plastic or Glass	Cool, 4 Degrees Celsius	48 Hours

8.5.2 Metals Preservation Requirements

Measurement	Volume (ml)	Container	Preservative	Holding Time
Dissolved	250	Plastic* or Glass	Filter on site first. HNO_3 – pH below 2	6 Months
Suspended	250	Plastic* or Glass	Filter on site. HNO_3 – pH below 2	6 Months
Total	500	Plastic* or Glass	HNO_3 – pH below 2	6 months
Dissolved (Mercury)	250	Plastic* or Glass	Filter on site first. HNO_3 – pH below 2	28 Days
Total (Mercury)	250	Plastic* or Glass	HNO_3 – pH below 2	28 Days

*Polyethylene with a polypropylene cap (no liner) is preferred.

8.5.3 Organics Preservation Requirements

Measurement	Volume (ml)	Container	Preservative	Holding Time
BOD	1000	Plastic or Glass	Cool, 4 Degrees Celsius	48 Hours
COD	50	Plastic or Glass	Cool, 4 Degrees Celsius. H_2SO_4 – pH below 2	28 Days
DOC	500	Plastic or Glass	Cool, 4 Degrees Celsius. Add H_3PO_4.	28 Days
Oil & Grease	1000	Glass only	Cool, 4 Degrees Celsius. H_2SO_4 – pH below 2.	28 Days
Phenolics	500	Glass only	Cool, 4 Degrees Celsius. HNO_3 – pH below 2.	28 Days
Semi-volatiles	1000	Glass only	Cool, 4 Degrees Celsius	7-14 Days

8.5.4 Inorganics & Non-Metallics Preservation Requirements

Measurement	Volume (ml)	Container	Preservative	Holding Time
Acidity	100	Plastic or Glass	Cool, 4 Degrees Celsius	14 Days
Alkalinity	250	Plastic or Glass	Cool, 4 Degrees Celsius	14 Days
Bromide	100	Plastic or Glass	None required	28 Days
Chloride	250	Plastic or Glass	None required	28 Days
Cyanides	500	Plastic or Glass	Cool, 4 Degrees Celsius. NaOH – pH over 12. 0.6g	14 Days (24 Hours when

			ascorbic acid (only in presence of residual chlorine)	sulfide is present)
Fluoride	250	Plastic or Glass	None required	28 Days
Iodide	100	Plastic or Glass	Cool, 4 Degrees Celsius	24 Hours
Nitrogen (Ammonia)	500	Plastic or Glass	Cool, 4 Degrees Celsius. H_2SO_4 – pH below 2	28 Days
Nitrate & Nitrite	250	Plastic or Glass	Cool, 4 Degrees Celsius. H_2SO_4 – pH below 2	28 Days
Nitrate	250	Plastic or Glass	Cool, 4 Degrees Celsius	48 Hours
Nitrite	250	Plastic or Glass	Cool, 4 Degrees Celsius	48 Hours
Sulfate	250	Plastic or Glass	Cool, 4 Degrees Celsius	28 Days
Sulfide	250	Plastic or Glass	Cool, 4 Degrees Celsius. Add 2 ml Zinc Acetate plus NaOH – pH over 9.	7 Days

9.0 PERSONNEL QUALIFICATIONS

All personnel who participate in field activities are required to obtain clearance in three mandatory health and safety programs: medical monitoring, respirator fit testing, and OSHA Hazardous Waste Operations and Emergency Response (HAZWOPER) 40-hour training. It is important for field personnel to familiarize themselves with other applicable SOP's such as Sampling Equipment Decontamination SOP FLD 02.00, Surface Water Sampling SOP FLD 01.00, Sample Custody and Labeling SOP FLD 11.00, and General Field Sampling Protocols SOP FLD 12.00. In addition, any personnel who will participate in sample preservation activities must read, understand, and sign the site-specific HASP and SAP/QAPP.

10.0 REFERENCES

Environmental Protection Agency. 1983. Sample Preservation. pp.xv-xx. In Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020. EPA Cincinnati, Ohio, USA.

United States Geological Survey. 2002. Processing of Water Samples (Version 2, 4/02). P. 89-94. In National Field Manual for the Collection of Water-quality data. USGS, Washington DC, USA.

Soil Sampling

APPROVED:

ESAT Region 8 QA Coordinator — Date 07/16/13

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ESAT Region 8 Task Lead — Date 7/16/13

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TABLE OF CONTENTS

1.0 PURPOSE

The purpose of this standard operating procedure (SOP) is to provide a guideline for the collection of representative soil samples in the field. The collection and analysis of soil samples serves to establish whether pollutants are present in the soils and helps determine the required action level(s) with regard to public and environmental health and welfare.

2.0 SCOPE AND APPLICATION

These are standard operating procedures which may be varied or changed as required, dependent upon site conditions, equipment limitations or limitations imposed by the procedure. In all instances, the ultimate procedures employed should be documented and included with the final report. Mention of trade names or commercial products does not constitute Techlaw, inc. endorsement or recommendation for use.

3.0 SUMMARY OF METHOD

Soil samples may be collected using a variety of methods and equipment. The methods and equipment used are dependent on the depth of the desired sample, the type of sample required (disturbed vs. undisturbed), and the soil type. Near-surface soils may be easily sampled using a spade, trowel, and scoop. Sampling at greater depths may be performed using a hand auger, continuous flight auger, a trier, a split-spoon, or a backhoe if necessary.

4.0 ACRONYMS AND DEFINITIONS

COC	Chain of Custody
GPS	Global Positioning System
HASP	Health and Safety Plan
HAZWOPER	Hazardous Waste Operations and Emergency Response
OSHA	Occupation Safety and Health Administration
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
SAP	Sampling and Analysis Plan
SOP	Standard Operating Procedure
EPA	United States Environmental Protection Agency
ESAT	Environmental Services Assistance Team

Chain of Custody (COC) – A chronological document that tracks transfer of samples between entities from collection to disposal

Composite Sampling – Sampling from several points or intervals and consolidating them into a larger sample

Discrete Sampling - Sampling from a single location

Health and Safety Plan (HASP) – A site-specific plan that outlines potential hazards and procedural/equipment recommendations

Standard Operating Procedure (SOP) - A set of written instructions that document a routine or repetitive activity followed by an organization (EPA, 2007)

5.0 HEALTH AND SAFETY

When working with potentially hazardous materials or in hazardous situations, personnel must understand and comply with the site-specific Sampling and Analysis Plan and Quality Assurance Project Plan (SAP/QAPP) and Health and Safety Plan (HASP) before the sampling event begins. More specifically, when sampling waste rock piles or fluvial deposit zones containing known or suspected hazardous substances, adequate personal protective equipment such as nitrile gloves, safety glasses, and protective footwear are necessary to prevent exposure.

