Final, Revision 0
Sampling and Analysis Plan/Quality Assurance Project Plan
2015 Sampling Events

Upper Animas Mining District
San Juan County, Colorado

Prepared For:
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Ecosystem Protection and Remediation – Program Support
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Final Revision 0	6/1/2015	Changed May event to June, updated background information, and updated the problem definition

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List of Abbreviations and Acronyms

ABA	Acid-Based Accounting
ARSG	Animas River Stakeholder Group
BERA	Baseline Ecological Risk Assessment
BLM	Bureau of Land Management
CA	Corrective Action
CDPHE	Colorado Department of Public Health and Environment
CFR	Code of Federal Regulations
CLP	Contract Laboratory Program
COPEC	Contaminant of Potential Ecological Concern
CPR	Cardiopulmonary Resuscitation
DO	Dissolved Oxygen
DOC	Dissolved Organic Carbon
DOI	United States Department of Interior
DQA	Data Quality Assessment
DQO	Data Quality Objectives
DRMS	Division of Reclamation, Mining and Safety
DS	Decision Statement
DSR	Data Summary Report
EDD	Electronic Data Deliverable
EDITQ	Equal Discharge Increment Technique
ESAT	Environmental Services Assistance Team
EPA	United States Environmental Protection Agency
gpm	Gallons per minute
GPS	Global Positioning System
HDPE	High-Density Polyethylene
Hg	Mercury
LCS/LCSD	Laboratory Control Spike/Laboratory Control Spike Duplicate
LEW	Left Edge Water
LIMS	Laboratory Information Management System
MDL	Method Detection Limit
MMI	Multi-Metric Index
MS/MSD	Matrix Spike/Matrix Spike Duplicate
OSHA	Occupational Safety and Health Administration
PE	Performance Evaluation
PQL	Practical Quantitation Limit
PSQ	Principal Study Question
QA/QC	Quality Assurance/Quality Control
QAO	Quality Assurance Officer
QAPP	Quality Assurance Project Plan
QMP	Quality Management Plan

REW	Right Edge Water
RI	Remedial Investigation
RPD	Relative Percent Difference
RPM	Remedial Project Manager
SAP	Sampling Analysis Plan
SAR	Sampling Activities Report
SGC	Sunnyside Gold Corporation
Site	Cement Creek and the Animas River
SOP	Standard Operating Procedure
SPLP	Synthetic Precipitation Leaching Procedure
TAL	Target Analyte List
TMDL	Total Maximum Daily Loads
TOPO	Task Order Project Officer
TU	Trout Unlimited
USFS	United States Forest Service
USFWS	United States Fish and Wildlife Service
USGS	United States Geological 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 sampling events scheduled in 2015 at the Upper Animas Mining District. Agency and contractor affiliations are also listed for each individual.

Paula Schmittdiel	EPA
Elizabeth Fagen	EPA
Steve Way	EPA
Dan Wall	EPA
Nicole Plescia	EPA
Lisa Richardson	BLM
Brent Lewis	BLM
Sherry Skipper	USFWS
Mary Blanchard	USFS
Randy Perlis	CDPHE
Kirsten Brown	DRMS
Pat Maley	SGC
Larry Perino	SGC
Peter Butler	ARSG
William Simon	ARSG
Steve Fearn	ARSG
Steven Auer	ESAT Field Task Lead
Mark McDaniel	ESAT Manager

A.4 Project and Task Organization

The following is a list of involved personnel, respective agencies and contract affiliations, and general responsibilities.

Managers:

Paula Schmittdiel	EPA	Remedial Project Manager
Elizabeth Fagen	EPA	Remedial Project Manager
Steven Way	EPA	On-Scene Coordinator
Mark McDaniel	ESAT	ESAT Team Manager

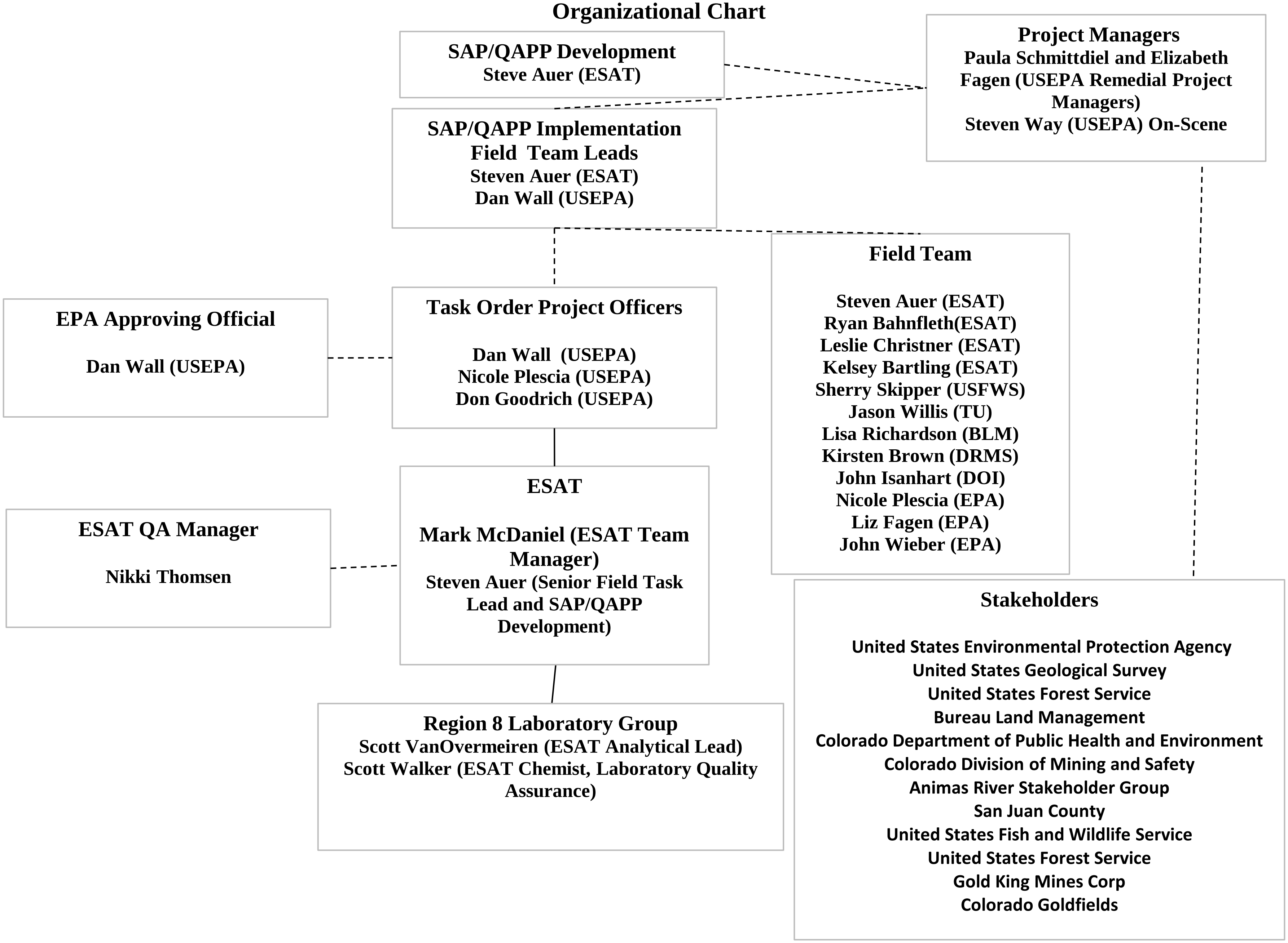
Field Team:

Steve Auer	ESAT	Field Task Lead
Ryan Bahnfleth	ESAT	Field Support
Leslie Christner	ESAT	Field Support
Kelsey Bartling	ESAT	Field Support
Sherry Skipper	USFWS	Field Support
Jason Willis	TU	Field Support
Lisa Richardson	BLM	Field Support
Kirsten Brown	DRMS	Field Support
John Isanhart	DOI	Field Support
Nicole Plescia	EPA	Field Support
Liz Fagen	EPA	Field Support
John Wieber	EPA	Field Support
Dan Wall	EPA	Field Support

Laboratory Group:

Scott VanOvermeiren	ESAT	Inorganic Task Lead
Scott Walker	ESAT	Analytical Support

Organizational Chart



SAP/QAPP Development
Steve Auer (ESAT)

Project Managers
Paula Schmittdiel and Elizabeth Fagen (USEPA Remedial Project Managers)
Steven Way (USEPA) On-Scene

SAP/QAPP Implementation Field Team Leads
Steven Auer (ESAT)
Dan Wall (USEPA)

Field Team

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Ryan Bahnfleth(ESAT)
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Steven Auer (Senior Field Task Lead and SAP/QAPP Development)

Stakeholders

United States Environmental Protection Agency
United States Geological Survey
United States Forest Service
Bureau Land Management
Colorado Department of Public Health and Environment
Colorado Division of Mining and Safety
Animas River Stakeholder Group
San Juan County
United States Fish and Wildlife Service
United States Forest Service
Gold King Mines Corp
Colorado Goldfields

Region 8 Laboratory Group
Scott VanOvermeiren (ESAT Analytical Lead)
Scott Walker (ESAT Chemist, Laboratory Quality Assurance)

A.5 Problem Definition

The Animas River flows through the town of Silverton in San Juan County, CO. This waterway is affected by flow which has come in contact with mineralized material, either naturally or as a result of mining activities, such as through the creation of mine adits. Affected water originates in the Animas River headwaters and its tributaries above Silverton and the upper reaches of the two major tributaries of the Animas River in this area, namely Cement Creek and Mineral Creek.

Recent investigations of the potential impacts of metals from Cement Creek on the Upper Animas River (the “Site”) have revealed significant sources of contamination upstream of Cement Creek at levels likely harmful to aquatic life (ESAT, 2015). A significant amount of information has been collected from the Animas River above Silverton over the last 20 years (New Mexico Environment Department 2013, Church et al., 2007, and Herron et al., 2000) however, it is not clear that the information is representative of current conditions. This sampling plan will provide current information regarding the nature and extent of contamination above the influence of Cement Creek.

Additionally, this SAP also includes preliminary nature and extent of contamination in pore water, sediment and fluvial deposits of tailings and waste rock in and adjacent to the Upper Animas River.

Recent sampling events by the United States Environmental Protection Agency (EPA) have also focused on determining the load allocation amongst known metal sources in Cement Creek. These investigations have led EPA to conclude that installation of a bulkhead in the Red and Bonita Mine is necessary to reduce metals loading in the Animas River. The installation of the bulkhead is planned for the Fall of 2015 and sentinel monitoring locations in the Cement Creek drainage have been selected to assess the effects of this action.

This SAP/QAPP will present a plan to collect sediment, surface water, pore water, and soil samples for laboratory analysis. The data collection effort will accomplish the following:

- Determine the baseline metals loads and concentrations at sentinel locations in Cement Creek prior to installation of the bulkhead in the Red and Bonita mine;
- Determine the loads and concentrations of metals in the Animas River headwaters and headwater tributaries during high-flow and low-flow conditions;
- Determine the nature and extent of soil, mine waste and tailings-derived metals contamination at select floodplain locations;

- Determine the preliminary nature and extent of sediment metals contamination in the Upper Animas River; and
- Determine the preliminary nature and extent of sediment pore water metals contamination in the Upper Animas River.

A.5.1 Introduction

EPA, in cooperation with participating stakeholders, will be sampling the Site as part of a Remedial Investigation (RI), and to characterize mine waste at select locations. Mobilization and watershed sampling activities are scheduled to be performed during the weeks of June 8, 2015; August 3, 2015; and September 28, 2015.

This SAP/QAPP has been prepared in accordance with the EPA "*Guidance on Systematic Planning Using the Data Quality Objectives Process (EPA QA/G-4)*, *Requirements for Quality Assurance Project Plans (QA/R-5)*", and the "*Guidance for Quality Assurance Project Plans (EPA QA/G-5)*", (EPA 2006; EPA 2001; EPA 2002). This SAP/QAPP is designed to guide field work that will include the collection of surface water, pore water, sediment, and soil samples as well as flow measurements, including field Quality Assurance/Quality Control (QA/QC) samples.

Laboratory analyses for the June high-flow event will include: total and dissolved Target Analyte List (TAL) metals and hardness (calculated from calcium and magnesium concentrations in the dissolved ratio), for surface water. Laboratory analyses for the September low-flow event will include: total and dissolved TAL metals and hardness, as well as anions and alkalinity for surface water, dissolved and total TAL metals for pore water, and total TAL metals including mercury (Hg) for sediment samples. Laboratory analyses for the summer event will include: total metals including Hg for sediment samples, and total metals including Hg, Synthetic Precipitation Leaching Procedure (SPLP), and Acid-Based Accounting (ABA) for mine waste/soil samples. All samples will be analyzed either directly or indirectly (by means of subcontractors) through the Environmental Services Assistance Team (ESAT) Region 8 Laboratory. Field-collected measurements of aqueous samples during both high and low-flow events will include temperature, pH, specific conductivity, Dissolved Oxygen (DO), and flow measurements.

Sampling procedures will adhere strictly to those outlined in the attached ESAT Region 8 Standard Operating Procedures (SOPs). Deviations from this SAP/QAPP will be documented in the Site-dedicated field notebook and reported in the Sampling Activities Report (SAR). Deviations that result in major modifications to this SAP/QAPP will be noted and incorporated into all addenda to this SAP/QAPP, which will be followed as applicable for subsequent sampling events. A brief addendum to this SAP/QAPP will be created for each subsequent sampling event by updating relevant figures, tables, and attachments. Data obtained from these investigations will be used in accordance with the provisions outlined in the Data Quality Objectives (DQOs).

A.5.2 Background

The discovery of gold and silver brought miners to the Silverton area and Animas Mining District in the early 1870's. The discovery of silver in the base-metal ores was the major factor in establishing Silverton as a permanent settlement. Between 1870 and 1890, the richer ore deposits were discovered and mined to the extent possible. Not until 1890 was any serious attempt made to mine and concentrate the larger low-grade ore bodies in the area. By 1900, there were twelve concentration mills in the valley sending products to the Kendrick and Gelder Smelter near the mouth of Cement Creek. Mining and milling operations slowed down circa 1905, and mines were consolidated into fewer and larger operations with facilities for milling large volumes of ore. After 1907, mining and milling continued throughout the basin whenever prices were relatively favorable.

Gladstone, located about eight miles upstream of Silverton on Cement Creek, is the site of an historic mining town developed in the 1880s commensurate with the onset of mining in the surrounding area. The town was the central location and railroad terminus for the milling and shipping of mine ores from the surrounding three-square-mile valley. The town declined in the 1920s and no remnants of the town currently remain. By the 1970's only one year-round productive mine (Sunnyside Mine) remained in the county. This mine ceased production in 1991, and has since undergone extensive reclamation efforts. Numerous historic and now abandoned mines exist within a two-mile radius of Gladstone. These include: the Upper Gold King 7 Level, American Tunnel, Grand Mogul, Mogul, Red & Bonita, Evelyne, Henrietta, Joe and John, and Lark mines.

Howardsville, located between Silverton and Eureka at the mouth of Cunningham Creek, was established in 1874 by the Bullion City Company. Howardsville became the base for many mines up Cunningham Gulch including: the Old Hundred Mine, Buffalo Boy, Green Mountain, Pride of the West, Shenandoah-Dives Mine, Gary Owen Mine, and Emma Mine (Herron et al., 2000). The Pride of the West mill was built in 1940 as a 50-ton capacity mill. It was expanded in 1967 by the Dixilyn Corporation to a 400-ton capacity mill (Church et al., 2007).

The town of Eureka is located approximately eight miles northeast of Silverton at the confluence of Upper Animas and Eureka Gulch. Some of the mines located up Eureka Gulch include: the Sunnyside Mine, the Clipper Mine, the Ben Franklin Mine, the Bavarian Mine, the Midway Mine, the Moonbeam Mine, and the Ransom Mine (Herron et al., 2000). The Sunnyside Flotation Mill in Eureka was built in 1917 with a 600 tons per day capacity. Two settling ponds were built in the Animas River Valley but when the mill was abandoned in 1949, the tailings dams were partially washed out and tailings were washed down the Animas River (Church et al., 2007).

Animas Forks, named for the three forks of the Animas River, is located twelve miles northeast of Silverton in San Juan County, Colorado and was first established in 1874. There were numerous mines located upstream of Animas forks. The town started to decline in 1910 when the Gold Prince Mill ceased operation and became a ghost town in the 1920's.

EPA along with the ESAT, Bureau of Land Management (BLM), United States Fish and Wildlife Service (USFWS), United States Geological Service (USGS), and United States Department of Interior (DOI) performed four sampling events from 2012 through 2014 to add to previously collected data. Over the five events, surface water, sediment, pore water, macroinvertebrates, stream flows and real-time water quality parameters were collected.

A.6 Project and Task Description

Sampling events will be conducted in 2015 to evaluate the extent of metals contamination in surface water, pore water and sediment in the Cement Creek drainage, several tributaries that feed into the Animas River, and the Animas River. The Site is near the headwaters of the Animas and select locations downstream to Bakers Bridge, including several tributaries (Mineral Creek, Burrows Creek, Placer Gulch, Grouse Gulch, Picayune Gulch, California Gulch, Eureka Gulch, Horseshoe Creek, Cinnamon Creek, Burns Gulch, Niagara Gulch, Minnie Gulch, Maggie Gulch, Hematite Gulch, and Arastra Creek) that feed into the Animas River. Additionally, samples of mine waste and tailings will be collected to evaluate the nature and extent of metals contamination in waste areas and their contribution and potential contribution to surface water. Additionally, fluvial deposits within floodplain areas will be characterized for metals at various depths and areas adjacent to the Animas River. Their actual or potential contribution to surface water will also be assessed. Data generated from the sampling events will be used in accordance with the established DQOs (Section A.7.2). The following data will be collected during the events:

- Real-time field water quality parameters – pH, conductivity, DO, temperature, and Global Positioning System (GPS) locations (if needed);
- Stream flows – using SonTek^{TM} flow meters, flumes (where necessary), and USGS gauging stations;
- Surface water samples, collected at predetermined sampling locations in streams and adit discharges for measurement of dissolved and total recoverable metals, alkalinity, and anions;

- Sediment samples – total recoverable metals and Hg;
- Pore-water samples – total and dissolved metals, hardness and pH;
- Soil samples (including SPLP and ABA methods) – total recoverable metals and Hg; and
- Photolog – Site photographs will be collected for all sampling locations.

A.7 Quality Objectives and Criteria

This section discusses the DQO process and how it was applied to this study. Specific areas addressed include the planning team and stakeholders, DQOs, and parameter metrics such as: precision, accuracy, representativeness, completeness, comparability and sensitivity. Data collected during these sampling events will be intended to achieve the following:

- Determine the baseline loads and concentrations of metals in Cement Creek prior to the installation of a bulk head in the Red and Bonita mine;
- Determine the concentrations and loads of metals in the Animas River and tributaries at the headwaters;
- Determine the nature and extent of metals contamination of sediment and sediment pore water in the Upper Animas River; and
- Determine the nature and extent of soil, mine waste, and tailings-derived metals contamination at select floodplain locations.

A.7.1 Planning Team and Stakeholders

The following sections list the members of the DQO planning team: 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.

A.7.1.1 DQO Planning Team

The following table includes the DQO planning team members, their respective organizations, and affiliation with that organization.

Table A.7-1 DQO Planning Team

Name	Organization	Area of Technical Expertise
Paula Schmittdiel	EPA Region 8	Remedial Project Manager
Elizabeth Fagen	EPA Region 8	Remedial Project Manager
Dan Wall	EPA Region 8	Toxicologist
Steven Way	EPA Region 8	On-Scene Coordinator
Lisa Richardson	BLM	Physical Scientist
Sherry Skipper	USFWS	Biologist
Steve Auer	ESAT	Biologist

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 this event is Paula Schmittdiel and Elizabeth Fagen, the EPA Region 8 Remedial Project Managers (RPMs) for this Site.

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.

Table A.7-2 Stakeholders

Organization	Represented By
EPA Region 8	Paula Schmittdiel
EPA Region 8	Elizabeth Fagen
EPA Region 8	Steve Way
ARSG	Peter Butler/William Simon/Steve Fearn
Gold King Mines Corp	Todd Hennis
BLM	Brent Lewis
CDPHE	Randy Perlis
Colorado Division of Reclamation	Kirstin Brown
SGC	Larry Perino
USFWS	Sherry Skipper
USFS	Mary Blanchard

A.7.2 Data Quality Objectives

The DQO process specifies project decisions, the data quality required to support those decisions, specific data types 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 that will be used to develop the data collection design. The final step of the process involves developing the data collection design based on the DQOs. A brief discussion of these steps and the corresponding application to this project are provided in the following sections.

A.7.2.1 Step 1: State the Problem

A substantial amount of data has been collected over the last 20 years from the Site. It is unclear if this information is representative of current conditions of the Site, particularly above the influence of Cement Creek. The water, which is affected by flow that has come into contact with mineralized material, results in increased metals concentrations and a lower pH of the water. This mineralized material is either a result of mining activities or naturally occurring. This affected water originates in the Animas headwaters and tributaries above Silverton and the upper reaches of the two major tributaries, Mineral Creek and Cement Creeks.

Cement Creek, the receiving stream for the discharge of the American Tunnel, Red and Bonita, Mogul, and Gold King Level 7, is unable to support aquatic life. Cement Creek is a major contributor of metals and acidity to the Animas River. With the planned installation of a bulkhead in the Red and Bonita mine, Cement Creek needs to be monitored to assess the effects of this action.

Further, there is little data available on the extent of contamination in pore water, sediment, and fluvial deposits of tailings and waste rock in certain areas within the floodplain of the Upper Animas River.

In order to address these data gaps and determine the extent of contamination upstream of Howardsville, sediment, surface water, pore water, and soil (from tailings and mine waste) samples will be collected for laboratory analysis.

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 will help determine appropriate data inputs and potential alternative actions. PSQs can be used to develop Decision Statements (DSs) when the potential alternative actions have been determined to resolve the problem. In situations where the outcomes may not lead to specific decisions or the information may be used to gain a greater understanding of existing data, estimation statements are more appropriate. Estimation statements are more applicable to the nature of the PSQs being investigated in these sampling efforts.

The PSQs are as follows:

PSQ 1 – What are the concentrations and loads of metals in the Animas River headwaters and headwater tributaries, during high-flow and low-flow conditions?

PSQ 2 – What is the current nature and extent of metal contamination in the Animas River headwater sediments and pore water?

PSQ 3 – What is the nature and extent of metal contamination in mine waste and tailings piles in the Animas River headwaters and headwater tributaries and are they contributing or do they have the potential to contribute to metals in surface water?

PSQ 4 – What is the nature and extent of metal contamination in select riparian areas of the Animas River?

PSQ 5 – What are the baseline metals loads and concentrations in Cement Creek prior to installation of a bulkhead in the Red and Bonita mine?

Estimation Statements

PSQ 1: What are the concentrations and loads of metals in the Animas River headwaters and headwater tributaries, during high-flow and low-flow conditions?

Surface water sampling for metals in conjunction with flows in the Animas River will be used to determine metals concentrations and loads in the Animas River during runoff and low-flow conditions in the Fall.

PSQ 2: What is the current nature and extent of metal contamination in the Animas River headwater sediments and pore water?

Results from sediment samples will be used to better quantify the metals concentrations in sediments and help to identify the potential source of these metals.

PSQ 3: What is the nature and extent of metal contamination in mine waste and tailings piles in the Animas River headwaters and headwater tributaries and are they contributing or do they have the potential to contribute to metals in surface water?

Identified waste piles will be sampled to determine bulk and leachable metals concentrations. These results in conjunction with loading measurements that bracket these waste piles will be used to determine their actual and/or potential contribution to surface water.

PSQ 4: What is the nature and extent of metal contamination in select riparian areas of the Animas River?

Composite samples will be collected in riparian areas within the Eureka tailings, Kittimack tailings, Minnie Gulch tailings, Forest Queen, and tailings near Grouse Gulch to determine bulk and leachable metals concentrations. Additionally, surface and pore water samples will be collected to determine the potential contribution of solid wastes in the riparian areas to surface water metals concentrations.

PSQ 5: What are the baseline metals loads and concentrations in Cement Creek prior to installation of a bulkhead in the Red and Bonita Mine?

Sentinel locations, potentially influenced by the installation of a bulkhead in the Red and Bonita mine, will be sampled prior to its installation. These results will serve as part of the baseline condition to monitor changes in water quality subsequent to its installation.

A.7.2.3 Step 3: Identify Information Inputs

The purpose of this step is to identify the data required to answer the PSQs listed in section A.7.2.2. The primary information and decision inputs will be data generated from field instruments and laboratory analyses, as well as established water quality for comparison.

Field parameters and non-sampling objectives include:

- Measuring temperature, pH, specific conductivity, and DO;
- Conducting flow measurements;
- Determining metals concentrations in sediments collected at Animas River and other tributary sampling locations;
- Determining metals concentrations, acid production potential and leachable metals in soil samples of mine waste areas and tailings-pile floodplain areas;
- Determining metals concentrations in pore water at select Animas River locations;
- Photographing the sampling locations and other notable observations; and
- Recording geospatial location data using GPS.

Analytical laboratory parameters for routine and opportunistic surface water samples will include:

- Total and dissolved TAL metals and hardness (calculated); and

- Analysis of alkalinity and anions for surface water samples collected during the low-flow event.

Laboratory method detection limits (MDLs) will be below selected standards and applicable benchmarks that were established in the Baseline Ecological Risk Assessment (BERA) (ESAT, 2015).

The following factors will be evaluated in the overall decision-making process:

- Comparison of surface water and pore water results to applicable standards or benchmarks and water quality criteria according to *Colorado Table Value Standards* or *National Recommended Ambient Water Quality Criteria*;
- Sediment analytical results will be compared to probable and threshold effect concentrations as described in *Development and Evaluation of Consensus-Based Sediment Quality Guidelines for Freshwater Ecosystems* (MacDonald et al., 2000);
- Comparison of analytical data to applicable historical data to evaluate changes in water chemistry and metals loading over time; and
- Comparison of analytical data of source areas to analytical data of background areas.

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. The scale of the decision making for the DS is defined by combining the population of interest with the study's spatial and temporal boundaries. Practical constraints that could interfere with sampling are also identified. Implementing this step helps ensure that the data are representative of the population.

A.7.2.4.1 Spatial Boundaries

The study area boundary comprises the Cement Creek watershed and Animas River, including several tributaries, upstream of Howardsville and downstream to Bakers Bridge. A strong emphasis is placed on known or suspected locations, or near the headwaters of the Animas River. The sampling locations for this event include: Mineral Creek, Burrows Creek, the North Fork of the Animas River, Placer Gulch, Grouse Gulch, Picayune Gulch, California Gulch, Eureka Gulch, Horseshoe Creek, Cinnamon Creek, Burns Gulch, Niagara Gulch, Minnie Gulch, Maggie Gulch, Hematite Gulch, and Arastra Creek, including subareas of each of those stations. The locations were selected in order to characterize previously uncharacterized mine and mine waste areas. Seeps, springs, or adits that contribute to flows into and out of the Animas River encountered in the field may also be sampled. Figures A.7-1 through A.7-4 shows the surface water sampling locations.

Finally, specific floodplain areas will be sampled to determine the nature and extent of contamination in floodplain areas, specifically: Eureka Tailings below Eureka, Kittimack Tailings, Minnie Gulch, Forest Queen, and Tailings near Grouse Gulch. Figures A.7-5 through A.7-7 show the sampling areas for the floodplain sampling and tailings areas.

A.7.2.4.2 Temporal Boundaries

Sampling activities will start in June 2015 and end in September 2015. Specific dates are dependent on river flow conditions and availability of accessibility to Animas River sample locations. The current schedule is as follows:

- June, 2015 High-flow sampling event, surface water;
- August, 2015 Summer sediment, mine waste, and tailings sampling; and
- September, 2015 Low-flow sampling event surface water, pore water, sediment.

A.7.2.5 Step 5: Develop the Analytic Approach

Total recoverable metals results and stream discharge measurements will be used to calculate loading of Site-related contaminants from contributing sources. The results from each of the inputs will be evaluated to determine the location of significant loading of Site-related contaminants into the Animas River. Concentrations of dissolved metals in surface water and pore water will be compared to applicable standards, benchmarks, and water quality criteria either with the *Colorado Table Value Standards* or *National Recommended Ambient Water Quality Criteria*. Concentrations of metals in sediments will be compared to *Consensus-Based Sediment Quality Guidelines for Freshwater Ecosystems* (MacDonald et al., 2000). Concentrations of metals in the soil will be compared to EPA *Ecological Soil Screening Levels* (EPA, 2010) or other relevant benchmarks. All analytical results will have detection limits suitable for comparison to these benchmarks and standards.

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. For this project, the number of samples and station locations are based largely on past investigations performed in years 2009 through 2014, and the data collected will build upon previously collected data in order to expand the understanding of the Site.

In order to mitigate the potential for false positive or false negative errors associated with field sampling, sample collection processes will be consistent with established and relevant SOPs. This includes collection of duplicate samples (and subsequent analysis using Relative Percent Difference (RPD) statistics), implementing a decontamination procedure (which may include the use of disposable sampling equipment), and the use of field blanks. For laboratory analysis of samples, QA/QC steps (such as the use of laboratory controls, matrix spikes and matrix spike duplicates (MS/MSDs), blanks, etc.) will be consistent with ESAT Region 8 reporting requirements.

A.7.2.7 Step 7: Develop Plan for Collecting Data

A judgmental sampling design as described in *Guidance for Choosing a Sampling Design for Environmental Data Collection*, December 2002 (EPA QA/G-5S) will be used to assist with identification and verification of the sources of Contaminants of Potential Ecological Concern (COPECs). Sampling locations were identified from the historical sampling locations and stations were refined study locations upstream of Howardsville (A55) in order to define the nature and extent of contamination in that area and identify any additional sources of contamination. Data collected from this event will assist with identifying the order and magnitude of contamination reaching the Upper Animas River and extent of migration to downstream locations through Bakers Bridge. Specific parameters of interest for the sampling events will follow the criteria listed in Section A.7.2.2 and are described in Tables A.7-5 through A.7-7. Analytical methods for the events are described in Section B.4 and management of the data is described in B.10 of this document.

A.7.2.8 Sampling Locations

Sampling locations, descriptions, and activities that will take place in 2015 are listed in Tables A.7-5 through A.7-7 and shown in Figures A.7-1 through A.7-7. Table A.7-8 shows the GPS coordinates for each of the sampling locations. Sampling locations will be verified to the sub meter using Trimble GPS handheld devices as needed.

A brief description of the sampling locations will be recorded in the Site-dedicated field notebook for each location sampled. Information will consist of sampling location identification number, date, time, access information, geographical observations, and any other pertinent information that will be useful in identifying the sampling location in the future. A photolog will be kept for locations and activities, and photo identifications as assigned by the camera will be logged in the Site-dedicated field notebook. If there are deviations from this SAP/QAPP or applicable SOPs, including the decision to not sample a location because conditions are either unsafe or there are accessibility problems, these changes will also be recorded in the Site-dedicated field notebook.

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

Tables A.7-3 and A.7-4 provide the MDLs and Practical Quantitation Limits (PQLs). In every case, the MDLs and PQLs fall well below the screening criteria, indicating that the analytical methods will be able to measure contaminant levels in the surface water samples with the required sensitivity.

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

The documentation of the data evaluation effort will be in the form of the worksheets prepared during validation. These worksheets will be an appendix to the SAR. The SAR will be prepared to identify problems that may affect data usability or require that the data be qualified. The SAR will discuss all precision, accuracy, representativeness, completeness, comparability, and sensitivity parameter results from the data validation and overall usability of the data for project objectives, which include the following:

Precision:
- 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:
- MS/MSDs: Percent Recovery (%R) criteria met?
- Laboratory control spike/laboratory control spike duplicates (LCS/LCSDs): %R criteria met?
- Initial and continuing calibration recoveries met?
- Interference check sample recoveries met?
- Inductively coupled plasma serial dilution recoveries met?

Representativeness:
- Sampling procedures and design: criteria met?
- Holding times and preservation: criteria met?
- Custody: all chain-of-custody forms complete and provided in data package?
- Blanks: contaminants present?

Completeness:
- The number of valid analytical results are comparable (90%) with the number determined necessary during establishment of DQOs.

Comparability:
- 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:
Method reporting limits met project objectives?

The data will be assessed for the following criteria:

- *Bias* – a 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, LCS/LCSD, blank spikes, MS/MSD, and method blanks;
- *Sensitivity* – the ability to discriminate between small differences in analyte concentration 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 Site;
- *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 20% for water samples and 35% for soil and sediment (EPA, 2010);
- *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;
- *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;
- *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.

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. Data usability and limitations will be evaluated by the RPM.

A.8 Special Training and Certifications

All field staff have completed the Occupational Safety and Health Administration (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. Field staff has completed American Red Cross Standard First Aid and adult Cardiopulmonary Resuscitation (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.

A.9 Documentation and Records

Field measurements will be recorded in a Site-dedicated field notebook at the time of data collection. A brief description of the stream flow measurements will also be recorded in the field notebook. Flow measurement data will be stored in the individual Flow Tracker unit and downloaded as soon as is practical. The data sheets from the data download will be printed, and scanned copies will be included in the SAR. Field notebooks, chain-of-custody forms, and other forms used for the Site investigation will be stored at the Region 8 EPA Laboratory Suite A127.

The documentation of the data evaluation efforts will be in the form of the worksheets prepared during validation. These worksheets will be an appendix to the SAR. The SAR will be prepared to identify problems that may affect data usability or require that the data be qualified. The SAR will discuss all precision, accuracy, representativeness, completeness, comparability, and sensitivity parameter results from the data validation and overall usability of the data for project objectives.

The laboratory will submit a data report to EPA containing all the analytical results for this sampling effort. The report will contain a case narrative that briefly describes the number of samples, analyses, and any analytical difficulties or QA/QC issues associated with the samples. The data report will also include signed chain-of-custody forms, analytical data, a QA/QC package, and raw data. Additional reporting requirements are outlined in the ESAT laboratory contract and the Final Revision 2 Quality Management Plan (QMP).

Peer review of the data package at a 100% frequency of reported versus raw data, will be performed by the analytical laboratory. The final report of the abbreviated data validation will be in a standard Contract Laboratory Program (CLP) format, including all laboratory and instrument QC results.

B. DATA GENERATION AND ACQUISITION

This section describes data generation and acquisition activities associated with these events, including process design, sampling and analytical methods, sample handling and custody, QC, equipment, and data use and management.

B.1 Sampling Process Design

The following sections describe the sampling methods to collect and analyze surface water, sediment, and soil samples. Appendix A provides copies of the applicable SOPs, outlining how field activities will be performed (including documentation protocols). Tables B.1-1 and B.1-2 provide the sampling checklist and field equipment checklist, respectively. Section A.8 provides the rationale for the sampling process outlined in this section.

Sampling will occur from the most downstream to the most upstream sampling locations to avoid cross-contamination in accordance with SOP# FLD-01 *Surface Water Sampling* (ESAT, 2012). Additionally, care will be taken at each sampling location to first collect the surface water samples before collecting the sediment samples in order to minimize the chances of contaminating the surface water with sediment particles.

The EPA RPM or her 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, or if sampling locations are inaccessible. Dan Wall is the EPA designee appointed for the studies. Any problems encountered and actions taken or deviations from this SAP/QAPP will be documented in the Site-dedicated field notebook.

B.1.1 Surface Water Sampling

Surface water samples will be collected during the June (high-flow) and September (low-flow) 2015 sampling events (Tables A.7-6 and A.7-7). Additionally, during the summer sampling event (August) surface water samples will be collected from select mine adit discharges and floodplain locations. For locations within the floodplain and fluvial depositional areas, surface water samples will be collected from right edge water (REW) and left edge water (LEW) nearest to the source areas where soil samples will be collected. Sampling activities will include measuring flow rates and In-Situ water quality measurements. Further, opportunistic samples may be collected on any observed adit discharge areas that are encountered during field activities. Tables A.7-5 through A.7-7 provides a name and description of each of the surface water sampling locations, as well as the sampling activities scheduled to occur.

B.1.2 Pore Water Sampling

Pore water will be collected during the September 2015 low-flow sampling event and during the summer sampling event. During the summer sampling event, pore water samples will be collected at the same locations as surface water locations and will be collected from REW and LEW of the Animas River. The data will be used for evaluating potential ecological impacts of the existing contaminants in the hyporheic zone of the streambed. The pH of the pore water will be measured by using the In-situ sample cup in accordance with ESAT SOP# FLD-09 *Water Quality Measurements with the In-Situ® Multi-Parameter Meter* (ESAT, 2012). Water samples will be analyzed for total recoverable and dissolved metals on the list of TAL metals. Analytical methods and detection limits are shown in Table A.7-3. Pore water samples will be collected using a

Push Point sampling device with a syringe. Specific procedures for using the Push Point sampler are included in *Pore Water Sampling* SOP# FLD-10 (ESAT, 2012).

B.1.3 Sediment Sampling

Sediment samples will be collected during the September 2015 low-flow sampling event and during the floodplain sampling event at the locations listed in Tables A.7-6 and A.7-7. Data will be used for determination of contaminant concentrations in streambed sediments. Samples will be analyzed for total recoverable metals and Hg. Analytical methods and detection limits are shown in Table A.7-4. Sediment samples will be collected using dedicated Teflon scoops that will be disposed of after use, in accordance with the protocols outlined in *Standard Operating Procedures for Shallow Stream Sediment Sampling,* FLD-06 (ESAT, 2012).

B.1.4 Soil Sampling

Soil samples will be collected during the August 2015 summer sampling event at the locations listed in Tables A.7-5 and A.7-7 for determination of metals contamination on waste deposits and tailings piles areas. Samples will be analyzed for total recoverable metals and Hg and SPLP. Samples will also be collected for the modified Sobek Method for ABA methodology. Analytical methods and detection limits are shown in Table A.7-4. Soil samples will be collected using dedicated Teflon scoops that will be disposed of after use, into eight-ounce glass jars.

Additionally, specific floodplain areas will be sampled to determine the nature and extent of contamination and the potential or actual contribution to surface water; specifically: Eureka Tailings, Kittimack Tailings, Minnie Gulch, Forest Queen, and tailings near Grouse Gulch. Both Eureka Tailings areas are subdivided subsections: one into three, the other into ten. The areas will be divided into river-right and river-left areas. Prior to soil sampling, pore water and surface water from river channel(s) at the downstream end of the subsection will be sampled and later analyzed for total and dissolved metals. Soil from each subsection will be sampled using thirty-point composite surface samples (zero to six inches) per each subsection. Each subsection will also have three to five test pits on a transect out from the river at the surface (zero to six inches) and one-foot depth intervals to groundwater. The exact number and location will be determined in the field.

The Kittimack Tailings area will be divided into two subsections with no further sectioning, using the same thirty-point composite surface sampling and test pit approach described above.

Minnie Gulch, Forest Queen, and the tailings near Grouse Gulch will not be subdivided, but will use the same thirty point composite surface sampling and test pit approach described above. All samples will be collected in accordance *Standard Operating*

Procedures for Soil Sampling, FLD-05 (ESAT, 2012), and analyzed for total recoverable metals and Hg and SPLP. Samples will also be collected for the modified Sobek Method for ABA methodology. Analytical methods and detection limits are shown in Table A.7-4.

B.1.5 Nature of Data Collected

As indicated in Section A.7, a variety of data will be collected during these events, some of which are critical to achieve the established DQOs and project objectives, and some of which are primarily for informational purposes or that will be used supplement critical data.

The following chart specifies each type:

Data Type	**Purpose**
Real-time water quality parameters (pH, temperature, conductivity and DO)	Critical
Stream flow measurements	Critical
Surface water (analyzed for dissolved metals and hardness, total recoverable metals, DOC, anions, and alkalinity)	Critical
Pore water (analyzed for total recoverable metals and dissolved metals and hardness)	Critical
Sediment (analyzed for total recoverable metals)	Critical
Soil (analyzed for total recoverable metals, including Hg)	Critical
GPS coordinates	Critical
Photolog	Informational
General field observations noted in logbook	Informational

B.1.7 Data Variability

As indicated in Section A.7, a variety of data will be collected during these events, some of which are critical to achieve the established DQOs and project objectives, and some of which are used primarily for informational purposes or that will be used to supplement critical data.

B.2 Sampling Methods

This section describes surface water, pore water, sediment, and soil sampling methods that will be employed during these sampling events as well as applicable SOPs, necessary equipment and support facilities.

B.2.1 Equipment and Support Facilities

Specific field equipment necessary for execution of this SAP/QAPP are included in Table B.1-2. During field activities, it is anticipated that additional support facilities and vehicles outside of the sampling vehicle will not be needed.

B.2.2 Sampling for Surface Water

Sampling will progress in a manner that will eliminate the potential for sediment disturbance in the stream bed that could result in cross-contamination of subsequent samples. Surface water samples will be collected by immersing the sample bottle several inches beneath the water surface with the mouth of the sample bottle facing upstream. To collect such a sample, the sample container will be inverted, lowered to the approximate sample depth and held at approximately a 45-degree angle with the mouth of the bottle facing downstream. The bottle will be rinsed three times with stream water from the sample location prior to collecting the sample.

If surface water samples cannot be collected directly into the sample container, a decontaminated one-liter bottle or cubitainer will be used to collect the sample. The bottle will be rinsed three times with water from the sample location prior to collecting the sample. Care will be taken to avoid excessive agitation when transferring samples to the sample containers.

For water samples from the Animas River, the Equal Discharge Increment Technique (EDITQ) will be used during the low-flow event and will require protocols specified in the "*Operators Manual for the US DH-81 Depth-Integrated Suspended-Sediment Sampler*" (FISP, No date). This technique will be employed during the low-flow event at all locations that warrant its use. It will be at the discretion of the field team lead to determine if the sampling location is suitable to perform this type of sampling using the DH-81. This process of using EDITQ will provide equal sample volumes at each stream vertical sample taken from the cross-section of the stream or river. **Attachment A** shows the filling time for the US DH-81 sampler using a one-liter bottle, which is used to calculate the transit rate. The transit rate is determined by multiplying the steam depth at the sampling vertical by two and dividing by the sampling time after determining the velocity of the stream. The resulting rate is in feet per second (FISP, no date). Table B.2-1 shows the filling time for the DH-81 sampler using a one-liter bottle.

B.2.3 Sampling for Pore Water

Pore Water samples will be collected by inserting a PushPoint® sampler into the hyporheic zone and purging until the pore water runs clear in accordance with *Pore Water Sampling,* SOP#FLD-10 (ESAT, 2012). The syringe used to extract the pore water will be rinsed three times prior to sample collection with water from the location. The water samples pulled into the syringe will be placed in a 250 mL High-Density Polyethylene (HDPE) sample bottle for total recoverable metals analysis and a 250 mL Nalgene filter bottles for dissolved metals analysis. After approximately 250 mL of water has been collected for each analysis, the sample for dissolved metals analysis will be filtered using a 0.45 micron filter. The push point pore water samplers will be decontaminated between uses in accordance with *Sampling Equipment Decontamination* SOP# FLD-02 (ESAT, 2012).

B.2.4 Sampling for Sediment

Sediment samples will be collected using dedicated Teflon scoops and placed in HDPE containers based on the protocols outlined in *Shallow Stream Sediment Sampling* SOP# FLD-06 (ESAT, 2012). Sampling locations that have not been previously recorded will be documented following *Global Positioning System (GPS) – Trimble GeoXT 2008 Series,* SOP# FLD-07 (ESAT, 2012) (Appendix A) and given an appropriate sample designation that is consistent with sampling location nomenclature for the Site.

Several sediment subsamples will be collected from a stretch of creek that is 50 meters upstream to 50 meters downstream of the actual sampling location, confirmed by GPS coordinates. These subsamples will then be combined and homogenized back at the laboratory. The goal is to obtain up to 60 mL of representative sediment from each sampling location and the required sample volume needed for the analyses of the sediment samples, which consists of 200 grams for total recoverable metals and total Hg analysis. No sampling equipment needs to be decontaminated since the Teflon scoops are not re-used across sampling locations.

B.2.5 Sampling for Soil

Soil will be collected at specific locations based on historical data used to determine mine waste deposits and tailings piles areas. Soil samples will be analyzed for total recoverable metals and Hg and SPLP. Samples will also be collected for the modified Sobek Method for ABA methodology. Soil samples will be collected using dedicated Teflon scoops. Sampling equipment will not need to be decontaminated since the Telfon scoops are not re-used across sampling locations. All samples will be collected in accordance *Standard Operating Procedures for Soil Sampling,* FLD-05 (ESAT, 2012). Analytical methods and detection limits are shown in Table A.7-4.

B.3 Sampling Handling and Custody

Sample designation will consist of a series of letters and numbers to indicate the station and location. Tables A.7-5 through A.7-8 show the naming convention used and the sample site descriptions. The sampling areas are as follows:

• A	Animas River
• ATS	American Tunnel Seep
• Bbridge	Bakers Bridge
• BG	Burrows Gulch
• CC	Cement Creek
• CG	California Gulch

DM	Sound Democrat/Silver Queen
EG	Eureka Gulch
FD	Fenn Drainage
FQ	Forest Queen
M	Mineral Creek
McC	McCarty
MTD	Mogul Tailings Drainage
TM	Treasure Mountain
UA	Upper Animas

The flood plain samples will be named in the field using the following naming convention:
Area-FP#-Media (A or B for each channel)-(Composite or Grab)-(depth (soil only))

Area

KT	Kittimack Tailings
MT	Minnie Tailings
ET	Eureka Tailings
ES	Eclipse Smelter

FP# for Floodplain with unique polygon number

Media

SW	Surface Water
PW	Pore Water
SO	Soil

Composite or Grab

30C	30 point composite
G	Grab

Depth for the collection depth of soil samples

The following are examples of this naming convention using Kittimack Tailings polygon 01 for surface water, pore water and soil:

Surface water
KT-FP01-SWa-G
Pore water
KT-FP01-PW-G

Soil

KT-FP01-SO-30C-6”

All samples will be preserved and transported back to the EPA Region 8 laboratory following chain-of-custody protocols. The samples will be relinquished to the sample custodian at the laboratory, together with the completed and signed chain-of-custody forms. **Attachment A** provides a blank chain-of-custody form. The sample custodian will inspect the coolers upon arrival to make sure that the proper temperature was maintained, that the sample containers are intact and sealed, and that the number of samples in the coolers match the information provided in the chain-of-custody forms. All the samples will be stored in an access-controlled sample cooler at the laboratory. An analytical chemist will log the samples in the Laboratory Information Management System (LIMS) upon receipt and will enter all analytical data into the SCRIBE database for permanent storage and archiving.

All field measurements and observations will be recorded in a bound notebook or on appropriate data sheets by field personnel at the time they are performed, in accordance with the *Contract Laboratory Program Guidance for Field Samplers* (EPA, 2011b). 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. Chain-of-custody forms will be filled out during the time of collection and will follow protocol provided in *Sample Custody and Labeling,* SOP# FLD-11 (ESAT, 2012).

B.3.1 Surface Water Sample Preservation

Surface water samples collected for analysis of total recoverable metals and dissolved metals will be acidified in the field using ultra-pure nitric acid to a pH less than two. Alkalinity and anions samples will not be chemically preserved and will be stored on ice. Samples will be stored in coolers on ice and kept at approximately four degrees Celsius for transport to the EPA Region 8 Laboratory in accordance with *Standard Operating Procedure for General Field Sampling Protocols* FLD-12 (ESAT, 2012).

The maximum holding times differ depending on the target analytes or compound, as follows (see also Table A.7-3): 180 days for total recoverable metals and dissolved metals, 28 days for DOC and anions, and 14 days for alkalinity.

B.3.2 Pore Water Sample Preservation

Pore water samples collected for analysis of total recoverable metals and dissolved metals will be acidified in the field using ultra-pure nitric acid to a pH of less than two. Samples will be stored in coolers on ice and kept at approximately four degrees Celsius for transport to the EPA Region 8 Laboratory in accordance with *Standard Operating Procedure for General Field Sampling Protocols* FLD-12 (ESAT, 2012). The maximum holding times are 180 days for all metals (see also Table A.7-3).