When traversing tailings piles, hazardous situations exist that require the sampling personnel to wear adequate safety equipment including gloves and non-slip footwear. Never perform sampling activities if it cannot be done so in a safe manner (tailing piles are too steep, lightning is occurring, etc).

6.0 CAUTIONS

There are cautions to be considered before deployment on a soil sampling event. If the samples are to be collected in an urban area at depth, the underground utility lines must be identified. In addition, if sampling at a remote waste rock pile, always use the buddy system when traversing steep gradients that may present fall hazards. Always review the site-specific HASP for potential safety hazards.

7.0 INTERFERENCES

There are two primary interferences or potential problems associated with soil sampling. These include cross-contamination of samples and improper sample collection. Cross-contamination problems can be eliminated or minimized through the use of dedicated sampling equipment. If this is not possible or practical, then decontamination of sampling equipment is necessary. Improper sample collection can involve disturbance of the matrix (i.e. walking on specific areas that will ultimately be sampled) resulting in compaction of the sample or inadequate homogenization of the samples where required, resulting in variable, non-representative results.

8.0 PERSONNEL QUALIFICATIONS

Any personnel involved with field sampling activities must be cleared for health and safety. Clearance includes medical monitoring, respirator fit testing, and Occupational Safety and Health Administration (OSHA) Hazardous Waste Operations and Emergency Response (HAZWOPER) 40-hour training. Personnel who will be collecting soil samples should familiarize themselves with this and other pertinent SOPs such as the Sample Equipment Decontamination SOP FLD 02.00, the Sample Preservation SOP FLD 03.00, the Sample Custody and Labeling SOP FLD 11.00, and the General Field Sampling Protocols SOP FLD 12.00.

9.0 EQUIPMENT

Equipment needed for collection of soil samples may include:

HASP Gear - Gloves, proper footwear, safety glasses, etc.

Mapping and Location Tools – Global Positioning System (GPS) units, site/local area maps, compass, tape measure, survey stakes, pin flags, camera, 2-way radios

Documentation Tools – Field log book, field data sheet, COC(s), labels, clear tape, pens, permanent marker, waterproof paper

Sampling Tools – Plastic, Teflon™, or other appropriate composition scoop (analysis dependent), shovel, spade, trowel, measuring cup or graduated cylinder, field scale, bucket auger, post hole auger, homogenization container w/ mixing tool, bucket, rinse bottle, purified water, paper towels

Sample Containers – Ziploc™ baggies, glass jars, labels, clear tape, pens, permanent marker, cooler(s), ice, thermometer

See Appendix A for a detailed list of soil sampling equipment.

10.0 STANDARDS AND REAGENTS

Reagents are not used for the preservation of soil samples. Decontamination solutions are specified in the Sampling Equipment Decontamination SOP# FLD-02 and the site-specific work plan.

11.0 PROCEDURES

11.1 Preparation

1. Determine the extent of the sampling effort, the sampling methods to be employed, and the types and amounts of equipment and supplies required.

2. Obtain the necessary sampling and monitoring equipment.

3. Decontaminate or pre-clean equipment, and ensure that it is in working order.

4. Prepare schedules, and coordinate with staff, client, and regulatory agencies where necessary. It is also important to obtain access agreements if sampling is to occur on private property.

5. Perform a general site survey prior to site entry in accordance with the site-specific HASP.

6. Use stakes, flagging, or buoys to identify and mark all sampling locations followed by a GPS point (see GPS Trimble® GeoXT 2008 series SOP FLD 07.00). Specific site factors, including extent and nature of contaminant should be considered when selecting sample location. If required, the proposed locations may be adjusted based on site access, property boundaries, and surface obstructions. All staked locations will be utility-cleared by the property owner

prior to soil sampling. It is the responsibility of the sampler to verify with the property owner that utility lines have been marked. If there is no property owner and there is concern for underground utility lines, it is the responsibility of the samplers to contact the state agency or contractor that can provide a marking service.

11.2 Sample Collection

In general, there are two primary ways to collect a soil sample. Composite sampling involves taking several subsamples from a designated sample location and consolidating into one larger sample. Discrete sampling is defined as taking one sample from a single location. Composite and discrete sampling can be achieved by the sample techniques listed below.

11.2.1 Surface Soil Samples

Collection of samples from near-surface soil can be accomplished with tools such as spades, shovels, trowels, and scoops. Remove surface material to the required depth and use a stainless steel or plastic scoop to collect the sample.

This method can be used in most soil types but is limited to sampling near-surface areas. Accurate, representative samples can be collected with this procedure depending on the care and precision demonstrated by the sample team member. A stainless steel scoop, lab spoon, or plastic spoon will suffice in most other applications. The use of a flat, pointed mason trowel to cut a block of the desired soil can be helpful when undisturbed profiles are required. Care should be exercised to avoid use of devices plated with chrome or other materials. Plating is particularly common with garden implements such as potting trowels. There are four depth classes that are typically used in Region 8: 0-2” range, 0-6” range, 6-12” range, and 12-18” range. The 0-2” and 0-6” range can usually be sampled with one of the tools listed above, but the deeper ranges generally require the use of one the tools described in sections 11.2.2 and 11.2.3.

The following procedure is used to collect surface soil samples:

1. Carefully remove the top layer of soil or debris to the desired sample depth with a pre-cleaned spade.

2. Using a pre-cleaned, stainless steel scoop, plastic spoon, or trowel, remove and discard a thin layer of soil from the area which came in contact with the spade.

3. If volatile organic analysis is to be performed, transfer the sample directly into an appropriate, labeled sample container with a stainless steel lab spoon or equivalent and secure the cap tightly. Place the remainder of the sample into a stainless steel, plastic, or other appropriate homogenization container, and mix thoroughly to obtain a homogenous sample representative of the entire sampling interval. Then, either place the sample into appropriate, labeled containers and secure the caps tightly; or, if composite samples are to be collected, place a sample from another sampling interval or location into the homogenization container and mix thoroughly. When compositing is complete, place the sample into appropriate, labeled containers and secure the

caps tightly.

4. Due to data quality requirements, some soil sampling events may require that each sub-sample of a composite be measured. This can be achieved two ways: by mass or by volume. Due to the remote nature of the sites in Region 8, it is recommended that composite samples are measured by volume. This requires the use of a measuring cup or graduated cylinder (of appropriate composition), placing the material into the measuring device to the desired volume, then adding the sub-samples to a larger sample container (plastic baggie for metals, glass jar for organics). Overall volume of sample will be dictated by analytical requirements.