B.3.3 Sediment Sample Preservation

The sediment samples will be immediately stored in coolers on ice, and kept at approximately four degrees Celsius and then transported to the EPA Region 8 Laboratory in accordance with *General Field Sampling Protocols* SOP# FLD-12 (ESAT, 2012). The maximum holdings times differ depending on the target analytes (see also Table A.7-4). The holding times are 180 days for all metals, except for Hg that has a holding time of 28 days.

B.3.4 Soil Sample Preservation

Soil samples will be stored in coolers on ice and kept at approximately four degrees Celsius and then transported to the EPA Region 8 Laboratory in accordance with *General Field Sampling Protocols* SOP# FLD-12 (ESAT, 2012). The maximum holdings times differ depending on the target analytes (see also Table A.7-4). The holding times are 180 days for all metals, except for Hg that has a holding time of 28 days. Holding times for SPLP analysis are 180 days prior to extraction and 180 days post extraction. There are no holding-time requirements for ABA.

B.4 Analytical Methods

Samples will be analyzed for total recoverable metals, dissolved metals, alkalinity, and anions. Tables A.7-3 and A.7-4 include the laboratory analytical instrumentation and methods to be used for sample analysis. These methods will be in accordance with EPA *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, also known as SW-846, Method 7473, Revision 0, January 1998. Additionally, sample analysis will be in accordance with Method 200.7 *Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry*, Revision 4.4, May 1994, and Method 200.8 *Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma-Mass Spectrometry*, Revision 5.4, May 1994 and Method 245.1, Revision 3.0 *Determination of Mercury in Water by Cold Vapor Atomic Absorption Spectrometry*.

Sample disposal of potentially hazardous waste will follow the protocol defined in *Collection, Analysis and Disposal of ESAT Laboratory Waste* SOP LAB01.00 (ESAT, 2007).

Field parameters measured at each sample site will include DO, temperature, pH, conductivity, and stream flow. Physical observations of the stream and any pertinent information regarding the surroundings, including the biota and abiota will be noted in the Site-dedicated field notebook. Photographs of the Site will be taken and will be documented as an appendix to the 2015 SAR.

In the event that problems are encountered in the field that may impact the implementation of this SAP/QAPP, the EPA RPM or her designee will be responsible for directing CAs. Any problems encountered and actions taken or deviations from this SAP/QAPP will be documented in the Site-dedicated field notebook and stakeholders will be notified of changes within 72 hours.

It is currently anticipated that ESAT will be used for sample analysis during this project. Laboratory instrumentation and method requirements for the ESAT laboratory are included in Tables A.7-3 and A.7-4.

B.4.1 Surface Water

Surface water samples collected will be submitted for analysis to the EPA Region 8 Laboratory, ESAT Analytical Chemistry department. Specific analysis for each event is presented in Tables A.7-5 through A.7-7. Samples will be collected, preserved, labeled, and stored in accordance with the *Field Sampling Protocols*, SOP # FLD-12 (ESAT, 2012). The sample protocols for surface water are provided in Tables B.5-1 and B.5-2.

B.4.2 Sediment

Sediment samples will be submitted for analysis to the ESAT analytical chemistry department at the EPA Region 8 Laboratory. Specific analysis for each event is presented in Tables A.7-5 through A.7-7. Samples will be collected, preserved, labeled, and stored in accordance with the *Field Sampling Protocols*, SOP # FLD-12 (ESAT, 2012). The sample protocols for sediment are provided in Table B.5-1.

B.4.3 Soil

Soil samples will be submitted for analysis to the ESAT analytical chemistry department at the EPA Region 8 Laboratory. Specific analysis for each event is presented in Tables A.7-5 through A.7-7. Samples will be collected, preserved, labeled, and stored in accordance with the *Field Sampling Protocols*, SOP # FLD-12 (ESAT, 2012). The sample protocols for sediment are provided in Table B.5-1.

B.5 Quality Control

Tables B.5-1 and B.5-2 provide acceptable laboratory QC criteria for ESAT and EPA Region 8. 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 process.

The calibration procedures for the field measurements to be performed using the In-Situ water quality meter are detailed in the *Water Quality Measurements with the In-Situ® Multi-Parameter Meter* SOP # FLD-09 (ESAT, 2012). If other multi-probes are used for this sampling event, the field sampling team will calibrate the probe according to the manufacturer's specifications listed in the owner's manual. The SOPs and procedures appended to this document also detail the associated QA/QC criteria for the field analyses and equipment.

Field QC samples will be collected on the following basis:

- Filter/container/preservative blank – minimum of one blank per 20 samples collected or one blank per day.
- Duplicates (collocated) – minimum of one duplicate per ten samples collected.
- Background soil samples – five samples will be collected to establish the background ambient metal concentration.

B.6 Instrument and Equipment Testing, Inspection and Maintenance

The following chart includes the equipment that will be used during execution of this SAP/QAPP that requires testing, inspection or maintenance.

Equipment/Instrument	Requirement	Schedule
In-Situ® Multi-Parameter Meter	Calibration, routine maintenance, scheduled service	In accordance with manufacturer's specifications, user's manual and applicable SOPs
Trimble®GeoXT™GPS	Service	As needed depending on equipment performance
Sontek®Flowtracker™	Calibration, routine maintenance, scheduled service	In accordance with manufacturer's specifications, user's manual and applicable SOPs
Laboratory analytical instrumentation	Calibration, routine maintenance, scheduled service	In accordance with manufacturer's specifications, user's manual and applicable SOPs

Maintenance and servicing schedules as well as applicable testing criteria are included in the applicable user's manuals as well as SOPs (attached to this document). Note that most spare parts for each piece of equipment are kept at the Region 8 Laboratory, including parts for field equipment as well as laboratory instrumentation. Spare parts are routinely available and are ordered during periodic maintenance activities to ensure they are on-hand when needed. Equipment and instrument calibration requirements and frequencies are detailed in the applicable SOPs and user's manuals (attached to this document).

Field equipment will be inspected, tested and routine maintenance will be performed prior to deployment in the field by contractor staff members at the EPA Region 8 Laboratory whom are knowledgeable of equipment operation and maintenance requirements. 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. Any repair or maintenance activities performed will be documented in the applicable equipment or instrument log book. Backup equipment will be deployed during these events in case of equipment or instrument failure in the field.

B.7 Instrument and Equipment Calibration and Frequency

As indicated in Section B.6, some laboratory instrumentation (analytical instrumentation) and field equipment (such as water quality meters and flow meters) will require periodic calibration to verify function. Calibration requirements, procedures, testing criteria and deficiency resolution procedures are included in applicable SOPs and user’s manuals, each of which are included in Appendix A of this document (for field equipment). SOPs and user’s manuals for laboratory analytical instrumentation are on-file and readily available at the 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 will be 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 supplies for this event will be purchased by the EPA from approved vendors, and stored in the field sampling room (or adjacent storage rooms at the Region 8 Laboratory). The week prior to the sampling event, an EPA or ESAT sampling team member will gather needed supplies and consumables, which will subsequently be verified by an ESAT team member. Supplies and consumables will be ordered, inspected upon receipt, accepted, tracked, and inventoried by the EPA field biologist at the 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)

Non-direct measurements were relied upon for preparation of project implementation. These measurements include previous sampling and analysis plans (EPA, 2013), historical data from ARSG, and past SARs.

B.10 Data Management

Specific management processes will be followed for data likely to be collected during field activities: field equipment calibration and maintenance entries, field logbook entries, chain-of-custody forms, electronically entered and logged data (such as GPS locations, flow measurements, etc.), and analytical data.

Field equipment calibration and maintenance logs – all field equipment calibration and maintenance activities will be documented in a logbook dedicated to each piece of equipment. Logbook entries will be signed and dated by the individual performing calibration or maintenance, or the individual responsible for coordination (such as the field task lead) if equipment is shipped to a manufacturer for repair or maintenance. Field logbooks will be stored with the appropriate piece of equipment. When new logbooks are needed, the former logbook will be stored at the Region 8 EPA Laboratory, Suite A127 until relinquished to EPA in accordance with ESAT Region 8 contract requirements.

Field logbook and datasheet entries – all field measurements and observations will be recorded in a bound notebook or on appropriate data sheets by the field personnel at the time they are performed. The personnel doing the recording will initial and date each logbook. Corrections to logbook entries will be made 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. Upon return to the Region 8 laboratory, all data that is hand-entered into field notebooks and datasheets will be transferred to electronic spreadsheets (such as Microsoft® Excel) by ESAT contract staff to prepare for uploading to a SCRIBE project (see below). ESAT field personnel will perform a 100% verification of spreadsheet entries against hand-entered field logbook and datasheet entries before uploading to SCRIBE. Original field notebooks and data sheets will be stored at the Region 8 EPA Laboratory, Suite A127 until relinquished to EPA in accordance with ESAT Region 8 contract requirements. Non-SCRIBE electronic files generated as a part of this process (i.e., spreadsheets, photographs, scanned logbooks) will be stored on the ESAT Region 8 contractor G drive.

Chain-of-custody forms – when possible, chain-of-custody forms will be generated prior to field activities using SCRIBE and will be filled out when samples are collected following the protocol outlined in *Sample Custody and Labeling* SOP# FLD-11 (ESAT, 2012). Otherwise, blank chain-of-custody forms will be used to collect sample information during field activities (Attachment A). Information entered on the forms during investigation activities will be entered into SCRIBE after returning to the 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 chain-of-custody 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.

Electronically entered or logged data – in some cases data may be recorded in the field directly on electronic field forms or using data loggers (such as GPS instrumentation or multi-probe data loggers). In these cases, upon return to the Region 8 Laboratory, all electronic data logs will be downloaded directly to a spreadsheet (or alternate electronic media depending on specific instrument software requirements), verified against the electronic form used in the field, and processed into an electronic form that can be uploaded directly to SCRIBE. Electronic field forms and data logs will be maintained on the ESAT Region 8 contractor G drive. In cases where information must be manually entered into SCRIBE, ESAT personnel will perform 100% verification between electronic documents and data logs and data manually entered into SCRIBE.

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 SOP# LAB-05.03, *Sample Receipt, Custody, Storage and LIMS Entry of Samples* (ESAT, 2014). 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, all 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 (ESAT, 2014). It is anticipated that more data may be collected in the field that supersedes existing or historical data that has already been published (such as GPS locations, etc.) for a specific sampling site. Therefore, before data are published or updated to SCRIBE projects, ESAT personnel will perform a 100% verification of each SCRIBE project against data collected in the field (hand-entered logbook data, electronic forms and data logs) 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 project manager will be immediately notified and an alternate publication date will be established. In the event that conditions preclude publication within that time period, the Task Order Project Officer (TOPO) 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 chain-of-custody procedures

The following individuals or their designees are authorized to perform any of the assessments listed above:

- EPA TOPO – Nicole Plescia
- EPA RPM – Paula Schmittdiel
- EPA RPM – Elizabeth Fagen

Assessment of field activities may occur at any time and without prior notice. Only authorized individuals may conduct the assessments and it is their role to issue any CA or response action to the situation. If minor problems are identified they will be addressed on-site prior to resuming work. If more significant problems are identified then a stop work order can be issued by the TOPO until the project manager or designee can resolve the problem.

C.1.2 Laboratory Assessments

System assessments of the designated laboratory may be performed by ESAT. The Quality Assurance Officer (QAO), or a designee, may perform a laboratory inspection.

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 TechLaw SOP# 02-06-05. The checklist requires examining the laboratory documentation on sample receiving, sample log-in, sample storage, chain-of-custody 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

CA 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 TechLaw 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

CA required as a result of the field data collection phase is initiated by the TechLaw field team leader and may result from log reports or field assessments. QC needs to be implemented both during the development of this SAP/QAPP and during sampling activities to ensure that CAs will not be required. CAs are initiated by ESAT if weaknesses or problems are uncovered as a result of field activities. 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
- Recalibration (or replacement) of field instruments
- Additional training of field personnel
- Reassignment of staff personnel
- Re-sampling

C.2 Reports to Management

The results of all laboratory assessments will be submitted to the appropriate ESAT project manager, task order manager, and laboratory assistance team, as well as the EPA Contracting Officer Representative and EPA QA personnel, if requested. An external assessment of the designated laboratory may also be conducted by EPA, at the Region’s discretion.

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 chain-of-custody forms and document sample condition (damage, temperature, etc.). Validation and verification of data will be performed by QA/QC personnel following *EPA National Functional Guidance for Inorganic Data*, (EPA, 2002) in order 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 the RPM 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 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 the RPM.

Abbreviated verification will be completed on ten percent of the analytical results for data that is electronically uploaded directly from the analytical instrumentation into the ESAT LIMS. This will be performed to ensure that data were produced in accordance with procedures outlined in this project plan. 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

The analytical data will be validated for ten percent of the results by either the acting EPA Region 8 Laboratory QAO or by a designated TechLaw QAO outside of the Region 8 ESAT office. The validation will include reviewing ten percent of the samples for

100% of the analytical analysis performed and reported. The following elements will be reviewed for compliance as part of the abbreviated data validation:

1. Holding Times
2. Calibration
3. Blanks
4. Spikes
5. Duplicates
6. LCSs
7. MS/MSDs
8. Post Digest Spike
9. Internal Control Standard
10. Dilution Sample
11. Reporting Limits
12. Analyte Identification
13. Analyte Quantification
14. Comparison of hard-copy results to the EDD

Data validation will conform to the EPA *Contract Laboratory Program National Functional Guidelines for Inorganic Data* 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 Regions choose to use additional qualifiers, a complete explanation of those qualifiers should accompany the data review.

U	The analyte was analyzed for, but 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 presented in the sample.
UJ	The analyte was analyzed for, but 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. If objectives have not been deployed, specify them 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 graphs of the data. 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 for summarizing and analyzing 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. In the event that 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.

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Environmental Services Assistance Team (2012) *Soil Sampling*. SOP# FLD-05

Environmental Services Assistance Team (2012) *Shallow Stream Sediment Sampling.* SOP# FLD-6

Environmental Services Assistance Team (2012) *Global Positioning System (GPS) – Trimble GeoXT 2008 series.* SOP# FLD-7

Environmental Services Assistance Team (2012) *Water Quality Measurements with the In-Situ® Multi-Parameter Meter*. SOP# FLD-9

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Tables

Table A.7-3 Water QC Criteria

Target Analytes	EPA Method[1]	Instrument	Fraction Evaluated	Sample Volume Req'd (ml)	Preservation	Holding Time	Laboratory MDL, ug/L	Laboratory PQL, ug/L	CDPHE Surface Water Regulations[3]	MCL[4]
Aluminum (Al)	200.7	ICP-OE	TR & Diss	Diss - 250ml TR - 500ml	HNO_3 to pH <2	180 days	20	50	750	-
Beryllium (Be)	200.7	ICP-OE	TR & Diss				2	5	-	4
Calcium (Ca)	200.7	ICP-OE	TR & Diss				100	250	NA	NA
Iron (Fe)	200.7	ICP-OE	TR & Diss				100	250	300 (WS)	300 (Diss only)
Chromium (Cr)	200.7	ICP-OE	TR & Diss				2	5	50 (TR)	100
Magnesium (Mg)	200.7	ICP-OE	TR & Diss				100	250	NA	NA
Manganese (Mn)	200.7	ICP-OE	TR & Diss				2	5	50 (WS)	50
Strontium (Sr)	200.7	ICP-OE	TR & Diss				2	10	NA	NA
Silica (SiO_2)	200.7	ICP-OE	TR & Diss				250	1000	NA	NA
Zinc (Zn)	200.7	ICP-OE	TR & Diss				10	20	65	5000
Calculated Hardness	2340B[2]	Calculated from 200.7, Ca & Mg	TR & Diss	-			-	-	(50)	-
Antimony (Sb)	200.8	ICP-MS	TR & Diss	Diss - 250ml TR - 500ml		180 days	0.5	1	-	6
Arsenic (As)	200.8	ICP-MS	TR & Diss				0.5	2	50	50
Cadmium (Cd)	200.8	ICP-MS	TR & Diss				0.1	0.2	3.5	5
Copper (Cu)	200.8	ICP-MS	TR & Diss				0.5	1	7	1000
Lead (Pb)	200.8	ICP-MS	TR & Diss				0.1	0.2	1.2	50
Nickel (Ni)	200.8	ICP-MS	TR & Diss				0.5	1	29	100
Selenium (Se)	200.8	ICP-MS	TR & Diss				0.5	1	4.6	50
Silver (Ag)	200.8	ICP-MS	TR & Diss				0.2	0.5	0.1	100
Thallium (Tl)	200.8	ICP-MS	TR & Diss				0.1	0.2	15	500
Dissolved Organic Carbon	415.3	Combustion/Non-dispersive IR	Diss	250 ml	Phosphoric acid, pH<2	28 days	1 mg/L	1 mg/L	-	-
Alkalinity	310.1	Mettler AT	Total	250 ml	Cooled to 4°C +/- 2	14 days	5	5	NA	NA
Chloride	300.0[6]	Ion Chromatography	Diss	250 ml	Cooled to 4°C +/- 2	28 days	1.0 mg/L	2.0 mg/L	250 mg/L	250 mg/L
Fluoride	300.0[6]		Diss				0.1 mg/L	0.2 mg/L	2 mg/L	4 mgL
Sulfate	300.0[6]		Diss				2 mg/L	5 mg/L	250 mg/L	250 mg/L

Diss = Dissolved metals fraction, i.e. source water filtered through 0.45 um filter prior to preservation (acidified).

TR = Total recoverable metals, source water, acidified (preserved).

MDL: Method Detection Limit, statistically determined from the deviation in a series of seven low level (3-5x the anticipated MDL) analyses, treated exactly as unknown samples for analysis. 40 CFR Chapter 1, Part 136, Appendix B

PQL: Practical Quantitation Level. Target analyte concentrations between PQL and MDL qualified as estimated, 'J', due to potential high variability. 40 CFR Parts 9, 141 and 142 [WH-FRL-6934-9]

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

[2] *Standard Methods for the Examination of Water and Wastewater*, 18th Edition, 1992

[3] *Colorado Department of Public Health and Environment Water Quality Control Commission, Regulation 31, The Basic Standards and Methodologgies for Surface Water (5 CCR 1002-31)*, Effective March 22, 2005. Three different standards apply to the waters in th

[4] MCL: Maximum Contaminant Level, a concentration set by the above CDPHE publication, Table III, "Drinking Water Supply".

[5] EPA's *Test Methods for Determining Solid Waste*, SW-846

[6] EPA's *Methods for Chemical Analysis of Water and Wastes*, June 2003

[7] Water supply limits for organic chemicals taken from CDPHE EWQCC, Reg. 31. Lower value of given range is reported.

Table A.7-4 Soil/Sediment Contaminants of Concern, Detection Limits and Methods

Target Analytes	EPA Digestion Method[1]	EPA Method[1]	Instrument	Fraction Evaluated	Sample Volume	Preservation	Holding Time	Laboratory PQL (mg/kg)	Sediment Screening Benchmark (mg/kg)
Aluminum (Al)	200.2	200.7	ICP-OE	TR	200 grams	Ice	180 days	5	25,519
Beryllium (Be)	200.2	200.7	ICP-OE	TR				0.5	NA
Calcium (Ca)	200.2	200.7	ICP-OE	TR				25	NA
Chromium (Cr)	200.2	200.7	ICP-OE	TR				0.5	43
Copper (Cu)	200.2	200.7	ICP-OE	TR				1	32
Iron (Fe)	200.2	200.7	ICP-OE	TR				25	188,400
Magnesium (Mg)	200.2	200.7	ICP-OE	TR				25	NA
Manganese (Mn)	200.2	200.7	ICP-OE	TR				0.5	631
Zinc (Zn)	200.2	200.7	ICP-OE	TR				2	121
Antimony (Sb)	200.2	200.8	ICP-MS	TR				0.1	2
Arsenic (As)	200.2	200.8	ICP-MS	TR				0.2	9.8
Cadmium (Cd)	200.2	200.8	ICP-MS	TR				0.02	1
Lead (Pb)	200.2	200.8	ICP-MS	TR				0.02	36
Nickel (Ni)	200.2	200.8	ICP-MS	TR				0.1	23
Selenium (Se)	200.2	200.8	ICP-MS	TR				0.1	NA
Silver (Ag)	200.2	200.8	ICP-MS	TR				0.05	1
Thallium (Tl)	200.2	200.8	ICP-MS	TR				0.1	NA
Mercury (Hg)	7473	7473	CVAA	Total			28 days	0.02	0.18
SPLP (Metals and Mercury)	1312	1312	ICP-MS	Total			180 Days prior to extraction and 180 days after extractiom	Laboratory Specific	N/A
Acid-Base Accounting (ABA)	NA	600/2-78-054	LECO-SC-144DR	Sulfer Forms			No holding time reqirements	5	NA

Notes:

TR - Total recoverable metals from bulk sediment

PQL - Practical Quantitation Limit

mg/kg - milligrams per kilogram

Sample containers should be plastic and capable of holding the required sample volume.

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

[2] *Development and Evaluation of consensus-Based Sediment Quality Guidelines for Freshwater Ecosystems.* Environ. Contam. Toxicol. Vol. 39, pp. 20-31. 2000.

[3] Not applicable - no established criteria for aluminum

[4] *Consensus-based sediment guidelines: Recommendations for use & application*. Wisconsin Dept. of Natural Resources, Madison, WI. Report No. WT-732-2003. December, 2003.

Table A.7-5 Sampling Locations and Activities
June 2015

		Station Name	Description	Lat	Long	Flow	WQP	SW metal (total and dissolved metals)
Cement Creek	Mogul	CC02D	Mogul at Flume	37.9098	-107.6384			
	Gold Point	[1]CC02E	Gold Point	37.90823	-107.638423			
	Red &Bonita	CC03B	CC abv R&B	37.897778	-107.645925			
		CC03D	R&B @ culvert	37.8968	-107.6449			
	American	CC19	American tunnel	37.890981	-107.64844			
	Silver Ledge	[1]CC14	Silver Ledge	37.87673	-107.644329			
	Gold King	CC06	Gold King 7 Level	37.894505	-107.640824			
		CC06B	2nd portal Gold King	37.894731	-107.639			
	CC and NF confluence	CC03	DS R&B, US of N Fork	37.895539	-107.647006			
		CC07	North Fork above CC	37.8951	-107.6468			
		CC18B	CC abv Amer Tunnel	37.894229	-107.647255			
	CC and SF confluence	CC18	On Cement Creek upstream of CC19, below mouth of the N Fork	37.891272	-107.649183			
		CC17	South Fork above Cement Creek	37.8894	-107.6506			
		CC21	Cement Creek below South Fork Cement Creek	37.889465	-107.654221			
	Cement Creek Mouth	CC48	Cement Creek Gauging Station	37.816274	-107.661599			
Mineral Creek	Mineral Creek Mouth	M34	Mineral Creek Gauge	37.8028	-107.6722	1	1	1
Burrows Creek	Burrows Creek	A07	BC abv NF - Burrows Creek above Animas	37.945684	-107.575561	1	1	1
		A07A	Abv Large Fault - Burrows Creek Above Fault	37.946768	-107.577851	1	1	1
		A07B	Blw London Mine - Burrows Creek Below London Mine	37.947633	-107.583285	1	1	1
		A07C	Abv London Mine - Burrows Gulch Above London Mine	37.947592	-107.587676	1	1	1
		BG1A	Burrow Gulch Above Mining	37.942949	-107.592706	1	1	1
		Waste Piles	DM3,4,5,6,7,8 if needed					
North Fork Animas	Lucky Jack	A01	Animas above Denver Lake	37.956067	-107.575928	1	1	1
		A02A	Animas above Lucky Jack Mine	37.954701	-107.57213	1	1	1
		A02B	Lucky Jack Mine Drainage	37.954002	-107.57321	1	1	1
		A02	Animas below Lucky Jack Mine	37.950179	-107.572776	1	1	1
		Waste Piles						
	Sentinel Stations for NF Animas	A03	Horseshoe Creek	37.950175	-107.572091	1	1	1
		A04	Animas above Horseshoe Creek	37.950861	-107.57345	1	1	1
		A05	NF animas blw Horseshoe	37.949223	-107.57347	1	1	1
		[2]A06	Unnamed Tributary below Horshoe Creek	37.947332	-107.573875	1	1	1
		A08	Animas below Burrows Creek	37.944025	-107.574582	1	1	1
Placer	Sound Democrat	DM26	Sound Democrat Mine Drainage	37.908431	-107.595128	1	1	1
	Silver Queen	DM25	Silver Queen Mine Drainage	37.907935	-107.596959	1	1	1
	Gold Prince	A23A	Gold Prince adit	37.908745	-107.600887	1	1	1
		A23	Placer Gulch below Gold Prince	37.910124	-107.599287	1	1	1
	Sunbank	A21	Placer below Sunbank Adit	37.919066	-107.590108	1	1	1
		[1]A21A	Comet Adit Lower	37.917106	-107.592415	1	1	1
		A22	Placer above Sunbank Adit	37.915211	-107.592282	1	1	1
		Waste Piles						
	Mountain Queen	A19	California Gulch above Mountain Queen	37.914256	-107.617238	1	1	1
		A19A	Mountain Queen Adit Discharge	37.916352	-107.613587	1	1	1
		A18	California Gulch below Mountain Queen	37.917416	-107.613125	1	1	1
		Waste Piles	Upper, Lower, Evening Star					
	Placer/CG Confluence	A20	Placer Gulch	37.929405	-107.586954	1	1	1
		A15	CalGulch below Vermillion and abv road	37.931669	-107.590424	1	1	1
California Gulch	Ida Mine	A17A	Consolidated discharge	37.931097	-107.604176	1	1	1
		CG3	California Gulch above DM11-16	37.929406	-107.604405	1	1	1
		Waste Piles						
	Burrows Mine	A17B	Adit Discharge in California Gulch - Old A17b	37.932239	-107.60217	1	1	1
		Waste Piles						
	Indian Chief	DM28	Mine in Upper California Gulch Mine Drainage (Indian Chief)	37.919707	-107.613579	1	1	1
		Waste Piles	DM28			1	1	1
	Vermillion Tunnel and Mine	A16	Vermillion Tunnel Mine Drainage	37.93391	-107.596642	1	1	1
		CG6	California Gulch below Vermillion Mine Tributary	37.932227	-107.596756	1	1	1
		CG5	Mine Drainage Trib - Vermillion Mine Discharge above the Road	37.934372	-107.600187	1	1	1
		CG4	Abv both Mine and Tunnel - California Gulch below DM11-16	37.931368	-107.599004	1	1	1
		Waste Piles	both Tunnel and Mine area					
	Bagley Mine/Ponds/Frisco Tunnel	A13	CG abv both Frisco and Pond	37.930548	-107.584345	1	1	1
		A11	CG blw Frisco and Pond - California Gulch below Bagley Mill Tailings	37.932789	-107.576914	1	1	1
		A12	Frisco tunnel - Bagley Tunnel Mine Drainage	37.932042	-107.581749	1	1	1
		CG9	Btw Frisco and Bagley Ponds - California Gulch below Bagley Adit Discharge	37.932068	-107.579373	1	1	1
		CG12A	Bagley Pond Discharge	37.933	-107.579	1	1	1
		Waste Piles	Mine and Pond Area					
	Columbus Mine	CG11	CG abv Columbus - California Gulch above Columbus Mine	37.932525	-107.572532	1	1	1
		A11A	Columbus Adit	37.932985	-107.571261	1	1	1
		UA5	Animas abv Columbus - Animas below Mining Complex	37.936431	-107.571539	1	1	1
		Waste Piles						
	Confluence Animas/CalGulch	A14	Below Confluence	37.929393	-107.567538	1	1	1
		A09	Animas above California Gulch	37.931813	-107.569842	1	1	1
		A10	California Gulch Mouth - California Gulch above Animas Confluence	37.931745	-107.570268	1	1	1
Animas Above Eureka	Tailings in floodplain	Tailings	Tailings above Grouse Gulch (#28)	-107.5587	37.9178			
	Cinnamon	A24	Cinnamon Creek	37.926707	-107.56161	1	1	1
	Grouse	[2]A25	Grouse Gulch	37.917869	-107.552321	1	1	1
	Picayne	[2]A26	Picayune Gulch	37.91139	-107.555845	1	1	1
	Treasure Mtn Mine	TM1	Treasure Mountain Adit (New Station)	37.913533	-107.568751	1	1	1
		Waste Piles						
	Silver Wing Burns Gulch	UA8	Animas above Burns Gulch	37.905867	-107.556755	1	1	1
		A27	Burns Gulch	37.905521	-107.556201	1	1	1
		A28	Animas below Burns Gulch	37.9046	-107.556673	1	1	1
		A29	Silverwing	37.903683	-107.555496	1	1	1
		A30	blw Burns and SilverWing	37.903039	-107.556737	1	1	1
		Waste Piles	Silver Wing (Mill area and Mine)					
	Niagara Trib	[2]A31	Animas above Niagara	37.885353	-107.562481	1	1	1
		[2]A32	Niagara Gulch	37.882931	-107.563434	1	1	1
Eureka Gulch	Concluence EG and South Fork	A36	South Fork above Eureka Gulch	37.88293	-107.592597	1	1	1
		A37	Eureka Gulch above South Fork	37.884834	-107.591341	1	1	1
		EG9	Downstream	37.883034	-107.586507	1	1	1
	Midway Mine	DM34	Midway Mine Drainage	37.885122	-107.589875	1	1	1
		Waste Piles						
	Terry Tunnel	A38	Terry Tunnel	37.892756	-107.603386	1	1	1
		A39	Eureka above Terry Tunnel	37.892522	-107.605327	1	1	1
		EG6	Eureka Gulch below Terry Tunnel	37.892081	-107.600552	1	1	1
		MC0	Mouth of McCarty	37.892	-107.6043	1	1	1
	Bavarian	DM33	Bavarian Mine Drainage	37.891604	-107.606477	1	1	1
	Ben Franklin	EG3A	Blw headwaters confluence. Flows into stope and daylights out of Ben Franklin Aditv(DM32)	37.894966	-107.60871	1	1	1
		DM32	Ben Franklin Prospect Draining Mine	37.894813	-107.608986	1	1	1
		EG5	Eureka Gulch below Ben Franklin Mine	37.894152	-107.606448	1	1	1
		ARD1	Drainage of BF waste pile	37.893821	-107.606795	1	1	1
		Waste Pile						
	Sunnyside Mine	EG2A	Eureka Gulch above Sunnyside Mine	37.898086	-107.610996	1	1	1
		EG4	Eureka Gulch below Lake Emma/Sunnyside Confluence	37.895689	-107.609867	1	1	1
		Waste Pile	check if still there					
	Clipper	EG1	Eureka Gulch Headwaters, above the Clipper	37.8946	-107.613838	1	1	1
		EG3	Eureka Gulch Headwaters above confluence	37.895311	-107.610435	1	1	1
		Waste pile						
	Ransom	DM69	Ransom Mine Discharge	37.882821	-107.577167	1	1	1
	Emma	EG2	Eureka Gulch below Lake Emma	37.897826	-107.61335	1	1	1
EG/UA confluence	EG/UA confluence	[3]A33	Animas above Eureka	37.881735	-107.564966	1	1	1
		[2]A34	Eureka Gulch above Animas	37.87948	-107.567451	1	1	1
		A35	Animas below Eureka	37.875752	-107.566027	1	1	1
Animas between Eureka and Howardsville	Eureka Tailings	Eureka Wetland	Need surficial sampling grid (from Maggie to above Eureka)					
	Forest Queen	A41	Forest Queen Adit	37.931745	-107.570262	1	1	1
		A41C	FQ drainage abv culvert	37.8659	-107.5678	1	1	1
		A40A	Animas abv FQ	37.8655	-107.5699	1	1	1
		FQ Tailings	Scattered Tailings					
	Kittimack	A40	Animas above Minnie Gulch (AKA Animas above Minnie)	37.863317	-107.571627	1	1	1
		A42	Minnie at Culvert (Minnie above Animas)	37.862128	-107.567246	1	1	1
		A43	Maggie Gulch above Animas	37.85424	-107.57163	1	1	1
		LA3	Animas below Maggie Gulch	37.853666	-107.577559	1	1	1
		A41A	A49? Animas below Minnie	37.861967	-107.573305	1	1	1
		KittiMack Tailings						
		Minnie Tailings						
	Howardsville	A45	Animas above Howardsville Tailings	37.842791	-107.590887	1	1	1
		A47	Hematite Gulch above Animas	37.836039	-107.598608	1	1	1
		A48	Cunningham abv Bridge	37.83513	-107.595077	1	1	1
		A54	Little Nation Mine Tributary	37.832101	-107.600545	1	1	1
		[2]A55	Animas 1/4 mile below A54	37.831087	-107.604696	1	1	1
Animas Below Howardsville	Animas Adjacent to Mayflower	A56	Animas Abv Arastra	37.82765815	-107.6242042	1	1	1
		[2]A58	Arasatra	37.825877	-107.625119	1	1	1
		[1]A60	Animas downstream of Arastra Creek and downstream of Bridge	37.827192	-107.626655	1	1	1
		A61	Animas below Arrastra	37.826169	-107.62972	1	1	1
		[1]A64	Animas downstream of Boulder and Aspen creeks	37.822921	-107.635217	1	1	1
		A65	Animas near wetland downstream f venture snowboard	37.820342	-107.642035	1	1	1
		A66	Animas at destroyed bridge	37.816235	-107.649617	1	1	1
	Animas near Silverton	A68	14th Street Gauge @ 13th Street Bridge	37.811202	-107.659167	1	1	1
		A72	Animas Gauge below Silverton	37.79027	-107.667578	1	1	1
	Below Canyon	Bakers Bridge	Actual location is Durango Riverside RV Park	37.45573315	-107.8011316	1	1	1
						103	103	103

[1] do not have access yet

[2] moved for access

[3] access was denied

Table A.7-6 Upper Animas Sampling Locations and Activities
August 2015

	Station Name	Description	Lat	Long	Sediment (Total Metals & Hg)	Soil/Waste (Total Metals&Hg)	SPLP (Solid Waste)	ABA (Solid Waste
Mogul	CC02D	Mogul at Flume	37.9098	-107.6384				
Gold Point	[1]CC02E	Gold Point	37.90823	-107.638423				
Red &Bonita	CC03B	CC abv R&B	37.897778	-107.645925				
	CC03D	R&B @ culvert	37.8968	-107.6449				
American	CC19	American tunnel	37.890981	-107.64844				
Silver Ledge	[1]CC14	Silver Ledge	37.87673	-107.644329				
Gold King	CC06	Gold King 7 Level	37.894505	-107.640824				
	CC06B	2nd portal Gold King	37.894731	-107.639				
CC and NF confluence	CC03	DS R&B, US of N Fork	37.895539	-107.647006				
	CC07	North Fork above CC	37.8951	-107.6468				
	CC18B	CC abv Amer Tunnel	37.894229	-107.647255				
CC and SF confluence	CC18	On Cement Creek upstream of CC19, below mouth of the N Fork	37.891272	-107.649183				
	CC17	South Fork above Cement Creek	37.8894	-107.6506				
	CC21	Cement Creek below South Fork Cement Creek	37.889465	-107.654221				
Cement Creek Mouth	CC48	Cement Creek Gauging Station	37.816274	-107.661599				
Mineral Creek Mouth	M34	Mineral Creek Gauge	37.8028	-107.6722				
Burrows Creek	A07	BC abv NF - Burrows Creek above Animas	37.945684	-107.575561	1			
	A07A	Abv Large Fault - Burrows Creek Above Fault	37.946768	-107.577851				
	A07B	Blw London Mine - Burrows Creek Below London Mine	37.947633	-107.583285	1			
	A07C	Abv London Mine - Burrows Gulch Above London Mine	37.947592	-107.587676	1			
	BG1A	Burrow Gulch Above Mining	37.942949	-107.592706	1			
	Waste Piles	DM3,4,5,6,7,8 if needed				6	6	6
Lucky Jack	A01	Animas above Denver Lake	37.956067	-107.575928	1			
	A02A	Animas above Lucky Jack Mine	37.954701	-107.57213	1			
	A02B	Lucky Jack Mine Drainage	37.954002	-107.57321				
	A02	Animas below Lucky Jack Mine	37.950179	-107.572776				
	Waste Piles					1	1	1
Sentinel Stations for NF Animas	A03	Horseshoe Creek	37.950175	-107.572091	1			
	A04	Animas above Horseshoe Creek	37.950861	-107.57345	1			
	A05	NF animas blw Horseshoe	37.949223	-107.57347	1			
	[2]A06	Unnamed Tributary below Horshoe Creek	37.947332	-107.573875				
	A08	Animas below Burrows Creek	37.944025	-107.574582	1			
Sound Democrat	DM26	Sound Democrat Mine Drainage	37.908431	-107.595128				
Silver Queen	DM25	Silver Queen Mine Drainage	37.907935	-107.596959				
Gold Prince	A23A	Gold Prince adit	37.908745	-107.600887				
	A23	Placer Gulch below Gold Prince	37.910124	-107.599287				
Sunbank	A21	Placer below Sunbank Adit	37.919066	-107.590108	1			
	[1]A21A	Comet Adit Lower	37.917106	-107.592415				
	A22	Placer above Sunbank Adit	37.915211	-107.592282	1			
	Waste Piles					3	3	3
Mountain Queen	A19	California Gulch above Mountain Queen	37.914256	-107.617238				
	A19A	Mountain Queen Adit Discharge	37.916352	-107.613587				
	A18	California Gulch below Mountain Queen	37.917416	-107.613125				
	Waste Piles	Upper, Lower, Evening Star				3	3	3
Placer/CG Confluence	A20	Placer Gulch	37.929405	-107.586954	1			
	A15	CalGulch below Vermillion and abv road	37.931669	-107.590424	1			
Ida Mine	A17A	Consolidated discharge	37.931097	-107.604176				
	CG3	California Gulch above DM11-16	37.929406	-107.604405				
	Waste Piles					1	1	1
Burrows Mine	A17B	Adit Discharge in California Gulch - Old A17b	37.932239	-107.60217				
	Waste Piles					1	1	1
Indian Chief	DM28	Mine in Upper California Gulch Mine Drainage (Indian Chief)	37.919707	-107.613579		1	1	1
	Waste Piles	DM28					1	1
Vermillion Tunnel and Mine	A16	Vermillion Tunnel Mine Drainage	37.93391	-107.596642				
	CG6	California Gulch below Vermillion Mine Tributary	37.932227	-107.596756				
	CG5	Mine Drainage Trib - Vermillion Mine Discharge above the Road	37.934372	-107.600187				
	CG4	Abv both Mine and Tunnel - California Gulch below DM11-16	37.931368	-107.599004				
	Waste Piles	both Tunnel and Mine area				2	2	2
Bagley Mine/Ponds/Frisco Tunnel	A13	CG abv both Frisco and Pond	37.930548	-107.584345	1			
	A11	CG blw Frisco and Pond - California Gulch below Bagley Mill Tailings	37.932789	-107.576914	1			
	A12	Frisco tunnel - Bagley Tunnel Mine Drainage	37.932042	-107.581749				
	CG9	Btw Frisco and Bagley Ponds - California Gulch below Bagley Adit Discharge	37.932068	-107.579373				
	CG12A	Bagley Pond Discharge	37.933	-107.579				
	Waste Piles	Mine and Pond Area				2	2	2
Columbus Mine	CG11	CG abv Columbus - California Gulch above Columbus Mine	37.932525	-107.572532				
	A11A	Columbus Adit	37.932985	-107.571261				
	UA5	Animas abv Columbus - Animas below Mining Complex	37.936431	-107.571539	1			
	Waste Piles					1	1	1
Confluence Animas/CalGulch	A14	Below Confluence	37.929393	-107.567538	1			
	A09	Animas above California Gulch	37.931813	-107.569842	1			
	A10	California Gulch Mouth - California Gulch above Animas Confluence	37.931745	-107.570268	1			
Tailings in floodplain	Tailings	Tailings above Grouse Gulch (#28)	-107.5587	37.9178		5	3	3
Cinnamon	A24	Cinnamon Creek	37.926707	-107.56161	1			
Grouse	[2]A25	Grouse Gulch	37.917869	-107.552321	1			
Picayne	[2]A26	Picayune Gulch	37.91139	-107.555845	1			
Treasure Mtn Mine	TM1	Treasure Mountain Adit (New Station)	37.913533	-107.568751				
	Waste Piles					1	1	1
Silver Wing Burns Gulch	UA8	Animas above Burns Gulch	37.905867	-107.556755				
	A27	Burns Gulch	37.905521	-107.556201				
	A28	Animas below Burns Gulch	37.9046	-107.556673	1			
	A29	Silverwing	37.903683	-107.555496				
	A30	blw Burns and SilverWing	37.903039	-107.556737	1			
	Waste Piles	Silver Wing (Mill area and Mine)				2	2	2
Niagara Trib	[2]A31	Animas above Niagara	37.885353	-107.562481	1			
	[2]A32	Niagara Gulch	37.882931	-107.563434	1			
Concluence EG and South Fork	A36	South Fork above Eureka Gulch	37.88293	-107.592597	1			
	A37	Eureka Gulch above South Fork	37.884834	-107.591341	1			
	EG9	Downstream	37.883034	-107.586507	1			
Midway Mine	DM34	Midway Mine Drainage	37.885122	-107.589875				
	Waste Piles							
Terry Tunnel	A38	Terry Tunnel	37.892756	-107.603386		1	1	1
	A39	Eureka above Terry Tunnel	37.892522	-107.605327				
	EG6	Eureka Gulch below Terry Tunnel	37.892081	-107.600552	1			
	MC0	Mouth of McCarty	37.892	-107.6043				
Bavarian	DM33	Bavarian Mine Drainage	37.891604	-107.606477				
Ben Franklin	EG3A	Blw headwaters confluence. Flows into stope and daylights out of Ben Franklin Aditv(DM32)	37.894966	-107.60871	1			
	DM32	Ben Franklin Prospect Draining Mine	37.894813	-107.608986				
	EG5	Eureka Gulch below Ben Franklin Mine	37.894152	-107.606448	1			
	ARD1	Drainage of BF waste pile	37.893821	-107.606795				
	Waste Pile					1	1	1
Sunnyside Mine	EG2A	Eureka Gulch above Sunnyside Mine	37.898086	-107.610996	1			
	EG4	Eureka Gulch below Lake Emma/Sunnyside Confluence	37.895689	-107.609867				
	Waste Pile	check if still there				1	1	1
Clipper	EG1	Eureka Gulch Headwaters, above the Clipper	37.8946	-107.613838	1			
	EG3	Eureka Gulch Headwaters above confluence	37.895311	-107.610435	1			
	Waste pile					1	1	1
Ransom	DM69	Ransom Mine Discharge	37.882821	-107.577167				
Emma	EG2	Eureka Gulch below Lake Emma	37.897826	-107.61335	1			
EG/UA confluence	[3]A33	Animas above Eureka	37.881735	-107.564966	1			
	[2]A34	Eureka Gulch above Animas	37.87948	-107.567451	1			
	A35	Animas below Eureka	37.875752	-107.566027	1			
Eureka Tailings	Eureka Wetland	Need surficial sampling grid (from Maggie to above Eureka)				50	25	25
Forest Queen	A41	Forest Queen Adit	37.931745	-107.570262				
	A41C	FQ drainage abv culvert	37.8659	-107.5678	1			
	A40A	Animas abv FQ	37.8655	-107.5699	1			
	FQ Tailings	Scattered Tailings				10	5	5
Kittimack	A40	Animas above Minnie Gulch (AKA Animas above Minnie)	37.863317	-107.571627	1			
	A42	Minnie at Culvert (Minnie above Animas)	37.862128	-107.567246	1			
	A43	Maggie Gulch above Animas	37.85424	-107.57163	1			
	LA3	Animas below Maggie Gulch	37.853666	-107.577559	1			
	A41A	A49? Animas below Minnie	37.861967	-107.573305	1			
	KittiMack Tailings					10	5	5
	Minnie Tailings					5	3	3
Howardsville	A45	Animas above Howardsville Tailings	37.842791	-107.590887	1			
	A47	Hematite Gulch above Animas	37.836039	-107.598608				
	A48	Cunningham abv Bridge	37.83513	-107.595077	1			
	A54	Little Nation Mine Tributary	37.832101	-107.600545				
	[2]A55	Animas 1/4 mile below A54	37.831087	-107.604696	1			
Animas Adjacent to Mayflower	A56	Animas Abv Arastra	37.82765815	-107.6242042				
	[2]A58	Arasatra	37.825877	-107.625119				
	[1]A60	Bridge	37.827192	-107.626655				
	A61	Animas below Arrastra	37.826169	-107.62972				
	[1]A64	Animas downstream of Boulder and Aspen creeks	37.822921	-107.635217				
	A65	Animas near wetland downstream f venture snowboard	37.820342	-107.642035				
	A66	Animas at destroyed bridge	37.816235	-107.649617				
Animas near Silverton	A68	14th Street Gauge @ 13th Street Bridge	37.811202	-107.659167				
	A72	Animas Gauge below Silverton	37.79027	-107.667578				
Below Canyon	Bakers Bridge	Actual location is Durango Riverside RV Park	37.45573315	-107.8011316				
					50	108	70	70