11.2.2 Sampling at Depth with Augers and Thin Wall Tube Samplers

This system consists of an auger, or a thin-wall tube sampler, a series of extensions, and a "T" handle (Figure 1, Appendix B). The auger is used to bore a hole to a desired sampling depth and is then withdrawn from the hole. The sample may be collected directly from the auger. If a core sample is to be collected, the auger tip is then replaced with a thin wall tube sampler. The sampling assembly is then lowered down the borehole, and driven into the soil to the completion depth. The system is withdrawn and the core is collected from the thin wall tube sampler.

Several types of augers are available; these include: bucket type, continuous flight (screw), and post-hole augers. Bucket type augers are better suited for direct sample recovery since they provide a large volume of sample in a short time. When continuous flight augers are used, the sample can be collected directly from the auger flights. The continuous flight augers are satisfactory for use when a composite of the complete soil column is desired. Post-hole augers have limited utility for sample collection as they are designed to cut through fibrous, rooted, swampy soil and cannot be used below a depth of three feet.

The following procedure is used for collecting soil samples with the auger:

1. Attach the auger bit to a drill rod extension, and attach the "T" handle to the drill rod.

2. Clear the area to be sampled of any surface debris (e.g., twigs, rocks, litter). It may be advisable to remove the first three to six inches of surface soil for an area approximately six inches in radius around the drilling location.

3. Begin augering, periodically removing and depositing accumulated soils onto a plastic sheet spread near the hole. This prevents accidental brushing of loose material back down the borehole when removing the auger or adding drill rods. It also facilitates refilling the hole, and avoids possible contamination of the surrounding area.

4. After reaching the desired depth, slowly and carefully remove the auger from the boring. When sampling directly from the auger, collect the sample after the auger is removed from the boring and proceed to Step 10.

5. Remove the auger tip from drill rods and replace with a pre-cleaned thin wall tube

sampler. Install the proper cutting tip.

6. Carefully lower the tube sampler down the borehole. Gradually force the tube sampler into soil. Care should be taken to avoid scraping the borehole sides. Avoid hammering the drill rods to facilitate coring as the vibrations may cause the boring walls to collapse.

7. Remove the tube sampler, and unscrew the drill rods.

8. Remove the cutting tip and the core from the device.

9. Discard the top of the core (approximately 1 inch), as this possibly represents borehole debris material collected before penetration of the layer of concern. Place the remaining core into the appropriate labeled sample container. Sample homogenization is not required.

10. If volatile organic analysis is to be performed, transfer the sample into an appropriate, labeled sample container with a stainless steel lab spoon, or equivalent and secure the cap tightly. Place the remainder of the sample into a stainless steel, plastic, or other appropriate homogenization container, and mix thoroughly to obtain a homogenous sample representative of the entire sampling interval. Then, either place the sample into appropriate, labeled containers and secure the caps tightly; or, if composite samples are to be collected, place a sample from another sampling interval into the homogenization container and mix thoroughly. When compositing is complete, place the sample into appropriate, labeled containers and secure the caps tightly.

11. If another sample is to be collected in the same hole, but at a greater depth, reattach the auger bit to the drill and assembly, and follow steps 3 through 11, making sure to decontaminate the auger and tube sampler between samples.

12. Abandon the hole according to applicable State regulations. Generally, shallow holes can simply be backfilled with the removed soil material.

11.2.3 Sampling at Depth with a Trier

The system consists of a trier, and a "T" handle. The auger is driven into the soil to be sampled and used to extract a core sample from the appropriate depth. The following procedure will be used to collect soil samples with a sampling trier:

1. Insert the trier (Figure 2, Appendix B) into the material to be sampled at a 0 to 45 angle from horizontal. This orientation minimizes the spillage of sample.

2. Rotate the trier once or twice to cut a core of material.

3. Slowly withdraw the trier, making sure that the slot is facing upward.

4. If volatile organic analysis is to be performed, transfer the sample into an appropriate,

labeled sample container with a stainless steel lab spoon, or equivalent and secure the cap tightly. Place the remainder of the sample into a stainless steel, plastic, or other appropriate homogenization container, and mix thoroughly to obtain a homogenous sample representative of the entire sampling interval. Then, either place the sample into appropriate, labeled containers and secure the caps tightly; or, if composite samples are to be collected, place a sample from another sampling interval into the homogenization container and mix thoroughly. When compositing is complete, place the sample into appropriate, labeled containers and secure the caps tightly.

11.2.4 Sampling at Depth with a Split Spoon (Barrel) Sampler

The procedure for split spoon sampling describes the collection and extraction of undisturbed soil cores of 18 or 24 inches in length. A series of consecutive cores may be extracted with a split spoon sampler to give a complete soil column profile, or an auger may be used to drill down to the desired depth for sampling. The split spoon is then driven to its sampling depth through the bottom of the augured hole and the core extracted.

When split spoon sampling is performed to gain geologic information, all work should be performed in accordance with ASTM D 1586-67 (reapproved 1974). The following procedures will be used for collecting soil samples with a split spoon:

1. Assemble the sampler by aligning both sides of barrel and then screwing the drive shoe on the bottom and the head piece on top.

2. Place the sampler in a perpendicular position on the sample material.

3. Using a well ring, drive the tube. Do not drive past the bottom of the head piece (past the full length of the sample barrel) or compression of the sample will result.

4. Record in the site logbook or on field data sheets the length of the tube used to penetrate the material being sampled, and the number of blows required to obtain this depth.

5. Withdraw the sampler, and open by unscrewing the bit and head and splitting the barrel. The amount of recovery and soil type should be recorded on the boring log. If a split sample is desired, a cleaned, stainless steel knife should be used to divide the tube contents in half, longitudinally. This sampler is typically available in 2 and 3 1/2 inch diameters. However, in order to obtain the required sample volume, use of a larger barrel may be required.

6. Without disturbing the core, transfer it to appropriate labeled sample container(s) and seal tightly.

11.3 Sample Sieving

Analytical methods may require that a sample be separated by particle size. A sieve is the most effective method of separating coarse and fine material from a soil sample. Sieving and random

sampling is also an effective method for soil sample homogenization (Schumacher et al, 1990). The site-specific SAP should be consulted when deciding what particle size of a soil sample should be submitted for analysis. Note that saturated soil samples should not be sieved. Those samples must first be dried before processing. Sieving procedures:

- Place sample in appropriate sized sieve (dry samples are optimal; wet samples will stick to the grid of a sieve).
- Use a catch pan that is of a material that won't compromise the integrity of the sample.
- Place lid on the sieve and shake vigorously to separate particle sizes.
- Transfer desired sample fraction to labeled container.
- Decontaminate thoroughly with brushes and/or compressed air before use on next sample.

12.**0 DATA RECORDS AND MANAGEMENT**

Once collected, samples are preserved, labeled, and stored for transport. A COC must accompany all samples during transport and transfer between entities. Sample labels should contain the following information:

- Site Identification
- Date sampled
- Sampler initials
- Time
- Analysis to be performed

In addition, soil characteristics may need to be documented when sampling. Below is a standardized list of soil characteristics and their corresponding Unified Soil Classification System identifiers.

- GW – well-graded gravels, gravel and sand mixtures, little or no fines
- GP – poorly graded gravels, gravel and sand mixtures, little or no fines
- GM – silty gravels, gravel, sand, silt mixtures
- GC – clayey gravels, gravel, sand, clay mixtures
- SW – well-graded sands, little or no fines
- SP – poorly-graded sands, little or no fines
- SM – silty sands, sand-silt mixtures
- SC – clayey sands, sand-clay mixtures
- ML – inorganic silts and very fine sands
- CL – inorganic clays of low to medium plasticity
- OL – organic silts and organic silty clays
- MH – inorganic silts, micaceous or diatomaceous fine sandy or silty soils
- CH – inorganic clays of high plasticity
- OH - organic clays of medium to high plasticity, organic silts
- Pt – highly organic soils

13.0 QUALITY CONTROL AND ASSURANCE

There are no specific quality assurance (QA) activities which apply to the implementation of these procedures. However, the following QA procedures apply:

1. All data must be documented on field data sheets or within site logbooks.

2. All instrumentation must be operated in accordance with operating instructions as supplied by the manufacturer, unless otherwise specified in the work plan. An instruction manual for the operation of the hand auger equipment is provided in Appendix C of this SOP. Equipment checkout and calibration activities must occur prior to sampling/operation, and they must be documented.

3. Always consult the SAP for duplicate sample frequency requirements.

4. Document any deviations from SOP's, work plan, SAP/QAPP, etc.

14.0 REFERENCES

EPA Guidance for Preparing Standard Operating Procedures, EPA QA/G-6, April 2007.

Mason, B.J., Preparation of Soil Sampling Protocol: Technique and Strategies. 1983 EPA-600/4-83-020.

Barth, D.S. and B.J. Mason, Soil Sampling Quality Assurance User's Guide. 1984 EPA-600/4-84-043.

U.S. EPA. Characterization of Hazardous Waste Sites. A Methods Manual: Volume II. Available Sampling Methods, Second Edition. 1984 EPA-600/4-84-076.

de Vera, E.R., B.P. Simmons, R.D. Stephen, and D.L. Storm. Samplers and Sampling Procedures for Hazardous Waste Streams. 1980 EPA-600/2-80-018.

ASTM D 1586-67 (reapproved 1974), ASTM Committee on Standards, Philadelphia, PA.

U.S. EPA, Environmental Response Team, standard operating procedures—Soil Sampling: U.S. Environmental Protection Agency, 1994, SOP #2012

Shumacher, B.A, Shines, K.C., Burton J.V., and Papp, M.L. (1990) "A Comparison of Soil Sample Homogenization Techniques."

Sample Custody and Labeling

APPROVED:

ESAT Region 8 QA Coordinator — Date: 06/06/12

ESAT Region 8 Team Manager — Date: 6/6/12

EPA Task Order Project Officer — Date: 7/10/12

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TABLE OF CONTENTS

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to assist field personnel in developing proper sample custody and sample identification methods for the collection of environmental samples. This includes the use of chain of custody (COC) forms and labels for samples collected in the field. These procedures are critical in ensuring the integrity of environmental samples.

2.0 SCOPE AND APPLICABILITY

To ensure the integrity of a sample collected in the field or generated in a laboratory setting, documentation is needed to chronicle all sample handling for collection or creation through analysis and/or disposal. Any sample that is collected in the field or generated in a laboratory setting will require that records are kept as it transfers from various entities. This is the basis for generation of a COC. Uniquely, labeling samples with information, such as sample location, date, time, preservation method, and analytical requirements, keeps samples organized. A COC is initiated for each sample, either at the time of sample collection or generation or as part of preparation for a sampling event. This SOP will cover the best practices for sample custody and the method of COC and label generation.

3.0 SUMMARY OF METHOD

Once a sample is collected, several steps need to be taken to ensure the required information is collected and maintained as it is transferred from the point of collection to the laboratory. If sample nomenclature and location is known before a field event, a COC will be generated before deployment into the field. When generating the COC, it is important to know the analytical fate of samples required for each sample location (e.g. total recoverable metal, dissolved metals, etc.). This information can be found in the site-specific Sampling and Analysis Plan (SAP) and other sampling event planning documents. Some software programs (e.g. Scribe) that generate COCs also have the ability to generate labels. Scribe is the Laboratory Information Management System (LIMS) used by the lab. It is important to keep in mind that it is not mandatory to generate COCs and labels before a sampling event, but it is preferred. If it is not known where samples will be collected or the nomenclature of the sites is unclear, sample containers can be labeled with permanent marker with tape placed over it, and a blank COC can be filled out at the time of sample collection. Once the method of custody is established, a specific person, known as the sample custodian, is then responsible for maintaining the integrity of the samples as they move from and within various locations.

4.0 ACRONYMS AND DEFINITIONS

CLP	Contract Lab Program
COC	Chain of Custody
EPA	United States Environmental Protection Agency
ERT	Environmental Response Team
ID	Identification
LIMS	Laboratory Information Management System
QAPP	Quality Assurance Project Plan
SAP	Sampling and Analysis Plan
SOP	Standard Operating Procedure

Chain of Custody (COC): A document used to chronologically track movement of samples between entities from collection to disposal.

Sampling and Analysis Plan (SAP): A site-specific document that describes the events to take place in the field.

Scribe: – A software tool developed by the United States Environmental Protections Agency (EPA) Environmental Response Team (ERT) to assist in the process of managing environmental data. Scribe captures sampling, observational, and monitoring field data.

Standard Operating Procedure (SOP): A set of written instructions that document a routine or repetitive activity followed by an organization (EPA, 2007).

5.0 HEALTH AND SAFETY

There are no specific health and safety hazards associated with sample custody and labeling, but these activities sometimes take place on-site during a sampling event. It is important for field personnel to familiarize themselves with the site-specific Health and Safety Plan before deployment to a site. In terms of personal interaction with the sample throughout the process of sample custody, there exists the possibility that the samples can leak. It is important to be aware of such hazards, especially when interacting with samples that are highly contaminated.

6.0 CAUTIONS

Samples sometimes require specific storage and maintenance, such as temperature preservation requirements. Proper storage of samples is critical in maintaining their integrity. Labeling is also critical in the process of sample custody. Samples usually are labeled with a series of letters and numbers that correspond to a site location, which sometimes are very similar to each other. Sample nomenclature will be designated in the approved SAP and will be followed in the field. Once a COC or label is generated, it is very important to have it reviewed for quality assurance purposes. Sample label and COC review is necessary to ensure that they match site documents.