Table A.7-7 Upper Animas Sampling Locations and Activities
September 2015

		Station Name	Description	Lat	Long	Flow	WQP	SW metal (total and dissolved metals)	Alk/Anions	Porewater (dissolved metals)	Sediment (Total Metals & Hg)
Cement Creek	Mogul	CC02D	Mogul at Flume	37.9098	-107.6384	1	1	1	1		
	Gold Point	[1]CC02E	Gold Point	37.90823	-107.638423	1	1	1	1		
	Red &Bonita	CC03B	CC abv R&B	37.897778	-107.645925	1	1	1	1		
		CC03D	R&B @ culvert	37.8968	-107.6449	1	1	1	1		
	American	CC19	American tunnel	37.890981	-107.64844	1	1	1	1		
	Silver Ledge	[1]CC14	Silver Ledge	37.87673	-107.644329	1	1	1	1		
	Gold King	CC06	Gold King 7 Level	37.894505	-107.640824	1	1	1	1		
		CC06B	2nd portal Gold King	37.894731	-107.639	1	1	1	1		
	CC and NF confluence	CC03	DS R&B, US of N Fork	37.895539	-107.647006	1	1	1	1		
		CC07	North Fork above CC	37.8951	-107.6468	1	1	1	1		
		CC18B	CC abv Amer Tunnel	37.894229	-107.647255	1	1	1	1		
	CC and SF confluence	CC18	On Cement Creek upstream of CC19, below mouth of the N Fork	37.891272	-107.649183	1	1	1	1		
		CC17	South Fork above Cement Creek	37.8894	-107.6506	1	1	1	1		
		CC21	Cement Creek below South Fork Cement Creek	37.889465	-107.654221	1	1	1	1		
	Cement Creek Mouth	CC48	Cement Creek Gauging Station	37.816274	-107.661599	1	1	1	1		
Mineral Creek	Mineral Creek Mouth	M34	Mineral Creek Gauge	37.8028	-107.6722	1	1	1	1		
Burrows Creek	Burrows Creek	A07	BC abv NF - Burrows Creek above Animas	37.945684	-107.575561	1	1	1	1		1
		A07A	Abv Large Fault - Burrows Creek Above Fault	37.946768	-107.577851	1	1	1	1		
		A07B	Blw London Mine - Burrows Creek Below London Mine	37.947633	-107.583285	1	1	1	1		1
		A07C	Abv London Mine - Burrows Gulch Above London Mine	37.947592	-107.587676	1	1	1	1		1
		BG1A	Burrow Gulch Above Mining	37.942949	-107.592706	1	1	1	1		1
		Waste Piles	DM3,4,5,6,7,8 if needed								
North Fork Animas	Lucky Jack	A01	Animas above Denver Lake	37.956067	-107.575928	1	1	1	1		1
		A02A	Animas above Lucky Jack Mine	37.954701	-107.57213	1	1	1	1		1
		A02B	Lucky Jack Mine Drainage	37.954002	-107.57321	1	1	1	1		
		A02	Animas below Lucky Jack Mine	37.950179	-107.572776	1	1	1	1		
		Waste Piles									
	Sentinel Stations for NF Animas	A03	Horseshoe Creek	37.950175	-107.572091	1	1	1	1		1
		A04	Animas above Horseshoe Creek	37.950861	-107.57345	1	1	1	1		1
		A05	NF animas blw Horseshoe	37.949223	-107.57347	1	1	1	1		1
		[2]A06	Unnamed Tributary below Horshoe Creek	37.947332	-107.573875	1	1	1	1		
		A08	Animas below Burrows Creek	37.944025	-107.574582	1	1	1	1		1
Placer	Sound Democrat	DM26	Sound Democrat Mine Drainage	37.908431	-107.595128	1	1	1	1		
	Silver Queen	DM25	Silver Queen Mine Drainage	37.907935	-107.596959	1	1	1	1		
	Gold Prince	A23A	Gold Prince adit	37.908745	-107.600887	1	1	1	1		
		A23	Placer Gulch below Gold Prince	37.910124	-107.599287	1	1	1	1		
	Sunbank	A21	Placer below Sunbank Adit	37.919066	-107.590108	1	1	1	1		1
		[1]A21A	Comet Adit Lower	37.917106	-107.592415	1	1	1	1		
		A22	Placer above Sunbank Adit	37.915211	-107.592282	1	1	1	1		1
		Waste Piles									
	Mountain Queen	A19	California Gulch above Mountain Queen	37.914256	-107.617238	1	1	1	1		
		A19A	Mountain Queen Adit Discharge	37.916352	-107.613587	1	1	1	1		
		A18	California Gulch below Mountain Queen	37.917416	-107.613125	1	1	1	1		
		Waste Piles	Upper, Lower, Evening Star								
	Placer/CG Confluence	A20	Placer Gulch	37.929405	-107.586954	1	1	1	1		1
		A15	CalGulch below Vermillion and abv road	37.931669	-107.590424	1	1	1	1		1
California Gulch	Ida Mine	A17A	Consolidated discharge	37.931097	-107.604176	1	1	1	1		
		CG3	California Gulch above DM11-16	37.929406	-107.604405						
		Waste Piles				1	1	1	1		
	Burrows Mine	A17B	Adit Discharge in California Gulch - Old A17b	37.932239	-107.60217	1	1	1	1		
		Waste Piles									
	Indian Chief	DM28	Mine in Upper California Gulch Mine Drainage (Indian Chief)	37.919707	-107.613579	1	1	1	1		
		Waste Piles	DM28			1	1	1	1		
	Vermillion Tunnel and Mine	A16	Vermillion Tunnel Mine Drainage	37.93391	-107.596642	1	1	1	1		
		CG6	California Gulch below Vermillion Mine Tributary	37.932227	-107.596756	1	1	1	1		
		CG5	Mine Drainage Trib - Vermillion Mine Discharge above the Road	37.934372	-107.600187	1	1	1	1		
		CG4	Abv both Mine and Tunnel - California Gulch below DM11-16	37.931368	-107.599004	1	1	1	1		
		Waste Piles	both Tunnel and Mine area								
	Bagley Mine/Ponds/Frisco Tunnel	A13	CG abv both Frisco and Pond	37.930548	-107.584345	1	1	1	1		1
		A11	CG blw Frisco and Pond - California Gulch below Bagley Mill Tailings	37.932789	-107.576914	1	1	1	1		1
		A12	Frisco tunnel - Bagley Tunnel Mine Drainage	37.932042	-107.581749	1	1	1	1		
		CG9	Btw Frisco and Bagley Ponds - California Gulch below Bagley Adit Discharge	37.932068	-107.579373	1	1	1	1		
		CG12A	Bagley Pond Discharge	37.933	-107.579	1	1	1	1		
		Waste Piles	Mine and Pond Area								
	Columbus Mine	CG11	CG abv Columbus - California Gulch above Columbus Mine	37.932525	-107.572532	1	1	1	1		
		A11A	Columbus Adit	37.932985	-107.571261	1	1	1	1		
		UA5	Animas abv Columbus - Animas below Mining Complex	37.936431	-107.571539	1	1	1	1		1
		Waste Piles									
	Confluence Animas/CalGulch	A14	Below Confluence	37.929393	-107.567538	1	1	1	1		1
		A09	Animas above California Gulch	37.931813	-107.569842	1	1	1	1		1
		A10	California Gulch Mouth - California Gulch above Animas Confluence	37.931745	-107.570268	1	1	1	1		1
Animas Above Eureka	Tailings in floodplain	Tailings	Tailings above Grouse Gulch (#28)	-107.5587	37.9178						
	Cinnamon	A24	Cinnamon Creek	37.926707	-107.56161	1	1	1	1		1
	Grouse	[2]A25	Grouse Gulch	37.917869	-107.552321	1	1	1	1		1
	Picayne	[2]A26	Picayune Gulch	37.91139	-107.555845	1	1	1	1		1
	Treasure Mtn Mine	TM1	Treasure Mountain Adit (New Station)	37.913533	-107.568751	1	1	1	1		
		Waste Piles									
	Silver Wing Burns Gulch	UA8	Animas above Burns Gulch	37.905867	-107.556755	1	1	1	1		
		A27	Burns Gulch	37.905521	-107.556201	1	1	1	1		
		A28	Animas below Burns Gulch	37.9046	-107.556673	1	1	1	1		1
		A29	Silverwing	37.903683	-107.555496	1	1	1	1		
		A30	blw Burns and SilverWing	37.903039	-107.556737	1	1	1	1		1
		Waste Piles	Silver Wing (Mill area and Mine)								
	Niagara Trib	[2]A31	Animas above Niagara	37.885353	-107.562481	1	1	1	1		1
		[2]A32	Niagara Gulch	37.882931	-107.563434	1	1	1	1		1
Eureka Gulch	Concluence EG and South Fork	A36	South Fork above Eureka Gulch	37.88293	-107.592597	1	1	1	1		1
		A37	Eureka Gulch above South Fork	37.884834	-107.591341	1	1	1	1		1
		EG9	Downstream	37.883034	-107.586507	1	1	1	1		1
	Midway Mine	DM34	Midway Mine Drainage	37.885122	-107.589875	1	1	1	1		
		Waste Piles									
	Terry Tunnel	A38	Terry Tunnel	37.892756	-107.603386	1	1	1	1		
		A39	Eureka above Terry Tunnel	37.892522	-107.605327	1	1	1	1		
		EG6	Eureka Gulch below Terry Tunnel	37.892081	-107.600552	1	1	1	1		1
		MC0	Mouth of McCarty	37.892	-107.6043	1	1	1	1		
	Bavarian	DM33	Bavarian Mine Drainage	37.891604	-107.606477	1	1	1	1		
	Ben Franklin	EG3A	Blw headwaters confluence. Flows into stope and daylights out of Ben Franklin Aditv(DM32)	37.894966	-107.60871	1	1	1	1		1
		DM32	Ben Franklin Prospect Draining Mine	37.894813	-107.608986	1	1	1	1		
		EG5	Eureka Gulch below Ben Franklin Mine	37.894152	-107.606448	1	1	1	1		1
		ARD1	Drainage of BF waste pile	37.893821	-107.606795	1	1	1	1		
		Waste Pile									
	Sunnyside Mine	EG2A	Eureka Gulch above Sunnyside Mine	37.898086	-107.610996	1	1	1	1		1
		EG4	Eureka Gulch below Lake Emma/Sunnyside Confluence	37.895689	-107.609867	1	1	1	1		
		Waste Pile	check if still there								
	Clipper	EG1	Eureka Gulch Headwaters, above the Clipper	37.8946	-107.613838	1	1	1	1		1
		EG3	Eureka Gulch Headwaters above confluence	37.895311	-107.610435	1	1	1	1		1
		Waste pile									
	Ransom	DM69	Ransom Mine Discharge	37.882821	-107.577167	1	1	1	1		
	Emma	EG2	Eureka Gulch below Lake Emma	37.897826	-107.61335	1	1	1	1		1
EG/UA confluence	EG/UA confluence	[3]A33	Animas above Eureka	37.881735	-107.564966	1	1	1	1		1
		[2]A34	Eureka Gulch above Animas	37.87948	-107.567451	1	1	1	1		1
		A35	Animas below Eureka	37.875752	-107.566027	1	1	1	1		1
Animas between Eureka and Howardsville	Eureka Tailings	Eureka Wetland	Need surficial sampling grid (from Maggie to above Eureka)								
	Forest Queen	A41	Forest Queen Adit	37.931745	-107.570262	1	1	1	1		
		A41C	FQ drainage abv culvert	37.8659	-107.5678	1	1	1	1		1
		A40A	Animas abv FQ	37.8655	-107.5699	1	1	1	1		1
		FQ Tailings	Scattered Tailings								
	Kittimack	A40	Animas above Minnie Gulch (AKA Animas above Minnie)	37.863317	-107.571627	1	1	1	1		1
		A42	Minnie at Culvert (Minnie above Animas)	37.862128	-107.567246	1	1	1	1		1
		A43	Maggie Gulch above Animas	37.85424	-107.57163	1	1	1	1		1
		LA3	Animas below Maggie Gulch	37.853666	-107.577559	1	1	1	1		1
		A41A	A49? Animas below Minnie	37.861967	-107.573305	1	1	1	1		1
		KittiMack Tailings									
		Minnie Tailings									
	Howardsville	A45	Animas above Howardsville Tailings	37.842791	-107.590887	1	1	1	1		1
		A47	Hematite Gulch above Animas	37.836039	-107.598608	1	1	1	1		
		A48	Cunningham abv Bridge	37.83513	-107.595077	1	1	1	1		1
		A54	Little Nation Mine Tributary	37.832101	-107.600545	1	1	1	1		
		[2]A55	Animas 1/4 mile below A54	37.831087	-107.604696	1	1	1	1		1
Animas Below Howardsville	Animas Adjacent to Mayflower	A56	Animas Abv Arastra	37.82765815	-107.6242042	1	1	1	1	1	1
		[2]A58	Arasatra	37.825877	-107.625119	1	1	1	1	1	1
		[1]A60	Animas downstream of Arastra Creek and downstream of Bridge	37.827192	-107.626655	1	1	1	1	1	1
		A61	Animas below Arrastra	37.826169	-107.62972	1	1	1	1	1	1
		[1]A64	Animas downstream of Boulder and Aspen creeks	37.822921	-107.635217	1	1	1	1	1	1
		A65	Animas near wetland downstream f venture snowboard	37.820342	-107.642035	1	1	1	1	1	1
		A66	Animas at destroyed bridge	37.816235	-107.649617	1	1	1	1	1	1
	Animas near Silverton	A68	14th Street Gauge @ 13th Street Bridge	37.811202	-107.659167	1	1	1	1	1	1
		A72	Animas Gauge below Silverton	37.79027	-107.667578	1	1	1	1	1	1
	Below Canyon	Bakers Bridge	Actual location is Durango Riverside RV Park	37.45573315	-107.8011316	1	1	1	1	1	1
						118	118	118	118	10	60

[1] do not have access yet
[2] moved for access
[3] access was denied

Table A.7-8
Sampling Location Coordinates for Fluvial Deposit Areas

Fluvial Deposit Area	Sample Location Identification Number	Latitude	Longitude
Kittimack and Minnie Tailings	KT-FP01	37.85564342300	-107.57619806200
	KT-FP02	37.85796245100	-107.57571300700
	KT-FP03	37.86006911300	-107.57432218200
	KT-FP04	37.86206208100	-107.57295651000
	MT-FP01	37.86281268700	-107.57253304800
	MT-FP02	37.86331717900	-107.57162694100
Eureka and Forest Queen Tailings	ET-FP01	37.86484337100	-107.57030651600
	ET-FP02	37.86703563600	-107.56835167400
	ET-FP03	37.86752456800	-107.56922112100
	ET-FP04	37.86813948700	-107.56745474200
	ET-FP05	37.86855906700	-107.56825388200
	ET-FP06	37.86930041800	-107.56669679400
	ET-FP07	37.86968486900	-107.56747121000
	ET-FP08	37.87072739000	-107.56648488100
	ET-FP09	37.87093066500	-107.56693148500
	ET-FP10	37.87245723400	-107.56592774100
	ET-FP11	37.87289197900	-107.56672078600
	ET-FP12	37.87442545400	-107.56655307100
	ET-FP13	37.87610467400	-107.56690281600
	ET-FP14	37.87765031600	-107.56628341500
	ET-FP15	37.87924954500	-107.56694664800
	ET-FP16	37.87938767600	-107.56582063100
	ET-FP17	37.88230147700	-107.56447088800
Eclipse Smelter and Mill Fines	ES-FP01	37.91778399900	-107.55817174100
	ES-FP02	37.91948531400	-107.55952208500
	ES-FP03	37.92181123700	-107.56081294300

Table B.1-1 Sampling Checklist

1. Make sure the necessary paperwork is in place for a field event: Approved LSR, SAP, and QAPP.

2. Coordinate sampling dates and times with members of the field team and talk with chemists involved in the project to see if your plans work for them. Coordinate sample delivery with outside laboratories.

3. Fill out the necessary paperwork: Comp Time forms and TAs if travel will be more than 50 miles from the laboratory. Be sure to have reservations made for airlines and hotels if necessary.

4. Make necessary arrangements with people outside of the Region VIII laboratory that are involved with the project. Arrange meeting times and places, vehicle needs, sampling teams, additional equipment needs, etc.

5. Inform any volunteers outside of the EPA laboratory group what will be involved with sampling - physical stressors, equipment to bring, lunch, water, etc.

6. Calibrate meters needed for fieldwork well-before leaving. Make sure:
    a. pH probes are filled.
    b. DO membranes are intact.
    c. Spare batteries, calibration logs, and pens are available for each meter.
    d. Replace pH and conductivity calibration standards with fresh solution.
    e. Condition new probes and replace damaged ones as needed. Buy new equipment from a scientific vendor if necessary.

7. Lay out needed sampling equipment in the field room (see attached list).

8. Check vehicles: fill with gas, top off windshield wiper fluid, equip with cell phones, walkie-talkies, and chargers.

9. Charge batteries for needed sampling equipment one or two nights before leaving: digital camera, hydrolab, GPS units, walkie-talkies, etc.

10. Pack vehicles the night before leaving. In the event of hot or cold weather, leave meters and deionized water in the field room and pack the day you leave.

11. In the event of a day-trip, calibrate meters the morning you leave.

Table B.1-2 Field Equipment List

Sample Containers:
- 250 ml HDPE containers
- Gallon cubitainers
- VOA vials
- Glass Amber (BNA, pest)
- 1 oz plastic (sed. metals)

Filter Apparatus:
- 250 or 500 ml filters
- Filter Stands
- Vacuum pump with spare
- Prefilters
- Teflon Tweezers

Preservatives:
- HNO_3 - metals
- H_2SO_4 - nutrients
- Phosphoric - TOC/DOC
- HCl - VOAs (pipet)
- $CaCO_3$ Acid Waste Container
- Ice/Snow

Paperwork:
- Flow forms with clipboard
- SAP / HSP
- Maps/Gazeteer
- Chains
- Tags
- Field Notebook(s)
- FedX Forms
- Pens
- Markers
- Custody Seals

Meters (w/logs):
- Flow meter-
    - Tape measure Wading rod
    - D batteries
    - Rebar
    - Forms
    - Calculator Stopwatch, bucket
    - Flumes- bubble level, shovel
- GPS Units (charged)-
    - Compass
    - Distance meter
- Digital Camera w/batteries
- Multimeters (charged)-
    - Cal standards
    - Cap & cal. cup
    - Membranes
    - Fill solutions
    - Control unit
    - Sonde
    - Logbook
    - Cables - long/short

Misc:
- Battery charger
- pH test strips
- Latex gloves
- Neoprene gloves
- Safety glasses
- Kimwipes
- Trash bags
- Plastic Bags
- Tape
- Bucket
- Coolers
- DI rinse bottles
- Cell Phones w/ charger
- Walkie-Talkies w/batteries
- Shovel
- Spare car keys
- Vehicle log & credit card
- Govt. purchase card
- DI water for blanks
- DI rinse bottles

Macroinvertebrate Samples:
- Sample containers
- D-Frame Dip Net
- Surber Sampler
- Sieve
- White Tray
- Rinse bottle
- Picking forceps/brush
- 95% Ethanol preservative
- Bucket
- Orange water gloves

Well Sampling:
- Solinst depth meter w/battery
- Grundfos well pump-
    - Control unit
    - Hose reel w/ pump
    - Discharge tube
    - Cooling sleeve
    - Screw driver
    - Wrenches
    - Metric hex keys
    - DI water
    - 3 to 2 prong electrical converter
- Generator- gas, ext. chord
- Well Bailors
- String or chord
- Long multimeter cable

Summer Field Gear:
- Backpacks
- Hiking Boots
- Hat
- GorTex Waders
- Wading Boots
- Rain Parka
- Wool Socks
- Layered Clothing
- Sunscreen
- Chapstick
- Bug Spray
- Sun Glasses
- Water/Food
- Pocket Knife

Winter Field Gear:
- Shovel/Ice Breaker
- Backpacks
- Snowshoes
- Hiking Poles
- Insulated Water Gloves
- Hat
- Gloves
- Balaclava
- Neoprene Waders
- Wading Boots
- Wool Socks
- Layered Clothing
- Sunscreen
- Chapstick
- Sunglasses
- Water/Food
- Pocket Knife

Field Meters (when not using multimeters):
- logbooks
- pH-
    - buffers
    - probe solutions batteries
- DO-
    - Spare membranes filling solution
    - Barometer
    - Calibration equip:
    - Winkler Bottle
    - Starch
    - 0.035N Na Thio
    - Buret/Pipet
    - Buret Holder
    - Flask w/ stir bar
    - Powder Pillows:
    - MnSO4
    - Alk Iodide-Azide
    - Sulfamic Acid
- Conductivity-
    - calibration stds

Table B.2-1

Filling Time for the DH-81 Sampler using a 1 liter Bottle

Velocity, ft/sec	Volume, mL	3/16-in nozzle	1/4-in nozzle	5/16-in nozzle
1.4	800		59	
1.6	800		52	
1.8	800		46	
2.0	800	74	41	27
2.2	800	67	38	24
2.4	800	61	35	22
2.6	800	57	32	20
2.8	800	53	30	19
3.0	800	49	28	18
3.2	800	46	26	17
3.4	800	43	24	16
3.6	800	41	23	15
3.8	800	39	22	14
4.0	800	37	21	13
4.2	800	35	20	13
4.4	800	33	19	12
4.6	800	32	18	12
4.8	800	31	17	11
5.0	800	29	17	11
5.2	800	28	16	10
5.4	800	27	15	10
5.6	800	26	15	9
5.8	800	25	14	9
6.0	800	25	14	9
6.2	800	24	13	9
6.4	800		13	8
6.6	800		13	8
6.8	800		12	8
7.0	800		12	8
7.2	800		12	
7.4	800		11	
7.6	800		11	

Table B.5-1
ESAT Region 8 - Metals QC 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 B.5-2
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 concentration

C_s = Sample extract, spiked concentration

S_a = Spike amount added

T = True (possibly certified) amount in standard solution

Hardness = (Ca, mg/L)*2.497 + (Mg, mg/L)*4.118

C_D = Duplicate sample concentration

C_L = Sample extract concentration, dilution factor corrected.

AT = Analyzed concentration for the known standard.

Figures

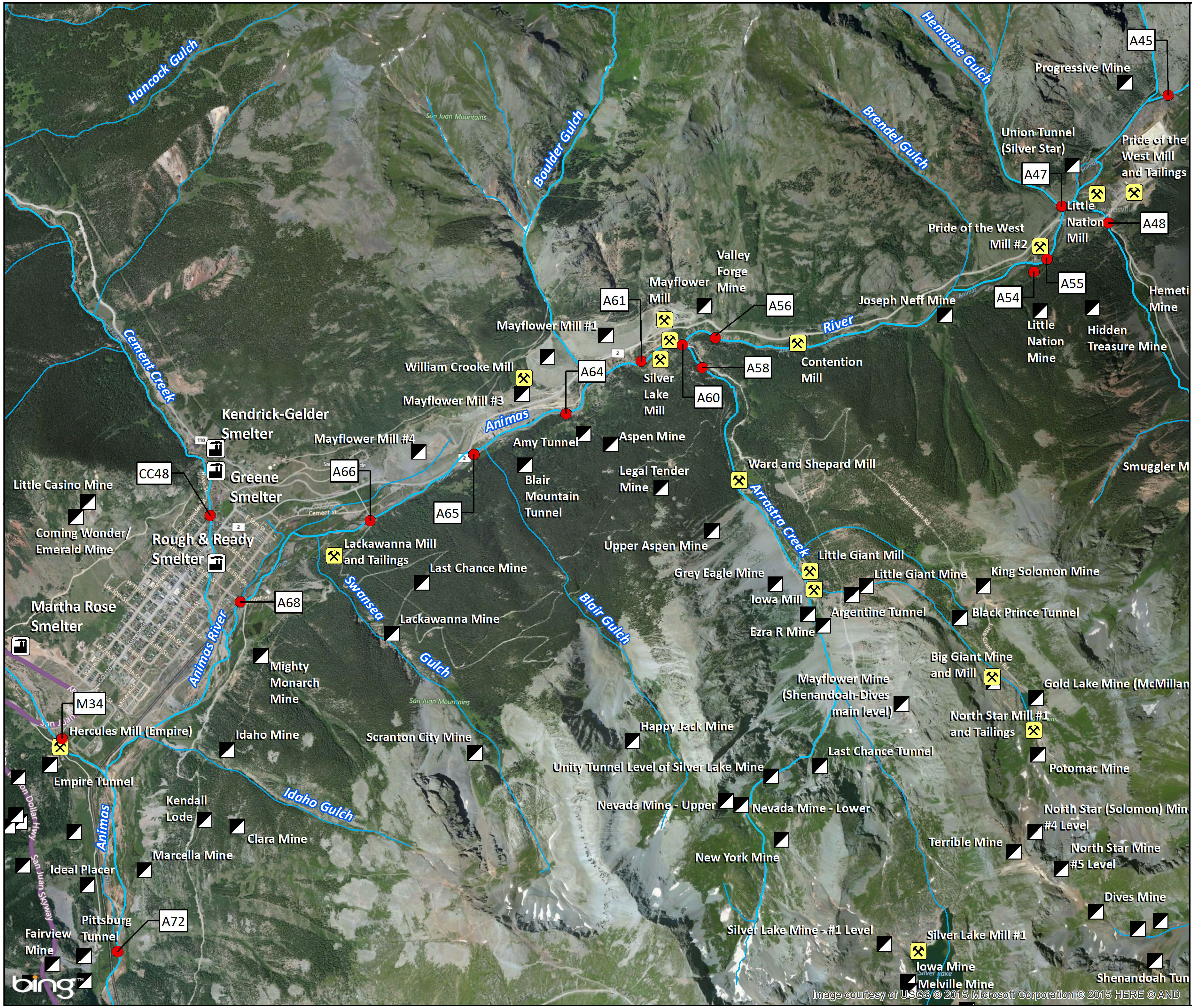


Figure A.7-1
Upper Animas Mining District
Silverton and Mayflower Mill Area

- ● Sample Locations
- ▣ Smelter Locations
- ⚒ Mill Locations
- ◩ Mine Locations
- ~ Rivers and Streams

Map Date: March 20, 2015

Data Sources:
Sample Locations: U.S. EPA (2015)
Rivers and Streams: CDOW (2004)
Smelter, Mine, & Mill Locations: USGS (2000)
Image: Microsoft Bing web service (2015)
Map Projection: UTM Zone 13 N, NAD83, Meters

0 2,000 4,000 Feet
0 400 800 Meters

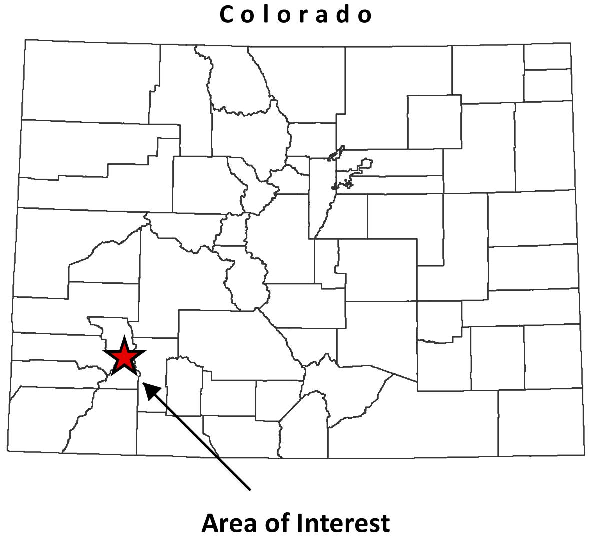
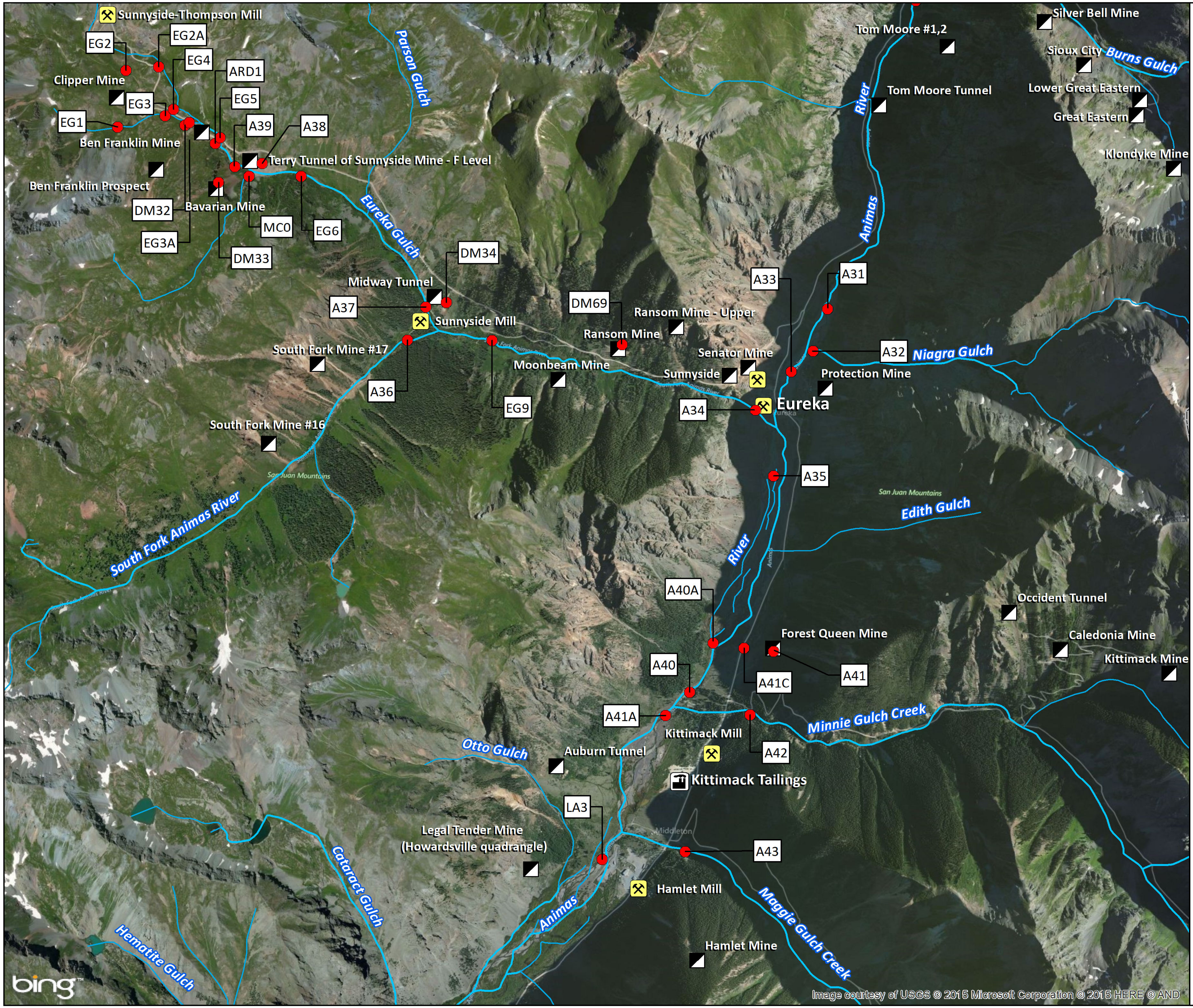


Figure A.7-2
Upper Animas Mining District
Upper Animas Eureka and Howardsville Area



● Sample Locations

▣ Smelter Locations

⚒ Mill Locations

◩ Mine Locations

〰 Rivers and Streams

Map Date: April 8, 2015

Data Sources:
Sample Locations: U.S. EPA (2015)
Rivers and Streams: CDOW (2004)
Smelter, Mine & Mill Locations: USGS (2000)
Image: Microsoft Bing web service (2015)
Map Projection: UTM Zone 13 N, NAD83, Meters

TechLaw

N
W E
S

0 2,000 4,000 Feet

0 400 800 Meters

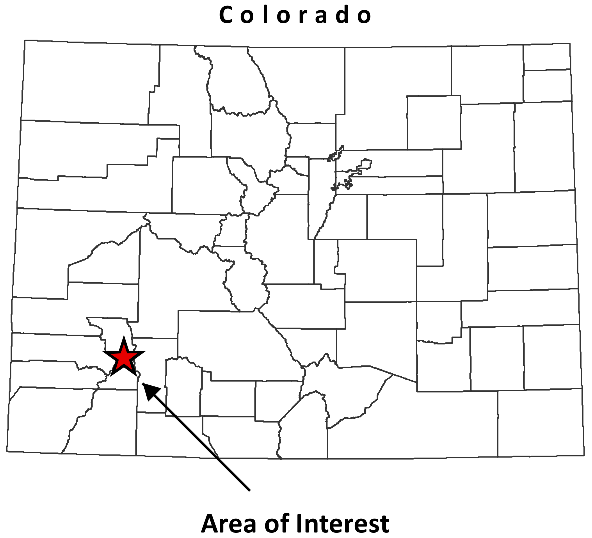


Figure A.7-3
Upper Animas Mining District
Upper Animas Headwaters Area

● Sample Locations

▣ Smelter Locations

☒ Mill Locations

◩ Mine Locations

〰 Rivers and Streams

⬚ Counties

Map Date: April 8, 2015

Data Sources:
Sample Locations: U.S. EPA (2015)
Rivers and Streams: CDOW (2004)
Counties: U.S. Census Bureau (2010)
Smelter, Mine & Mill Locations: USGS (2000)
Image: Microsoft Bing web service (2015)
Map Projection: UTM Zone 13 N, NAD83, Meters

0 2,000 4,000 Feet

0 400 800 Meters

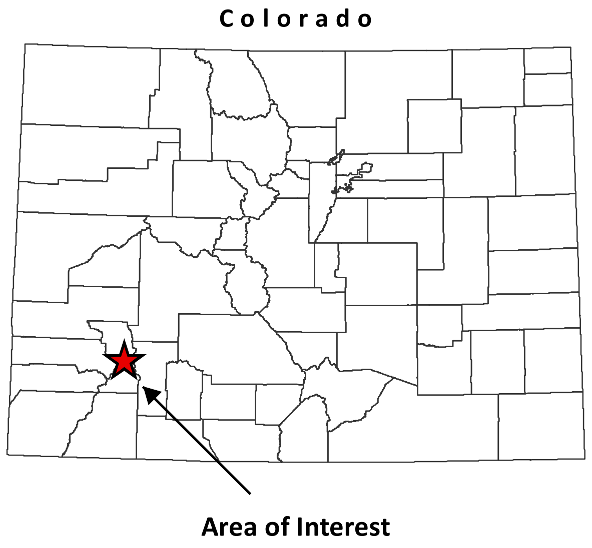


Figure A.7-4
Upper Animas Mining District
Upper Cement Creek Area

● Sample Locations
⚒ Mill Locations
◩ Mine Locations
〰 Rivers and Streams
⬚ Counties

Map Date: April 8, 2015

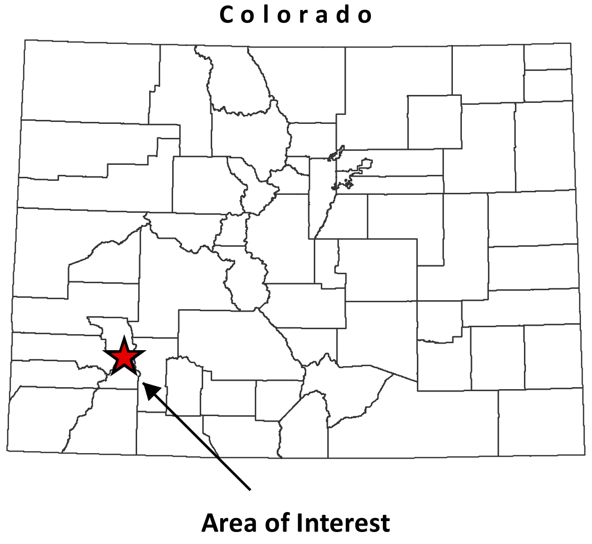
Data Sources:
Sample Locations: U.S. EPA (2015)
Rivers and Streams: CDOW (2004)
Counties: U.S. Census Bureau (2010)
Mine & Mill Locations: USGS (2000)
Image: Microsoft Bing web service (2015)
Map Projection: UTM Zone 13 N, NAD83, Meters

TechLaw

N
W E
S

0 1,000 2,000 Feet

0 200 400 Meters



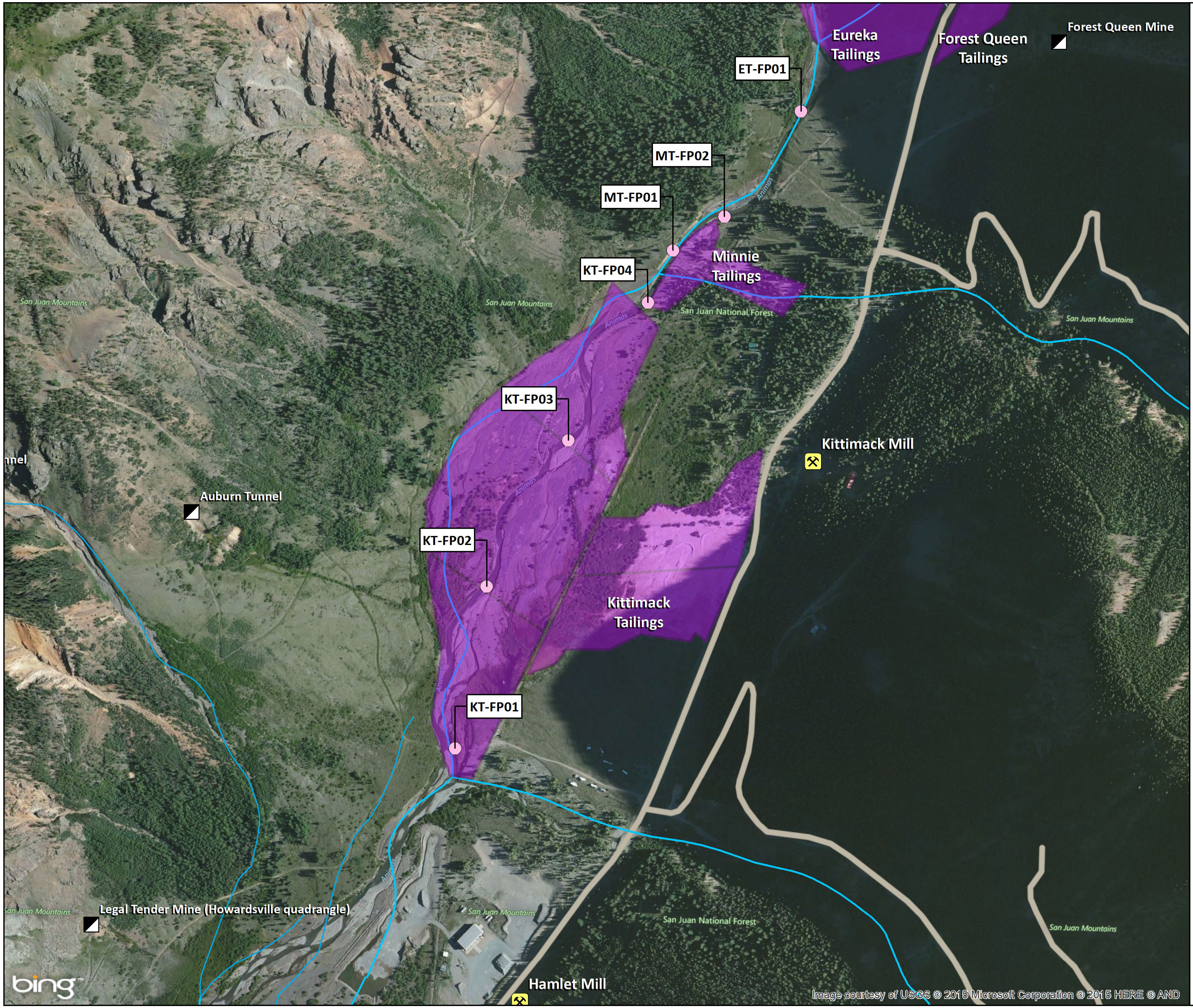


Figure A.7-5
Upper Animas Mining District
Kittimack & Minnie Tailings Fluvial Deposits

●	Sample Locations
▨	Floodplain Sampling Area
⚒	Mill Locations
◩	Mine Locations
~	Rivers and Streams

Map Date: April 8, 2015

Data Sources:
Fluvial Samples & Wetlands: U.S. EPA (2015)
Rivers and Streams: CDOW (2004)
Smelter, Mine & Mill Locations: USGS (2000)
Image: Microsoft Bing web service (2015)
Map Projection: UTM Zone 13 N, NAD83, Meters

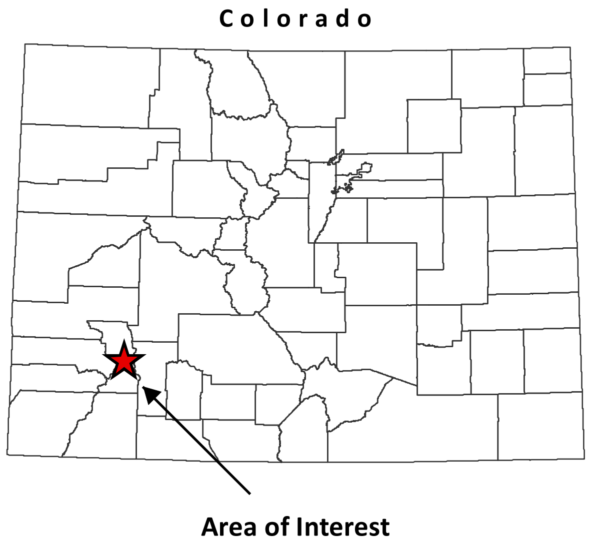


Figure A.7-6
Upper Animas Mining District
Eureka & Forest Queen Tailings Fluvial Deposits

● Sample Locations

▨ Floodplain Sampling Area

▪ Smelter Locations

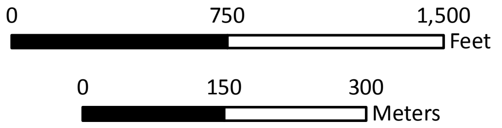
⚒ Mill Locations

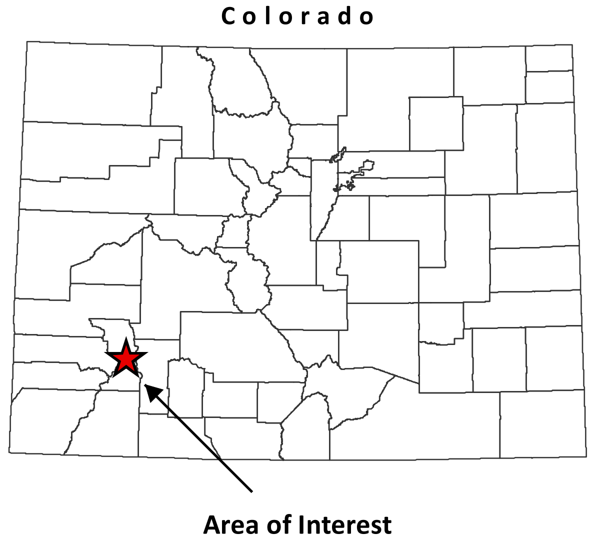
◩ Mine Locations

〰 Rivers and Streams

Map Date: April 8, 2015

Data Sources:
Fluvial Samples & Wetlands: U.S. EPA (2015)
Rivers and Streams: CDOW (2004)
Smelter, Mine & Mill Locations: USGS (2000)
Image: Microsoft Bing web service (2015)
Map Projection: UTM Zone 13 N, NAD83, Meters





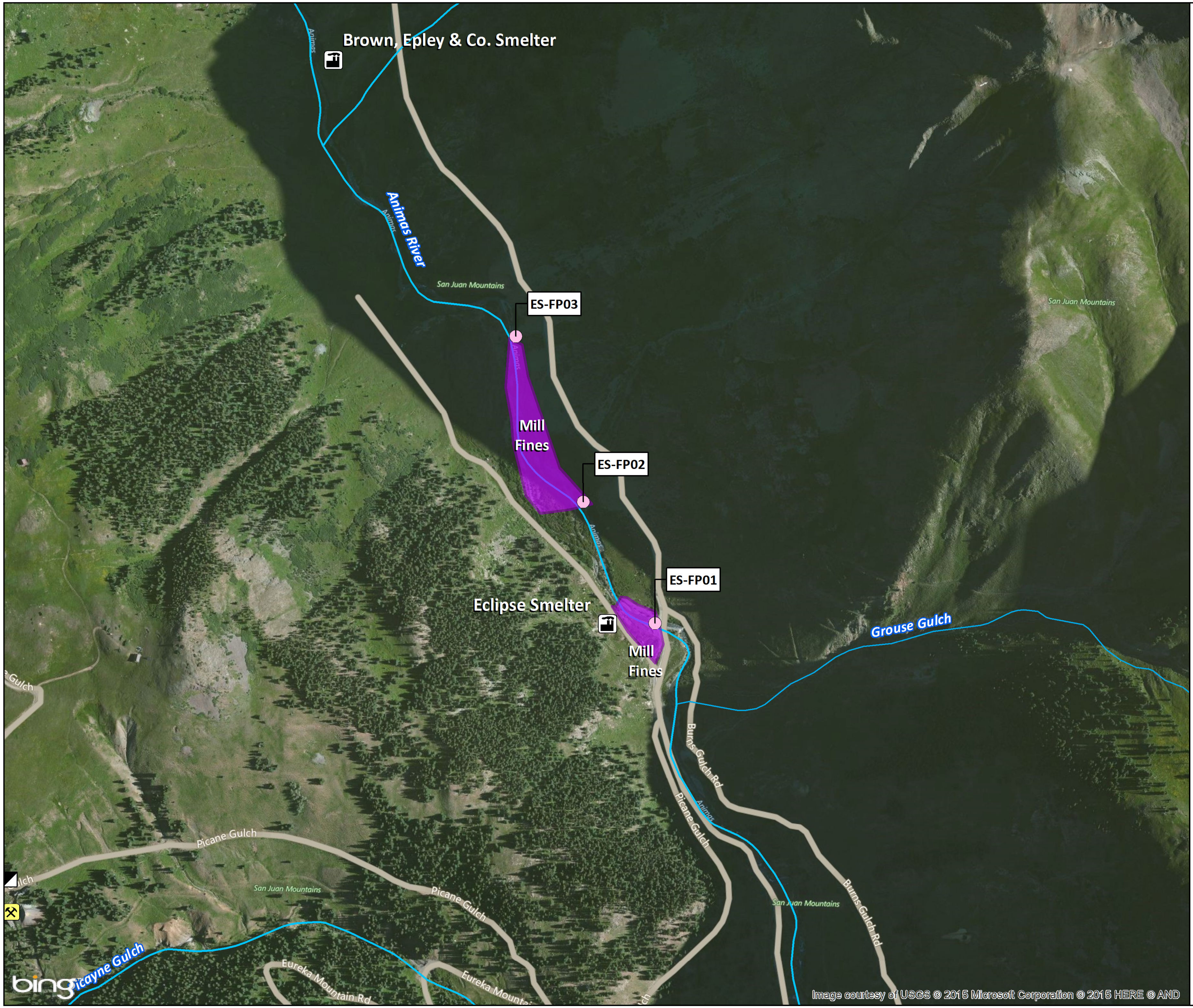


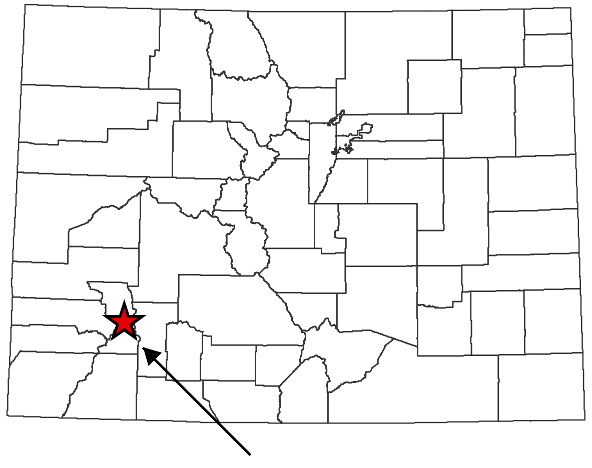
Figure A.7-7
Upper Animas Mining District
Eclipse Smelter & Mill Fines Tailings Fluvial Deposits

●	Sample Locations
🟪	Floodplain Sampling Area
▣	Smelter Locations
⚒	Mill Locations
◩	Mine Locations
〰	Rivers and Streams

Map Date: April 8, 2015

Data Sources:
Fluvial Samples & Wetlands: U.S. EPA (2015)
Rivers and Streams: CDOW (2004)
Smelter, Mine & Mill Locations: USGS (2000)
Image: Microsoft Bing web service (2015)
Map Projection: UTM Zone 13 N, NAD83, Meters

Colorado



Area of Interest

Appendix A
Standard Operating Procedures

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

This document was prepared by the ESAT Region 8 Team and is intended to provide documentation of administrative, analytical and quality control procedures used in the daily performance of ESAT support services. This is a controlled document and may only be provided to a third party, such as consultants or other government agencies, at the direction of EPA and if all said third party recipients agree that the contents of this document remain confidential. If the document is provided as a controlled document, the user agrees to surrender the document upon request of EPA or ESAT Region 8. If the document is provided as an uncontrolled document, the user understands that subsequent revisions may not be provided.

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

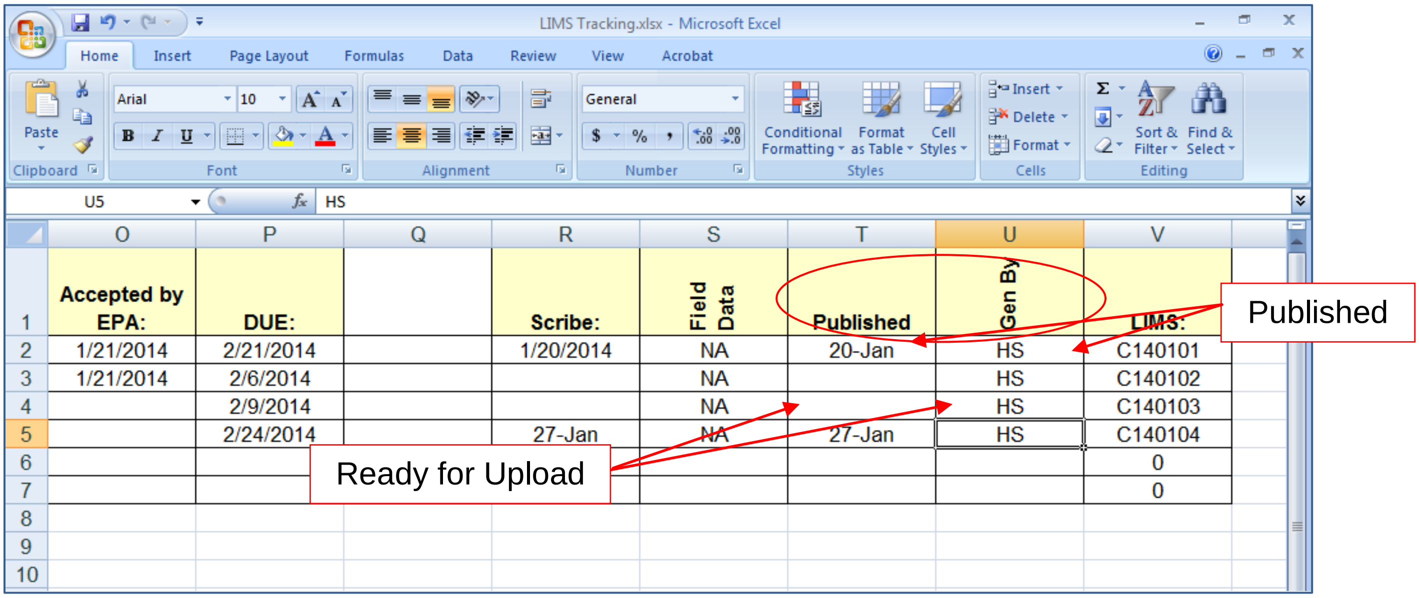
The analysts and/or Data Package Coordinator posts EDDs to the appropriate Task Order (TO) and project folder on the network drive upon completion. Prior to uploading or publishing a project to Scribe, review the LIMS Tracking spreadsheet to ensure that the

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



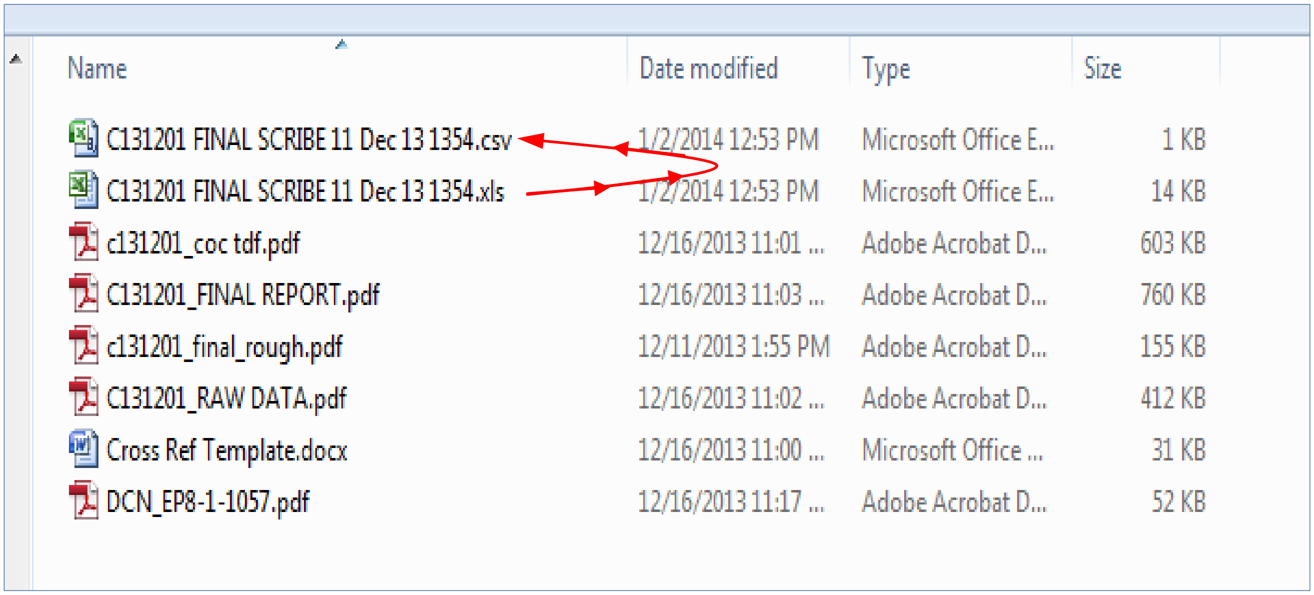
	O	P	Q	R	S	T	U	V
1	Accepted by EPA:	DUE:		Scribe:	Field Data	Published	Gen By	LIMS:
2	1/21/2014	2/21/2014		1/20/2014	NA	20-Jan	HS	C140101
3	1/21/2014	2/6/2014			NA		HS	C140102
4		2/9/2014			NA		HS	C140103
5		2/24/2014		27-Jan	NA	27-Jan	HS	C140104
6								0
7								0
8								
9								
10								

Published

Ready for Upload

- 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



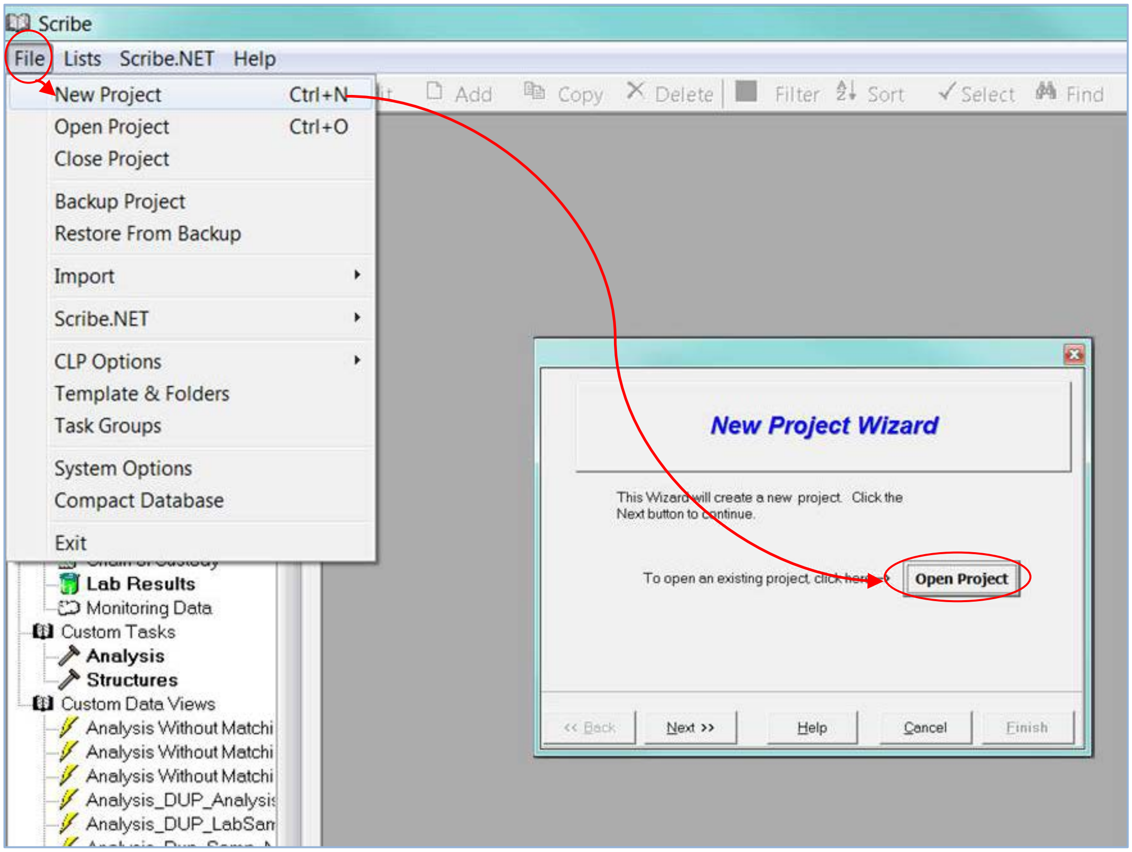
Name	Date modified	Type	Size
C131201 FINAL SCRIBE 11 Dec 13 1354.csv	1/2/2014 12:53 PM	Microsoft Office E...	1 KB
C131201 FINAL SCRIBE 11 Dec 13 1354.xls	1/2/2014 12:53 PM	Microsoft Office E...	14 KB
c131201_coc tdf.pdf	12/16/2013 11:01 ...	Adobe Acrobat D...	603 KB
C131201_FINAL REPORT.pdf	12/16/2013 11:03 ...	Adobe Acrobat D...	760 KB
c131201_final_rough.pdf	12/11/2013 1:55 PM	Adobe Acrobat D...	155 KB
C131201_RAW DATA.pdf	12/16/2013 11:02 ...	Adobe Acrobat D...	412 KB
Cross Ref Template.docx	12/16/2013 11:00 ...	Microsoft Office ...	31 KB
DCN_EP8-1-1057.pdf	12/16/2013 11:17 ...	Adobe Acrobat D...	52 KB

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"



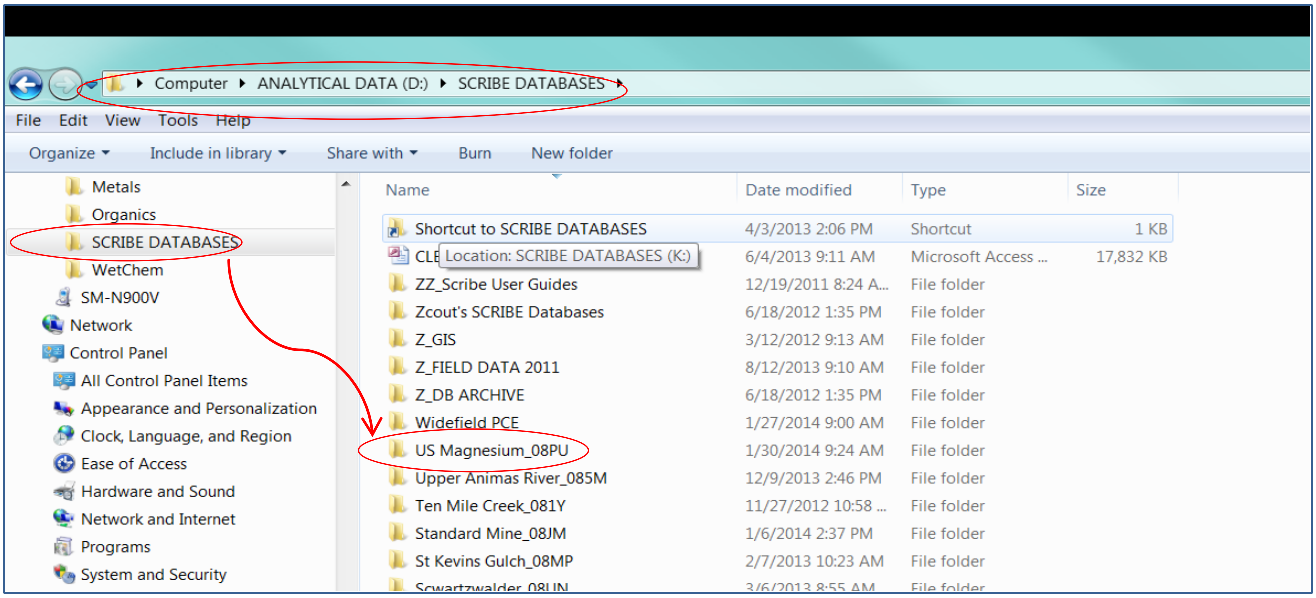
This Wizard will create a new project. Click the Next button to continue.

To open an existing project, click here

- 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.

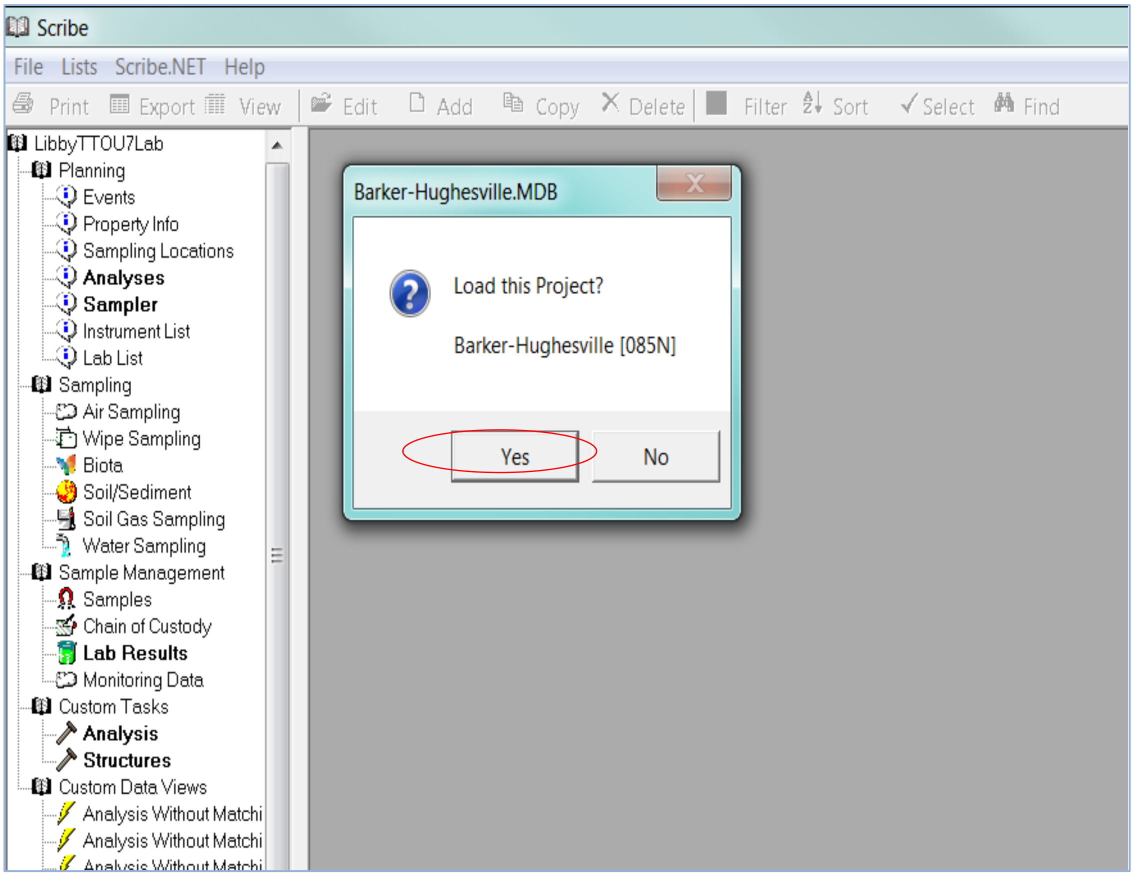
Figure 5.4 Scribe Database Project Pathway



Name	Date modified	Type	Size
Shortcut to SCRIBE DATABASES	4/3/2013 2:06 PM	Shortcut	1 KB
CLE Location: SCRIBE DATABASES (K:)	6/4/2014 9:11 AM	Microsoft Access ...	17,832 KB
ZZ_Scribe User Guides	12/19/2011 8:24 A...	File folder	
Zcout's SCRIBE Databases	6/18/2012 1:35 PM	File folder	
Z_GIS	3/12/2012 9:13 AM	File folder	
Z_FIELD DATA 2011	8/12/2013 9:10 AM	File folder	
Z_DB ARCHIVE	6/18/2012 1:35 PM	File folder	
Widefield PCE	1/27/2014 9:00 AM	File folder	
US Magnesium_08PU	1/30/2014 9:24 AM	File folder	
Upper Animas River_085M	12/9/2013 2:46 PM	File folder	
Ten Mile Creek_081Y	11/27/2012 10:58 ...	File folder	
Standard Mine_08JM	1/6/2014 2:37 PM	File folder	
St Kevins Gulch_08MP	2/7/2013 10:23 AM	File folder	
Scwartzwalder_08UN	3/6/2013 8:55 AM	File folder	

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

Figure 5.5 Scribe Database Load Project

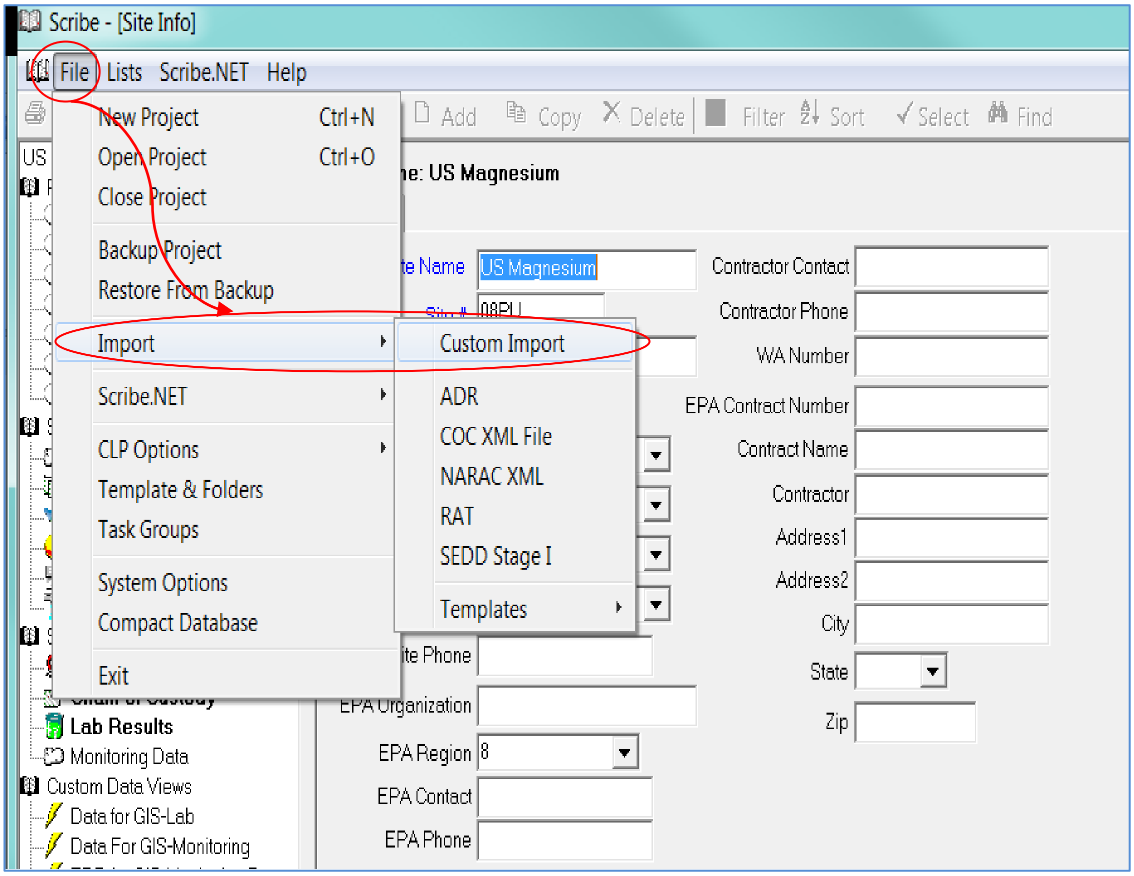


Load this Project?

Barker-Hughesville [085N]

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

Figure 5.6 Scribe "File/Import/Custom Import"

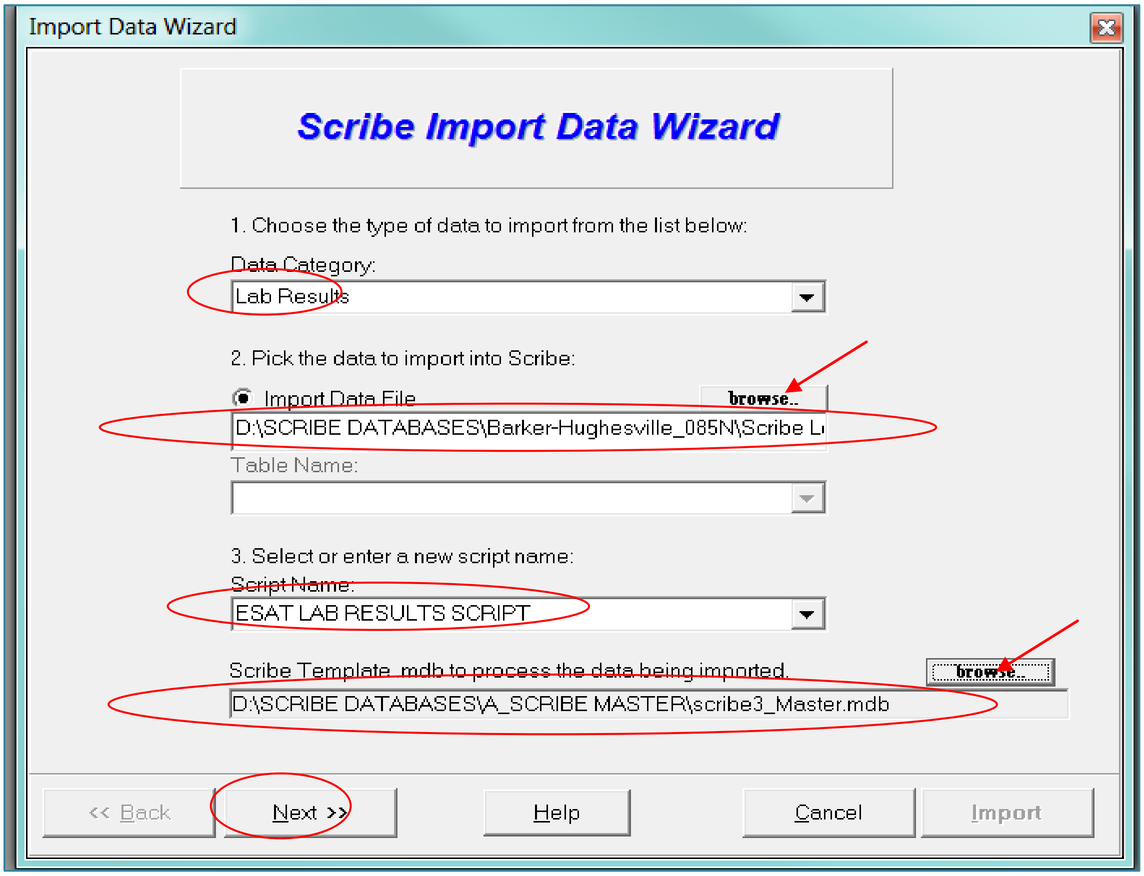


- Select "Lab Results" from the drop down arrow list in the Data Category field
- Select the correct EDD using the "browse" button above the Import Data File field

  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"



1. Choose the type of data to import from the list below:

Data Category: Lab Results

2. Pick the data to import into Scribe:

● Import Data File
D:\SCRIBE DATABASES\Barker-Hughesville_085N\Scribe L

Table Name:

3. Select or enter a new script name:

Script Name: ESAT LAB RESULTS SCRIPT

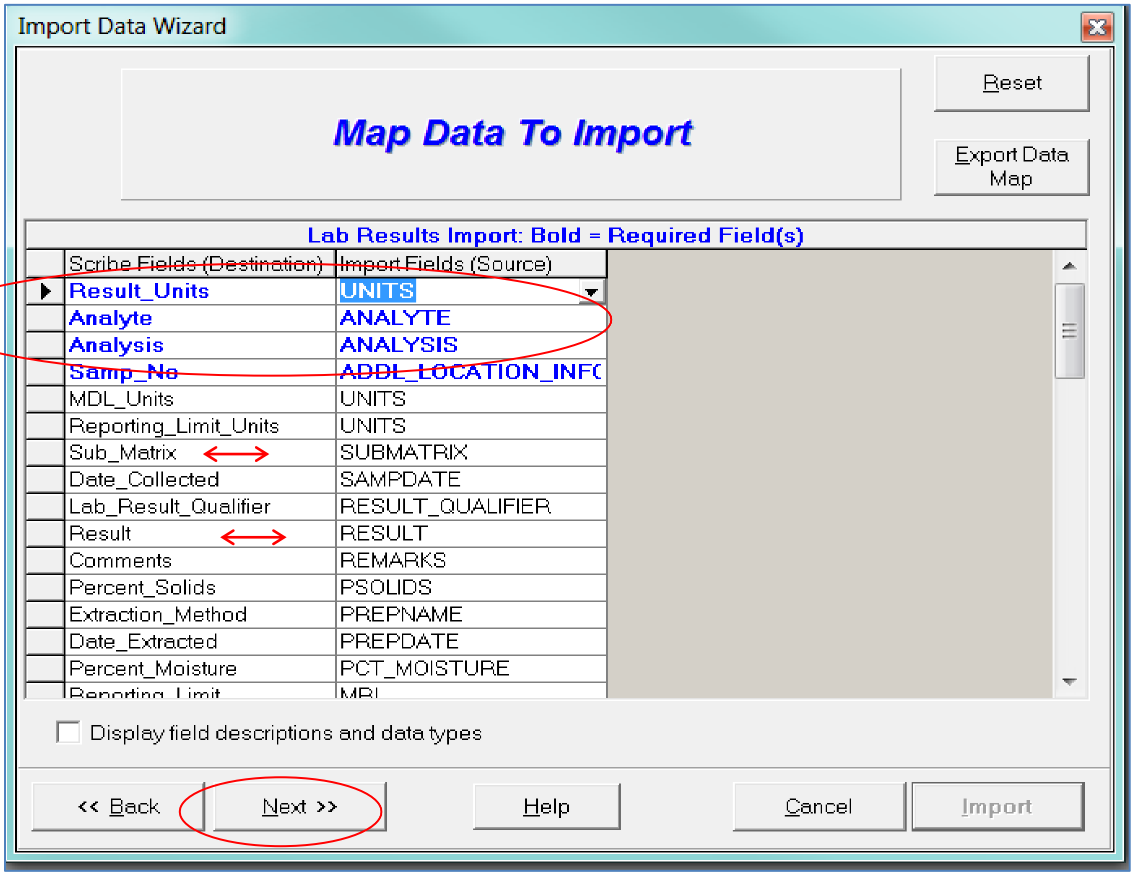
Scribe Template .mdb to process the data being imported.
D:\SCRIBE DATABASES\A_SCRIBE MASTER\scribe3_Master.mdb

- 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"



Lab Results Import: Bold = Required Field(s)

Scribe Fields (Destination)	Import Fields (Source)
Result_Units	**UNITS**
Analyte	**ANALYTE**
Analysis	**ANALYSIS**
Samp_No	**ADDL_LOCATION_INFC**
MDL_Units	UNITS
Reporting_Limit_Units	UNITS
Sub_Matrix	SUBMATRIX
Date_Collected	SAMPDATE
Lab_Result_Qualifier	RESULT_QUALIFIER
Result	RESULT
Comments	REMARKS
Percent_Solids	PSOLIDS
Extraction_Method	PREPNAME
Date_Extracted	PREPDATE
Percent_Moisture	PCT_MOISTURE
Reporting_Limit	MRL

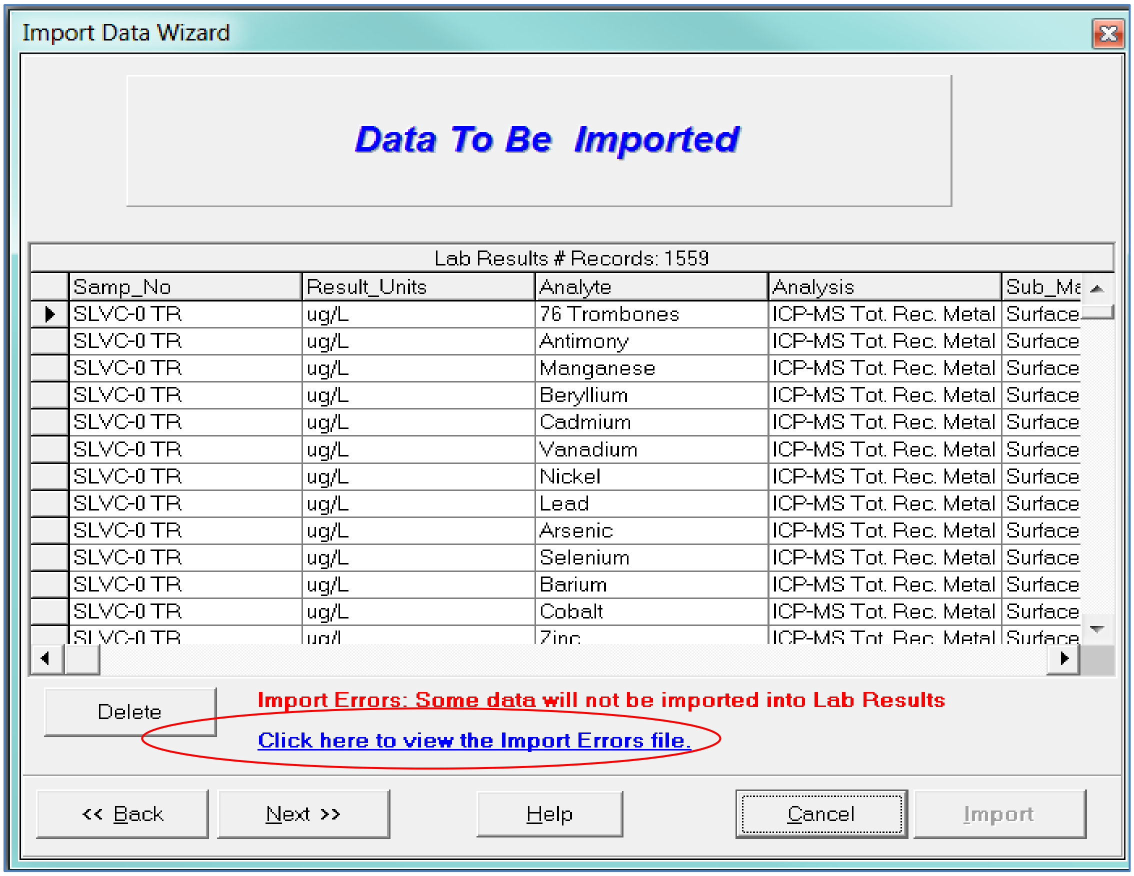
☐ Display field descriptions and data types

- 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



Import Data Wizard

Data To Be Imported

Lab Results # Records: 1559

Samp_No	Result_Units	Analyte	Analysis	Sub_Ma
SLVC-0 TR	ug/L	76 Trombones	ICP-MS Tot. Rec. Metal	Surface
SLVC-0 TR	ug/L	Antimony	ICP-MS Tot. Rec. Metal	Surface
SLVC-0 TR	ug/L	Manganese	ICP-MS Tot. Rec. Metal	Surface
SLVC-0 TR	ug/L	Beryllium	ICP-MS Tot. Rec. Metal	Surface
SLVC-0 TR	ug/L	Cadmium	ICP-MS Tot. Rec. Metal	Surface
SLVC-0 TR	ug/L	Vanadium	ICP-MS Tot. Rec. Metal	Surface
SLVC-0 TR	ug/L	Nickel	ICP-MS Tot. Rec. Metal	Surface
SLVC-0 TR	ug/L	Lead	ICP-MS Tot. Rec. Metal	Surface
SLVC-0 TR	ug/L	Arsenic	ICP-MS Tot. Rec. Metal	Surface
SLVC-0 TR	ug/L	Selenium	ICP-MS Tot. Rec. Metal	Surface
SLVC-0 TR	ug/L	Barium	ICP-MS Tot. Rec. Metal	Surface
SLVC-0 TR	ug/L	Cobalt	ICP-MS Tot. Rec. Metal	Surface
SLVC-0 TR	ug/L	Zinc	ICP-MS Tot. Rec. Metal	Surface

Import Errors: Some data will not be imported into Lab Results

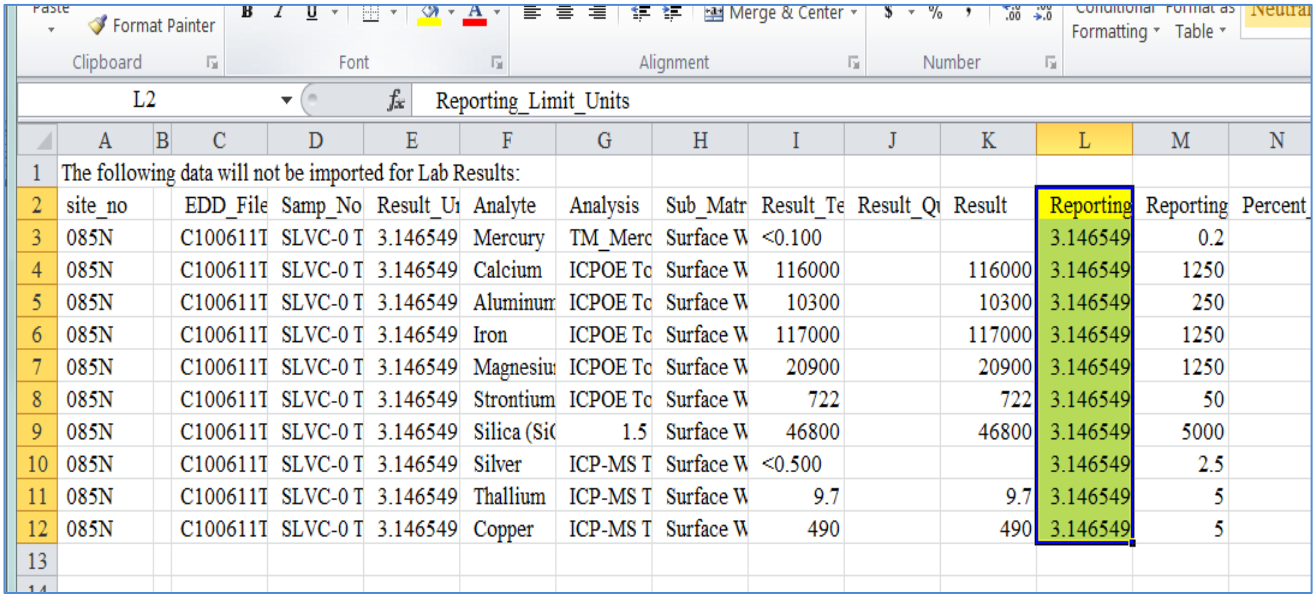
Click here to view the Import Errors file.

- 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

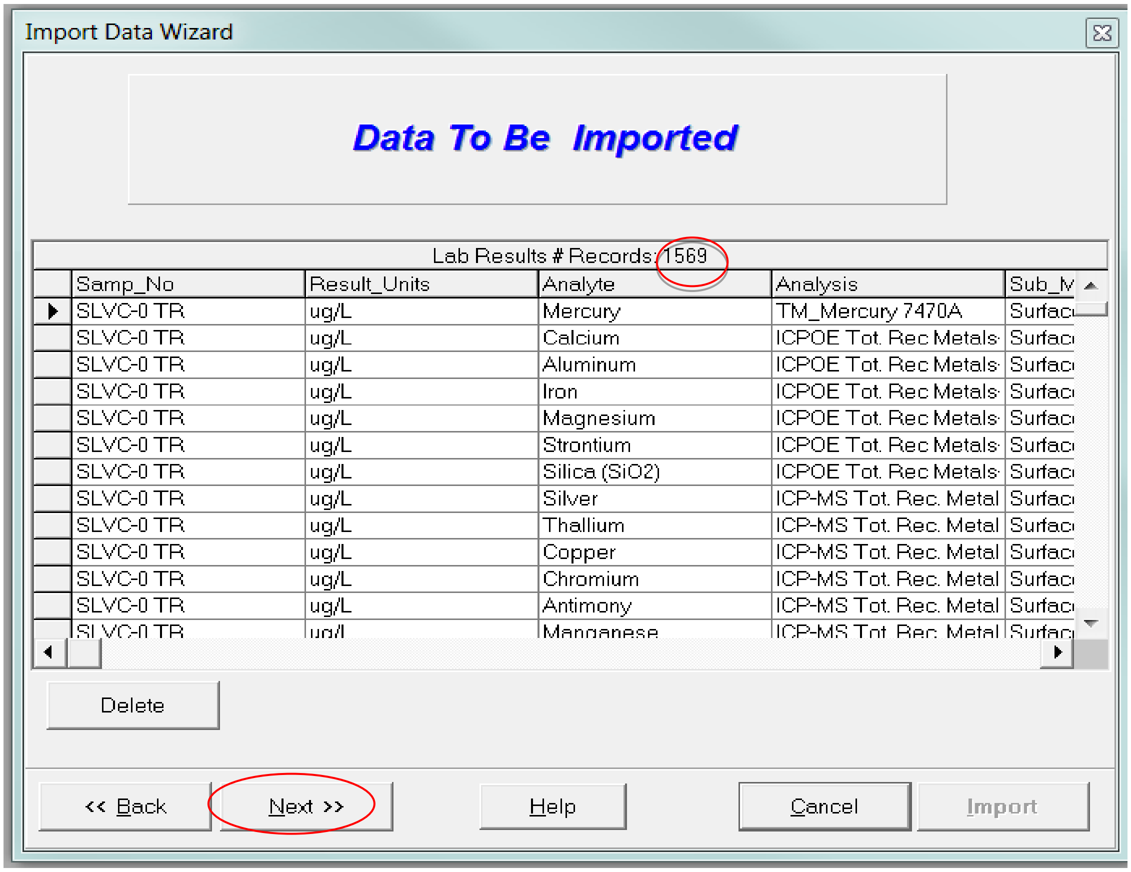


L2 Reporting_Limit_Units

	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 Tc	Surface W	116000		116000	3.146549	1250	
5	085N		C100611T	SLVC-0 T	3.146549	Aluminum	ICPOE Tc	Surface W	10300		10300	3.146549	250	
6	085N		C100611T	SLVC-0 T	3.146549	Iron	ICPOE Tc	Surface W	117000		117000	3.146549	1250	
7	085N		C100611T	SLVC-0 T	3.146549	Magnesiu	ICPOE Tc	Surface W	20900		20900	3.146549	1250	
8	085N		C100611T	SLVC-0 T	3.146549	Strontium	ICPOE Tc	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"

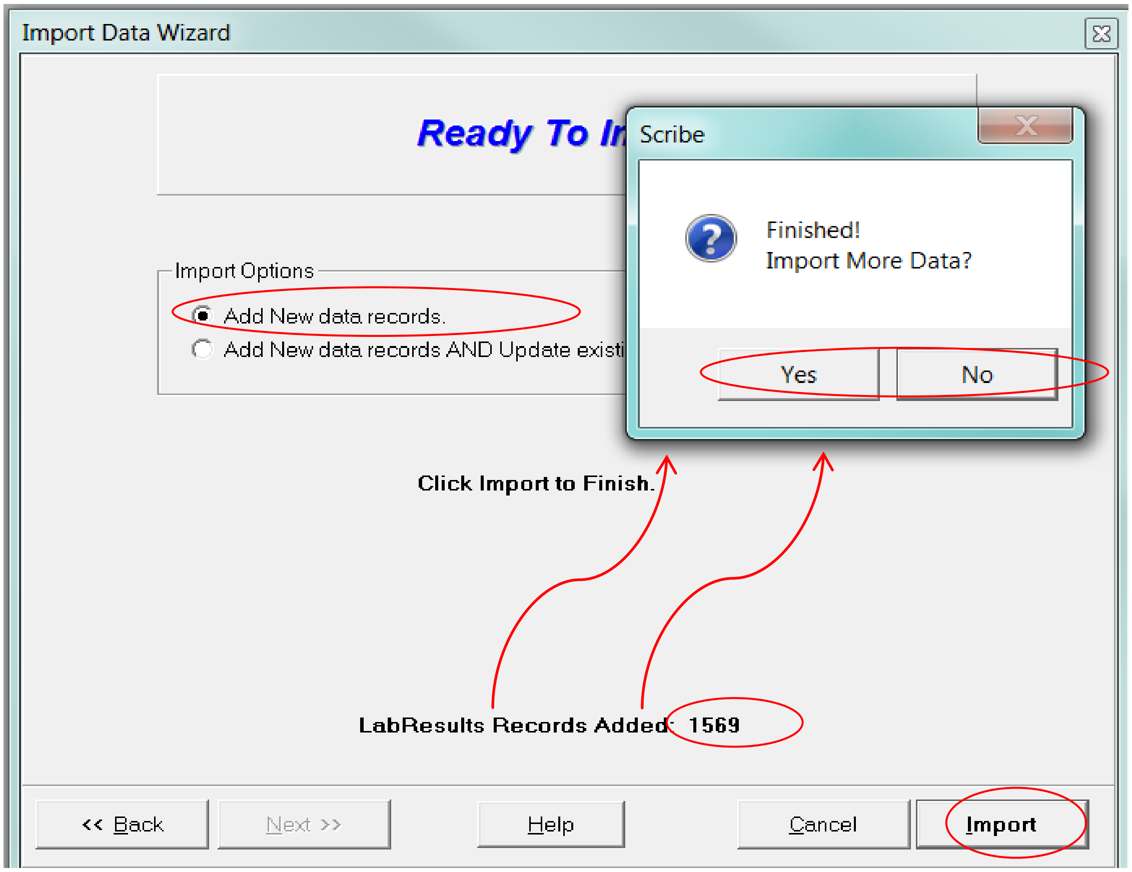


Lab Results # Records: 1569

Samp_No	Result_Units	Analyte	Analysis	Sub_M
SLVC-0 TR	ug/L	Mercury	TM_Mercury 7470A	Surfac
SLVC-0 TR	ug/L	Calcium	ICPOE Tot. Rec Metals	Surfac
SLVC-0 TR	ug/L	Aluminum	ICPOE Tot. Rec Metals	Surfac
SLVC-0 TR	ug/L	Iron	ICPOE Tot. Rec Metals	Surfac
SLVC-0 TR	ug/L	Magnesium	ICPOE Tot. Rec Metals	Surfac
SLVC-0 TR	ug/L	Strontium	ICPOE Tot. Rec Metals	Surfac
SLVC-0 TR	ug/L	Silica (SiO2)	ICPOE Tot. Rec Metals	Surfac
SLVC-0 TR	ug/L	Silver	ICP-MS Tot. Rec. Metal	Surfac
SLVC-0 TR	ug/L	Thallium	ICP-MS Tot. Rec. Metal	Surfac
SLVC-0 TR	ug/L	Copper	ICP-MS Tot. Rec. Metal	Surfac
SLVC-0 TR	ug/L	Chromium	ICP-MS Tot. Rec. Metal	Surfac
SLVC-0 TR	ug/L	Antimony	ICP-MS Tot. Rec. Metal	Surfac
SLVC-0 TR	ug/L	Manganese	ICP-MS Tot. Rec. Metal	Surfac

- 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?"



Import Data Wizard

Ready To In

Import Options

● Add New data records.

○ Add New data records AND Update existi

Scribe

Finished!
Import More Data?

Click Import to Finish.

LabResults Records Added: 1569

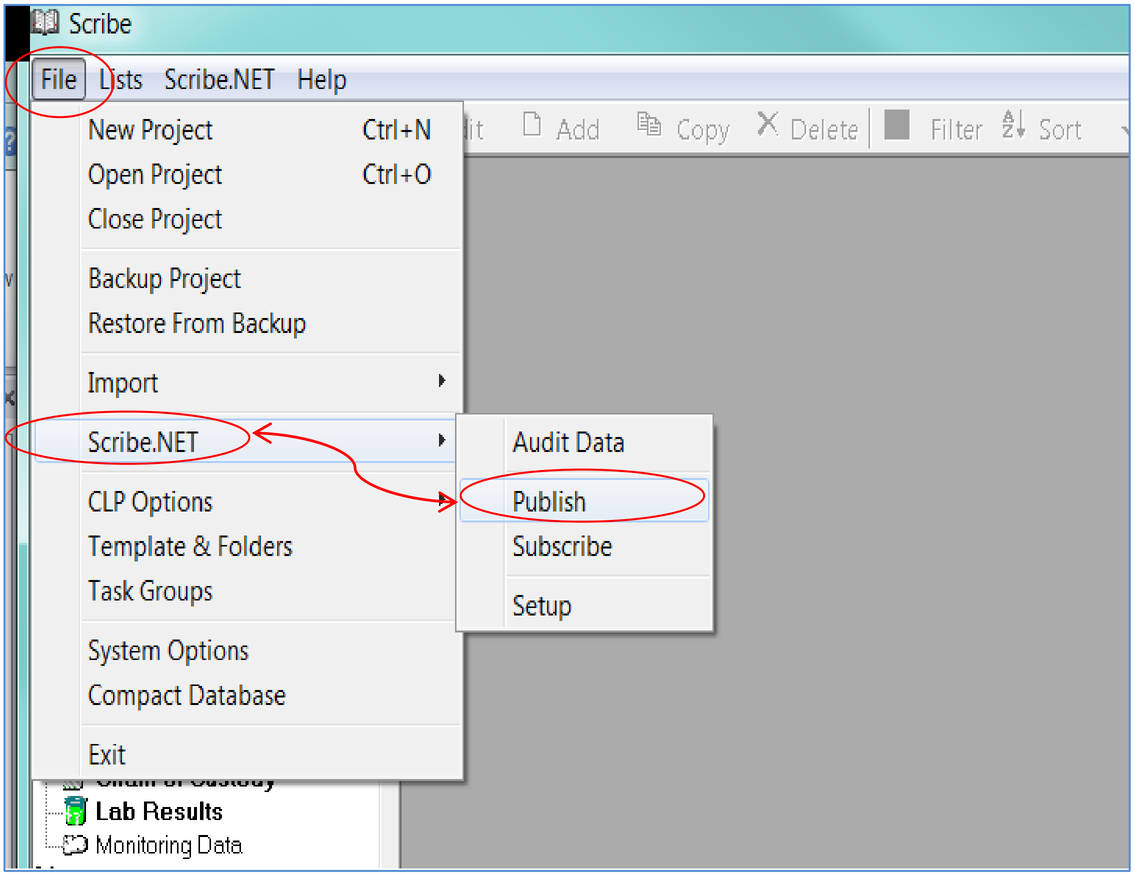
- Continue with all completed EDDs (those noted as ready to upload in the LIMS Tracking spreadsheet)

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"

This wizard will Publish your Scribe project to Scribe.NET. Click Next to Continue.

Network: Connected

- 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

Scribe.NET Publisher Wizard

Enter your Publisher ID or Select a Publication ID from the list below.
Then Click Publish to publish the Scribe project to Scribe.NET.

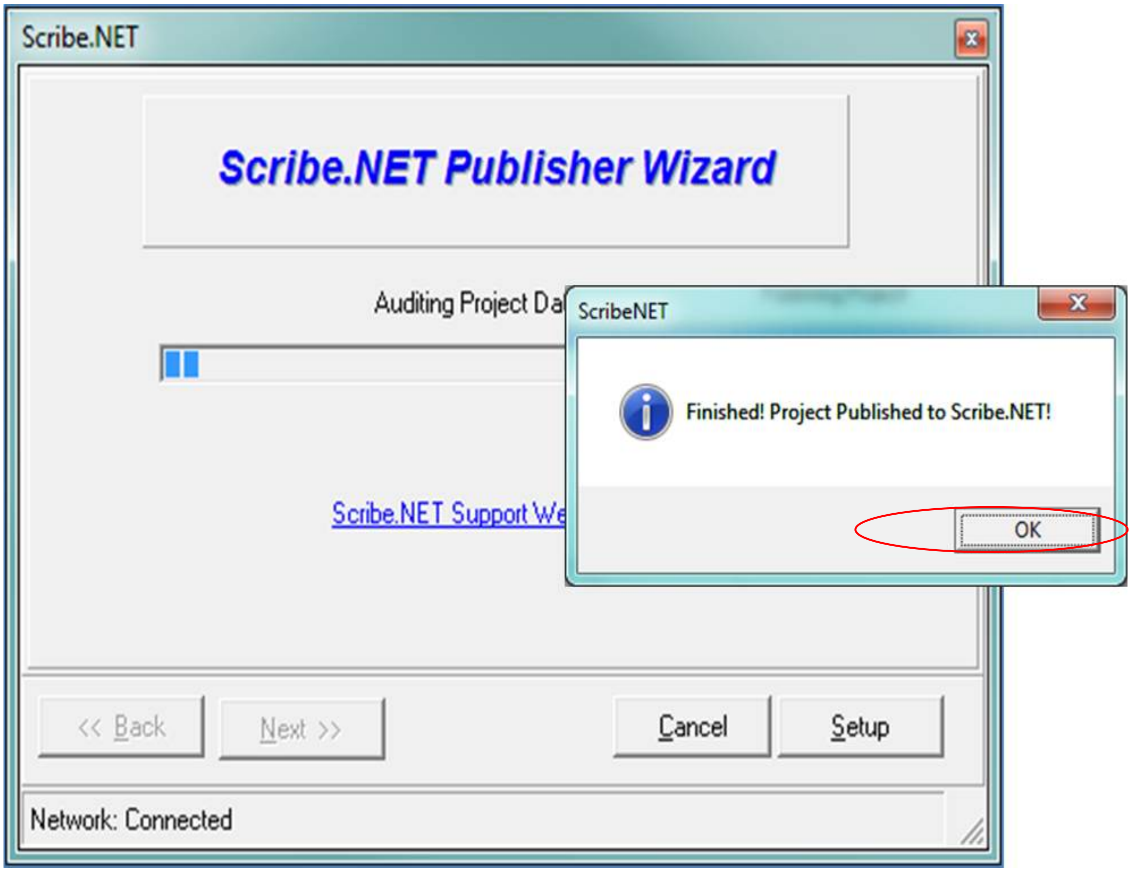
Publisher ID: USEPA Region 8

Password: *****

Network: Connected

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

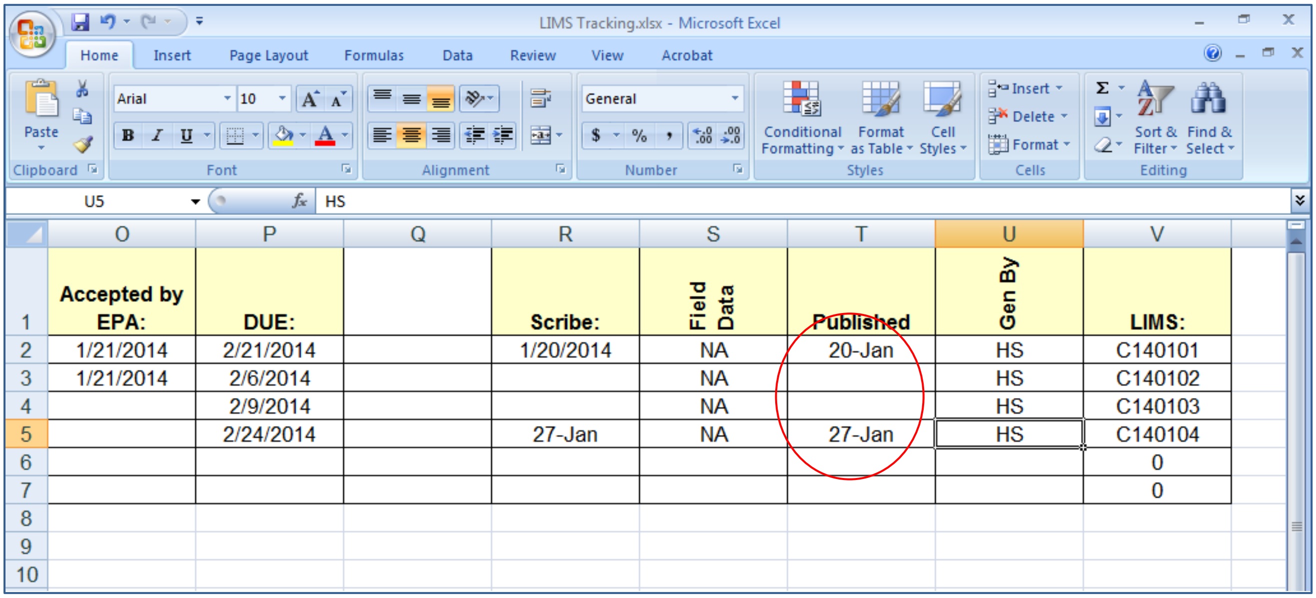
- **Figure 5.16 Scribe.NET "Project Published"**



Finished! Project Published to Scribe.NET!