7.0 INTERFERENCES

Once a COC and group of labels are reviewed and deployed, it is critical that the proper label ends up on the correct sample container. There will be more than one subsample collected at the majority of sampling locations in the region. This means that sample numbers can be very close in nomenclature, which puts more emphasis on attention to detail when labeling the sample containers. If the wrong label is attached to a sample, it may result in improper preservation, improper analysis, or rejection by the analytical laboratory.

8.0 PERSONNEL QUALIFICATIONS

It is critical that field personnel have proper clearance and health and safety training. Anyone who performs sample custody activities should also familiarize themselves the site-specific SAP and Quality Assurance Project Plan (QAPP), as well as with applicable SOPs: Surface Water Sampling SOP FLD 1.00, Groundwater Sampling SOP FLD 04.00, Soil Sampling SOP FLD 5.00, Pore Water Sampling SOP FLD 10.00, and Shallow Stream Sediment Sampling SOP FLD 06.00.

9.0 EQUIPMENT AND SUPPLIES

Below is a list of equipment and supplies required for COC activities (refer to the site specific SAP for additional items that maybe needed:

- Scribe software
- A SAP that details sample locations and analytical requirements
- Printer (that accepts corresponding labels)
- Blank COC pages in case of unexpected opportunistic sampling
- Permanent marker for preliminary labeling
- Clear tape for label protection from moisture
- Printable labels
- Field Logbook

10.0 STANDARDS AND REAGENTS

There are no standards or reagents associated with this SOP.

11.0 PROCEDURES

The following sections outline the general procedures for sample custody and labeling, filling out COCs with the proper information, and relinquishing samples. See Attachment A for an example of a blank COC and Attachment B for an example of a sample label.

11.1 Generating a Blank COC and Sample Labels

There are several types of data management software that can be used to generate COCs and labels. Scribe is used at the EPA Region 8 laboratory. Some training is required before an individual can use Scribe; however, once the basics of Scribe are understood, it can be used to generate COCs and labels for any type of sample or analysis. A COC that is generated prior to deployment should have the following information:

- Site Identification
- Analysis to be performed
- Preservation
- Tag Identification

The following information should not be filled out until sampling occurs:

- Date
- Time
- Sampler identification
- Comments describing anomalies

Labels can be produced with the same information found in the COCs.

11.2 Populating COC Fields and Affixing Labels

Sample containers should always be marked with a permanent marker with the site identification (ID), time of collection, analysis to be performed, date, and sampler initials prior to sample collection. Once samples have been collected, and a safe place to fill out COC and labels is established, field personnel should fill out the pre-populated COCs and labels with information such as date, time of collection, sampler initials, and comments. It is imperative that the information written on the sample container in permanent marker is the same information on the sample labels and the COC. The same information should also be recorded in a site-dedicated field logbook.

Once the labels have been verified to have the correct information, they should be affixed to the sample containers. Always be sure to double check that the proper label is placed on the corresponding sample container by cross-referencing it with the markings. Once the label is affixed to the sample container, place clear packing tape over the label and wrap completely around the container. This will prevent moisture from dissolving the label adhesive and blurring the writing. It also prevents holes, knicks, or tears from rendering the label unreadable.

11.3 Review/Custody Transfer

Once sample information is written on the COC and labels, and the label IDs have been verified against the permanent marker ID on the container, they are then ready for transfer of custody. Whether the samples are going to the EPA Region 8 lab or a Contract Lab Program (CLP) laboratory partner, the samples must be properly shipped at the required temperature (4°C for water and sediment samples) and done so in a way that containers are not compromised. In order to not compromise the integrity of the samples, the handler needs to make sure the cooler or other transporting vessel is not dropped, exposed to moisture or extreme weather, or in any other way disturbed. A signed copy of the COC intended for the receiving laboratory (samples IDs and event information should not be viewable to the lab) must be included in the shipping container. If samples are returning to the Region 8 Laboratory, they should be properly stored on ice in the field until delivered to the lab. To protect against sample contamination, place the ice in the coolers in plastic bags. When at the lab, samples should be placed in the walk-in coolers located in the sample receiving room. A signed copy of the COC is given to the sample receiving coordinator. In order to ensure samples are transferred to the correct party with the appropriate information and communication, a mutual signing of the COC by the sampler or transport agency and the sample coordinator can be arranged.

12.0 DATA RECORDS AND MANAGEMENT

As mentioned earlier, a COC should have information such as site ID, sample location, sample time, sample date, sampler initials, analytical requirements, sample matrix, preservative type, and a comments field. A sample label should have information such as sample location, time, date, matrix, preservative, and sampler initials. Any other field observations that require an explanation should be noted in the field forms or site-dedicated field notebook. Data such as sample ID, time, date, field parameters, (pH, temperature, conductivity, and dissolved oxygen) and sampler initials will eventually be entered into Scribe.

13.0 QUALITY CONTROL AND ASSURANCE

Proper sample custody and labeling requires a number of quality control and assurance steps. A COC generated in Scribe should always be crossed-checked by another person with the sample list found in the SAP. Completed COCs and labels should also be compared for accuracy before being relinquished to the receiving analytical laboratory. Any incorrect information on a COC or label may cause the lab to reject the shipment.

14.0 REFERENCES

EPA Guidance for Preparing Standard Operating Procedures, EPA QA/G-6, April 2007.

15.0 ATTACHMENTS

Attachment A: Example Chain of Custody Form

TechLaw
ESAT Region 8 Laboratory
16194 W 45th Drive
Golden, CO 80403
303.312.7047

US EPA CLP
Chain-of-Custody

Page 1 of 3

EVENT: 2011_eCOC Template

Sample #	Tag	Location	Sub Location	Sample Type	Collection	Matrix	Analyses	Preservation	Sample Date	Sample Time	Sampler	Remarks

Relinquished By (DATE):

Relinquished By:

Cooler Temp:_____
ICE: Y N
pH: Y N
Cust. Seals: Y N
COC/Labels Agree: Y N
Containers Intact: Y N

Received By (DATE/TIME):

Received By:

Attachment B: Example Sample Label

General Field Sampling Protocols

APPROVED:

ESAT Region 8 QA Coordinator — Date: 04/04/12

ESAT Region 8 Team Manager — Date: 6/6/12

EPA Task Order Project Officer — Date: 7/10/12

ESAT Region 8 Task Lead — Date: 6/8/12

DCN: EP8-7-7051

TABLE OF CONTENTS

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide general field sampling guidelines that will assist Environmental Protection Agency (EPA) and Environmental Services Assistance Team (ESAT) personnel in choosing sampling strategies, location, and frequency for proper assessment of site characteristics. This SOP is applicable to all field activities that involve sampling.