- 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



U5 | HS

	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

Surface Water Sampling

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

This document has been prepared for the Environmental Protection Agency by the TechLaw, Inc. ESAT Region 8 Team and is intended to provide documentation of administrative, analytical and quality control procedures used in the daily performance of EPA and ESAT support services.

TABLE OF CONTENTS

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to guide field personnel on general surface water sampling procedures. Not all situations are accounted for; therefore site reconnaissance is a key factor in determining sampling techniques that may be utilized. Always consult the site-specific Sampling and Analysis Plan and Quality Assurance Project Plan (SAP/QAPP) before deployment to a surface water sampling event.

2.0 SCOPE AND APPLICABILITY

This SOP is applicable to the collection of representative liquid samples from streams, rivers, lakes, ponds, lagoons, and surface impoundments utilizing direct method sampling procedures. This method may be varied or changed as required, dependent upon site conditions, equipment limitations or other procedural limitations. These are the preferred methodologies and should be implemented as closely as possible. Any deviations from these procedures should be discussed with site managers in order to confirm that data objectives are being met. All procedures employed should be documented and associated with the final report. Mention of trade names or commercial products does not constitute TechLaw, Inc. endorsement or recommendation for use.

This SOP is to be used in conjunction with other relevant and applicable documents which may include:

- Water Quality Measurement Procedures
- Sample Preservation Procedures
- Sediment Sampling Procedures
- Pore Water Sampling Procedures
- Field Sampling Procedures

The following documentation should be included to assist in preparing for and conducting surface water sampling activities:

- Health and Safety Plan (HASP)
- Sampling and Analysis Plan (SAP)
- Quality Assurance Project Plan (QAPP)
- Any other site-specific planning documents

3.0 SUMMARY OF METHOD

This SOP is intended to provide guidance on collection of surface water. Sampling situations vary widely, therefore, no universal sampling procedure can be recommended. However, sampling of aqueous liquids from the above mentioned sources is generally accomplished through use of the direct method technique (filling sample containers directly from the source) and transfer devices (collecting the sample with a container and then transferring to another container). This allows for the collection of representative samples of surface water from streams, creeks, rivers, lakes, ponds, and other impoundments. Note that for certain types of sampling, transfer devices are not appropriate. Volatile Organic Analysis and Semi-Volatile Organic Analysis samples should always be sampled directly if possible. Also, proper decontamination or conditioning of transfer devices must occur in order to avoid cross-contamination.

4.0 ACRONYMS AND DEFINITIONS

°C	Degrees Celsius
COC	Chain of Custody
EPA	United States Environmental Protection Agency
ESAT	Environmental Services Assistance Team
EDI	Equal Discharge Increment
EWI	Equal Width Increment
GPS	Global Positioning System
HASP	Health and Safety Plan
HAZWOPER	Hazardous Waste Operations and Emergency Response
HDPE	High Density Polyethylene
LDPE	Low Density Polyethylene
mL	Milliliter
OSHA	Occupational Health and Safety Administration
SAP	Sampling and Analysis Plan
SOP	Standard Operating Procedure
QAPP	Quality Assurance Project Plan
QA/QC	Quality Assurance/Quality Control
VCF	Ventrical at Centroid of Flow

Equal Discharge Increment (EDI): A surface water sampling strategy that requires isokintetic water sampling from locations in a moving body of water that have the same discharge rate.

Equal Width Increment (EWI): A surface water sampling strategy that requires isokinetic water sampling from established equidistant intervals in a moving body of water.

Global Positioning System (GPS): A geospatial referencing tool that is used for mapping and identification.

Sampling and Analysis Plan (SAP): A site-specific document that details 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).

Quality Assurance Project Plan (QAPP): A site-specific document that specifies quality assurance activities and data quality objectives.

Ventrical at Centroid of Flow (VCF): A sampling strategy similar to EDI sampling where only one location along a transect is used to collected the isokinetic water sample.

5.0 HEALTH AND SAFETY

When working with potentially hazardous materials or in hazardous situations, personnel must understand and comply with the site-specific SAP/QAPP and HASP before the sampling event begins. More specifically, when sampling streams or surface impoundments containing known or suspected hazardous substances, adequate personal protective equipment such as nitrile gloves, safety glasses, and waders are necessary to prevent contact with contaminants during sampling. When entering a stream, hazardous situations may exist requiring the use of adequate personal safety equipment including personal floatation devices and non-slip footwear. When conducting sampling from a boat in an impoundment or flowing waters, appropriate boating safety procedures should be followed.

6.0 CAUTIONS

Only collect surface water samples if it can be done so safely. Many unsafe conditions exist on streams, rivers, ponds, and other surface water impoundments. Consult the site HASP before performing any sample collection.

7.0 INTERFERENCES

There are three primary interferences or potential problems with surface water sampling. These include cross-contamination of samples, improper sample collection, and improper sample preservation.

1. 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. Refer to the Sampling Equipment Decontamination SOP #FLD-02.00.

2. Improper sample collection can involve disturbance of the stream substrate and/or sampling in an obviously disturbed area. To minimize potential for unrepresentative samples, consider sampling from downstream to upstream to avoid sampling where the substrate has been disturbed. Site sampling often involves multiple sampling procedures being deployed simultaneously. Therefore, well-organized team member coordination is essential to prevent improper sample collection.

3. Sample preservation is a critical component of sample collection. If the wrong preservative is used, the sample must be re-collected if possible. Improperly preserved samples cannot be analyzed. Refer to the Sample Preservation SOP #FLD-03.00 for proper field sample preservation guidelines.

8.0 PERSONNEL QUALIFICATIONS

Any personnel who are involved with field sampling activities must be cleared for health and safety. Clearance includes medical monitoring, respirator fit testing, and Occupational Health and Safety Administration (OSHA) Hazardous Waste Operations and Emergency Response (HAZWOPER) 40-hour training. Personnel who will be collecting surface water samples should familiarize themselves with this and other pertinent SOPs including: Sample Equipment Decontamination SOP #FLD-02.00; Sample Preservation SOP #FLD-03.00; Water Quality Measurements with the In-Situ® Multi-Parameter Meter SOP #FLD-09.00; Sample Custody and Labeling SOP #FLD-11.00; and General Field Sampling Protocols SOP #FLD-12.00.

9.0 EQUIPMENT AND SUPPLIES

Equipment needed for collection of surface water samples may include:

HASP Required Gear - personal floatation device, waders/gloves, proper footwear, safety glasses, insulating clothing for cold water, etc.

Mapping & Location Tools - GPS units, site/local area maps, compass, tape measure, survey stakes, pin flags, camera, 2-way radios

Documentation Supplies - field log book, field data sheet, Chains of Custody (COCs), labels, clear tape, pens, permanent marker, waterproof paper

Sampling Tools - plastic or other appropriate composition transfer device, bucket, rinse bottles, purified water, paper towels, filtering equipment, vacuum pump tool, vacuum pump stand, preservative, Ziploc™ plastic bags, cooler(s), ice, thermometer, preservative waste containment

Sample Containers - High-density polyethylene/Low Density Polyethylene (HDPE/LDPE) or other appropriate composition containers.

See Table 9.0-1 for a detailed list of surface water sampling equipment.

10.0 STANDARDS AND REAGENTS

Reagents will be utilized for preservation of samples and for decontamination of sampling equipment (refer to SOP's #FLD-02.00 and #FLD-03.00). The preservatives required are specified by the analysis to be performed and will be specified in the SAP/QAPP but usually include nitric acid (Total Recoverable and Dissolved Metals samples) and phosphoric acid (Dissolved and Total Organic Carbon samples). Field sampling personnel should also be aware of any special sampling considerations, contamination issues, and sample compositing and mixing methods that could affect the sampling efforts. Appropriate regional guidance and procedures should be consulted for detailed sample collection, preservation, handling and storing, equipment decontamination, and Quality Assurance/Quality Control (QA/QC) procedures. Field sampling personnel should preserve and immediately cool all water samples to 4 degrees Celcius (°C) (±2°C) upon collection and samples should remain <6°C until the time of analysis (do not freeze water samples).

11.0 PROCEDURES

11.1 Preparation

1. Determine the extent of the sampling effort, the sampling methods required, and the types and amounts of equipment and supplies needed. Use the site-specific SAP/QAPP for guidance to determine which kind of samples need to be collected.

2. Obtain the necessary sampling and monitoring equipment and ensure it is in working order.

3. Decontaminate equipment according to the procedures outlined in SOP #FLD-02.00, or use triple rinsed, dedicated disposable sample containers (non-filtering and not pre-

preserved).

4. Prepare scheduling and coordinate with staff, clients, and regulatory agencies where appropriate. It is also important to obtain site 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, GPS markers, or photos to identify and mark all sampling locations. If required, the proposed locations may be adjusted based on site access, property boundaries, and surface obstructions.

Generally, factors to consider in the selection of a device for sampling liquids in streams, rivers, lakes, ponds, lagoons, and surface impoundments are:

a. Can the sample be collected directly from the source? (i.e. is a transfer device needed for sample collection?)

b. What is the desired depth at which you wish to collect the sample?

c. What is the overall depth and flow direction of river or stream?

d. What type of analysis will be run (total recoverable metals, dissolved metals, alkalinity and anions, etc.)?

11.2 Sample Container Composition

A sample container should be selected based on analysis to be performed. A triple rinse should be performed on any sample container being used for direct sample collection. Filtered sample bottles or pre-preserved bottles should never be rinsed first.

11.3 Sample Collection

1. Direct collection is the optimal procedure for sample collection. After rinsing, the sample should be collected in a well-mixed area, as close to the middle of the stream as possible, in-between the streambed and the surface. The sample should be capped immediately.

2. Preserve the sample if appropriate. Refer to the site-specific SAP and SOP #FLD-03.00 for correct methods. The sample should be clearly labeled (and bagged in order to keep samples from the same location together) before being placed in a cooler on ice.

3. Record all pertinent site data (usually date, time, pH, conductance, dissolved oxygen, temperature, site ID, and anomalies) in the field logbook, field data sheets and/or sample container labels.

4. Complete the COC record. Refer to SOP #FLD-11.00 for guidelines on sample

custody and labeling documentation.

5. Attach custody seals to cooler prior to shipment where applicable. Refer to SOP #FLD-11.00 for guidelines on sample custody and labeling.

6. If a non-direct method was used for sampling (i.e. a bucket from a bridge or a dip sampler), decontaminate or condition all sampling equipment prior to the collection of additional samples with that sampling device as required by SOP #FLD 02.00. Sections 11.3.4 and 11.3.5 describe a few of the non-direct sampling methods that may be useful in Region 8.

7. If sampling on private property, sample receipts will be provided to property owners for all samples taken and removed from the property.

11.3.1 Direct Method

For streams, rivers, and lakes, the direct method may be utilized to collect water samples from the surface directly into the sample container. For shallow stream stations, collect the sample under the water surface while pointing the sample container upstream; the container must be upstream of the collector. Samples should be collected prior to all other activities as specified in the SAP/QAPP to avoid disturbing the substrate. Rinse the sample container three times (unless it's filtered or pre-preserved) with site water before procuring a sample. When using the direct method, do not use pre-preserved sample bottles as the collection method may dilute the concentration of preservative necessary for proper sample preservation.

11.3.2 Dip Sampling

Dip sampling is useful in situations where a sample is to be recovered from an outfall pipe or from a bridge where direct access is limited. The long handle or rope on such a device allows access from a discrete location. Sampling procedures are as follows:

1. Assemble the device in accordance with the manufacturer's instructions.

2. Extend the device to the sample location and collect the sample by dipping the sampler into the water at the sampling location.

3. Triple rinse the sampler with the site water

4. Retrieve the sampler and transfer the sample to the appropriate (triple rinsed) sample container

11.3.3 Synoptic Sampling

Synoptic sampling is a strategy used for evaluating a surge of water as it moves downstream. In general, the flow rate should be determined before executing a synoptic sampling event. Flow measurements are used to calculate when a surge of water will pass by a certain point or sample location. Floating visual objects may also be used to accurately determine when a surge of water passes by a sampling location. The synoptic sampling strategy is useful in determining where contaminants may be entering a watershed through seeps, fens, or other inflows that may not be visible. Below is a general guideline on synoptic sampling:

- Identify sample locations
- Conduct flow measurements; or test a floating object (ping pong ball, tangerine, etc.) to see if it will float through to the last sampling location (some objects may get caught in eddies or shallow areas)
- Calculate when the surge of water will pass through each sampling location
- Position sampling personnel or equipment where a sample can be captured at the required time
- Process samples

11.3.4 Large Stream or River Sampling

There are several techniques for sampling large rivers or streams. The most commonly used are Equal-Width Increment (EWI) sampling, Equal Discharge Increment (EDI) sampling, Single Vertical at Centroid of Flow sampling (VCF), dip sampling (section 11.3.2), direct method sampling (section 11.3.1), discrete sampling, and pump sampling (USGS, 2006). Below is a brief description of the sampling methods that have not yet been mentioned in this document:

<u>EWI Method</u> – A stream cross-section is divided into equal width intervals, and samples are collected by lowering and raising a sampler through the water column at the center of each interval. This produces a discharge weighted sample that is proportional to stream flow. This method cannot be used if the stream flow is less than what is required to fill the sampler during the isokintetic (constant rate) motion.

<u>EDI Method</u> – The objective of this method is to obtain a discharge weighted sample that represents the entire flow through a cross-section by obtaining a series of samples. For this method, the flow in the cross-section must be divided into points of equal discharge. Equal volume and depth integrated samples are collected at the center of the equal discharge interval along the cross-section. Flow measurements or historical data is necessary to determine interval number (usually more than 4, but less than 20) and location. This method may also require additional personnel to sample a large cross-section. If conducted properly, both the EWI and EDI methods should produce identical results.

<u>VCF Method</u> – This method is a simple version of the EDI method, but only one sample is collected at flow center (usually a smaller river or stream). This is to be used if the sample location is known to be homogenous and is warranted by the sampling plan objectives. Flow data should be obtained to determine where the flow center is located.

11.3.5 Shallow Stream and Still Water Sampling

Shallow streams and still water such as a pond are common locations to sample in Region 8. Below are three methods (in addition to ones mentioned above) that may be required.

<u>Discrete (point) Sampling</u> – This method is achieved by lowering a sample container to a specific depth in a body of water then opening and closing the container to obtain the sample.
<u>Pump Sampling</u> – This method is used to collect single point samples using a suction-lift or submersible pump. These are not used for collecting isokinetic samples (EWI, EDI, or VCF). Using the pump method is limited by electrical needs in remote areas. Always consult the SAP for sampling objective requirements.

<u>Syringe Sampling</u> – A 50 mL syringe can be used to collect sample from very shallow locations to avoid contact with substrate.

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

13.0 QUALITY CONTROL AND ASSURANCE

1. The following general QA procedures apply:

2. All data must be documented on field data sheets or within site logbooks.

3. In general, concurrent (duplicate) sample collection at a frequency of 10% is required for most sampling activities. Blanks at a frequency of one per day are also generally required. Consult the corresponding SAP/QAPP for specific QA/QC sampling frequency. Below is a list of typical QA/QC sample types and the inaccuracy they are intended to detect:

    - <u>Field blank</u> – checks cross-contamination during sample collection, preservation, and shipment as well as in the laboratory.
    - <u>Equipment blank</u> – equipment contamination due to inadequate decontamination procedures
    - <u>Temperature blank</u> – provides an accurate temperature measurement of field samples upon arrival to the laboratory and establishes whether the temperature range has been maintained while in transit
    - <u>Trip blank</u> – checks contamination of samples during handling, storage, and shipment from the field to the laboratory; carried through the same sampling and handling

protocols as field samples and placed in the cooler for the duration of the trip.

- MS/MSD – checks accuracy and precision of organic or inorganic analyses in specific sample matrices. They are collected from areas know or suspected to be contaminated.
- Sequential replicates – samples are pulled one after another to detect variability among field activities (collection, preservation, handling, etc.)
- Split samples – division of one sample into two, then submitting for identical analysis in order to detect variability in the process from collection to analysis
- Concurrent or collocated replicate samples (often referred to as “duplicate” samples) – two samples collected at the same location at the same time, intended to detect variability inherent in collection, processing, and handling procedures; Relative percent difference is usually calculated from these samples.

4. All instrumentation must be operated in accordance with operating instructions as supplied by the manufacturer unless otherwise specified in the work plan or SAP/QAPP. Equipment calibration activities must be conducted and documented prior to sampling and/or operation of equipment.
5. Document any deviations from SOPs, work plan, SAP/QAPP, etc.

14.0 REFERENCES

Environmental Protection Agency (EPA) Guidance for Preparing Standard Operating Procedures, EPA QA/G-6, April 2007.

U.S. Geological Survey. 1977. National Handbook of Recommended Methods for Water Data Acquisition. Office of Water Data Coordination, Reston, Virginia. (chapter updates available)

U.S. Geological Survey 2006. National Field Manual for the Collection of Water Quality Data. Chapter A4, version 2.0, Office of Water Quality.

Environmental Protection Agency (EPA), 1984. Characterization of Hazardous Waste Sites - A Methods Manual: Volume II. Available Sampling Methods, Second Edition. EPA/600/4-84-076.

Environmental Protection Agency (EPA), Environmental Response Team, Standard Operating Procedures - Surface Water Sampling: U.S. Environmental Protection Agency, 1994, SOP #2013.

Table 9.0-1 Surface Water Sampling Equipment

Category	**Item**	**Use**	**Comment**
Health and Safety	Gloves	Protection from absorption of contaminants	Nitrile or neoprene are recommended
Health and Safety	Waders	Slip/contaminant protection, warmth	Any type are acceptable
Health and Safety	Safety Glasses	Eye protection	Sunglasses for UV protection
Health and Safety	Layered Clothing	Protection from hypothermia	Polyester base layers only
Mapping/Location	GPS unit	Sample station locating	Pre-loaded with site locations
Mapping/Location	Maps	Location identification	Most current information required
Mapping/Location	Two-way radios	Communication	Extra batteries or charger required
Documentation	Field Logbook	Site data and conditions documentation	Waterproof pages
Documentation	Field Data Sheets (waterproof)	Marsh McBirney Flows	
Documentation	Chain of Custody	Sample handling/identification	Generated using Scribe
Documentation	Labels	Sample identification	Generated using Scribe
Documentation	Clear tape & Scissors	Label protection	
Sampling Tools	Bucket/Transfer Device	Sample transfer (if required)	Decontaminated between samples
Sampling Tools	Vacuum Pump	Dissolved sample processing	
Sampling Tools	Vacuum Stand	Dissolved sample processing	
Sampling Tools	Ziploc baggies	Sample containment	
Sampling Tools	Water Chemistry meters	In-situ water quality data gathering	Temp, pH, dissolved oxygen, conductivity
Sampling Tools	Flow measurement equipment	Flow measurements	
Sampling Tools	Cooler	Sample containment	Samples to be kept at 4°C
Sampling Containers	250 ml HDPE/LDPE	Total recoverable metals samples	
Sampling Containers	250 ml HDPE/DLPE filtered bottles	Dissolved Metals/ DOC samples	Single use only
Sampling Containers	500 ml HDPE/LDPE	Alkalinity+Anions samples	
Sampling Containers	VOA Vials	Volatile and Semi-Volatile organics analysis	
Reagents	Nitric Acid(HNO3) ampules	For preserving metals samples	
Reagents	Phosphoric Acid (H3PO4) ampules	For preserving DOC samples	
Reagents	Hydrochloric (HCl) Acid	For preserving VOA samples	
Reagents	CaCO3 Acid waste Containment	Ampule/acid waste disposal	Usually in a 1 liter cubitainer
Reagents	pH and Conductivity standards	Calibration of water quality equipment	Quantity for each day of sampling

Sampling Equipment Decontamination

APPROVED:

ESAT Region 8 QA Coordinator

06/06/12
Date

ESAT Region 8 Team Manager

6/6/12
Date

EPA Task Order Project Officer

7/10/12
Date

ESAT Region 8 Task Lead

6/8/12
Date

DCN: EP8-7-7061

This document has been prepared for the Environmental Protection Agency by the TechLaw, Inc. ESAT Region 8 Team and is intended to provide documentation of administrative, analytical and quality control procedures used in the daily performance of EPA and ESAT support services.

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

<u>Code of Federal Regulations (CFR)</u>: The codification of the general and permanent rules published in the Federal Register by the executive departments and agencies of the Federal Government

<u>Contamination Reduction Corridor (CRC)</u>: 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.

<u>Contamination Reduction Zone (CRZ)</u>: An area between the exclusion zone and contamination reduction corridor where preliminary decontamination activities occur.

<u>Department of Transportation (DOT)</u>: A government agency that oversees and regulates transportation functions.

<u>Exclusion Zone (EZ)</u>: The area at a site where work is being performed.

<u>Health and Safety Plan (HASP)</u>: A site-specific document that identifies safety hazards and proper safety procedures. This normally includes hospital route maps and material safety data sheets.

<u>Occupational Safety and Health Administration (OSHA)</u>: 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:

Mechanical

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.

Air Blasting

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.

<u>Station 4: Low-Pressure Rinse</u>
Rinse sampling equipment with distilled/deionized water with a low-pressure sprayer.

<u>Station 5: Nitric Acid Sprayers (required only if metals are a contaminant of concern)</u>
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.

<u>Station 6: Organic Solvent Sprayers</u>
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

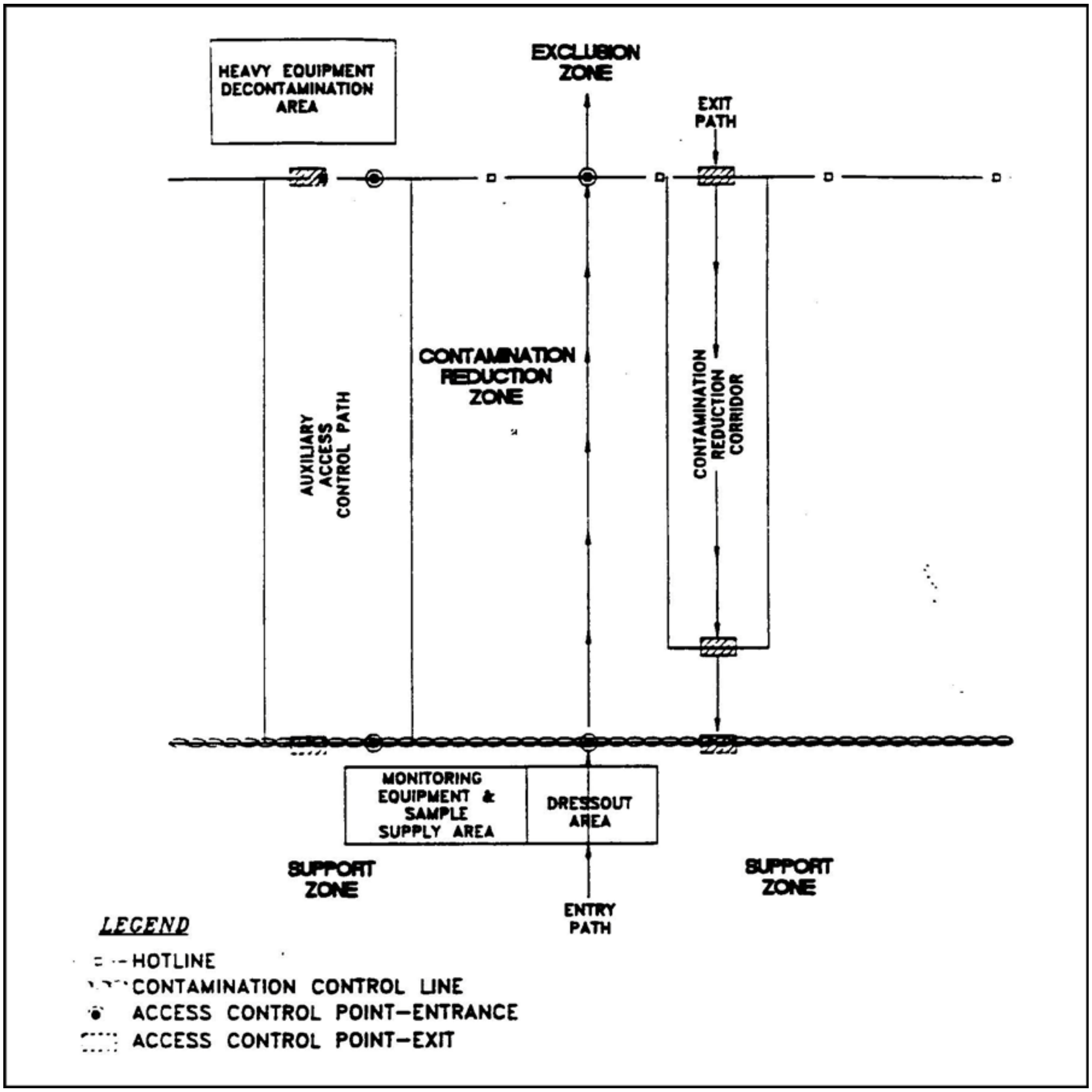
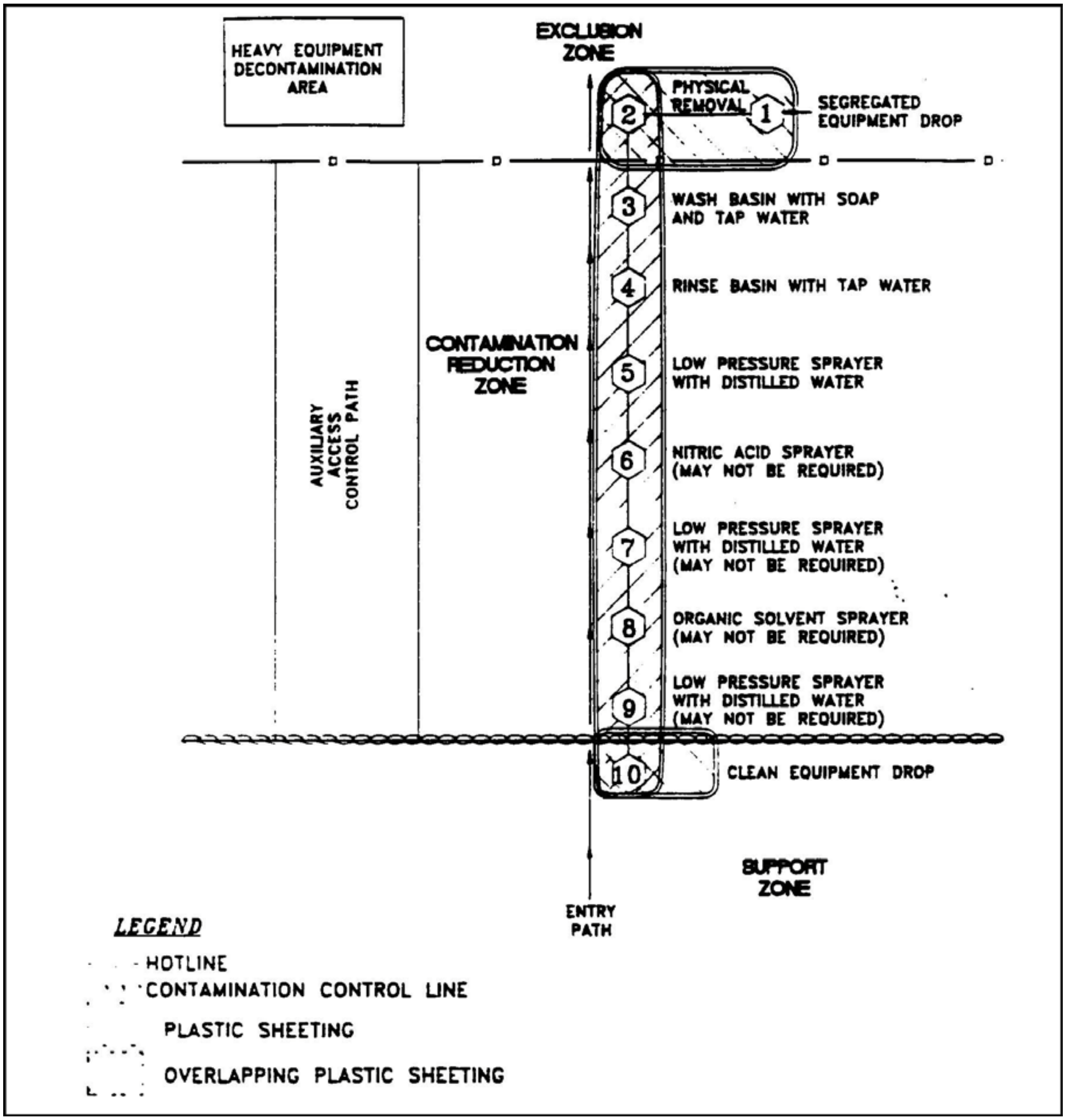


Figure 2 - Contamination Reduction Zone Layout (2)



Sample Preservation

APPROVED:

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04/04/12
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7/10/12
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This document has been prepared for the Environmental Protection Agency by the TechLaw, Inc. ESAT Region 8 Team and is intended to provide documentation of administrative, analytical and quality control procedures used in the daily performance of EPA and ESAT support services.

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:

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This document has been prepared for the Environmental Protection Agency by the TechLaw, Inc. ESAT Region 8 Team and is intended to provide documentation of administrative, analytical and quality control procedures used in the daily performance of EPA and ESAT support services.

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

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Shallow Stream Sediment Sampling

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TABLE OF CONTENTS

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide field personnel a set of guidelines for the proper collection of stream sediment samples.

2.0 SCOPE AND APPLICABILITY

This SOP is applicable to the collection of shallow stream sediment samples. Analysis of sediment may be biological, chemical, or physical in nature and may be used to determine the following:

- Toxicity
- Biological availability and effects of contaminants
- Benthic biota
- Extent and magnitude of contamination
- Contaminant migration pathways and source
- Fate of contaminants
- Grain size distribution

The methodologies discussed in this SOP are applicable to the sampling of sediment in lotic environments. They are generic in nature and may be modified in whole or part to meet the handling and analytical requirements of the contaminants of concern, as well as the constraints presented by site conditions, equipment limitations and requirements of the site-specific Sampling and Analysis Plan (SAP). However, if modifications occur, they should be documented in field data sheet/field notebook and discussed in reports summarizing field activities and analytical results. For the purposes of this procedure, sediments are those mineral and organic materials situated beneath an aqueous layer in rivers and streams. Mention of trade names or commercial products for use in sediment sample collection does not constitute endorsement or recommendation for use.

3.0 SUMMARY OF METHOD

Sediment samples may be collected using a variety of methods and equipment, depending on the depth of the aqueous layer, the portion of the sediment profile required (surface vs. subsurface), the type of sample required (disturbed vs. undisturbed), contaminants present, and sediment type. Sediment is collected from beneath an aqueous layer directly, using a hand held device such as a shovel, trowel, or plastic scoop. Following collection, sediment is transferred from the sampling device to an appropriate sample container. If composite sampling techniques are employed, multiple grabs are placed into a container constructed of inert material, homogenized, and transferred to sample containers appropriate for the analyses requested.

The homogenization procedure should not be used if sample analysis includes volatile organics. In this case, if sediment is to be analyzed for volatile organics then the sample must be transferred to the appropriate sample container directly after collection. The sample bottle is filled completely and tapped lightly to get the trapped air out of the bottle. If the sediment settles in the bottle creating airspace then additional sediment should be collected. Repeat this step as many times necessary in order to have the sample bottle completely filled without having any air gaps.

4.0 ACRONYMS AND DEFINITIONS

COC	Chain of Custody
GPS	Global Positioning Systems
EPA	United States Environmental Protection Agency
ESAT	Environmental Services Assistance Team
HASP	Health and Safety Plan
HAZWOPER	Hazardous Waste Operations and Emergency Response
HDPE	High-Density Polyethylene
OSHA	Occupational Health and Safety
QA	Quality Assurance
SAP/QAPP	Sampling and Analysis Plan/Quality Assurance Project Plan
SOP	Standard Operating Procedure

Chain of Custody (COC): A chronological document that tracks movement 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.

Global Positioning System (GPS): A geospatial referencing tool that is used for mapping and identification

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.

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).

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 United States Environmental Protection Agency (EPA), Occupational Safety and Health Agency (OSHA), and corporate health and safety procedures. More specifically, when sampling sediment from water bodies, physical hazards must be identified and adequate precautions must be taken to ensure the safety of the sampling team. The team member collecting the sample should not get too close to the edge of the water body, where bank failure may cause loss of balance. To prevent this, the person performing the sampling should be on a lifeline, and be wearing adequate protective equipment. If sampling from a vessel, appropriate protective measures and procedures must be implemented.

6.0 CAUTIONS

Only collect sediment samples if it can be done so safely. Many unsafe conditions exist on streams and rivers. Also, review the SAP/QAPP or any other planning documents for analytical requirements and equipment selection. Consult the site HASP before performing any sample collection.

7.0 INTERFERENCES

Substrate particle size and organic matter content are a direct consequence of the flow characteristics of a water body. Contaminants are more likely to be concentrated in sediments typified by fine particle size and high organic matter content. This type of sediment is most likely to be collected from depositional zones. In contrast, coarse sediments with low organic matter content do not typically concentrate pollutants and are generally found in erosion zones.

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 Occupational Safety and Health Administration (OSHA) Hazardous Waste Operations and Emergency Response (HAZWOPER) 40-hour training. In addition to this, any personnel who will participate in sediment sampling activities must read, understand, and sign the site specific HASP and the associated SAP/QAPP. Additionally, field personnel would benefit from understanding relevant SOPs including Sampling Equipment Decontamination SOP FLD 02.00, Sample and Labeling SOP FLD 11.00, and the General Field Sampling Protocols SOP FLD 12.00.

9.0 EQUIPMENT AND SUPPLIES

Equipment needed for collection of sediment samples may include:

> Health and Safety Plan (HASP) - Personal floatation device, life line, neoprene waders/gloves, proper footwear, safety glasses, insulating clothing for cold water, etc.
>
> Mapping & Location Tools - GPS units, site/local area maps, tape measure, compass, survey stakes, pin flags, camera, and 2-way radios.
>
> Documentation - Field log book, field data sheet, chain of custody (COC), labels & clear tape, pens/sharpie, waterproof paper.
>
> Sampling Tools - Plastic or other appropriate composition scoop, shovel, spade, trowel, homogenization container with mixing tool, rinse bottle, purified water, and paper towels.
>
> Sample Containers -High-density polyethylene (HDPE) or other appropriate composition containers (50 mL and 1 liter [bulk] are frequently used), amber glass jars (organics analysis) labels, clear tape, pens, permanent marker, sealable plastic bags, cooler(s), and ice.

See Table 9.0-1 for a detailed list of sediment sampling equipment.

10.0 STANDARDS AND REAGENTS

Reagents are not used for preservation of sediment samples.

11.0 PROCEDURES

11.1 Sample Preservation, Containers, Handling and Storage

Chemical preservation of solids is not recommended. Cooling to 4 degrees Celsius (ºC) is recommended for sediment samples. HDPE containers with Teflon^{TM} lined caps are typically used for sediment samples. Sample container size is typically 50 milliliter (mL) for metal analysis and 1 liter for sediment toxicity testing. However, the sample volume is a function of the analytical requirements and will be specified in the SAP/QAPP. If analysis of sediment from a discrete depth or location is desired, sediment is transferred directly from the sampling device to a labeled sample container(s) of appropriate size and construction for the analyses requested. Transfer is accomplished with a decontaminated stainless steel or plastic lab spoon or equivalent.

If composite sampling techniques or multiple grabs are employed, equal portions of sediment from each location are deposited into a stainless steel, plastic, or other appropriate composition containers. The sediment is then homogenized thoroughly, to obtain a composite sample that is representative of the area and is then transferred to a labeled container. Transfer of sediment is accomplished with a stainless steel or plastic lab scoop or equivalent. Samples for volatile organic analysis must be transferred directly from the sample collection. It is important that when collecting sediment for volatile organic compounds analysis, the sample container is filled completely full and the sample container is tapped lightly to ensure all air is purged from the sample. This is done to minimize loss of contaminant due to volatilization.

All sampling devices should be decontaminated following procedures described in the Sample Equipment Decontamination SOP FLD 02.00. The sampling device should remain in its wrapping until it is needed. Each sampling device should be used for only one sample. Although disposable sampling devices for sediment are generally impractical due to cost and the large number of sediment samples which may be required, such devices may prove efficient and effective for difficult terrain/remote locations. Sampling devices should be cleaned in the field using the decontamination procedure described in the Sampling Equipment Decontamination SOP FLD 02.00.

11.2 Preparation

Determine the objective(s) and extent of the sampling effort. Obtain access to private property if sample locations are located within private boundaries. The sampling methods to be employed, and the types and amounts of equipment and supplies required will be a function of site characteristics and objectives specified in the SAP and QAPP.

- Obtain the necessary sampling and monitoring equipment.
- Prepare schedules, and coordinate with staff, client, and regulatory agencies where appropriate.
- Decontaminate or pre-clean equipment, and ensure that it is in working order.

- Perform a general site survey prior to site entry in accordance with the site-specific HASP.
- Use stakes, flagging, or buoys in addition to using a GPS (Refer to SOP FLD 07.00) to identify and mark all sampling locations. Specific site factors including flow regime, basin morphology, sediment characteristics, depth of overlying aqueous layer, contaminant source, and extent and nature of contamination should be considered when selecting sample locations. If required, the proposed locations may be adjusted based on site access, property boundaries, and surface obstructions.

11.3 Sample Collection

Selection of a sampling device is most often contingent upon the depth of water at the sampling location and the physical characteristics of the sediment to be sampled. The following procedure consists of sampling surface sediment with a scoop, trowel or shovel from beneath a shallow aqueous layer:

For the purpose of this method, surface sediment is considered to range from 0 to 1 inch in depth and a shallow aqueous layer is considered to range from 0 to 12 inches in depth. Collection of surface sediment from beneath a shallow aqueous layer can be accomplished with tools such as spades, shovels, trowels, and scoops. Although this method can be used to collect both unconsolidated/consolidated sediment, it is limited somewhat by the depth and movement of the aqueous layer. Deep and rapidly flowing water may render this method less accurate than other methods such as utilizing a handheld dredge or coring device. However, representative samples can be collected with this procedure in shallow sluggish water provided care is demonstrated by the sample team lead. A plastic scoop will suffice in most applications. Care should be exercised to avoid the use of devices plated with chrome or other materials.

The following procedure will be used to collect sediment with a scoop, shovel, or trowel:

1. Using a decontaminated sampling implement, collect the desired thickness and volume of sediment from the sampling area.

2. Transfer the sample into an appropriate sample or homogenization container. Ensure that non-dedicated containers have been adequately decontaminated.

3. Surface water should be decanted from the sample or homogenization container prior to sealing or transfer; care should be taken to retain the fine sediment fraction during this procedure.

11.3.1 Composite Sampling

Composite sampling consists of taking several sub-samples from a location and consolidating them into a larger sample. If data quality objectives dictate that each sub-sample of a composite be measured, it can be done two ways; by mass or by volume. For remote site field sampling activities (such as ones that typically occur in Region 8), it is recommended that sub-samples be measured by volume. This can be done with a graduated beaker/measuring cup or cylinder. Place the sub-sample in the measuring device, record the measurement, and transfer the sub-sample into a larger container where the complete composite sample will be processed.

Composite representative sample collection can also be accomplished without measurement of sub-samples. For sediment collection that does not require sub-sample measurement, larger amounts of sample can be collected in areas where sediment is more readily available. This method is used very frequently in high-gradient streams such as those found in the region.

11.3.2 Discrete Sampling

Discrete sampling consists of taking a sample from a single location. This method requires that the selected location for a sediment sample have sufficient amount of material for the analytical requirements. In general, sediment samples in a stream are difficult to obtain from a single location; therefore composite samples are more commonly collected.

12.0 DATA RECORDS AND MANAGEMENT

Once collected, samples are labeled and stored for transport (at 4°C). 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

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 SAP and QAPP. Equipment inspection must occur prior to sampling, and they must be documented.

3. QA samples should be sampled at a standardized frequency. Field duplicates are generally sampled at a rate of 1:20.

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 EPA600/4-84-076.

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.

U.S. EPA, Environmental Response Team, standard operating procedures—Sediment Sampling: U.S. Environmental Protection Agency, 1994, SOP #2016.

Table 9.0-1: Sediment Sampling Equipment

Category	**Item**	**Use**	**Comment**
Health and Safety	Gloves	Protection from absorption of contaminants	Nitrile or neoprene are recommended
Health and Safety	Waders	Slip/contaminant protection	Any type are acceptable
Health and Safety	Safety Glasses	Eye protection	Sunglasses for UV protection
Health and Safety	Layered Clothing	Protection from hypothermia	Polyester base layers only
Mapping/Location	GPS unit	Sample station locating	Pre-loaded with site locations
Mapping/Location	Maps	Location identification	Must contain most current information
Mapping/Location	Two-way radios	Communication	Extra batteries or charger required
Documentation	Field Logbook	Site data and conditions documentation	Waterproof pages
Documentation	Chain of Custody	Sample handling/identification	Pre-printed using Scribe
Documentation	Labels	Sample identification	Pre-printed using Scribe
Documentation	Clear tape & Scissors	Label protection	
Sampling Tools	Bucket/Transfer Device	Sample transfer (if required)	Can also be used for sample homogenization
Sampling Tools	Sediment Scoop	Sediment sampling	Select based on analysis
Sampling Tools	Cooler	Sample containment	Cool to 4°C
Sampling Containers	Amber glass jars	Volatile Organics Analysis sample containment	Tight cap seal
Sampling Containers	50 ml HDPE (widemouth)	Metals analysis sample containment	
Reagents	10% Nitric Acid (HNO3) solution	For decontamination of metals sampling equipment	Pre-mixed at lab
Reagents	10% Hydrochloric Acid (HCl) solution	For decontamination of organics sampling equipment	Pre-mixed at lab

Global Positioning System (GPS) - Trimble GeoXT 2008 Series

APPROVED:

ESAT Region 8 QA Coordinator — 07/16/13
Date

ESAT Region 8 Team Manager — 7/16/13
Date

EPA Task Order Project Officer — 7/16/13
Date

ESAT Region 8 Task Lead — 7/16/13
Date

DCN: EP8-7-7061

This document has been prepared for the Environmental Protection Agency by the TechLaw, Inc. ESAT Region 8 Team and is intended to provide documentation of administrative, analytical and quality control procedures used in the daily performance of EPA and ESAT support services.

TABLE OF CONTENTS

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide a standard approach for United States Environmental Protection Agency (EPA) and Environmental Services Assistance Team (ESAT) Region 8 personnel to use a Global Positioning System (GPS) during field activities.

2.0 SCOPE AND APPLICATION

This SOP is specifically intended for application by EPA and ESAT personnel who conduct field work using a GPS.

3.0 SUMMARY OF METHOD

This SOP covers the use of TerraSync on Trimble GeoXT 2008 Series GPS machines as well as the Laser RangeFinder and Pathfinder Office. This SOP is based on manufacturers' instructions.

4.0 ACRONYMS AND DEFINITIONS

EPA	United States Environmental Protection Agency
ESAT	Environmental Services Assistance Team
GIS	Geographic Information System
GPS	Global Positioning System
HASP	Health and Safety Plan
SOP	Standard Operating Procedure

Geographic Information System (GIS): A system of hardware and software used for storage, retrieval, mapping, and analysis of geographic data.

Global Positioning System (GPS): A navigational system involving satellites and computers that can determine the latitude and longitude of a receiver on Earth by computing the time difference for signals from different satellites to reach the receiver.

Terrasync: Software used in GPS units that captures and stores spatial data collected in the field.

Trimble: A private GPS Company that provides equipment and technical support, including the 2008 GeoXT GPS units and TerraSync software.

5.0 HEALTH AND SAFETY

There are no health and safety issues requiring mention in this SOP; however, refer the applicable site-specific Health and Safety Plan (HASP) any time field work is conducted.

6.0 EQUIPMENT

Trimble GeoXT 2008 Series GPS unit with TerraSync
LTI TruPulse 360 Laser RangeFinder
TruPulse 200B/36B Cheat Notes card
Tape measure

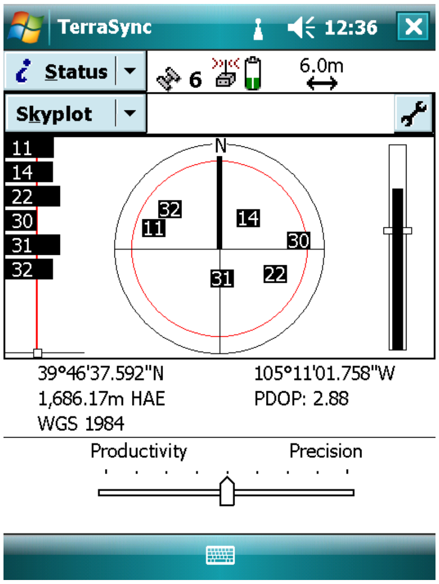
Flags
Computer and/or external hard drive

7.0 TERRASYNC

7.1 Starting TerraSync

To begin, press the green button on the Trimble unit. Tap **Start**, then **TerraSync** to open the GPS application (left). If not available, tap **Programs**, then select **TerraSync**.

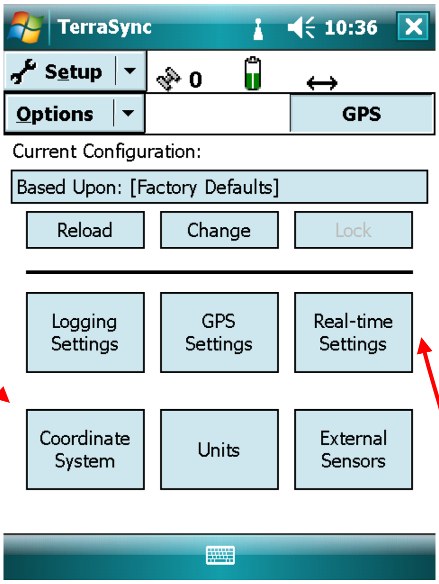
TerraSync will open with the **Status-Skyplot** page and begin to acquire satellite information. A minimum of four satellite locks are necessary to obtain a position. More satellites and proper satellite geometry increase accuracy.



39°46'37.592"N
1,686.17m HAE
WGS 1984
105°11'01.758"W
PDOP: 2.88

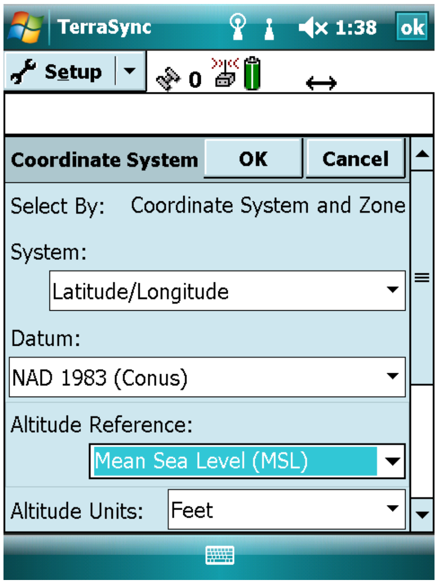
7.2 TerraSync Setup Menu

As TerraSync begins to acquire satellites, make sure the unit is properly configured. Tap the upper drop down menu and choose **Setup**. From the **Setup** page, tap **Coordinate System** and ensure that your screen matches the figure below. If a different coordinate system is required, use the dialog shown below to make necessary changes. Keep defaults if unsure. Select **OK** to return to setup menu.



Current Configuration:

Based Upon: [Factory Defaults]



Select By: Coordinate System and Zone

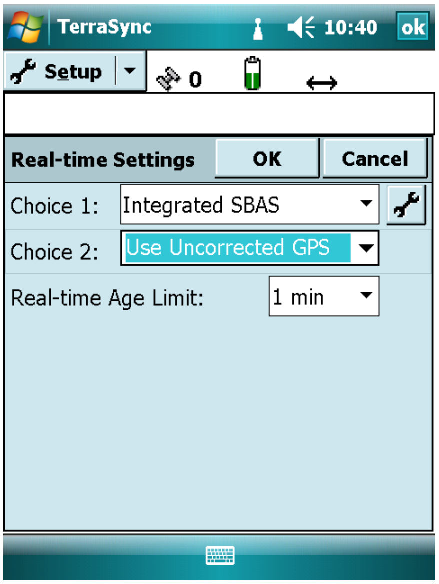
System: Latitude/Longitude

Datum: NAD 1983 (Conus)

Altitude Reference: Mean Sea Level (MSL)

Altitude Units: Feet

Next, choose **Real-Time Settings.** These settings will allow you to receive real time differentially corrected readings from satellite-based radio transmitters, if available. If not available, you will be using uncorrected GPS signals that can be corrected later by ESAT Region 8 GIS personnel. Please ensure that your screen matches the figure on the right. Select **OK** to return to setup menu.

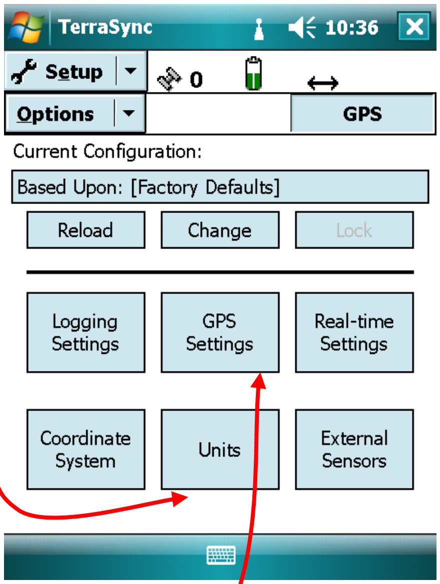


Choice 1: Integrated SBAS

Choice 2: Use Uncorrected GPS

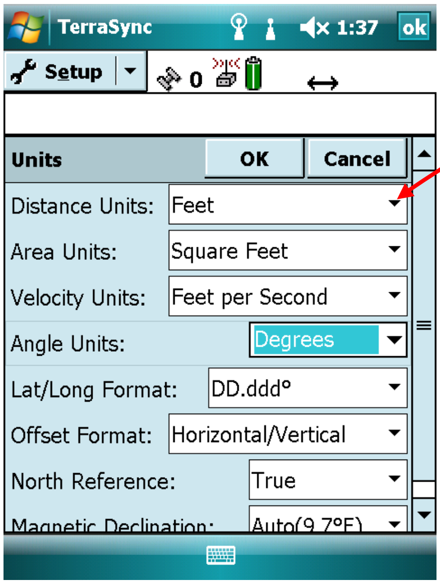
Real-time Age Limit: 1 min

The **Units** menu allows you to choose which units will be used for display in the **Map** section (feet). Make any necessary changes. Select **OK** to return to setup menu.



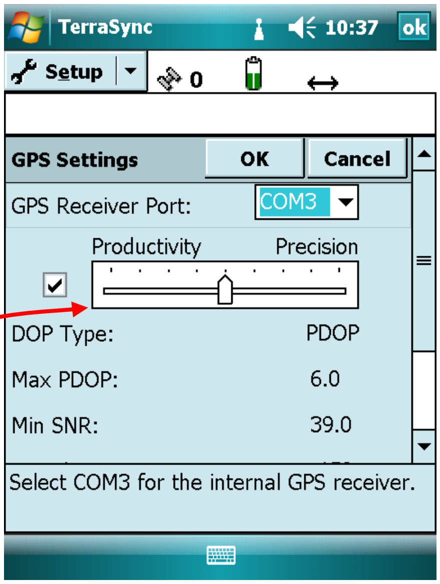
Current Configuration:

Based Upon: [Factory Defaults]



Distance Units: Feet
Area Units: Square Feet
Velocity Units: Feet per Second
Angle Units: Degrees
Lat/Long Format: DD.ddd°
Offset Format: Horizontal/Vertical
North Reference: True
Magnetic Declination: Auto(9.7°E)

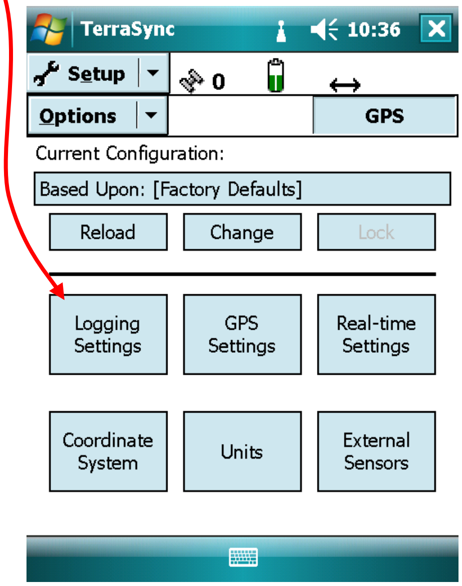
Under the **GPS Settings** menu, you can control the level of accuracy that your unit measures by adjusting the slider bar. Keep the defaults unless you are sure that you are willing to sacrifice precision for productivity or vice versa. Make sure that the **Productivity/Precision Bar** is set in the middle to give you a PDOP of 6.0, a minimum SNR of 39, and a minimum elevation of 15 degrees. Keep the settings for Velocity Filter and NMEA Output set to "Off." Select **OK** to return to setup menu.



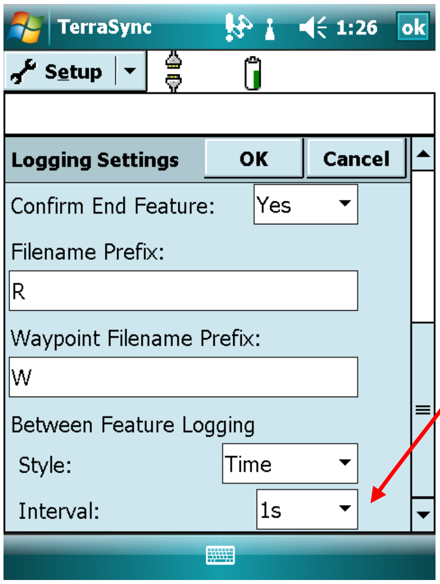
GPS Receiver Port: COM3
☑
DOP Type: PDOP
Max PDOP: 6.0
Min SNR: 39.0

Select COM3 for the internal GPS receiver.

The **Logging Settings** menu allows you to adjust how you will log your data. In this dialog box, you will set your logging interval to either 1 or 5 seconds, depending on the data being collected (the default used by ESAT is 1 second; however, 5 seconds is better suited for high volume data collection, such as a large polygon or area). You will also set your antenna height to the height at which you will be collecting your data. Normally, this will be set at 1 meter (approximately 3.5 feet or the height an average person would hold the unit). If you wish to change Antenna Height, click the following icon located just right of the Antenna Height figure: 🔧

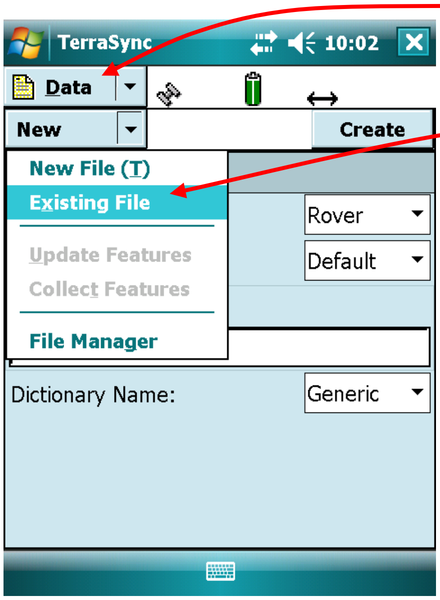


Current Configuration:
Based Upon: [Factory Defaults]



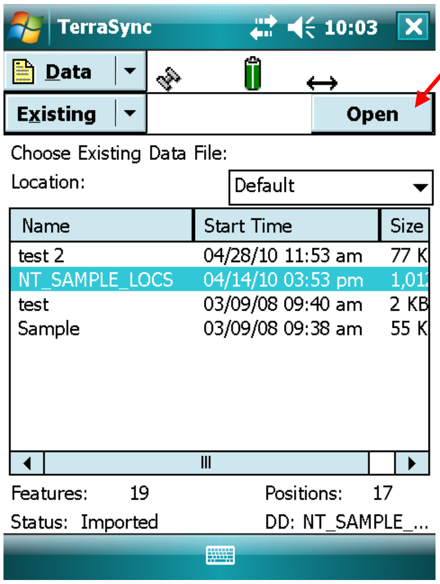
Confirm End Feature: Yes
Filename Prefix: R
Waypoint Filename Prefix: W
Between Feature Logging
Style: Time
Interval: 1s

7.3 TerraSync Data Menu



Dictionary Name: Generic

Select **Data** from the main drop down menu. The default will be to create a new data file. Click the drop down labeled **New** and choose **Existing File**. From this menu, select the appropriate file and click on the **Open** button. Then confirm antenna height.

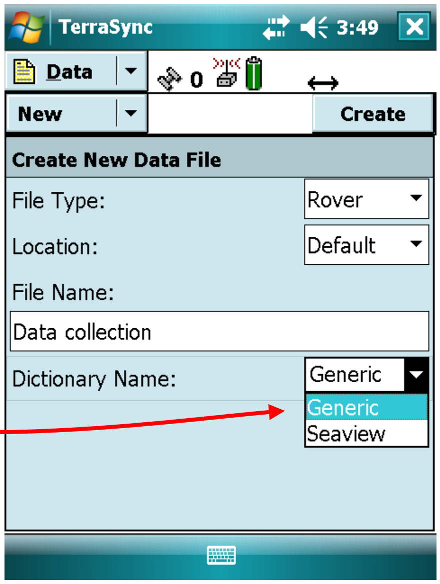


Choose Existing Data File:
Location: Default

Name	Start Time	Size
test 2	04/28/10 11:53 am	77 K
NT_SAMPLE_LOCS	04/14/10 03:53 pm	1,01
test	03/09/08 09:40 am	2 KB
Sample	03/09/08 09:38 am	55 K

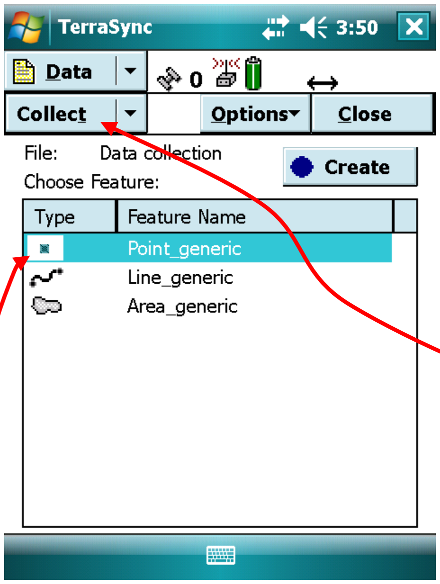
Features: 19 Positions: 17
Status: Imported DD: NT_SAMPLE_...

If you wish to create a new file or cannot find the appropriate existing file, click the **New** tab. Rename the new file to a project specific name. Select the proper data dictionary. Choose **Generic** if a data dictionary does not exist for your project. Tap **Create** and confirm antenna height at 1 meter (~3.5 feet) in subsequent pop-up.



Create New Data File
File Type: Rover
Location: Default
File Name:
Data collection
Dictionary Name: Generic

The features you are able to collect are now available in a list (below left). Click the **Options** tab and select **Log Later**. Use the **Options** tab to select a logging interval appropriate for the type of data you are collecting (below right).



File: Data collection

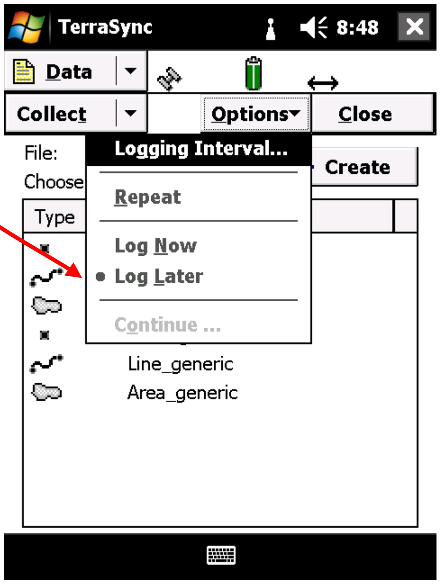
Choose Feature:

Point_generic

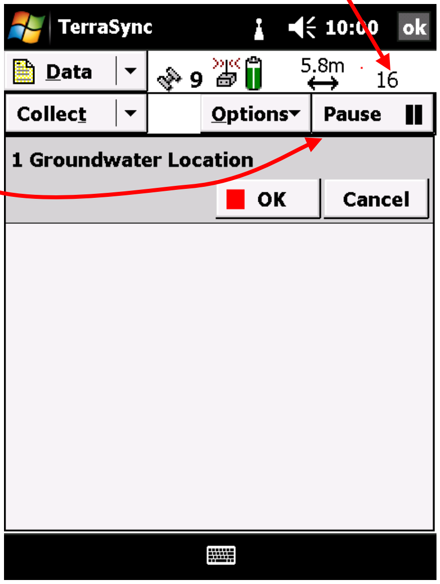
Line_generic

Area_generic

Ensure that this Tab has **Collect** selected.

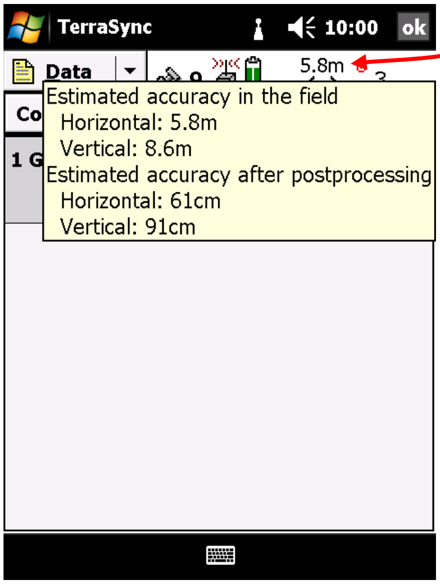


Select **Point**, **Line**, or **Area,** then tap the **Create** button. When ready to collect data, tap the **Log** button (button will show pause if not currently logging, as shown here). Attribute any and all necessary fields (tap keyboard icon to enter description). This can be done before, during, or after collecting your data. Make sure to collect at least 30 positions for each point. Your positions count is displayed above the **Log** button.



1 Groundwater Location

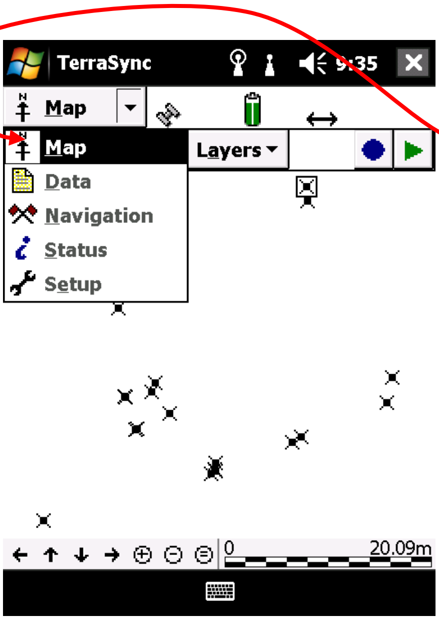
Estimated accuracy is listed here. By tapping this number more detail will be shown (right).



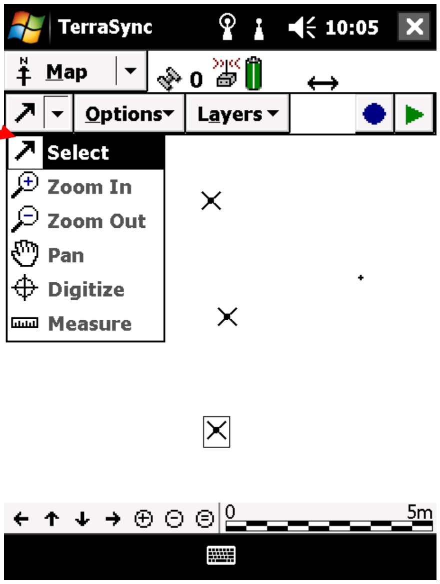
Estimated accuracy in the field
Horizontal: 5.8m
Vertical: 8.6m
Estimated accuracy after postprocessing
Horizontal: 61cm
Vertical: 91cm

When ready, click **OK** to finish feature collection. You will be returned to the main data collection page. Continue collecting data until finished. Data will be saved as you go. When finished with data collection, exit out of TerraSync and turn off unit (Green button).

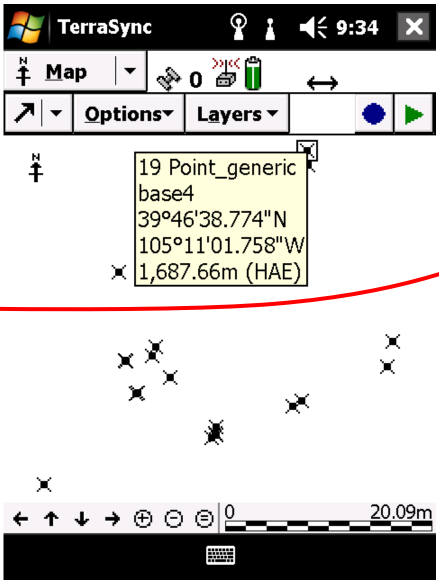
7.4 TerraSync Map Menu



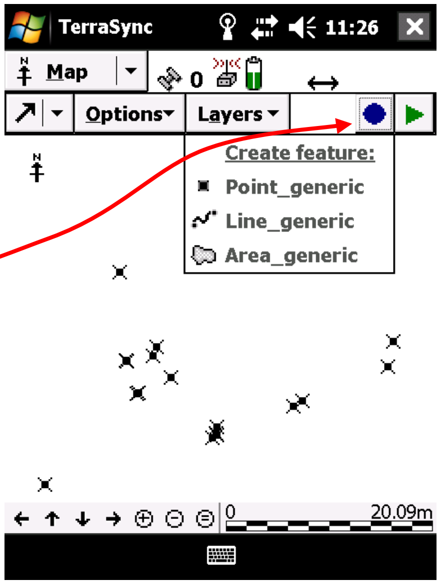
To view data that has been collected, choose the **Map** section. There are multiple tools to help with the display of data shown here.



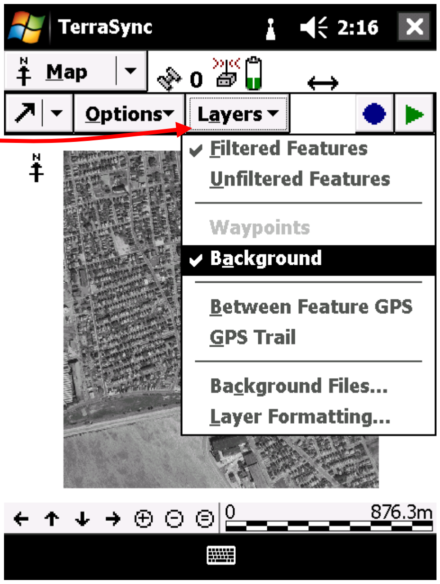
Tapping any point will display the attributes of a point, including a point's latitude and longitude. Logging GPS positions can be accomplished in the **Map** section. After selecting and setting up the proper data file in the **Data** section, choose **Map.** Tap **Create Feature** button, then tap the arrow key on the top right to begin logging points. When enough points have been collected, click **Ok**.



19 Point_generic
base4
39°46'38.774"N
105°11'01.758"W
1,687.66m (HAE)

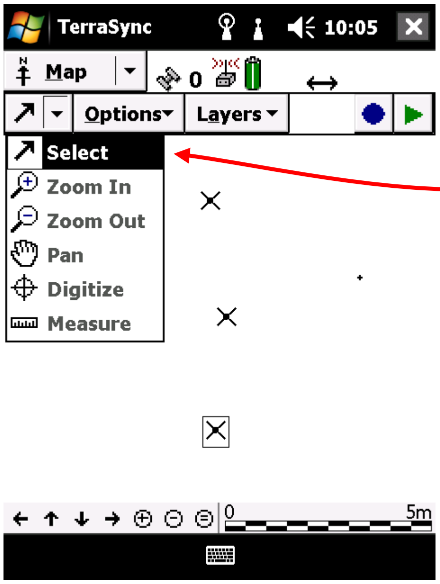


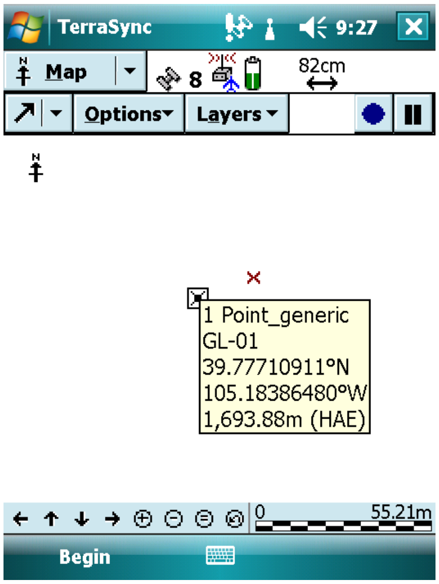
The on-screen display in the **Map** section can be enhanced with an aerial or topographical image. This must be set up prior to field collection with ESAT. If available select **Layers** and tap **Background** to enable the image (See Below).



7.5 Navigation Using TerraSync

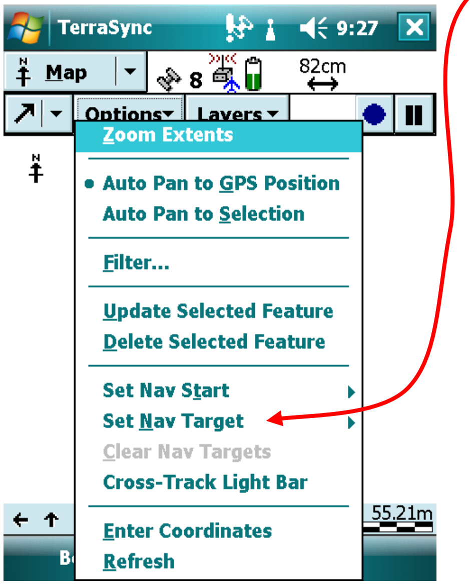
TerraSync and your Trimble unit can help you to navigate to a specific point if that point has already been loaded into the data. In the **Map** tab, use the selection arrow to select the point of interest (below, right).

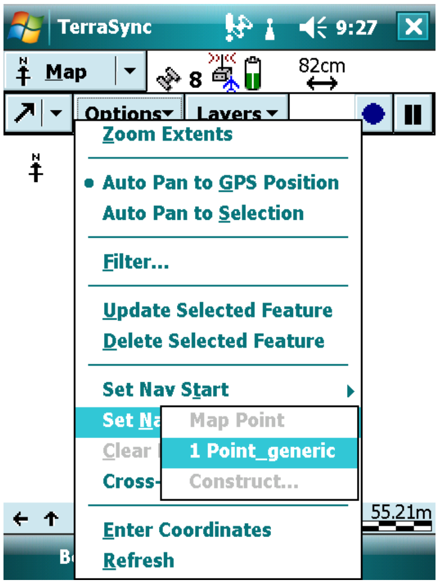




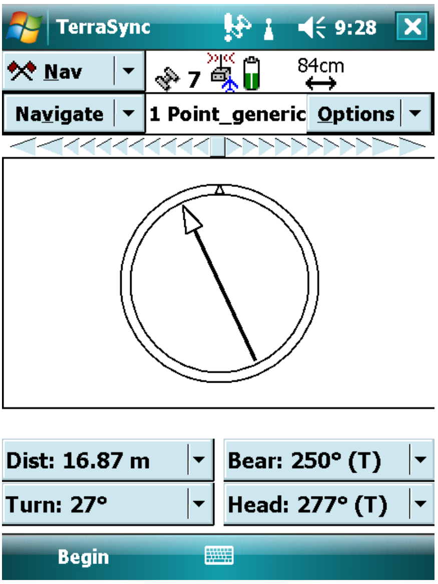
1 Point_generic
GL-01
39.77710911°N
105.18386480°W
1,693.88m (HAE)

Tap the **Options** tab and then select **Set Nav Target.** Next, select the highlighted feature in the drop down menu (below). On the map, the point you are navigating to will have a crossed flag symbol like this:

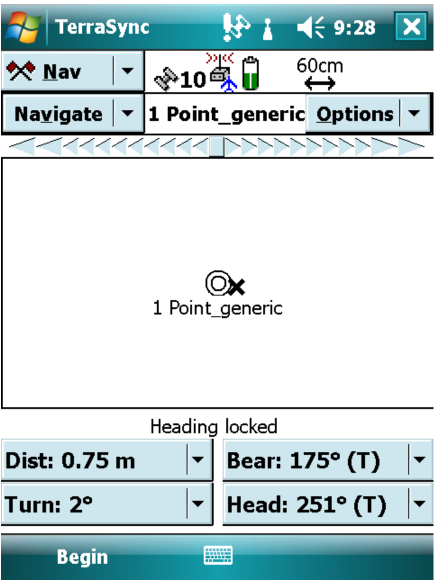




Once you have selected the point feature of interest, you can switch to the **Nav** menu on the main drop down list (left). The arrow guides you in the direction of the point of interest giving distance and bearing. You have to be moving for this to work. This navigation menu will change once you are close (right). You are the **X.**

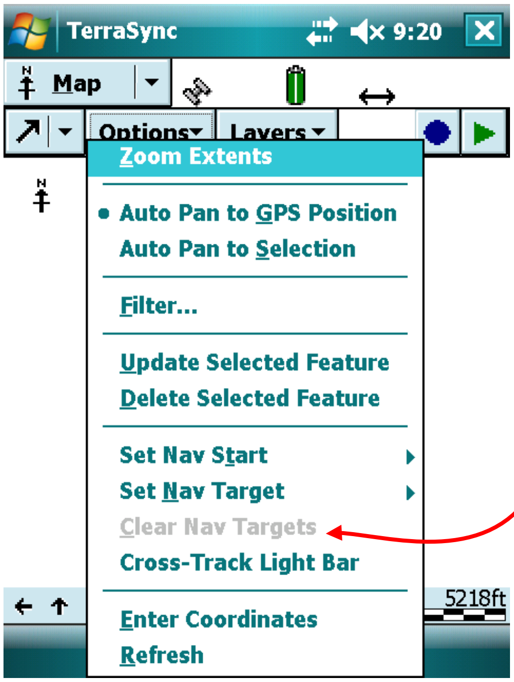


Dist: 16.87 m
Bear: 250° (T)
Turn: 27°
Head: 277° (T)



Heading locked
Dist: 0.75 m
Bear: 175° (T)
Turn: 2°
Head: 251° (T)

Once finished navigating, go back to the **Map** section by going to the **Nav** menu and clicking **Map**. In the **Map** section, click **Options** and select **Clear Nav Targets.**



8.0 LASER RANGEFINDER

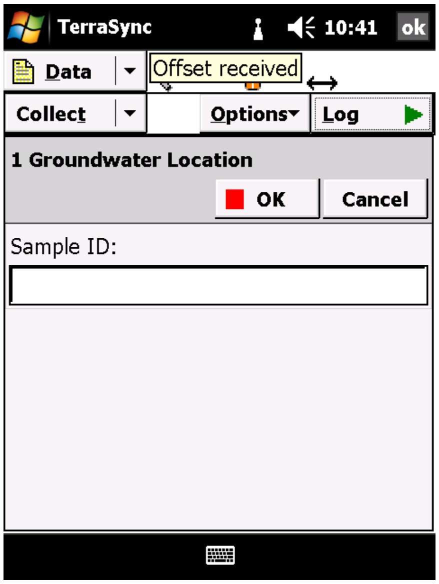
The LTI TruPulse 360 Laser RangeFinder is a measurement device that emits a laser to a distance of up to 1,000 meters and returns various information, such as vertical distance, horizontal distance, slope distance, inclination, azimuth, and height.

8.1 Enabling the Laser RangeFinder for Offset Point Collection

The Trimble units have been configured with the Laser setup and are ready to work. Laser #1 is setup for GPS units 1 & 2. Laser #2 is setup for GPS units 3 & 4. Before using the laser on your GPS device, you need to turn on Bluetooth on the Laser Rangefinder. To do this, look into the Laser eye piece and turn on the laser by clicking the **FIRE** button on top of the laser. Press the down arrow for four seconds. Press the down arrow once to get to the Blue Tooth option. Select **FIRE**. You have now enabled Blue Tooth on the laser. It should read **Bt_on**. Press **Fire** to return to main menu. Once the laser is enabled, it should be ready to work when you open a data file in the **Data** section of TerraSync.

8.2 Using the Laser RangeFinder in TerraSync

To use the laser once setup is complete, follow the same steps in data collection and begin logging points. After naming your sample ID, begin logging points. Aim the Laser RangeFinder at your target and push the **FIRE** button. You will be given notice that the offset has been received. Make sure that the **Offset received** notification appears. Sometimes your laser shot will not register and you will need to repeat your shot. When finished with the laser, it will turn off by itself.



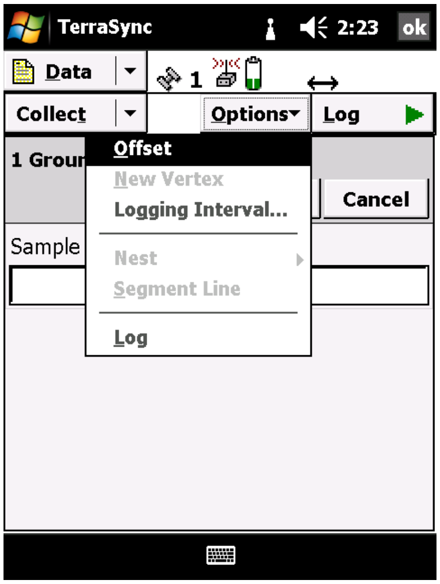
Offset received

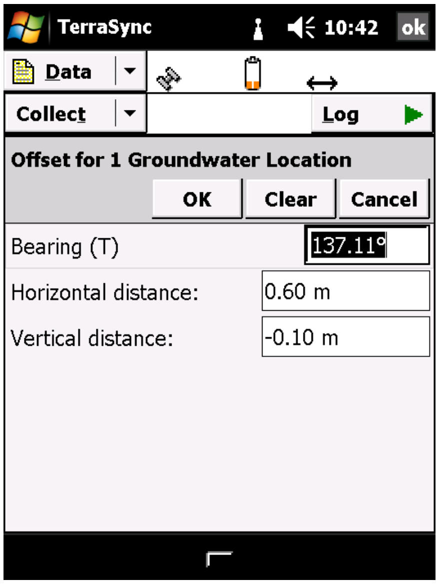
1 Groundwater Location

Sample ID:

REFER TO SECTION 8.3 TROUBLESHOOTING THE LASER RANGEFINDER IF YOU EXPERIENCE ANY DIFFICULTIES IN THE SETUP PROCESS.

To confirm the offset distance, you can tap the **Options** button and then click **Offset** to view the offset information that has been gathered.





Bearing (T) 137.11°

Horizontal distance: 0.60 m

Vertical distance: -0.10 m

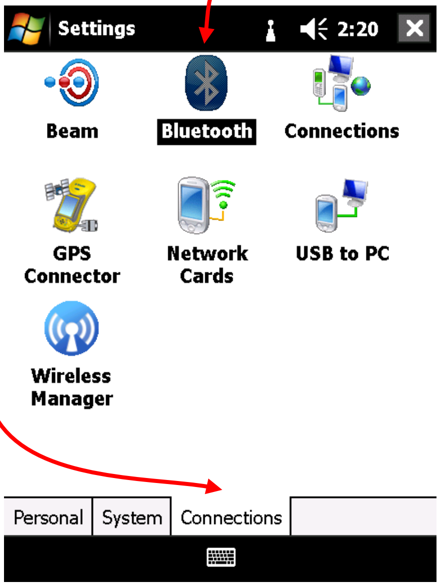
Your offset has been captured. Click **OK** and then click **OK** again to capture the offset point.

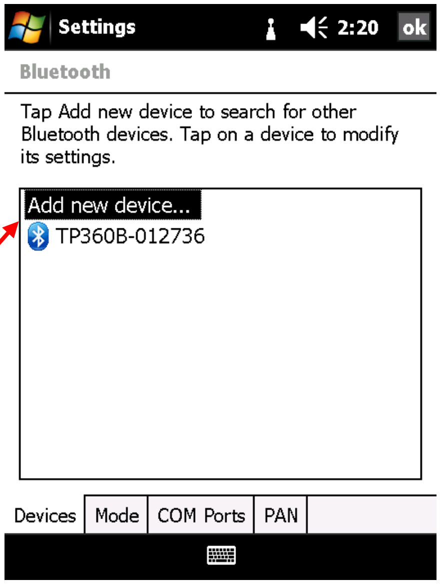
For additional setting and configuration options for the laser, refer to the TruPulse 200B/36B Cheat Notes card. This card can be found in the Laser Box.

8.3 Troubleshooting the Laser RangeFinder

If enabling the laser did not work as described above, you will have to reestablish and reconfigure the Bluetooth connection to the Trimble unit. This is common so do not panic! At times, this process may seem to loop on itself. Follow these instructions and you will be ready to use the laser in very little time.

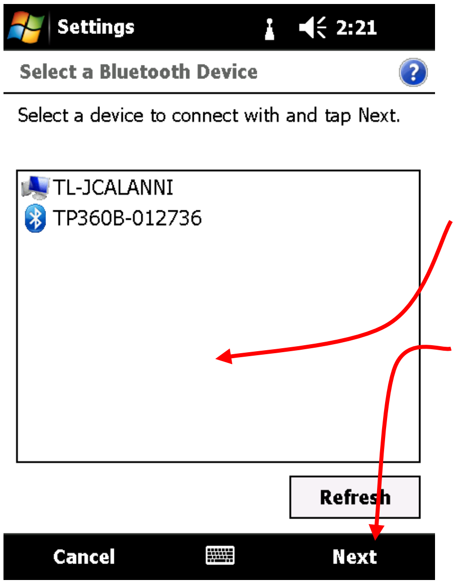
First ensure that the laser's Bluetooth is turned on.
To activate the laser on your GPS device, tap **Start** and select **Settings.** At the bottom of the **Settings** page, select the **Connections** tab, then tap the **Bluetooth** icon.





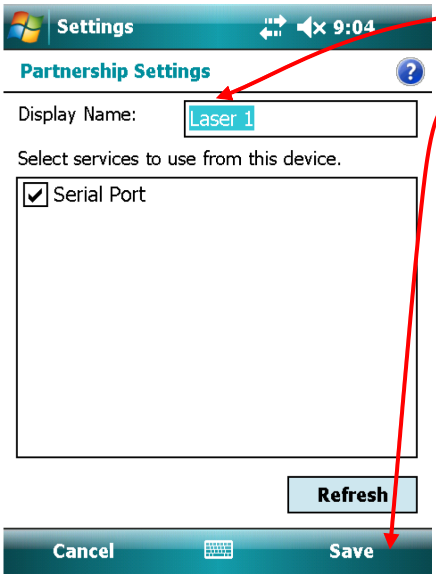
Tap Add new device to search for other Bluetooth devices. Tap on a device to modify its settings.

Choose **Add new device.**



Select a device to connect with and tap Next.

Select the Bluetooth Icon with label TP360B-012736. This number code is for Laser 1 (used with GPS unit 1 and 2); if using Laser 2 (for use with GPS units 3 and 4) the number code will be TP360B-017564. Tap **Next**. You will be prompted to enter the following pass code: 1111

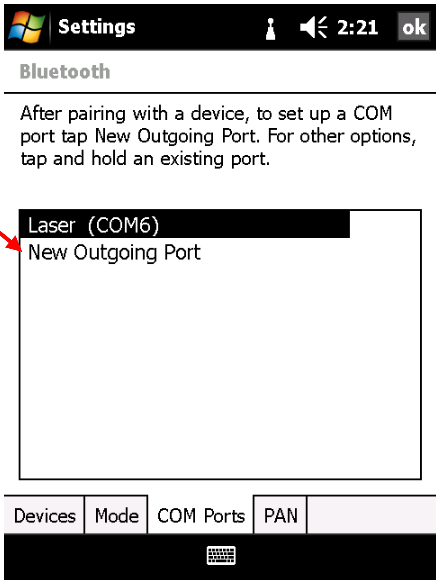


Display Name: Laser 1

Select services to use from this device.

☑ Serial Port

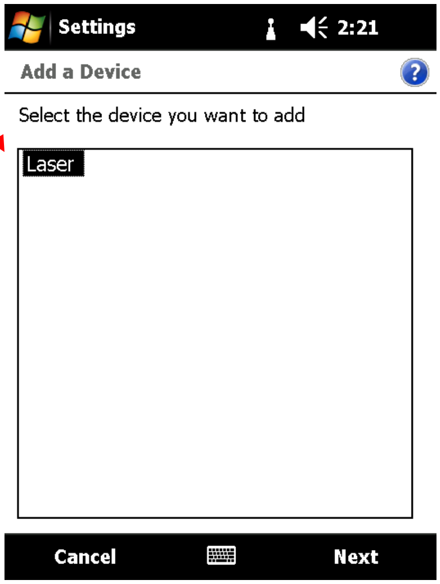
Check **Serial Port**, rename the **Display Name** “Laser,” and click **Save** (left). In the next screen, choose **COM Ports** and select **New Outgoing Port**.



After pairing with a device, to set up a COM port tap New Outgoing Port. For other options, tap and hold an existing port.

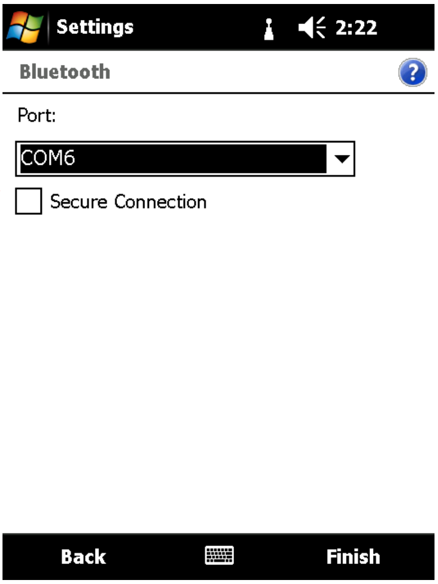
Laser (COM6)
New Outgoing Port

Select **Laser** and tap **Next**. On the next screen, choose a COM Port from the drop down. ***Do not check Secure Connection*****.** Tap **Finish. Close this application**.



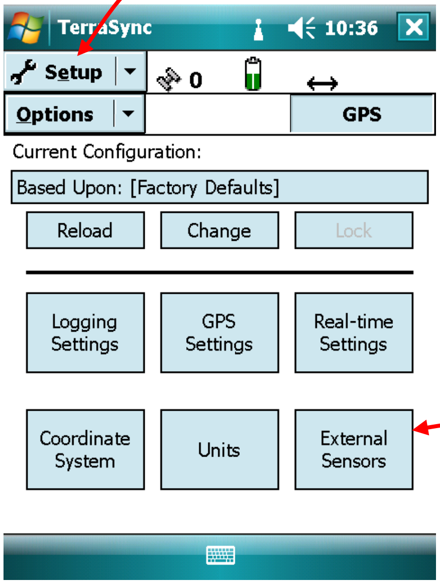
Select the device you want to add

Laser



Port: COM6

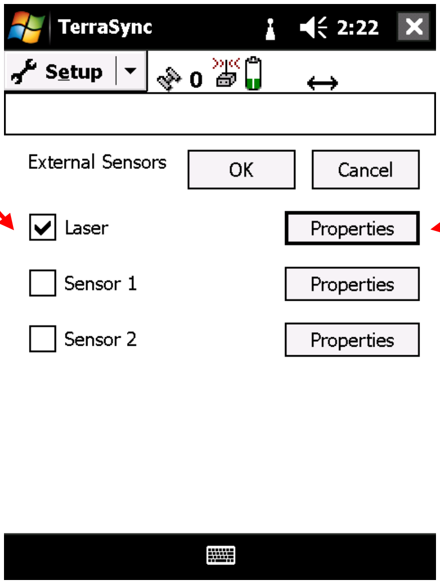
☐ Secure Connection



Current Configuration:
Based Upon: [Factory Defaults]

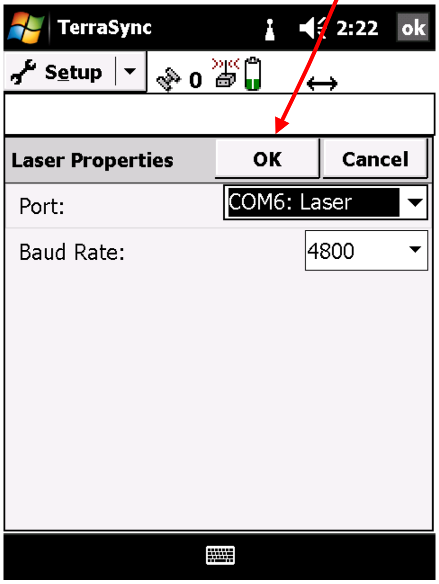
Back in TerraSync, tap the **Setup** menu and choose **External Sensors** to link to the Laser Rangefinder.

Check the box next to **Laser** to enable. Next tap the **Properties** box to setup the correct COM Port.



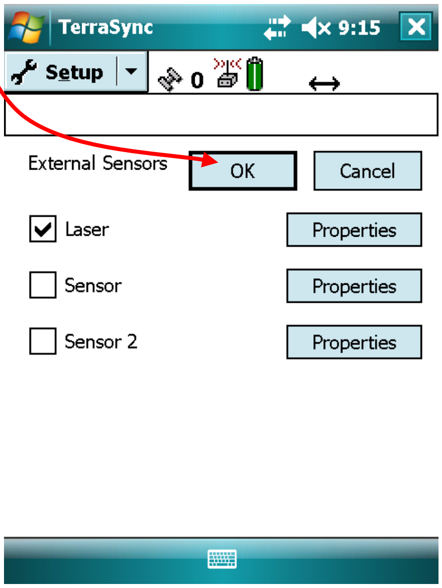
☑ Laser
☐ Sensor 1
☐ Sensor 2

Tap the drop down box next to **Port** and choose the COM Port that you assigned to the **Laser**. Tap **OK** and then tap **OK** on the next screen. You are now ready to take offset points using the Laser Rangefinder.



Port: COM6: Laser

Baud Rate: 4800



☑ Laser

☐ Sensor

☐ Sensor 2

9.0 TROUBLESHOOTING

If the unit does not show any satellites the GPS receiver may have been disconnected. Simply go into the **Setup** menu and click the **GPS** button to connect to the receiver. Sometimes the units go into deep hibernation and will not turn on using the on/off button. To fix this, the power button and tiny grey reset button need to be depressed simultaneously. Use a pen to press the reset button (inset on the upper left of the Trimble keypad).

9.1 Contact numbers for assistance

Trimble Support	1.800.728.5066

10.0 REFERENCES

Trimble Navigation Limited. Trimble Mapping and GIS Products: New Post-Processing Engine. 2009. Westminster, CO, USA.

Trimble Navigation Limited. TerraSync Software Reference Manual. 2008, Version 3.21, Revision A. Westminster, CO, USA.

Trimble Navigation Limited. GeoExplorer 2008 Series User Guide. 2008, Version 1.00, Revision B. Westminster, CO, USA.

FlowTracker® Operation

APPROVED:

ESAT Region 8 QA Coordinator

04/04/12
Date

ESAT Region 8 Team Manager

6/6/12
Date

EPA Task Order Project Officer

7/10/12
Date

ESAT Region 8 Task Lead

6/8/12
Date

DCN: EP8-7-7061

TABLE OF CONTENTS

1.0 GETTING STARTED: OPERATIONAL OVERVIEW

NOTE: This screw cap must be unscrewed during transport of the FlowTracker.

Figure 1.0-1 – FlowTracker with 2D Probe

1.1 System Components

1.1.1 Figure 1.0-1 Flow Tracker with 2D Probe

Figure 1.0-1 shows the FlowTracker with all major components labeled**.**

- **Probe –** The probe contains the acoustic elements used to measure velocity. See the *FlowTracker Principles of Operation* for more information.

- **Probe cable –** The probe is mounted from a 200-cm (80-in) flexible cable. The probe cable is custom built and highly noise-sensitive; it must never be modified.

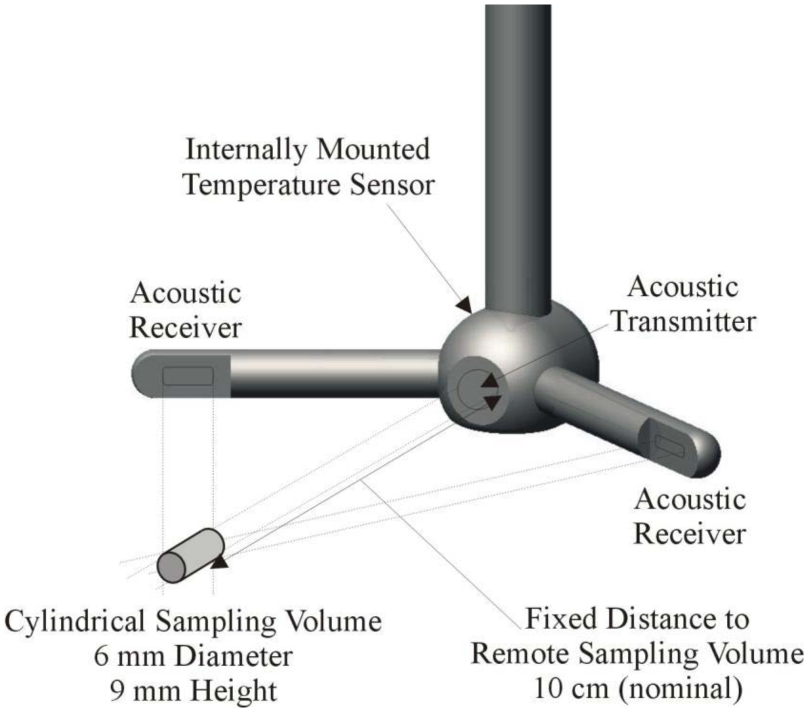
- **Handheld controller** – The handheld controller contains the processing electronics, batteries, keypad, and liquid crystal display (LCD) screen. The controller is designed to withstand temporary submersion, but is not intended for underwater operation.