2.0 SCOPE AND APPLICABILITY

These are standard (i.e., typically applicable) operating procedures which may be varied or changed as required, dependent on site conditions, equipment limitations or limitations imposed by the procedure. In all instances, the ultimate procedures employed should be documented and associated with the final report.

3.0 SUMMARY OF METHOD

Sampling is the selection of a representative portion of a larger population, area or body. Through examination of a sample, the characteristics of the larger entity from which the sample was drawn can be inferred. In this manner, sampling can be a valuable tool for determining the presence, type, and extent of contamination by hazardous substances in the environment. The sampling design is a fundamental part of data collection for scientifically based decision making. A well-developed sampling design plays a critical role in ensuring that data are sufficient to draw the conclusions needed. The goals of a sampling design can vary widely. Typical objectives of a sampling design for environmental data collection are:

- To support a decision about whether contamination levels exceed a threshold of unacceptable risk
- To determine whether certain characteristics of two populations differ by some amount
- To estimate the mean characteristics of a population or the proportion of a population that has certain characteristics of interest
- To identify the location of “hot spots” (areas having high levels of contamination) or plume delineation
- To characterize the nature and extent of contamination at a site
- To monitor trends in environmental conditions or indicators of health

A well-planned sampling design is intended to ensure that resulting data are adequately representative of the target population and defensible for their intended use. Representativeness may be considered as the measure of the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition. Throughout the sampling design process, the efficient use of time, money, and human resources are critical considerations. A good design should meet the needs of the study with a minimum expenditure of resources. If resources

4.0 ACRONYMS AND DEFINITIONS

EPA	United States Environmental Protection Agency
ESAT	Environmental Services Assistance Team
DOT	Department of Transportation

HAZWOPER	Hazardous Waste Operations and Emergency Response
IATA	International Air Transport Association
MI	Multi-increment
OSHA	Occupational Safety and Health Administration
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
SAP	Sampling and Analysis Plan
SOP	Standard Operating Procedure

Occupational Safety and Health Administration (OSHA): A regulatory agency that governs health and safety standards in the United States.

Standard Operating Procedure (SOP): A set of written instructions that document a routine or repetitive activity followed by an organization (EPA, 2007).

Quality Assurance Project Plan (QAPP): A site-specific document that specifies quality assurance activities and data quality objectives.

5.0 HEALTH AND SAFETY

When working with potentially hazardous materials, follow U.S. Environmental Protection Agency (EPA), Occupational Safety and Health Administration (OSHA) and corporate health and safety procedures. Always review the site Health and Safety Plan (HASP) before beginning work at any site.

6.0 CAUTIONS

In general, health and safety of field team members and sample/data integrity are the two main concerns during a field sampling event. Field personnel must understand sampling procedures and be familiar with health and safety protocols before deployment to a site. Always consult the HASP before entering a site.

7.0 INTERFERENCES

The nature of the object or materials being sampled may be challenging to characterize. If a material is homogeneous, it will generally have a uniform composition throughout. In this case, any sample increment can be considered representative of the material. On the other hand, heterogeneous samples present problems to the sampler because of spatial and temporal changes in the material. Samples of hazardous materials may pose a safety threat to both field and laboratory personnel. Proper health and safety precautions should be implemented when handling this type of sample. Environmental conditions, weather conditions, or non-target chemicals may cause problems and/or interferences when performing sampling activities or when sampling for a specific parameter. Refer to the specific SOPs for sampling techniques.

8.0 PERSONNEL QUALIFICATIONS

All personnel who participate in field activities are required to obtain clearance in three mandatory health and safety programs: medical monitoring, respirator fit testing, and OSHA Hazardous Waste Operations

and Emergency Response (HAZWOPER) 40-hour training. In addition, any personnel who will participate in sampling activities must read, understand, and sign the site-specific HASP and associated Sampling and Analysis Plan/Quality Assurance Project Plan (SAP/QAPP).

9.0 EQUIPMENT AND SUPPLIES

The equipment required to collect samples must be determined on a site-specific basis. Due to the wide variety of sampling equipment available, refer to the specific SOPs for sampling techniques which include lists of the equipment required for sampling.

10.0 STANDARDS AND REAGENTS

Reagents may be utilized for preservation of samples and for decontamination of sampling equipment. The preservatives required are specified by the analysis to be performed. Decontamination solutions are specified in the Sampling Equipment Decontamination SOP FLD 02.00.

11.0 PROCEDURES

11.1 Types of Samples

In relation to the media to be sampled, two basic types of samples can be considered: the environmental sample and the hazardous sample.

Environmental samples are those collected from streams, ponds, lakes, wells, and are off-site samples that are not expected to be contaminated with high levels of hazardous materials. They usually do not require the special handling procedures typically used for concentrated wastes. However, in certain instances, environmental samples can contain elevated concentrations of pollutants and in such cases would have to be handled as hazardous samples.

Hazardous or concentrated samples are those collected from drums, tanks, lagoons, pits, waste piles, fresh spills, or areas previously identified as contaminated, and require special handling procedures because of their potential toxicity or hazard. These samples can be further subdivided based on their degree of hazard; however, care should be taken when handling and shipping any wastes believed to be concentrated regardless of the degree. The importance of making the distinction between environmental and hazardous samples is two-fold:

1. Personnel safety requirements: Any sample thought to contain enough hazardous materials to pose a safety threat should be designated as hazardous and handled in a manner which ensures the safety of both field and laboratory personnel. Personnel handling potentially hazardous substances should always wear proper Personal Protective Equipment.

2. Transportation requirements: Hazardous samples must be packaged, labeled, and shipped according to the International Air Transport Association (IATA) Dangerous Goods Regulations or Department of Transportation (DOT) regulations and U.S. EPA guidelines.

11.2 Sample Collection Techniques

In general, two basic types of sample collection techniques are recognized, both of which can be used for either environmental or hazardous samples.

Grab (Discrete) Samples
A grab sample is defined as a discrete aliquot representative of a specific location at a given point in time. The sample is collected all at once at one particular point in the sample medium. The representativeness of such samples is defined by the nature of the materials being sampled. In general, as sources vary over time and distance, the representativeness of grab samples will decrease.

Composite (Multi-Increment) Samples
Multi-increment (MI) or composite sampling is a structured sampling protocol that reduces data variability and increases sample representativeness. The objective of MI sampling is to obtain a single sample for analysis that has a mean analyte concentration representative of the decision unit. The decision unit size is site-specific and represents the smallest area on which to base a decision or conclusion. Samples are collected from multiple locations within the decision unit and composited so the samples are spatially representative of the decision unit. The decision unit must be defined so that the results are relevant to explicitly articulated sampling objectives. Note that establishment of decision units is necessary to develop any effective sampling approach, whether using MI or discrete sampling.