- **Keypad** – The FlowTracker keypad is designed for quick, efficient entry of data collection parameters and commands.

- **LCD screen** – The LCD screen displays instructions and real-time data.

- **External power/communication connector** – A waterproof connector on the bottom of the handheld controller connects to an external power/communication cable. This is used to download data from the FlowTracker to a personal computer (PC). During data collection, the connector is sealed with a dummy cap. ***WHEN TRANSPORTING FLOWTRACKER, THIS SCREWCAP MUST BE UNSCREWED*.**

1.1.2 Figure 1.0-2 2D Side Looking FlowTracker Probe and Sampling Volume



2.0 ACRONYMS AND DEFINITIONS

°C	Degrees Celcius
°F	Degrees Fahrenheit
%Q	Section Discharge
σV	Standard error of velocity
Bnd	Boundary QC variable
cfs	Cubic feet per second
cm	centimeter
dB	Decibel
EPA	United States Environmental Protection Agency
ESAT	Environmental Services Assistance Team
ft	feet
ft/s	feet per second
Hz	Hertz
in	inch
LCD	Liquid Crystal Display
m	meter
mA	Milliampere
MB	Megabyte
m/s	Meters per Second
PC	Personal Computer
ppt	parts per thousand
QC	Quality Control
SNR	Signal-to-Noise Ratio

Averaging time: The time (in seconds) in which the FlowTracker records data at each measurement location. This is a United States Geological Survey specified value of 40 seconds (Blanchard, 2004).

External control: The FlowTracker can be controlled by an external computer using the RS232 serial interface. This is accessed from the external power/communication connector. External control is used to download data from the internal recorder to a PC for further analysis, display, and archiving.

Keypad interface: The FlowTracker is controlled from the keypad on the handheld controller. The LCD screen is used to display command options and real-time data.

Measurement location: At each measurement location, the FlowTracker records one second velocity data for the specified averaging time, location, and water depth parameters (to document the data set), and a variety of statistical and quality control data.

Ping: A single estimate of the 2D or 3D water velocity.

Ping rate: The number of pings per second (Hz). The FlowTracker ping rate is 10 Hz.

Quality control data: In addition to velocity, the FlowTracker records several quality control (QC) parameters. These include signal-to-noise ratio (SNR), standard error of velocity, boundary adjustment, the number of spikes filtered from data, and velocity angle.

Salinity: Water salinity (in ppt) is a user-supplied value that is used for sound speed calculations. Note: If using the system in salt water, a zinc anode should be installed on the probe for corrosion protection.

Sample: A sample refers to the mean of 10 pings to produce a measurement of the 2D or 3D water velocity. A sample includes velocity and signal strength data. The FlowTracker records one sample per second.

Signal strength: This refers to the strength of the reflected acoustic signal. It is a function of the acoustic conditions of the water – primarily the amount and type of suspended material (scatterers) present. This is most commonly accessed as a SNR.

Signal-to-noise ratio (SNR): SNR is the ratio of the received acoustic signal strength to the ambient noise level. It is expressed in logarithmic units (dB) and is the most important QC data for the FlowTracker.

Sound speed: Speed of sound in water (in m/s) is used to convert the Doppler shift to velocity.

Temperature: Water temperature (°C) is measured by the internal temperature sensor. Temperature is used for sound speed calculations.

3.0 USING THE KEYPAD INTERFACE, MENUS AND DISPLAY

This section describes FlowTracker's keypad interface, its menus, and the LCD display.

3.1 On/Off Switch

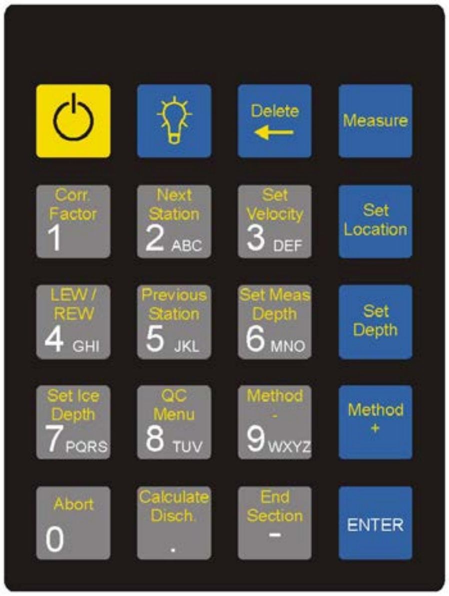
The **On/Off** power button is in the upper left hand corner of the keypad.

- To turn the system on, hold the button for one second until the LCD screen turns on.
- To turn the system off, hold the button for four seconds until the LCD screen resets.
- The FlowTracker draws a small amount of current when off (< 1 mA). If the system is to be left idle for a long period (more than 1 month), remove the batteries to prevent unnecessary draining and potential battery leakage.

NOTE: ***It is important to always return to the Main Menu before turning the system off to ensure all data has been properly saved.***

3.2 Keypad

3.2.1 Figure 2.2-1 – FlowTracker Keypad



The function of each key is described below. Many keys have multiple functions.

Keys are described starting at the top left, moving right and down by rows.

Numbers (0-9)

- These keys are used to enter the following information when prompted by the FlowTracker: Menu selections, filename and extension, station location, depth, and other information.

Letters (A-Z)

- These keys are used to enter text for the filename and for comments in the file.
- Text entry is done in the same manner as for mobile phones. For file names, the text entry assumes numbers first (i.e., for “C” press **2** button four times **2** – **A** – **B** – **C**); for all other text, it assumes letters first (i.e., for “C” press **2** button three times **A** – **B** – **C**).

NOTE: It is important to always return to the Main Menu before turning the system off to ensure all data has been properly saved.

Backlight

This key turns the LCD backlight on/off.

- If the backlight is already on, it turns the backlight off.
- The backlight will turn off automatically after 1 minute.
- Minimize backlight use to increase battery life (the backlight doubles power consumption).

Delete
This key deletes the last character when entering parameters and is used for deleting stations from a file.

Measure
This key starts a measurement.

- It works identically in both **Discharge** and **General** data collection modes.
- It is only active if the FlowTracker is displaying the current station information screen. It is not active when displaying data from a previous measurement.

Corr. Factor
This key is used to enter special correction factors.

- The correction factor is used only in the **Discharge** mode.
- The correction factor is only used for certain situations.

Next Station
This key is used to view the next station when scrolling through completed stations.

- It works identically in both **Discharge** and **General** data collection modes.
- It is not active during a measurement, or from the time **Measure** is used to start a station until the time the last measurement for that station is completed.
- In **Discharge** mode, stations are sorted/displayed by location (regardless of the order in which they were collected).
- In **General** mode, stations are displayed in the order they were collected.

Set Velocity
This key is used to enter a user-estimated velocity. It is active only in **Discharge** mode for stations using the **Input V** method.

Set Location
This key is used to set the measurement location.

- It sets the location value (**Loc**) in **Discharge** mode.
- It sets both the location 1 and location 2 values (**L1/L2**) in **General** mode.
- It can be used either for the current station (before the measurement) or to edit data from a previous station.

LEW/REW
This key is used to specify the starting or ending edge of water.

- It is used only in the **Discharge** mode.
- **LEW** means “Left Edge of Water” and **REW** means “Right Edge of Water.” Your position is based on your body orientation when facing downstream.

Prev. Station

This key is used to view the previous station when scrolling through completed stations.

- It is not active during a measurement, or from the time **Measure** is used to start a station until the time the last measurement for that station is complete.
- In **Discharge** mode, stations are sorted/displayed by location (regardless of the order in which they were collected).
- In **Discharge** data collection mode, it will scroll past station 1 to the starting edge and starting gauge information.

Set Measurement Depth

This key is used to set the measurement depth.

- It sets the measurement depth value (**MDep**).
- It can be used either for the current station (before the measurement) or to edit data from a previous measurement.

Set Depth

This key is used to set the water depth (**Dep**). It can be used either for the current station (before the measurement) or to edit data from a previous station.

Set Ice Depth

This key is used to set the ice depth.

- It is active only in **Discharge** mode.
- It sets the ice depth value (**Idep**) as measured from the water surface to the bottom of the ice.
- It can be used either for the current station (before the measurement) or to edit data from a previous station.

QC Menu

This key is used to access the **QC Menu** during data collection.

- It works identically in both **Discharge** and **General** data collection modes.
- The **QC Menu** includes the following functions:

    - Input supplemental data (gauge height, rated flow, and comments).
    - Accessing **QC Settings** and **Discharge Settings.**
    - Changing the averaging time for each measurement.
    - Display raw velocity data.
    - Run the automatic QC test.

Method –

This key sets the method used for calculating the mean velocity in discharge measurements.

- It is active only in **Discharge** mode.
- This key changes the method to the *previous* method in the list.

Method +
This key sets the method used for calculating the mean velocity in discharge measurements.

- It is active only in **Discharge** mode.
- This key changes the method to the *next* method in the list.

Abort
This key is used to stop data collection during a measurement.

- After pressing **Abort**, it can take 1-2 seconds to stop data collection.
- The FlowTracker will display a message saying that data collection was aborted and how much data were collected.
- You can accept the aborted measurement (using the mean values of the data that were collected) or repeat the measurement.

Calculate Disch.
This key tells the FlowTracker to perform the final discharge calculation.

- It is active only in **Discharge** mode.
- It is active only when all stations have been completed and the **End Section** key has been pressed to enter the ending edge information.
- It is active only when the ending edge screen is displayed.
- The system will display a series of discharge screens showing final discharge data.

End Section
This key is used to end a series of measurements. In **General** mode, it ends the series of measurements and displays the summary of all data collected.

- It is active only when the current station (not yet measured) is displayed.
- It takes a few seconds for the file to be closed before displaying the summary.
- In **Discharge** mode, this indicates that all stations have been collected.
- It ends the section and displays the ending edge screen. After this data has been entered, the final discharge calculation can be done.
- It is active only when the current station (not yet measured) is displayed.

ENTER
This key serves several functions and works identically in both **Discharge** and **General** data collection modes.

- It is used to complete the entry of any user parameter.
- It is used to toggle between multiple display screens when available.
- It is used to acknowledge a FlowTracker system message.

3.3 Main Menu

When turned on, the FlowTracker displays a wake up screen showing the firmware version and the date/time from the system clock (continually updating).

<u>Example:</u> FlowTracker 3.0 2006/06/01 08:10:25 Press Enter For Main Menu

Pressing **Enter** displays the **Main Menu**.

Main Menu 1:Setup Parameters 2:System Functions 3:Start Data Run
From the **Main Menu**, press the appropriate key to access the desired function.

Press **1** for the **Setup Parameters Menu.**

Press **2** for the **System Functions Menu**.

Press **3** to start a data run.

NOTE: It is important to always return to the Main Menu before turning the system off to ensure all data has been properly saved.

3.3.1 Setup Parameters Menu (Main Menu <1>)

Setup parameters determine how the FlowTracker collects data. This section describes each item in the **Setup Parameters Menu**.

1: Units English
2: Avg. Time (40)
3: Mode Discharge
0=Exit or Enter=More

4: QC Settings
5: Discharge Settings
6: Salinity (0.00)
0=Exit or Enter=More

7: Language English
8: No Correction
0=Exit or Enter=More

The **Setup Parameters Menu** is shown above (three screens are needed to show all options). Press **Enter** to cycle through the screens. To change a setting, press the appropriate menu number.

3.3.2 Units System (Setup Parameters <1>)

The **Units** option defines the units system used for display and output data (set as **English**). The units system does not affect internal calculations or storage (internal units are metric).

To change the units system, press **1** from the **Setup Parameters Menu**, and then press **1** for **English** units.

3.3.3 Averaging Time (Setup Parameters <2>)

The **Avg Time** (averaging time) option specifies the amount of data (in seconds) to be collected at each measurement site. Averaging time is specified in 1-second intervals from 10 to 1000 seconds. Forty (40) seconds is the approved time.

To change the averaging time, press **2** from the **Setup Parameters Menu**.

3.3.4 Data Collection Mode (Setup Parameters <3>)

The **Mode** option determines the procedure when collecting a series of measurement stations.

To change the data collection mode, press **3** from the **Setup Parameters Menu**, and then press **1** for **Discharge** mode. In **Discharge** mode, a sequence of measurements is used to calculate river discharge (cfs).

3.3.5 QC Settings (Setup Parameters <4>)

The **QC Settings** menu sets universal QC criteria for **Discharge** mode.

To access the **QC Settings** menu, press **4** from the **Setup Parameters Menu**, and then:

- Press **1** to set the **SNR Threshold (>10dB)**
- Press **2** to set the **σV Threshold (<0.03 ft/s)**
- Press **3** to set the **Spike Threshold (<10%)**
- Press **4** to set **Max Velocity Angle (<20 degrees)**

3.3.6 Discharge Settings (Setup Parameters <5>)

The **Discharge Settings** menu specifies settings for the discharge calculations and the QC criteria used for **Discharge** measurements.

To access the **Discharge Settings** menu, press **5** from the **Setup Parameters Menu**. You can also access **Discharge Settings** by pressing **3** from the **QC Menu** (although options **1**, **2**, and **3** are not available when a data file is open).

- Press **1** to set the discharge **Equation**.
- Press **1** to select **Mid Section**. Selecting this option sets **Repeat Depth** and **Repeat Velocity** to **NO**.

The section discharge (**%Q**) displayed for each station can be calculated based on either the user-supplied **Rated** discharge or the total **Measured** discharge.

Select the desired reference, **Rated** or **Measured**.

The default is **Rated**. If no **Rated** value is specified, the **Measured** value is used.

- Press **8** to select the **Methods Displayed**.

The FlowTracker supports several methods to determine mean velocity. The method determines the number and location of velocity measurements.

Scroll through available methods using the **Method+** and **Method–** keys.

- Press **1** to toggle **2-6-8 Methods** (**0.6**, **0.2/0.8**, **0.8/0.2**, **0.2/.6/.8**, **0.8/.6/.2**) on/off.

3.3.7 Salinity (Setup Parameters <6>)

The **Salinity** option specifies the salinity value used to compute sound speed.

To change salinity, press **6** from the **Setup Parameters Menu**, and then enter the desired salinity value in ppt.

Sound speed is used in Doppler velocity calculations (see *FlowTracker Principles of Operation* for details).

Salinity is specified in parts per thousand (ppt). Fresh water has a salinity of 0; seawater typically has a salinity of about 35 ppt.

As a rule of thumb, a 12-ppt error in the value of salinity will result in a 1% error in sound speed, which results in a 2% error in velocity data.

Salinity should be specified as accurately as possible for each location.

NOTE***: When using the FlowTracker in salt water, a sacrificial zinc anode should be installed on the probe for corrosion protection.***

3.3.8 Language (Setup Parameters <7>)

The FlowTracker firmware can operate in five different languages. Keypad labels are changed for compatibility with each language; keypad overlays are available on request for each language (and are included with each system shipped outside the United States and Canada).

To change the language, press **7** from the **Setup Parameters Menu**, and then press **1** for **English**.

3.3.9 Mounting Correction (Setup Parameters <8>)

Mounting Correction specifies how to account for the device used to hold the FlowTracker in the water. The mount may have a small impact on velocity measurements. Specifying the proper mounting correction allows the FlowTracker to account for the effect of the mount.

To change the Mounting Type, press **8** from the **Setup Parameters Menu**:

- Press **1** for **No Correction** (the default setting).
- Press **2** for **Custom**. When prompted, enter a **Mounting Correction** for the mount being used.
- Contact SonTek for help in determining the appropriate **Mounting Correction**. The standard recommendation is 1.0%.

3.4 System Functions Menu (Main Menu <2>)

The **System Functions Menu** provides access to items that should be checked periodically but do not directly affect how data is collected. This section describes those items.

1: View Data File
2: Recorder Status
3: Format Recorder
4: Temperature Data
5: Battery Data
6: Raw Velocity Data
7: Auto QC Test
8: Show Config.
9: Set System Clock
0: Exit or Enter=More

Three screens are needed to show all items above. Press **Enter** to switch between screens. To change a setting, press the number shown.

3.4.1 View Data File (System Functions <1>)

The **View Data File** option allows you to view data files stored on the internal recorder.

To view a data file stored on the internal recorder, press **1** in the **System Functions Menu**.

The FlowTracker will display a menu of the files on the internal recorder. At most, three files are displayed on each screen.

- Use **Enter** to scroll through the entire recorder directory (three files at a time).
- Press **1**, **2**, or **3** to select the desired file.
- Press **0** to exit the **View Data File** option without loading a file.

After loading the file, the FlowTracker displays a summary of the data file.

- Press **Enter** to move between the different summary display screens.
- Press **Previous Station** from any file summary screen to view station data.
- Use **Next Station** and **Previous Station** to scroll through station data (no new measurements can be added). The same station display screens are available as

those used during data collection. Press **Enter** to scroll through the station data screens.

- Pressing **Next Station** from the last station returns you to the file summary screens.
- When done, press **0** to exit and return to the main menu. The **0** key is only active when the display is showing the summary data. If you have scrolled back to a previous station to view data, you will not be able to exit until you scroll forward to the summary data screen.

3.4.2 Check Recorder Status (System Functions <2>)

The **Recorder Status** option displays the number of files stored on the internal recorder and the number of files remaining.

To view the status of the recorder, press **2** from the **System Functions Menu**. Press any key to return to the main menu. The FlowTracker will have either a 2-MB or 4-MB recorder.

3.4.3 Format Recorder (System Functions <3>)

The **Format Recorder** option erases all data on the internal recorder.

To format the recorder, press **3** from the **System Functions Menu**.

The system will prompt you to enter **123** to confirm your decision to format (erase) the recorder. Entering any other value will abort the formatting process.

Be certain that all data has been downloaded before formatting the recorder; data cannot be recovered after the recorder is formatted.

Recorder formatting takes 20-60 seconds to complete.

3.4.4 Temperature Data (System Functions <4>)

The **Temperature Data** option displays the current temperature sensor data.

To view temperature data, press **4** from the **System Functions Menu**. Press any key to return to the main menu.

The temperature sensor is in the probe head and is accurate to +/- 0.1°C.

3.4.5 Battery Data (System Functions <5>)

The **Battery Data** option shows the current battery voltage and an estimate of remaining capacity.

To view battery data, press **5** from the **System Functions Menu**. Press any key to return to the main menu.

An estimate of remaining capacity (as a percent of total) is given for three battery types. The FlowTracker does not know what types of batteries are installed. Typical capacities:

- Alkaline: ≈25 hours
- NiMH: ≈15 hours
- NiCad: ≈7 hours

Battery capacity estimates are based on voltage and are only approximate.

Cold weather can greatly affect battery voltage and capacity; always check battery voltage after the system has acclimated to outside temperatures.

3.4.6 Raw Velocity Data (System Functions <6>)

The **Raw Velocity Data** option shows a continuous display of raw velocity and SNR data.

To display raw data, press **6** from the **System Functions Menu**.

- This can also be accessed by pressing **5** from the **QC Menu**.
- Press any key to exit the raw velocity display.
- This can be done as a quick test before data collection or to check stream conditions during data collection. Data displayed during this function are not recorded.
- Velocity and SNR data are updated once per second.
- Velocity data can be expected to show notable variations (most of which are real), and should be indicative of the general conditions in the water.
- SNR is primarily a function of the amount of particulate matter in the water. For good conditions, SNR should be at least 10 dB. The system can operate effectively with SNR as low as 2-3 dB, although the noise in individual velocity measurements will increase.

3.4.7 Automatic QC Test (System Functions <7>)

The Auto QC Test is an automated version of the *BeamCheck* software. This test can be run in three ways:

- When you are prompted to run this test at the start of each data file.
- When you press **7** from the **System Functions** menu.
- When you press **6** from the **QC Menu**.

Follow the instructions on the LCD. When the test is done and while a data file is open, results are recorded with the file. Place the probe in moving water well away from any underwater obstacles. The FlowTracker collects data for about 30 seconds. The data are analyzed based on several criteria:

Noise level
- Measured electronics noise level is compared to reference data. Any significant deviation causes a warning.
- A large change in noise level may indicate damage to the probe.

SNR
- The SNR is checked as sufficient for reliable data collection.
- Each beam SNR is compared to be sure all beams perform equally.
- A warning is issued for low SNR or if beam SNR values differ.

Peak shape
- The shape of the sampling volume curve is compared to the expected shape. Any significant deviation causes a warning.
- This criterion can only be checked with sufficient SNR (> 7 dB).

Peak location
- The physical location of the sampling volume is compared to the expected location. Any significant deviation causes a warning.
- This criterion can only be checked for sufficient SNR (> 7 dB).
- If any warnings are issued, you are given the option to repeat the test.

It is recommended that the test be repeated at least once after you verify that the probe and sampling volume are well away from any underwater obstacles.

If multiple warnings are received, run *BeamCheck* from a PC to evaluate FlowTracker performance in more detail.

3.4.8 Show System Configuration (System Functions <8>)

The **Show Config** option displays basic system configuration information.

To display system configuration information, press **8** from the **System Functions Menu**. Press any key to return to the main menu. Displayed items include:

- Firmware version number
- Serial number
- Probe type

Side XY 10cm indicates a side-looking 2D probe.

3.4.9 Set System Clock (System Functions <9>)

The **Set System Clock** option displays the date and time (continually updated) from the FlowTracker’s internal clock.

To change the date/time, press **9** from the **System Functions Menu,** and then follow the menu options to change the date or time.

3.5 QC Menu

The **QC Menu** is accessed only during data collection by pressing the **QC Menu** key (in either **Discharge** or **General** mode). It provides access to several functions.

Input supplemental data

- Supplemental data is provided to allow you to further document that data set.
- Up to 20 different supplemental data records can be included with each file.
- Each record can include gauge height, rated flow, user comments, a time stamp, and a location stamp.

The time stamp and location stamp are automatically generated with any gauge height, rated flow, or comment entries, but can be edited. Data in all records can be viewed and modified following the on-screen instructions in the **QC Menu**.

- Modify **QC Settings**
- Modify **Discharge Settings** (**Discharge** mode only)
- Change the averaging time (**Avg Time**) used for each measurement
- Display **Raw Velocity Data** from the FlowTracker without recording to a file
- One possible use for this function is to use the FlowTracker to locate the bottom of a slush layer under ice
- Run and record an additional **Auto QC Test**
-

4.0 ESTABLISHING A FLOW DISCHARGE STATION

4.1 Basic FlowTracker Data Collection Process - Using the Keypad Interface

Pressing **3** from the main menu starts a data collection run. Press **1** to **Name** the data file. Enter the station ID using the keypad.

Find the previously established sample location and visually inspect the site for hazards both above and below the surface of the water. To minimize QC errors, spend time engineering the stream above your tag line by removing rocks, sticks, moss, and any other obstacles that may channelize water currents or cause back flow. Once you begin flow measurement, you may not move obstacles.

1. Stretch the tag line across the stream perpendicular to the stream flow.
2. The user proceeds through a series of measurement locations (a minimum of 20 stations and up to 100 stations can be recorded with each file).
3. At the start of data collection, you are prompted for a file name. Name this file for the Station Identification.

4. The operator will next be prompted to **Run** or **Skip** the **Auto QC Test**. The **Auto QC Test** is essentially a field version of *BeamCheck* but is particular to the present measurement environment. This information is stored with each discharge measurement file and is displayed on the discharge measurement summary.
5. It is a requirement of all United States Environmental Protection Agency (EPA) and Environmental Services Assistance Team (ESAT) personnel to conduct an **Auto QC Test** prior to each discharge measurement.
6. Press **1** to **Run Test**.
7. The FlowTracker will collect 20 pings and either report that “All Results are Good” or that the test completed with warnings.
8. If a warning exists, Press **1** to **End Test** or Press **2** to **Repeat Test**. If the test is being repeated, move to a different place in the cross section free of potential boundary interference.
9. At this point of the procedure you are prompted to enter the **Starting Edge** of the cross section to be measured and will soon be collecting velocity data.
10. Press **LEW/REW** (#4) to toggle between the edges, and set the starting edge. The Right or Left edge of water is determined by your body orientation when facing downstream.
11. Press **Set Location** to enter the distance on the tagline for the starting edge of water. Press **Enter** when finished. You will not take a measurement at the Starting Location.
12. Press **Set Depth** to enter the depth of water at the particular location on the tagline. Press **Enter** when finished.
13. Press **Next Station** (#2) to continue the measurement.
14. The following sequence will occur at each successive location of the cross section. Generally, 20 - 25 evenly-spaced observations per cross section are sufficient to define the natural variability of the channel.
15. Press **Set Location** to enter the location on the tagline of the velocity observation. Press **Enter** when finished. The default observation spacing is one foot. Each successive observation sends you to the next point on the tagline based on the spacing of previous observations.
16. Press **Set Depth** to enter the depth of water at the observation point. Press **Enter** when finished. Each successive observation copies the depth of the previous point.
17. Depending on the depth of water at the observation point, the measurement method may differ in order to calculate mean velocity. Depths below 2 feet will use the $6/10^{ths}$ method (the default setup on the FlowTracker). If the depth is above 2 feet, the $2/10^{ths-}$ $8/10^{ths}$ method is used. To toggle between the various methods, press **Method+**.
18. It is possible to recover if you mistakenly press **Measure** with an incorrect parameter or **Method**. Press **Abort** to terminate the measurement (or let the measurement finish), and press **2** to repeat the measurement. Until one measurement is accepted at a station, you have the ability to change all parameters. After one measurement

has been accepted, **Method** can no longer be changed (although other parameters can still be changed).

19. Press **1** to **Accept** the data and move to the next station or location in the measurement series (e.g., advancing from the 2/10ths to 8/10ths observation depth).
20. Press **2** to **Repeat** the measurement, especially if QC issues arise. When a measurement is repeated, data are not lost. However, you will no longer be able to view the *old* data from the keypad interface. The *old* data are still recorded. Later, the data can be extracted in the raw data file (*.dat) and the measurement summary file (*.sum), but not in the discharge summary file (*.dis).
21. Once a station is completed, the FlowTracker displays the next station. Location, depth, and method data for new stations are predicted using previous stations. If a multiple measurement method was used (e.g., 2/10ths and 8/10ths water depth), the next station will use the same method in the opposite order (i.e.; 8/10ths then 2/10ths depth).
22. Summary velocity and QC data are displayed at the end of each measurement. QC data is automatically reviewed. Values outside expected boundary limits generate a warning to the user. You are allowed to repeat individual measurements if desired.
23. You can scroll through previous stations to view data and edit station information.
24. When done, press **End Section**. For Discharge measurements, enter ending-edge information to be shown the final discharge data.
25. Press **Calculate Disch.** to finish the measurement. Press **Enter** to advance through the multiple screens. Any stations with a discharge greater than 10% must be re-evaluated. Do not end section and return to add stations on both sides of the station with greater than 10% discharge until all QC errors are satisfied.

NOTE: ***It is important to always return to the Main Menu before turning the system off to ensure all data has been properly saved.***

5.0 EXPLANATION OF QC CRITERIA

5.1 Accessing QC Criteria

All QC criteria can be modified or disabled. All discharge measurements for EPA and ESAT field events should conform to the following criteria and are entered as the defaults in the ESAT FlowTrackers. To access QC settings:

From the Main Menu, press 1 for Setup Parameters.

From Setup Parameters, select 4 for QC Settings.

- Select 1 to specify SNR Threshold.
- Select 2 to specify σV Threshold.
- Select 3 to specify Spike Threshold.
- Select 4 to specify Max Velocity Angle.

From Setup Parameters, select 5 for Discharge Settings.

- Select 4 to specify Max Section Discharge.
- Select 5 to specify Max Depth Change.
- Select 6 to specify Max Location Change.

To disable any QC criteria, set that parameter to a value of 0.

5.1.1 Table 1-1 Quality Control (QC) Parameters

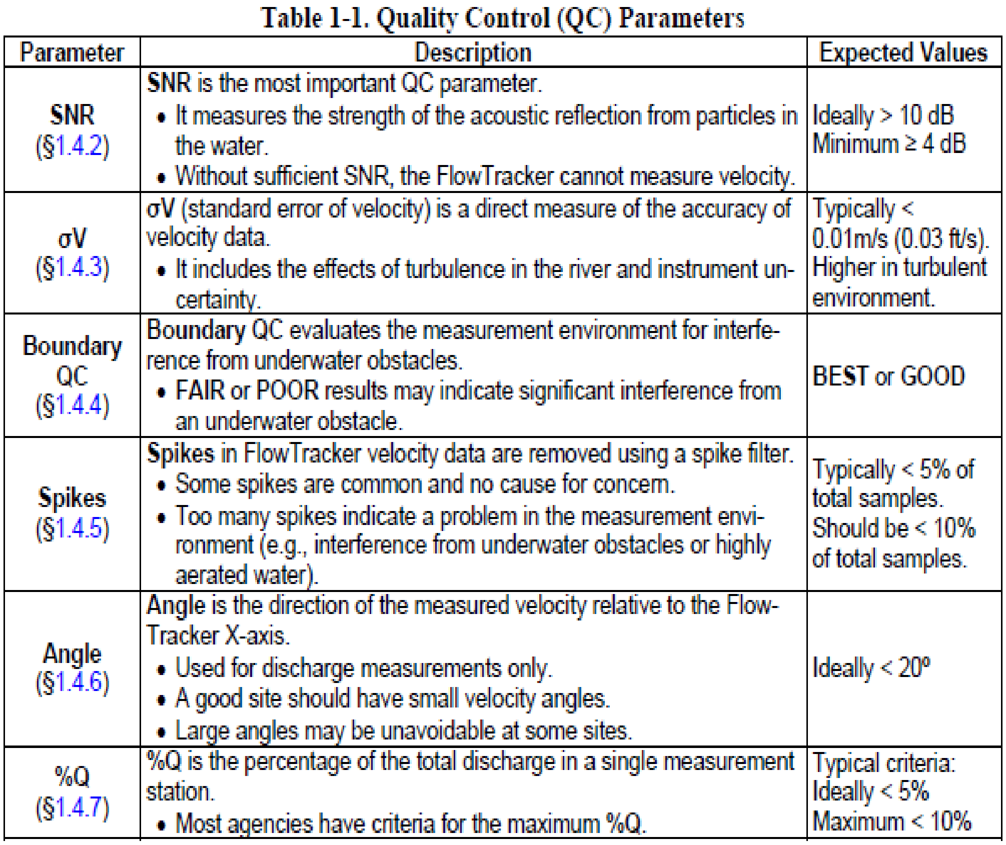


Table 1-1. Quality Control (QC) Parameters

Parameter	Description	Expected Values
SNR (§1.4.2)	**SNR** is the most important QC parameter. • It measures the strength of the acoustic reflection from particles in the water. • Without sufficient SNR, the FlowTracker cannot measure velocity.	Ideally > 10 dB Minimum ≥ 4 dB
σV (§1.4.3)	**σV** (standard error of velocity) is a direct measure of the accuracy of velocity data. • It includes the effects of turbulence in the river and instrument uncertainty.	Typically < 0.01m/s (0.03 ft/s). Higher in turbulent environment.
Boundary QC (§1.4.4)	**Boundary** QC evaluates the measurement environment for interference from underwater obstacles. • FAIR or POOR results may indicate significant interference from an underwater obstacle.	BEST or GOOD
Spikes (§1.4.5)	**Spikes** in FlowTracker velocity data are removed using a spike filter. • Some spikes are common and no cause for concern. • Too many spikes indicate a problem in the measurement environment (e.g., interference from underwater obstacles or highly aerated water).	Typically < 5% of total samples. Should be < 10% of total samples.
Angle (§1.4.6)	**Angle** is the direction of the measured velocity relative to the FlowTracker X-axis. • Used for discharge measurements only. • A good site should have small velocity angles. • Large angles may be unavoidable at some sites.	Ideally < 20°
%Q (§1.4.7)	%Q is the percentage of the total discharge in a single measurement station. • Most agencies have criteria for the maximum %Q.	Typical criteria: Ideally < 5% Maximum < 10%

5.1.2 Table 1-2 QC Warning Messages

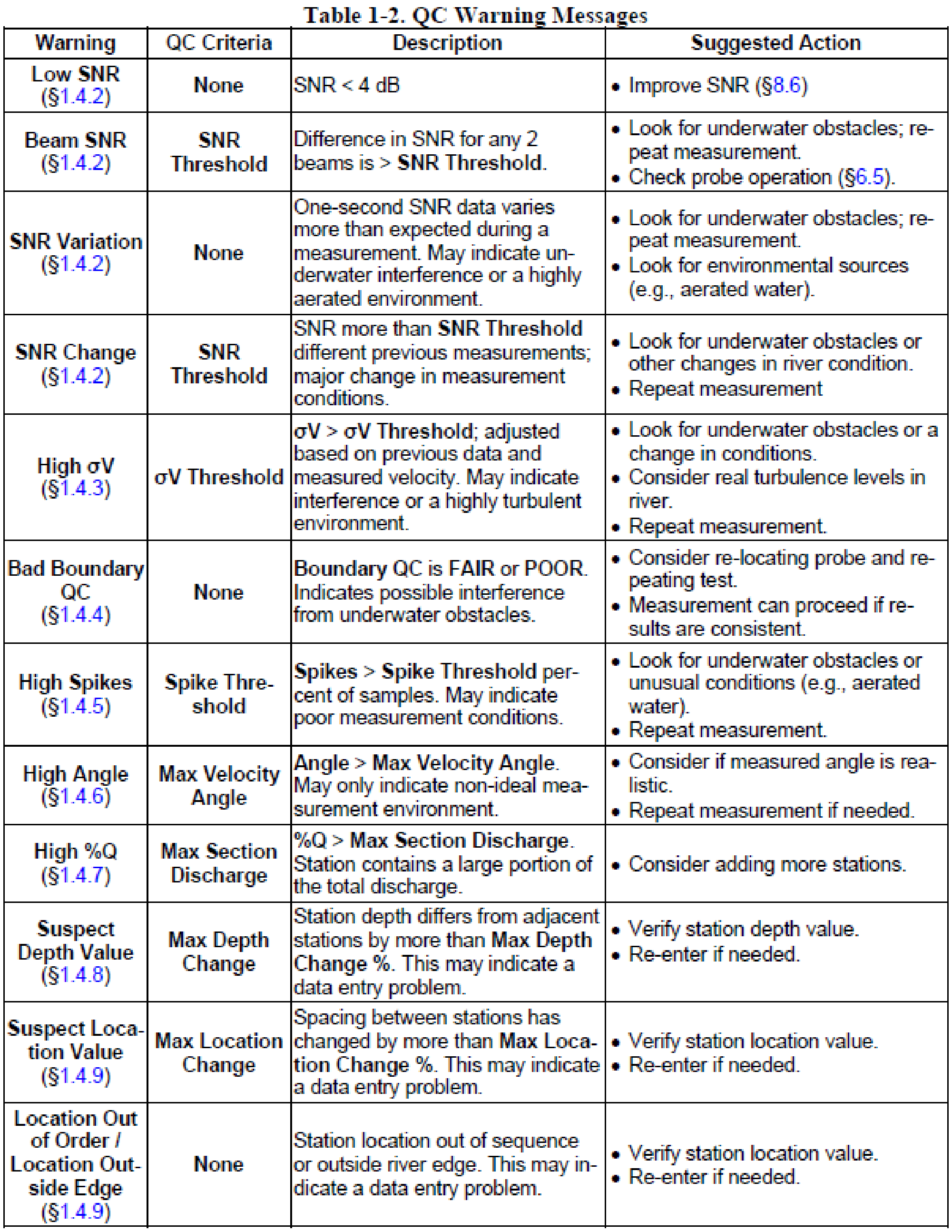


Table 1-2. QC Warning Messages

Warning	QC Criteria	Description	Suggested Action
Low SNR (§1.4.2)	None	SNR < 4 dB	• Improve SNR (§8.6)
Beam SNR (§1.4.2)	SNR Threshold	Difference in SNR for any 2 beams is > **SNR Threshold**.	• Look for underwater obstacles; repeat measurement. • Check probe operation (§6.5).
SNR Variation (§1.4.2)	None	One-second SNR data varies more than expected during a measurement. May indicate underwater interference or a highly aerated environment.	• Look for underwater obstacles; repeat measurement. • Look for environmental sources (e.g., aerated water).
SNR Change (§1.4.2)	SNR Threshold	SNR more than **SNR Threshold** different previous measurements; major change in measurement conditions.	• Look for underwater obstacles or other changes in river condition. • Repeat measurement
High σV (§1.4.3)	σV Threshold	**σV** > **σV Threshold**; adjusted based on previous data and measured velocity. May indicate interference or a highly turbulent environment.	• Look for underwater obstacles or a change in conditions. • Consider real turbulence levels in river. • Repeat measurement.
Bad Boundary QC (§1.4.4)	None	**Boundary** QC is **FAIR** or **POOR**. Indicates possible interference from underwater obstacles.	• Consider re-locating probe and repeating test. • Measurement can proceed if results are consistent.
High Spikes (§1.4.5)	Spike Threshold	**Spikes** > **Spike Threshold** percent of samples. May indicate poor measurement conditions.	• Look for underwater obstacles or unusual conditions (e.g., aerated water). • Repeat measurement.
High Angle (§1.4.6)	Max Velocity Angle	**Angle** > **Max Velocity Angle**. May only indicate non-ideal measurement environment.	• Consider if measured angle is realistic. • Repeat measurement if needed.
High %Q (§1.4.7)	Max Section Discharge	**%Q** > **Max Section Discharge**. Station contains a large portion of the total discharge.	• Consider adding more stations.
Suspect Depth Value (§1.4.8)	Max Depth Change	Station depth differs from adjacent stations by more than **Max Depth Change %**. This may indicate a data entry problem.	• Verify station depth value. • Re-enter if needed.
Suspect Location Value (§1.4.9)	Max Location Change	Spacing between stations has changed by more than **Max Location Change %**. This may indicate a data entry problem.	• Verify station location value. • Re-enter if needed.
Location Out of Order / Location Outside Edge (§1.4.9)	None	Station location out of sequence or outside river edge. This may indicate a data entry problem.	• Verify station location value. • Re-enter if needed.

5.2 Signal-to-Noise Ratio (SNR)

SNR is a measure of the strength of the reflected acoustic signal relative to the ambient noise level of the FlowTracker. SNR is the most important QC data provided by the FlowTracker. An error may indicate interference from an underwater obstacle or a potential problem with the probe. At the first alert, repeat the measurement (perhaps after moving probe location). Some errors are unavoidable. Field conditions, such as turbulent water, may affect SNR and are unavoidable.

- SNR is reported in logarithmic units (dB). It is recorded with each one-second velocity sample. Mean values are recorded for each measurement location.
- For the best operating conditions, SNR should be greater than 10 dB.
- The FlowTracker can operate reliably with SNR as low as 4 dB, although the noise in individual measurements will increase.
- The FlowTracker displays an alert at the end of a measurement if SNR of any beam is <4.0 dB. SNR data are displayed during the measurement and with the measurement summary. For 2D systems, if the SNR of either beam is low, this will affect all velocity data even if the other beam shows higher SNR values.

Low SNR indicates a lack of suspended material in the water. For clear water, seeding material can be introduced to increase SNR. Seeding is typically only required in large laboratory tanks. Most field applications have sufficient natural scattering material.

The SNR data shown during data collection is the mean of the primary receivers (depending on the operating mode of the FlowTracker – either General or Discharge). Individual receiver data can be accessed after downloading the data file to a PC.

If the problem persists, run *BeamCheck* from a PC to evaluate FlowTracker operation in more detail. When the variation of SNR during a measurement (as measured by the standard deviation SNR data) is greater than a fixed threshold of 5 dB:

- This may indicate interference from an underwater obstacle, a highly turbulent environment, or highly aerated water.
- At the first alert, repeat the measurement (perhaps after moving probe location).
- If the problem persists, evaluate the measurement environment. In some cases, large variations may be unavoidable and may not impact the quality of velocity data.

SNR Threshold is checked with each measurement and when the **End Section** key is pressed in **Discharge** mode.

- You are notified of any stations that exceed the above criteria.
- If desired, you can go back and delete suspect stations and repeat the measurements.

5.3 Standard Error of Velocity (σV)

Standard error of velocity (**σV**) is a direct measure of the accuracy of the mean velocity data.

σV can be directly interpreted as the accuracy of the mean velocity.

σV is calculated by dividing the standard deviation of one-second samples by the square root of the number of samples.

σV is normally dominated by real variations in the flow and will vary depending on the measurement environment. When **σVx** is greater than the standard error threshold for that measurement:

- This may indicate interference from an underwater obstacle, a highly turbulent environment, or highly aerated water.
- At the first alert, repeat the measurement (perhaps after moving probe location).
- If the problem persists, evaluate the measurement environment. In some cases, large variations may be unavoidable (e.g., in highly turbulent waters).

σV Threshold is checked with each measurement and when the **End Section** key is pressed in **Discharge** mode.

- You are notified of any stations that exceed the above criteria.
- If desired, you can go back and delete suspect stations and repeat the measurements.

5.4 Boundary Adjustment (Boundary QC)

The FlowTracker has a potential for acoustic interference from underwater objects. The **Boundary QC** test looks for interference with underwater objects that are in or close to the FlowTracker sampling volume. The system tries to avoid this interference, but you must be aware of system limitations.

- Reflections can occur from the bottom, the water surface, or submerged objects (e.g., rocks).
- If the sampling volume is on top of or beyond an underwater object, velocity data will be meaningless.
- When working in very shallow water or near underwater obstacles (with the sampling volume within 15 cm (6 in) of the obstacle), acoustic reflections can potentially affect velocity data.
- At each measurement location, the FlowTracker looks for these conditions, and if necessary, adapts its operation to avoid interference.
- For most locations, any required changes do not affect system performance.
- In some environments, changes may result in a lower maximum velocity. The FlowTracker records any changes required to avoid acoustic interference. It reports this as **Boundary QC**. This value describes the effect (if any) of boundary adjustments on performance.

The **Boundary QC** variable (**Bnd**) can have the following values (0 and 1 are the most common).

0 (BEST): No boundary adjustments were necessary, or if necessary, they have minimal impact on system performance. Maximum velocity is at least 3.5 m/s (11 ft/s).
1 (GOOD): Minor boundary adjustments were necessary with moderate impact on system performance. Maximum velocity is at least 2.5 m/s (8 ft/s).

2 (FAIR): Larger boundary adjustments were necessary with notable impact on system performance. Maximum velocity is at least 1.2 m/s (4 ft/s).

3 (POOR): Major boundary adjustments were necessary with significant impact on system performance. Maximum velocity is less than 1.2 m/s (4 ft/s). The FlowTracker will still provide good performance for lower flows.

If the **Boundary QC** results are **FAIR** or **POOR**, this indicates possible interference, and the FlowTracker will issue an alert before the measurement is made. You are prompted to consider moving the probe to avoid this interference. If the probe is moved, repeat the boundary test prior to data collection. If repeated **Boundary QC** tests do not give improved results, you can proceed with the measurement but should carefully evaluate velocity data.

5.5 Spike Filtering

Spikes in velocity data occur with any acoustic Doppler velocity sensor such as the FlowTracker. Spikes may have a variety of causes – large particles, air bubbles, or acoustic anomalies.

- Velocity data from each FlowTracker measurement are evaluated to look for spikes.
- The number of spikes is displayed and recorded at the end of each measurement.
- The number of velocity spikes present in data is evaluated as follows.

The **Spike Threshold** (default 10%) is used as follows.

- If the number of spikes is a greater percentage of the total number of points than specified by the **Spike Threshold**, a warning is given.
- This may indicate interference from an underwater obstacle, a highly turbulent environment, or highly aerated water.
- At the first alert, repeat the measurement (perhaps after moving probe location).
- If the problem persists, evaluate the measurement environment. A large number of spikes may be unavoidable but may not overly impact the quality of velocity data.

Spike Threshold is checked with each measurement and when the **End Section** key is pressed in **Discharge** mode.

- You are notified of any stations that exceed the above criteria.
- If desired, you can go back and delete suspect stations and repeat the measurements.

5.6 Velocity Angle

In **Discharge** mode, an additional QC parameter is provided – *velocity angle*. Velocity angle is defined as the direction of flow relative to the upstream direction. The measured velocity angle is evaluated to ensure reliable data collection.
For an ideal discharge measurement site, flow should be perpendicular to the tag line used to define the cross section.

- The FlowTracker's X-axis is always held perpendicular to the tag line.
- An angle of 0 degrees means flow direction is perpendicular to the tag line (as desired for an ideal measurement location).
- A good measurement site will typically show some flow variations but with all angles less than about 20-30 degrees.
- The **Max Angle** criterion (default 20º) is used as follows:

    - When measured angle is greater than **Max Angle**, a warning is given.
    - Evaluate the measurement site to verify the measured angle is reasonable.
    - Consider repeating the measurement if the angle does not appear reasonable (perhaps after moving probe location).

Max Angle is checked with each measurement and when the **End Section** key is pressed.

- You are notified of any stations that exceed the above criterion.
- If desired, you can go back and delete suspect stations and repeat the measurements.

5.7 Maximum Section Discharge

Most agencies monitoring discharge expect that no individual station should contain more than a certain percentage of the total discharge. The **Max Sect. Q** criterion (default 10%) alerts you if this standard is exceeded.

If the station discharge exceeds **Max Sect. Q** percent of the rated flow, a warning is issued, and you are prompted to consider adding another station on either side of the station in exceeding the 10% rule.

Max Sect. Q is also checked when the **End Section** key is pressed.

- You are warned if any station exceeds **Max Sect. Q** percent of total measured discharge.
- **If warned, you must go back and add more stations!**
- Add stations on each side of the station with the Maximum Section Discharge warning.

5.8 Maximum Depth Change

The **Max Depth Change** criterion (default 50%) is intended to avoid data entry errors. It is assumed that depth changes between stations will be gradual. If the entered depth is different from a reference by more than **Max Depth Change** (and at least 0.20 m; 0.66 ft), an alert is issued to be sure the depth was not incorrectly entered.

- If only the previous station is available, the newly entered depth is compared to the depth from the previous station.
- If depth data are available on both sides of this station, the newly entered depth is compared to an interpolated depth between the two adjacent stations.

- You are prompted to verify the depth value or re-enter the depth.
- This criterion can be adjusted or disabled by setting **Max Depth Change** to 0.

Max Depth Change is also checked when the **End Section** key is pressed. You are notified of any stations that exceed the **Max Depth Change** criterion and are given the option to review all stations and modify any incorrectly entered data.

5.9 Maximum Location Change

The **Max Location Change** criterion (default 100%) is intended to avoid data entry errors.
It is assumed that the spacing of adjacent stations will be nearly constant across the river.
If spacing between stations has changed by more than **Max Location Change**, an alert is issued to be sure the location was not incorrectly entered.

- A 100% **Max Location Change** means the new station spacing is more than two times the previous station spacing. Any time a station location is changed, the location is compared to adjacent value(s) to see if the station is out of order. Collecting an out-of-order station is allowed. However, when an out-of-order station is entered, we verify the location value since the station is sorted into the correct place within the stream.

Any time a station location is changed, the location is compared to the starting edge location. If the new location is outside the starting edge, a warning is given.

6.0 BATTERY POWER

The FlowTracker uses eight AA batteries (alkaline, NiMH, or NiCad).

	Alkaline	NiMH (rechargeable)	NiCad (rechargeable)
New battery voltage	12.0 V	10.2 V	10.0 V
Approximate operating life	25 hours	15 hours	7 hours

To check FlowTracker battery level and estimated battery capacity:

- Press 5 from the **System Functions Menu**. Battery life estimates are at approximately 20°C (70°F). Cold weather greatly reduces battery capacity. Check battery capacity with the system acclimated to the outside temperature.

The batteries are accessed from the back of the FlowTracker handheld controller (Figure 1.0-1). To change the batteries, use the following steps:

- Turn the system off.
- Remove the six screws holding the battery compartment lid to the main housing.
- Remove the old batteries from the battery holder.
- Install the new batteries matching the orientation shown on the battery holder.
- Do not mix old and new batteries.

- Do not mix different types of batteries.
- Secure the battery compartment lid using the six screws.
- Turn the system on and check the battery voltage level to ensure proper installation.

To avoid draining batteries when system is not in use, always turn the system off before storing the system.

If the system will not be used for more than one month, remove the batteries

7.0 HEALTH AND SAFETY

When working in potentially hazardous situations, personnel must understand and comply with the site-specific Sampling and Analysis Plan/Quality Assurance Project Plan and Health and Safety Plan before the sampling event begins. More specifically, when entering a stream, hazardous situations exist that warrant the person performing the measurements wear adequate safety equipment, including a personal floatation device and waders with slip-resistant footwear.