The MI sampling strategy improves the reliability and defensibility of sampling data by reducing their variability compared to conventional discrete sampling strategies. The data distribution for MI replicate samples tends to be normally distributed, as contrasted to the positively skewed distribution seen with discrete samples. Fewer non-detect results can be expected using MI, thus mitigating problems caused by using censored data sets and lessening the chance of missing significant contamination. In addition, levels of statistical confidence and decision uncertainty that would require a large number

11.3 Types of Sampling Strategies

It is important to select an appropriate sampling approach for accurate characterization of site conditions. Prior to undertaking any sampling program, it is necessary to establish appropriate measurement and system Data Quality Objectives. Refer to the U.S. Environmental Protection Agency (EPA) Soil Sampling Quality Assurance User's Guide (listed in Section 14.0 References) for guidance in establishing Data Quality Objectives, statistical sampling methodologies and protocols for each of the sampling approaches. Each approach is defined below.

Judgmental or Biased Sampling
Judgmental or Biased sampling is used primarily for documenting an observed release to the groundwater, surface water, air or soil exposure pathways. This form of sampling is based on the subjective selection of sampling locations where contamination is most likely to occur. Locations are based on relative historical site information and on-site investigation (site walk-over) where contamination is most likely to occur.

There is no randomization associated with this sampling approach because samples are primarily collected at areas of suspected highest contaminant concentrations. Any statistical calculations based on the results of this sampling technique will be biased.

Random Sampling

Random sampling, used for the characterization of a heterogeneous non-stratified waste, involves arbitrary collection of samples within a defined area. This method is most effective and accurate if the chemical heterogeneity of the waste remains constant from batch to batch. The easiest method for Random Sampling is to divide the area for sampling into an imaginary grid, assign a series of numbers to the units of the grid, and select the numbers or units to be sampled through the use of a random-numbers table which can be found in the text of any basic statistics book. Note that haphazardly selecting sample numbers or units is not a suitable substitute for a randomly selected sample.

Stratified Random Sampling

Stratified random sampling, used for the characterization of a heterogeneous stratified waste, involves arbitrary collection of samples within a defined area and strata. This method is most effective and accurate if the chemical heterogeneity of the waste remains constant from batch to batch. The easiest method for stratified random sampling is to divide the area for sampling into an imaginary grid, assign a series of numbers to the units of the grid, and select the numbers or units to be sampled through the use of a random-numbers table which can be found in the text of any basic statistics book. A random sample is then collected from each strata at the selected numbers or units on the grid. Note that haphazardly selecting sample numbers or units is not a suitable substitute for a randomly selected sample.

Systematic Grid Sampling

Systematic grid sampling involves dividing the area of concern into smaller sampling areas using a square or triangular grid. Samples are then collected from the intersection of the grid lines or nodes. The origin and direction for placement of the grid should be selected by using an initial random point. The distance between nodes is dependent upon the size of the site or area of concern and the number of samples to be collected. Generally, a larger distance is used for a large area of concern.

Systematic Random Sampling

Systematic random sampling involves dividing the area of concern into smaller sampling areas. Samples are collected within each individual grid cell using random selection procedures.

Search Sampling

Search sampling utilizes a systematic grid or systematic random sampling approach to define areas where contaminants exceed clean-up criteria. The distance between the grid lines and number of samples to be collected are dependent upon the acceptable level of error (i.e., the chance of missing a hot spot). This sampling approach requires that assumptions be made regarding the size, shape, and depth of hot spots.

Transect Sampling

Transect sampling involves establishing one or more transect lines, parallel or non-parallel, across the area of concern. If the lines are parallel, this sampling approach is similar to systematic grid sampling. The advantage of transect sampling over systematic grid sampling is

the relative ease of establishing and relocation transect lines versus an entire grid. Samples are collected at regular intervals along the transect line at the surface and/or at a specified depth(s). The distance between the sample locations is determined by the length of the line and the number of samples to be collected.

11.4 Quality Assurance Project Plans (QAPP)

A Quality Assurance Project Plan (EPA, 2006) is required when it becomes evident that a field investigation is necessary. It should be initiated in conjunction with, or immediately following, notification of the field investigation. This plan should be clear and concise and should detail the following basic components, with regard to sampling activities:

- Objective and purpose of the investigation
- Basis upon which data will be evaluated
- Information known about the site including location, type and size of the facility, and length of operations/abandonment
- Type and volume of contaminated material, contaminants of concern (including concentration), and basis of the information/data
- Technical approach including media/matrix to be sampled, sampling equipment to be used, sample equipment decontamination (if necessary), sampling design and rationale, and SOPs or description of the procedure to be implemented
- Project management and reporting, schedule, project organization and responsibilities, manpower and cost projections, and required deliverables
- QA objectives and protocols including tables summarizing field sampling and QA/QC analysis and objectives

Note that this list of QAPP components is not all-inclusive and that additional element(s) may be added or altered depending on the specific requirements of the field investigation. It should also be recognized that although a detailed QAPP is quite important, it may be impractical in some instances. Emergency responses and accidental spills are prime examples of such instances where time might prohibit the development of site-specific QAPPs prior to field activities. In such cases, investigators would have to rely on general guidelines and personal judgment, and the sampling or response plans might simply be a strategy based on preliminary information and finalized on site. In any event, a plan of action should be developed, no matter how concise or informal, to aid investigators in maintaining a logical and consistent order to the implementation of their task.

11.5 Legal Implications

The data derived from sampling activities are often introduced as critical evidence during litigation of a hazardous waste site cleanup. Legal issues in which sampling data are important may include cleanup cost recovery, identification of pollution sources and responsible parties, and technical validation of remedial design methodologies. Because of the potential for involvement in legal actions, strict adherence to technical and administrative SOPs is essential during both the development and implementation of sampling activities.

Technically valid sampling begins with thorough planning and continues through the sample collection and analytical procedures. Administrative requirements involve thorough, accurate documentation of all sampling activities. Documentation requirements include maintenance of a chain of custody, as well as accurate records of field activities and analytical instructions. Failure to observe these procedures fully and consistently may result in data that are questionable, invalid and non-defensible in court, and the consequent loss of enforcement proceedings.

12.0 DATA RECORDS AND MANAGEMENT

There are many data parameters and custody records that require attention to detail. Refer to the specific SOPs for data management activities that are associated with sampling techniques.

13.0 QUALITY CONTROL/QUALITY ASSURANCE (QC/QA)

Refer to the specific SOPs for the type and frequency of QA/QC samples to be analyzed, the acceptance criteria for the QA/QC samples, and any other QA/QC activities which are associated with sampling techniques.

14.0 REFERENCES

EPA Guidance for Preparing Standard Operating Procedures, EPA QA/G-6. April 2007.
EPA Guidance on Systematic Planning using the Data Quality Objectives Process (QA/G-4). February 2006.

EPA Guidance on Choosing a Sampling Design for Environmental Data Collection (QA/G-5S). December, 2002

Attachment 1
Blank Field Datasheet

Project Field Datasheet Number _____ of _____

2017-2018 Southern Ute Indian Tribe Wild Plant Sampling

Field Personnel (Identify Field Team Manager and Samplers):

Data Recorder/Scribe (Name and Contact Information):

Weather Conditions:

Sampling Area Location Information (General Coordinates):

Sampling Equipment Decontaminated Prior to Use (Yes/No)

Sample Information

Sample ID	Plant Genus/Species	# of sub-samples	Date	Time (MST)	Notes

Photograph Log:

Attachment 2
Blank Chain of Custody Form

COC From Number _____ of _____ Page _____ of _____

Bonita Peak Mining District Risk Assessment 2017-2018 Southern Ute Indian Tribe Wild Plant Tissue Sampling Chain of Custody Form

This chain of custody form is used to provide sample information and relinquish custody of plant tissue and soil samples to the analytical laboratory. Refer to the wild plant collection sampling and analysis plan/QAPP for detailed instructions on how to use this form. The completed original version of this form will be sealed with respective samples when sending samples by currier service.

Sample Custodian Contact Information	Analytical Laboratory Contact Information
Name and affiliation:	Laboratory name: EPA Region 8 Laboratory
	Contact name and affiliation: Scott Walker / ESAT
Address:	Address:
	16194 West 45th Dr. Golden, CO 80403
Phone number:	Contact phone number: (303) 312-7726 or (303) 312-7700
Email address:	Contact email address: walker.scott@Epa.gov

Plant code	Plant part code	Unique sample no.	Sample date	Sample time	Impacted or ref. location	Soil sample ID	Notes
-	-						
-	-						
-	-						
-	-						
-	-						
-	-						
-	-						
-	-						
-	-						
-	-						

Relinquished By			Received By		
Date	Time	Name/affiliation	Date	Time	Name/affiliation

Attachment 3
Equipment List

Attachment 3 Equipment List

Task	Item	Quantity	Notes
Sample Documentation and Collection			
	Copy of the wild plant SAP/QAPP	1	Suggest using Rite in the Rain All-Weather Paper
	Blank field datasheets	1 sheet per 14 samples	Suggest using Rite in the Rain All-Weather Paper
	Blank chain of custody sheets	1 per 10 samples	Suggest using Rite in the Rain All-Weather Paper
	Bound field notebook	1	Suggest using Rite in the Rain Env. Field Book
	GPS units with extra batteries	1	
	Digital camera w with batteries and data card	1	
	Nitrile gloves	1 pair per sample per sampler	Suggest getting a couple of different hand sizes
	Ceramic or stainless steel chef knife	2	One working with a backup
	Plastic or similar non-metallic garden trowel	2	One working with a backup
	Full size spade shovel (non-metallic not available)	1	
	Plastic scoops for soil samples	1 to 2 per collection area	New, clean and certified
	Limb or pruning saw (non-metallic not available)	2	One working with a backup
	Ceramic or stainless steel utility scissors	2	One working with a backup
	Garden shears (non-metallic not available)	2	One working with a backup
	Plastic or similar non-metallic cutting boards	3	
	Soft bristle brushes	3	
	A-frame ladder	1	May be need to reach fruits and seeds
	Distilled, deionized, or reverse osmosis water	Couple gal. per day	To rinse vegetation; obtain from store or laboratory
	Laboratory water wash bottles	2	To rinse vegetation
	Plastic washing bowls	4	To rinse vegetation
	1-gallon Ziploc baggies	2 per sample	Double bag sample container
	Brown paper bags	As needed	New and clean
	4 oz glass jars for soil samples	1 to 2 per collection area	New, clean and certified
	250 mL HDPE bottles for equipment blank samples	As needed	New, clean and certified
	Fine and extra fine point Sharpie permanent markers for sample labels	Many	Useless when they get wet so bring extras
	Blue and black pens, paper clips, clipboards, scissors	Many	Note that Rite in the Rain paper is not compatible with most types of pens
	Sample labels	1 per sample	High quality, self adhesive
	Clear packaging tape	Few rolls	To cover sample labels
	Paper towels	Few rolls	
	Coolers with lids	At least two	To store samples and extra ice
	Pre-frozen ice packs or bagged wet-ice	At least 20 lbs.	Ice packs preferred
Equipment Decontamination and Cleanup			
	Nitrile gloves	1 pair per decon. session	Suggest getting a couple of different hand sizes
	Alconox powdered or Liquinox liquid detergent	1/4 lb. box or 1 L bottle	Specially made for metals decon with no residue
	Laboratory water wash bottles	1	For detergent; can also use any similar plastic squeeze bottles
	Carboy of tap water with spigot	1 five gallon carboy	Initial decon. rinse; also used for general cleanup
	Distilled, deionized, or reverse osmosis water	Couple gal. per day	Final decon. Rinse and make equipment blank samples
	Non-metallic hard and soft brittle brushes	Couple of sets	New and clean
	Drop cloth/plastic sheeting	1 roll	Useful to cover ground and create a clean decon. work station
	Plastic trash bags	Couple	For trash and storing decontaminated equipment
	5 gallon plastic buckets with lids	3	For a wash basin and to store wash water/reinstate for off-site disposal
Health and Safety and Hygiene/Cleanup			
	First aid kit	1	
	Eyewash bottle	1	Saline filled
	Work gloves	Couple pairs	
	Safety glasses/goggles		
	Hard hats		
	Hand soap	1 bar/bottle	
	Carboy of tap water with spigot	1 five gallon carboy	Also used for equipment decontamination
	Sunblock	1 tube	
Sample Shipping			
	Copy of the wild plant SAP/QAPP	1	
	Completed chain of custody forms	For all samples being shipped	Original forms - sample custodian retains photocopies
	Photocopier	1	To photocopy completed COC forms
	1-gallon Ziploc baggies	Few per cooler	Use to bag wet ice, organize samples, and seal COC form
	Coolers with lids	As needed	Medium sized, ~40 quart coolers are ideal for shipping
	Temperature blank bottle with lid	1 per cooler	Label "Temp. blank", fill with cold water, and tightly cap
	Pre-frozen ice packs or bagged wet-ice	8 to 10 lbs. per cooler per 24-hrs hold time	Ice packs preferred
	Clear packaging tape	Few rolls	To secure and seal cooler lid
	Air bill shipping forms	1 per shipment	FedEx or similar