8.0 REFERENCES

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Water Quality Measurements with the In-Situ® Multi-Parameter Meter

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This document has been prepared for the Environmental Protection Agency by the TechLaw, Inc. ESAT Region 8 Team and is intended to provide documentation of administrative, analytical and quality control procedures used in the daily performance of EPA and ESAT support services.

TABLE OF CONTENTS

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to document best practices in the use of the In-Situ® multi parameter water quality meter. For optimal performance, refer to the In-Situ® Multi-Parameter Troll® 9500 Operator's Manual and the Rugged-Reader® Operator's Manual.

2.0 SCOPE AND APPLICABILITY

This SOP is applicable only for In-Situ® water quality meters that are currently in use by United States Environmental Protection Agency (EPA) and Environmental Services Assistance Team (ESAT) field personnel. Water quality parameters may include pH, dissolved oxygen (DO), specific conductance, and temperature readings.

3.0 SUMMARY OF METHOD

Water quality measurements are a critical component of field sampling activities. Water quality meters are versatile tools for various measurements of water quality. For field sampling purposes, water quality meters are calibrated on site before use to account for barometric pressure. The main parameters for surface water quality are pH, DO, specific conductance, and temperature. These parameters are built in to the In-Situ® Rugged Reader® Troll® device and it's Pocket Situ 4 Software. Once calibrated, sampling teams have up to 24-hours to record water quality readings if barometric pressure conditions remain constant (see section 11.2). Data is recorded in a site-dedicated field logbook.

4.0 ACRONYMS AND DEFINITIONS

mg/L	milligram/liter
mL	milliliter
µS/cm	microsiemens/centimeter
DO	Dissolved Oxygen
EPA	United States Environmental Protection Agency
ESAT	Environmental Services Assistance Team
GPS	Global Positioning System
HASP	Health and Safety Plan
HAZWOPER	Hazardous Waste Operations and Emergency Response Standard
OSHA	Occupational Safety and Health Administration
PPE	Personal Protective Equipment
SAP/QAPP	Sampling and Analysis Plan/Quality Assurance Project Plan
SOP	Standard Operating Procedure

Health and Safety Plan (HASP): A site-specific plan that outlines safety hazards and hazard mitigation practices.

Sampling and Analysis Plan/Quality Assurance Project Plan (SAP/QAPP): Site-specific documents that outline sampling and data quality objectives for a field sampling project.

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/Quality Assurance Project Plan (SAP/QAPP) and Health and Safety Plan (HASP) before the sampling event begins. When taking water quality readings in streams or surface impoundments containing known or suspected hazardous substances, adequate personal protective equipment (PPE) such as nitrile gloves, safety glasses, and waders, are necessary to prevent exposure. If entering a stream, safety equipment, including a personal floatation device and non-slip footwear should be worn in addition to general PPE. When taking measurements from a vessel in an impoundment or flowing waters, appropriate safety procedures and practices should be followed.

6.0 CAUTIONS

To ensure safe and reliable operation of the equipment, the manufacturer's directions for transport, cleaning, decontamination, storage, and operation should be followed. See the operations manuals for additional guidance (Multi-parameter Troll® 9550 and the Rugged Reader®)

7.0 INTERFERENCES

Improper calibration of the instrument can lead to erroneous readings or equipment malfunction. For each calibration standard, be sure to condition the instrument/probe with a rinse of the appropriate standard before calibration. Additionally, the hand held Rugged-Reader® unit should be fully charged before deployment to the field. If the unit fails to recognize the probe, make sure to check the batteries located in the upper part of the probe just below the interface cable connection. Note that batteries do not operate optimally in cold weather conditions.

8.0 PERSONNEL QUALIFICATIONS

Any personnel involved with field sampling activities must be cleared for participation by their organization's health and safety officer. Clearance includes medical monitoring, respirator fit testing, and Occupational Safety and Health Administration (OSHA) Hazardous Waste Operations and Emergency Response Standard (HAZWOPER) 40-hour training. Personnel who will be collecting surface water quality data must be familiar with this and any other relevant SOPs, including the Sample Equipment Decontamination SOP FLD 02.00, Sample Preservation SOP FLD 03.00, Surface Water Sampling SOP FLD 01.00, Sample Custody and Labeling SOP FLD 11.00, and General Field Sampling Protocols SOP FLD 12.00.

9.0 EQUIPMENT AND SUPPLIES

Equipment needed for collection of surface water quality data may include:

<u>Water quality collection device</u> – Multi-parameter Troll® 9550 and the Rugged-Reader®

<u>PPE</u> - personal floatation device, waders, gloves, proper footwear, safety glasses, insulating clothing for cold water, 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 – field logbook or field data sheet(s)

Calibration standards - see Section 10.0, Standards and Reagents

10.0 STANDARDS AND REAGENTS

Certified standards are required for calibration of the Rugged Reader® In-Situ® water quality multi-parameter meter. pH calibration can be done at one to three points using buffer solutions at pH 4, 7, or 10. Specific conductance is calibrated with one point, usually at 1000 µS/cm. In addition to certified standards, it is best practice to also have spent standard, referred to as rinse, to use as probe conditioner in order to equilibrate the sensor. Refer to the Sampling Equipment Decontamination SOP FLD 02.00.

11.0 PROCEDURES

Commonly used procedures for the use of the Rugged Reader® In-Situ® water quality meter are provided in the following sections. Note that this equipment has many other functions, but only the most frequently used procedures for EPA Region 8 field activities will be discussed. For further information on potential uses and capabilities, consult the Rugged-Reader® and Troll® 9550 Operations Manuals.

11.1 Pre-Deployment Activities

Before field deployment, it is important to check the equipment for functionality. The batteries to the Rugged Reader® should be charged at least 24 hours prior to use. The Troll® 9500 requires two D-cell batteries. Calibration frequency varies between sensor types on the multi-parameter meter and environmental conditions. Consult calibration frequency requirements mandated by sampling event protocols or project data quality objectives. It is recommended that water quality sensors be stored, calibrated, and maintained in accordance with the In-Situ® multi-parameter Troll® 9500 operations manual. Sufficient time for thermal stabilization of standards to ambient conditions should be considered before calibration. To reduce the time for stabilization, all calibration standards and the In-Situ® equipment should be stored at the same temperature before starting calibration.

11.2 Field Water Quality Measurements – Calibration

Many sampling sites in EPA Region 8 are located at various elevations with differing barometric pressures, and as a result, re-calibration of the water quality sensors will occur at a greater frequency than manufacturer recommendations. Upon arrival on a site, it is recommended that a 500mL bottle of tap water be used to begin aeration of the optical DO sensor. Specific conductance and pH calibration can occur while the water aerates. In general, most mine effluent sites have pH values between 4.0 and 7.0; therefore, only a 2-point calibration with 4.0 and 7.0 pH buffer solutions is necessary. If deployed to a site where pH values are completely unknown, perform a 3-point calibration (adding the 10.0 calibration). Below are calibration procedures for pH, specific conductance, and DO measurements.

11.2.1 pH Calibration

Connect the Troll® 9550 to the Rugged-Reader® with the interface cable and power on the unit. From the starting screen, tap **Start** (located on the upper right corner of the screen) with the stylus. The Start menu is the main access point to all programs, files, and settings loaded on the Rugged-Reader®. If this is the first time the Troll® 9550 is connected to the Rugged-Reader®, follow the instructions for using the Connection Wizard in the Rugged-Reader® Operations Manual (for subsequent uses, tap on the COM1-19200 line in the navigation window). If the Troll® 9550 is not recognized in the navigation window you may have to 'find' the device. Do this by tapping the COM port (labeled as COM1-19200) and then tap **Find** located in the lower right hand corner. The information for the Troll® 9550 should appear in the Rugged-Reader® navigation screen. If there is no connection to the Troll® 9550, check cable connections and/or battery status. Once the Troll® 9550 connection appears as a line in the navigation window, tap to open the **Parameters** option. Tap this twice in order to bring up the sensors that are currently connected to the probe (this should always be pH, specific conductance, DO, temperature, and barometric pressure). From this menu, tap **pH**. Three options will appear at the bottom of the screen, select **Calibrate**.

1. This will bring up the calibration setup menu. Select number of calibration points desired (usually 2). Select **Next**.
2. Start the pH calibration with the lowest standard. Use the pH 4.0 standard rinse to condition the sensor, empty the cup, and then place certified pH 4.0 standard in the calibration cup up to the fill level line. Press **Run**.
3. The sensors will take a few moments to stabilize for temperature and pH. Once stabilized, the screen will immediately prompt for the next standard. Follow the same procedure by first conditioning the sensor with the pH standard rinse, then fill the calibration cup with the certified standard. Press **Run** again.
4. Once the final calibration has stabilized, the user will be given the option to view the calibration report. Click **Yes** to view the report and record the final calibration results in the instrument-dedicated calibration logbook.

11.2.2 Specific Conductance Calibration

To calibrate for specific conductance, select the **Conductivity** option from the parameters menu by tapping it once, then select **Calibrate** by tapping it once. This will bring up the calibration setup menu.

1. This menu allows the user to select the standard intended for use. If the standard being used does not appear as one of the options, select **Other** to input the desired standard. Tap **Next**.
2. Condition the sensors by saturating with specific conductance rinse of similar concentration, and then fill the calibration cup with the certified standard. Make sure to fill the cup all the way to the fill line.
3. Once the calibration is stabilized, the user is given the option to view the calibration reports. Select **Yes** and record the new calibration results in the instrument-dedicated calibration logbook.

11.2.3 Dissolved Oxygen Calibration

Finally, the optical DO sensor should be calibrated. If the other parameters were calibrated first, there should have been sufficient time to saturate with oxygen (10-15 minutes). From the parameters menu, tap once on the **Rugged Dissolved Oxygen** icon, then once again on the calibration option at the bottom of the screen.

1. The first screen allows the user to edit the barometric setting. Select **Yes**.
2. The next screen will give the user options as to how the barometric setting should be calculated into the DO calibration. Select the **Use Vented Cable** option, and then tap **OK**.
3. The following screen gives the user the choice of 1) restoring default settings and calibrating, 2) restoring defaults and not calibrating, and 3) calibration only. Select the **Calibrate Only** option, and then tap **Next**.
4. Place the DO probe in the saturated tap water, then tap the **Run** button. The sensor will take a few moments to equilibrate for temperature and DO.
5. Once stabilized, the unit will prompt for a zero oxygen calibration. To skip this, simply tap the **Next** button. To finish the calibration, tap the **Finish** button.
6. The user will be given the option to see the calibration reports. Select **Yes** and record the calibration information in the instrument dedicated logbook.

11.3 Field Water Quality Measurements – Data Collection

Once calibration is complete, the user will be returned to the parameters screen. To begin collecting water quality data, tap the **Parameters** icon once, then the **Profiler** option once. The next screen will ask the user to assign a test name. Enable the keyboard icon on the bottom of the screen and type in the Location ID for the site that is to be sampled. Once finished entering the Test Name, tap **OK**. The screen will then display temperature, pH, specific conductance, DO, and barometric pressure data. Tap the icon on the bottom right of the screen that says **Continuous**. Allow several minutes for the readings to stabilize. Note: make sure the readings are within a certain range of expectation. Refer to previous readings at the site in the logbook to verify correct data ranges. Once the readings have stabilized, tap the icon **Stop Log**. Before exiting out of the screen, record the readings in a project dedicated field logbook. Data should be recorded in the following units for each parameter: pH, standard units; DO, mg/L; specific conductance, µS/cm; and temperature, °C. Once the data has been recorded in the logbook, tap the icon **Close** on the bottom right of the screen. To begin collecting data at a different site, repeat these steps found here in section 11.3.

11.4 Troubleshooting

When problems are noted during the calibration procedure, check the following:

1. Make sure the parameter shown on the screen is the same parameter to which the unit is being calibrated.
2. Make sure the proper standard is being used for its corresponding calibration value (i.e. make sure not to calibrate specific conductance with a pH standard).
3. Verify that the standards used have not been contaminated.
4. Make sure the equipment and solutions are at the correct temperature for the

calibration being performed.

5. Be sure to fill the calibration cup according to directions in the Troll® 9550 operations manual.
6. Make sure the pH reference electrode was filled with the proper pH filling solution.
7. Check the multi-probe housing and sensors for physical damage (cracked or bent electrodes) and fouling (tarnished, soiled, color change, or otherwise coated electrodes).
8. Check the multi-probe battery status. Recharge the unit if there are only one or two bars showing in the battery icon in the opening screen. There is also a battery voltage indicator under the parameters screen in the Pocket-Situ 4 software. If the Troll® 9550 batteries are running low, quit the Pocket Situ 4 software and replace the D-cell batteries.
9. If the above troubleshooting tips and maintenance procedures do not solve the problem, contact In-situ® technical support at 1-970-498-1500 or toll free at 1-800-466-1500 (Option 3).
10. If problems persist, refer to the In-situ® operator's manual. All In-situ® cases will include a copy of the operator's menu.

12.0 DATA RECORDS AND MANAGEMENT

All data and measurements must be recorded in the site-dedicated field logbooks. This data will be entered into a spreadsheet and published in Scribe. Any recording errors should be struck out with a single line through the incorrect value, initialed by the recorder, and then re-recorded with the correct value. All calibration or repair data should be recorded in the equipment-dedicated field logbook.

13.0 QUALITY CONTROL AND ASSURANCE

EPA and ESAT Region 8 personnel who use these instruments will calibrate the In-situ® each day before use and will perform maintenance procedures as needed. If personnel feel uncertain about the quality of measurement data in the field, performance of the instrument will be checked using a known calibration standard. Corrective actions will be determined on a case by case basis with all circumstances being considered. Data will be flagged as needed in the final data package.

14.0 REFERENCES

EPA Guidance for Preparing Standard Operating Procedures, EPA QA/G-6, April 2007.

In-Situ, Inc. Multi-Parameter TROLL® 9500 Operator's Manual. Rev. 007, January 2009.

In-Situ, Inc. Rugged Reader® Operator's Manual. October 2007.

Pore Water Sampling

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TABLE OF CONTENTS

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to describe the procedures, methods, and considerations to be used when obtaining a pore water sample using PushPoint® samplers.

2.0 SCOPE AND APPLICABILITY

This document describes procedures for pore water sampling using PushPoint® samplers and is based on the Operators Manual and Applications Guide provided by MHE Products (Ver. 2.01,2/15). It is intended to be used by field personnel when collecting and handling samples in the field. All deviations from this SOP must be noted in the site-dedicated field logbook.

3.0 SUMMARY OF METHOD

Sediment pore water is collected using a pore water extractor, called a PushPoint® (Figure 1) which is made out of stainless steel tubing developed by MHE Products. The sampling end of the PushPoint® is inserted into the sediment to the desired depth, and pore water is extracted using a syringe or peristaltic pump.

4.0 ACRONYMS AND DEFINITIONS

COC	Chain of Custody
DOC	Dissolved Organic Compounds
DOT	United States Department of Transportation
DQO	Data Quality Objective
EPA	United States Environmental Protection Agency
ESAT	Environmental Services Assistance Team
HASP	Health and Safety Plan
HAZWOPER	Hazardous Waste Operations and Emergency Response
IATA	International Air Transportation Association
OSHA	Occupational Safety and Health Administration
QA/QC	Quality Assurance/Quality Control
QAPP	Quality Assurance Project Plan
SAP	Sampling and Analysis Plan
SOP	Standard Operating Procedure
SVOC	Semi-Volatile Organic Compound
VOA	Volatile Organic Analysis
VOC	Volatile Organic Compound

Health and Safety Plan (HASP): A site-specific document outlining potential safety hazards and hazard mitigation techniques.

Occupational Safety and Health Administration (OSHA): An agency that regulates 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).

5.0 HEALTH AND SAFETY

Proper safety precautions must be observed when collecting pore water samples. Refer to Environmental Services Assistance Team (ESAT) site-specific Health and Safety Plans (HASPs) for guidelines on safety precautions. These guidelines, however, should only be used to complement the judgment of an experienced professional. When following this SOP, minimize exposure to potential health hazards in the field by using personal protective equipment (protective clothing, eye wear and gloves). Address chemicals that pose specific toxicity or safety concerns and follow any other relevant requirements, as appropriate.

6.0 SAMPLE MANAGEMENT

The following precautions should be considered when collecting pore-water samples:

- Special care must be taken not to contaminate samples. This includes storing samples in a secure location to preclude conditions which could alter the properties of the sample. Samples shall be custody sealed during long-term storage or shipment.
- Collected samples are in the custody of the sampler or sample custodian until the samples are relinquished to another party (see SOP FLD-11.00, or current version, “Sample Custody and Labeling”).
- If samples are transported by the sampler, they will remain under his/her custody or be secured until they are relinquished.
- Shipped samples must conform to all United States Department of Transportation (DOT) and/or International Air Transportation Association (IATA) hazardous materials shipping requirements.
- Documentation of field sampling is done in a bound logbook.
- Chain of custody (COC) documents must be filled out and remain with the samples until custody is relinquished until analysis is complete (or samples are disposed).
- All shipping documents, such as bills of lading, etc., shall be retained by the project leader and stored in a secure place.

7.0 INTERFERENCES

The following sections describe potential interferences when sampling for trace level contaminants. For decontamination procedures, see the Sampling Equipment Decontamination SOP FLD 02.00.

7.1 Potential Volatile Organic Analysis (VOA) Sampling Interferences

Pore water samples for volatile organic compound (VOC) and semi-volatile organic compounds (SVOC) analysis must be collected in 40-ml amber glass vials with Teflon® septa. The vials may be preserved with concentrated hydrochloric acid or they may be unpreserved. Preserved samples have a two week holding time, whereas, unpreserved samples have only a seven day holding time. Normally, either preserved or unpreserved vials can be used, but there are instances where the use of unpreserved vials is preferred. For example, if the surface water sample contains a high concentration of dissolved calcium carbonate, there may be an effervescent reaction between the hydrochloric acid and the water, producing large numbers of fine bubbles. This will render the sample unacceptable. In this case, unpreserved vials should be used and arrangements must be confirmed with the laboratory to ensure that they can accept the unpreserved vials and meet the shorter sample holding times.

Samples for VOC and SVOC analysis must be collected using either stainless steel or Teflon® equipment. Samples should be collected with as little agitation or disturbance as possible. The vial should be filled so that there is a meniscus at the top of the vial, and absolutely no bubbles or headspace should be present in the vial after it is capped. After the cap is securely tightened, the vial should be inverted and tapped on the palm of one hand to see if any undetected bubbles are dislodged. If a bubble or bubbles are present, the vial should be refilled. Care should be taken not to flush any preservative out of the vial during topping off. If bubbles are still present after attempting to refill and cap the vial, a new vial should be obtained and the sample re-collected.

7.2 Potential Dissolved Metals or Dissolved Organic Compound Sampling Interferences

If a dissolved metals or Dissolved Organic Compounds (DOC) pore water sample is to be collected, in-line filtration or post-collection filtrations are acceptable approaches. The in-line filter apparatus uses disposable, high capacity filter cartridges (barrel-type) or membrane to filter the sample. The high capacity, barrel-type filter works well due to the higher surface area associated with this configuration. Post-collection filtration involves two approaches. The first approach is to take the sample water and filter it through a .45 micron filter apparatus. The second approach involves the use of a syringe with a .45 micron acrodisc filter attached to end of syringe.

Potential differences could result from variations in filtration procedures used to process water samples for the determination of trace element concentrations. A number of factors associated with filtration can substantially alter "dissolved" trace element concentrations, including filter pore size, filter type, filter diameter, filtration method, volume of sample processed, suspended sediment concentration, suspended sediment grain-size distribution, concentration of colloids and colloidally-associated trace elements, and concentration of organic matter. Therefore, consistency of sample technique and filter characteristics is critical in the comparison of short-term and long-term results.

7.3 Special Precautions for Trace Contaminant Pore Water Sampling

1. A clean pair of new, non-powdered, disposable gloves will be worn each time a different location is sampled, and the gloves should be donned prior to handling sampling equipment and sampling. The gloves should not come in contact with the media being sampled and should be changed any time during sample collection when their cleanliness is compromised.

2. Sample containers for samples suspected of containing high concentrations of contaminants shall be stored separately from samples suspected of only having trace levels of contaminants.

3. All background or control samples shall be collected and placed in separate ice chests or shipping containers. Sample collection activities shall proceed progressively from the least suspected contaminated area to the most suspected contaminated area. Samples of waste or highly contaminated media must not be placed in the same ice chest as environmental (i.e., containing low contaminant levels) or background samples.

4. Samplers must use new, verified, certified clean disposable equipment, or pre-cleaned non-disposable equipment.

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 pore water samples must be familiar with this SOP and any other relevant SOPs, including the Sample Equipment Decontamination SOP FLD 02.00, Sample Preservation SOP FLD 03.00, Water Quality Measurements with the In-Situ® Multi-Parameter Meter SOP FLD 09.00, Sample and Labeling SOP FLD 11.00, and General Field Sampling Protocols SOP FLD 12.00.

9.0 EQUIPMENT AND SUPPLIES

The PushPoint® sampler consists of a tubular 3/16 stainless steel body with a screened zone at one end and a sampling port at the other. It comes with a guard rod that is nested in the tube during deployment in order to provide structural support and to prevent plugging and deformation of the screened zone (Figure 1). The screened zone consists of a series of interlaced machined slots that form a short screened zone with approximately 20% open area. Additional filters can be placed over the screened zone if additional screening is needed. Pore water is collected through the opposite end of the PushPoint® sampler through peristaltic flexible tubing using a syringe through the sampling port. Tygon® tubing is the preferred tubing to be used with PushPoint® samplers. However, other tubing can be used, if allowed by data quality objectives (DQOs) for the specific application. PushPoint® samplers can be custom made to any width or length.

There are many modifications that can be incorporated into the procedure to satisfy DQOs for a specific application. The procedures discussed in the following sections provide guidance on the basic operation of the PushPoint® and issues to consider when deploying the PushPoint® sampler to collect pore water.

Other equipment used in the process of pore water sample collection includes syringes, flanges, tubing, sample bottles or containers, and filters.

10.0 STANDARDS AND REAGENTS

Reagents will be used for preserving samples and for decontaminating sampling equipment (refer to the Sampling Equipment Decontamination SOP FLD 02.00 and Sample Preservation SOP FLD 03.00). The preservatives required are determined by the analysis to be performed and will be specified in the Sampling and Analysis Plan (SAP)/Quality Assurance Project Plan (QAPP), but usually include nitric acid (total recoverable and dissolved metals samples), hydrochloric acid (VOC samples), and phosphoric acid (DOC samples). The sampler should also be aware of any special sampling considerations, contamination issues, and sample compositing and mixing methods that could affect their sampling efforts. Appropriate regional guidance and procedures should be consulted for detailed sample collection, preservation, handling and storing, equipment decontamination, and quality assurance/quality control (QA/QC) procedures. The sampler should preserve and immediately cool all water samples to 4°C (±2°C) upon collection and samples should remain cooled until the time of analysis (do not freeze water samples).

11.0 PROCEDURES

It is critical in the collection of pore water to avoid surface water intrusion. Water will flow in a path of least resistance. If space is created around the sides of the PushPoint® sampler during deployment, surface water may flow down the outside of the tube to the screened area and into the intended sample. Therefore, the PushPoint® can be used with a sampling platform or flange (Figure 2), especially when collecting pore water near the sediment-surface water interface. However, if pore water is collected from deep in the sediments or in cobble-bottom streams, a flange may not be necessary. Additionally, it is important to note that a platform is only useful in specific situations when you are sampling multiple holes and specific depths and when sampling at shallow depths where the integrity of the hole may be a concern. It is critically important to collect samples from the hyporheic zone, or the area beneath the streambed where shallow groundwater is mixed with surface water (this area is critical to benthic macro-invertebrates and fish spawning activity). When inserted though the sampling platform, or flange, the flange should fit securely around the PushPoint® to eliminate surface water intrusion from around the PushPoint® body during sample collection.

The flange can be made of any material that will not cross contaminate the intended sample. If full scan analytical analysis is required, the flange should be made of inert material such as stainless steel or Teflon®. The size of the flange depends on the volume of pore water to be collected. If large volumes of pore water are to be collected, use a large flange size. If it is not practical to use a large flange, then multiple PushPoints® with smaller flanges can be deployed and smaller volumes can be collected from several PushPoints® for a composite sample. If multiple PushPoints® are deployed, they should be spaced at least 30 cm apart.

11.1 PushPoint® Sampler Basic Operation

The PushPoint® sampler should be inserted into the sediment as carefully as possible (Figure 2). When deploying the PushPoint®, care must be taken not to disturb the sampling area. If the sampler is wading in the water body, the sampler should lean out and insert the PushPoint® as far as possible away from where he/she is standing in order to reduce potential effects of the sampler on the integrity of the pore water sample. Depth of penetration of the PushPoint® into the sediment depends on the objectives of the specific investigation. Once depth is established for sample collection, be sure to measure and record the sampling depth in the logbook.

After the PushPoint® has been deployed, carefully remove the guard rod and attach the sample tubing (Figure 3). The other end of the sample tubing can be connected to the sample withdrawing device, such as a peristaltic pump or syringe (Figure 4). Before collecting a pore water sample, be sure to purge out all air and surface water from the PushPoint® sampler and sample tubing with the appropriate amount of pore water. At least three volumes of pore water (until water is clear) should be purged before sample collection.

11.2 Peristaltic Pump/Vacuum Jug Collection

The peristaltic pump/vacuum jug can be used for sample collection because it allows for sample collection without the sample coming in contact with the pump head tubing. This is accomplished by placing a Teflon® transfer cap assembly onto the neck of a clean standard 1-liter amber glass container. Teflon® tubing (3-inch outside diameter) connects the container to both the pump and the sample source. The pump creates a vacuum in the container, thereby drawing the sample into the container without it coming into contact with the pump head tubing.

Because the sample is exposed to a vacuum and is agitated as it enters the vacuum jug, this method cannot be used for collection of VOC samples. An alternative method for collecting VOC samples involves filling the Teflon® tubing with sample by running the pump for a short period of time. Once the tubing is full of water, the tubing is removed from the PushPoint® and, after the tubing is disconnected from the pump head tubing, the water is allowed to drain, by gravity, into the sample vials. Alternatively, without disconnecting the tubing from the pump head, the contained sample can be pushed out of the tubing and into the sample vials by reversing the peristaltic pump at low speed.

For samples that are collected for metals analyses, or other analysis not affected by the silastic tubing, it is permissible to collect the sample directly from the discharge of the pump head tubing after an adequate purge has been completed. When collecting samples in this manner, there are several considerations to be aware of. The pump head tubing (silastic, etc.) must be changed after each sample and a rinsate blank must be collected from a representative piece of the pump head tubing (only one blank per investigation). Also, precautions must be taken to ensure that the end of the discharge tubing is not allowed to touch the ground, or other surface, in order to maintain the integrity of the sample when it is collected in this manner.

11.3 Syringe

Syringes, in conjunction with PushPoint® samplers, can be used to collect pore water samples if the integrity of the sample analysis will not be compromised. The tubing from the sampling port of the PushPoint® can be directly attached to a syringe and a pore water sample can be manually withdrawn from the sediment. The syringe can be used as the final sample container or the pore water can be transferred to another container, depending on project objectives and analytical requirements.

11.4 Sample Handling and Preservation Requirements

1. Pore water will typically be collected from sediments using a PushPoint® and placed directly into the sampling containers. A syringe may then be used to transfer the sample from the sampling container into the appropriate container.

2. When transferring the pore water sample from a collection device, make sure that the device does not come in contact with the final sample containers. The syringe used in the sample transfer is the only piece of equipment that should be in contact with the transfer vessel and the final sample container.

3. Place the sample into the appropriate labeled container. Samples collected for VOC

analysis must not have any headspace (see Section 7.1). All other sample containers must be filled with an allowance for ullage.

4. All samples requiring preservation must be preserved as soon as practically possible after sample collection. If preserved VOA vials are used, these will be preserved with concentrated hydrochloric acid prior to departure for the field investigation. All other chemical preservatives required for the remaining suite of analytes will be specified in the site-specific SAP. The adequacy of sample preservation will be checked after the addition of the preservative for all samples, except for the samples collected for VOC analysis. If it is determined that a sample is not acceptably preserved, additional preservative should be added to achieve adequate preservation. Preservation requirements for surface water samples will be specified in the site-specific SAP/QAPP and the Sample Preservation SOP FLD 03.00.

12.0 DATA RECORDS AND MANAGEMENT

Once collected, samples are preserved, labeled, and stored for transport. A chain of custody form must accompany all samples during transport and transfer between entities. Sample labels should contain the following information:

- Site identification
- Date sampled
- Location identification
- Sampler initials
- Time
- Analysis to be performed
- Preservative

Any other pertinent data should be recorded in the site dedicated field logbook.

13.0 QUALITY CONTROL AND ASSURANCE

The following general QA procedures apply:

1. All data must be documented on field data sheets or within site logbooks.

2. In general, concurrent (duplicate) sample collection at a frequency of 10% is required for most sampling activities. Field blanks at a frequency of one per day are also generally required. Consult the corresponding SAP/QAPP for specific QA/QC sampling frequency. Below is a list of usual pore water QA/QC sample types and the inaccuracy they are intended to detect:

   - Duplicate sample – two samples collected at the same location at the same time, intended to detect variability inherent in collection, processing, and handling procedures
   - Field blank – checks cross-contamination during sample collection, preservation, and shipment as well as in the laboratory.
   - Rinsate blank – detects equipment contamination due to inadequate decontamination

procedures

3. All instrumentation should be operated in accordance with operating instructions as supplied by the manufacturer unless otherwise specified in the work plan or SAP/QAPP. Equipment calibration activities should be conducted and documented prior to sampling and/or operation of equipment.
4. Document any deviations from SOPs, work plan, SAP/QAPP, etc.

14.0 REFERENCES

EPA Guidance for Preparing Standard Operating Procedures, EPA QA/G-6, April 2007.

MHE Products, PushPoint® Sampler (US Pat. # 6,470,967), Operators Manual and Applications Guide, Ver. 2.01, 2/15.

15.0 FIGURES

Figures 1-4 show equipment operation and basic sampling techniques.

Figure 1 - Pore Water PushPoint®

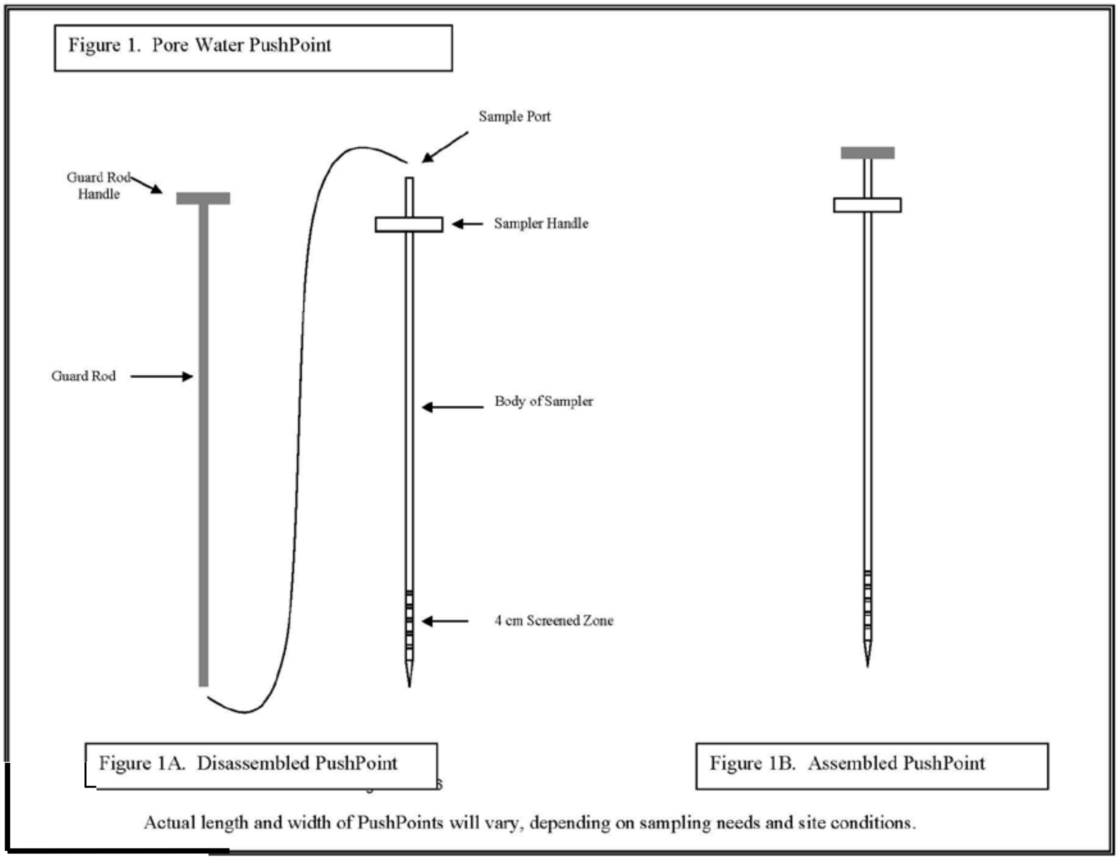


Figure 1. Pore Water PushPoint

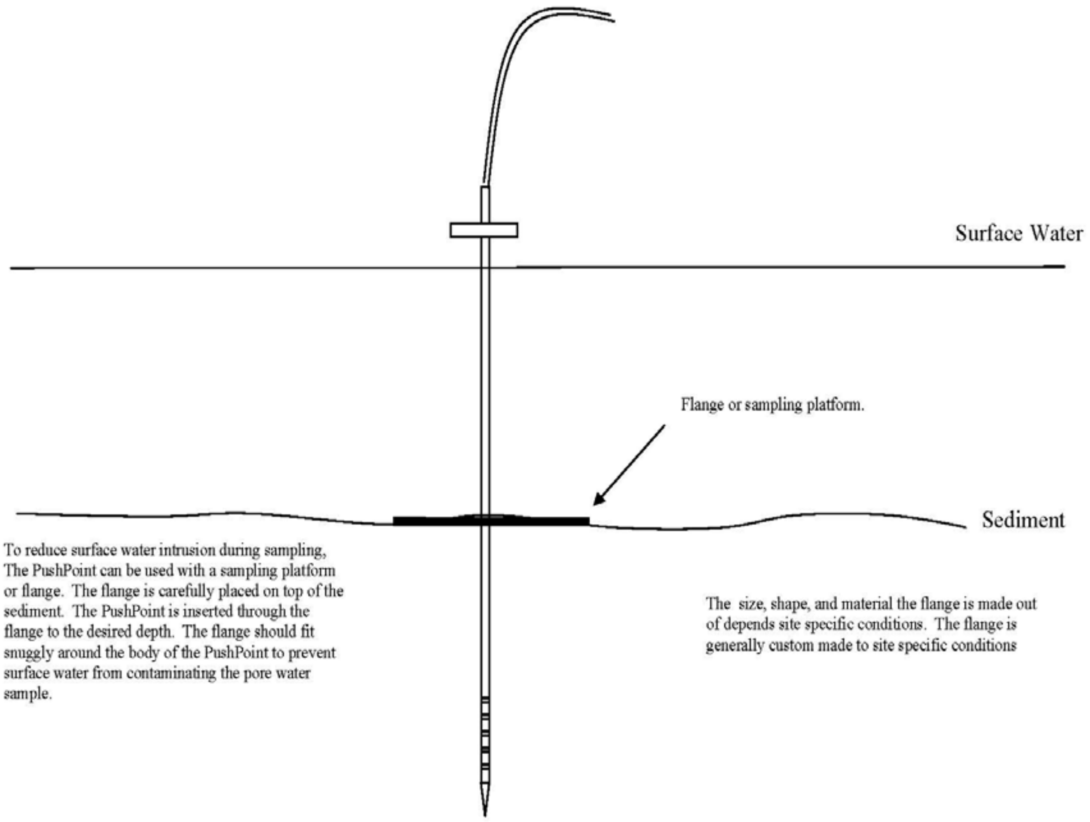
Figure 1A. Disassembled PushPoint

Figure 1B. Assembled PushPoint

Actual length and width of PushPoints will vary, depending on sampling needs and site conditions.

Figure 2 - PushPoint® deployed with a Sampling Platform

Figure 2. PushPoint deployed with a Sampling Platform



To reduce surface water intrusion during sampling, The PushPoint can be used with a sampling platform or flange. The flange is carefully placed on top of the sediment. The PushPoint is inserted through the flange to the desired depth. The flange should fit snuggly around the body of the PushPoint to prevent surface water from contaminating the pore water sample.

The size, shape, and material the flange is made out of depends site specific conditions. The flange is generally custom made to site specific conditions

Figure 3 - PushPoint® Being Deployed into the Sediment

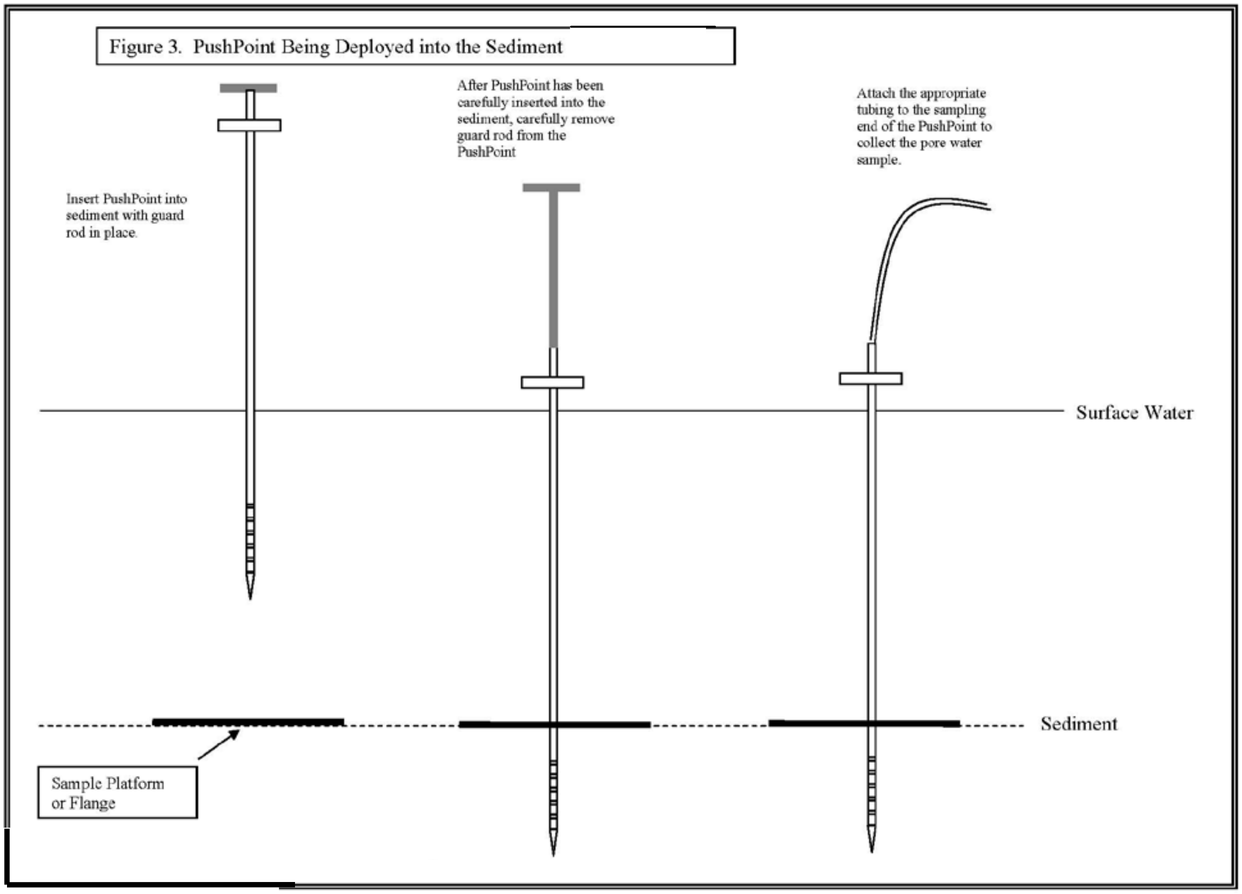
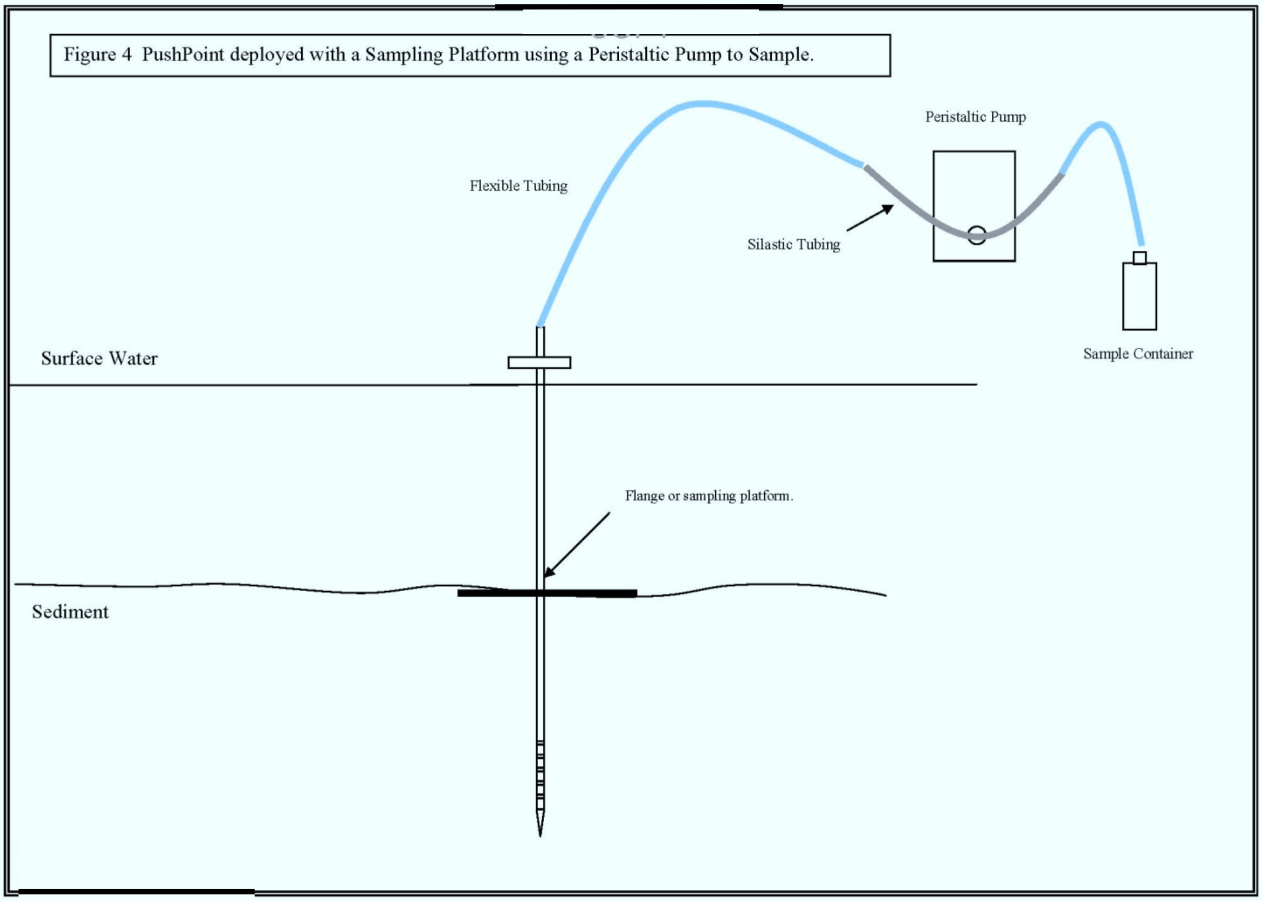


Figure 4 - PushPoint® deployed with a Sampling Platform using a Peristaltic Pump to Sample



Sample Custody and Labeling

APPROVED:

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06/06/12
Date

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6/6/12
Date

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7/10/12
Date

ESAT Region 8 Task Lead

6/8/12
Date

DCN: EP8-7-7061

This document has been prepared for the Environmental Protection Agency by the TechLaw, Inc. ESAT Region 8 Team and is intended to provide documentation of administrative, analytical and quality control procedures used in the daily performance of EPA and ESAT support services.

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

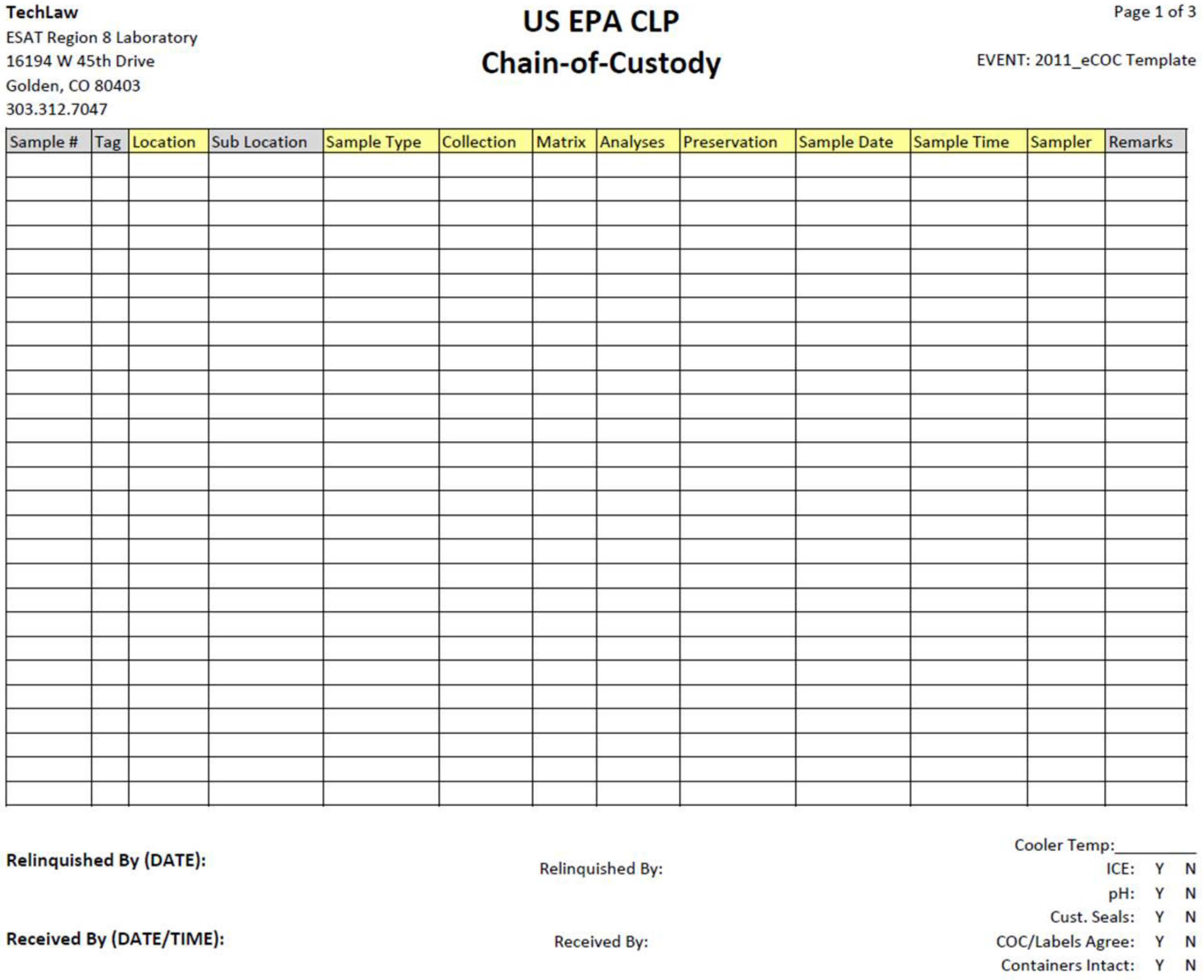
Proper sample custody and labeling requires a number of quality control and assurance steps. A COC generated in Scribe should always be crossed-checked by another person with the sample list found in the SAP. Completed COCs and labels should also be compared for accuracy before being relinquished to 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



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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):

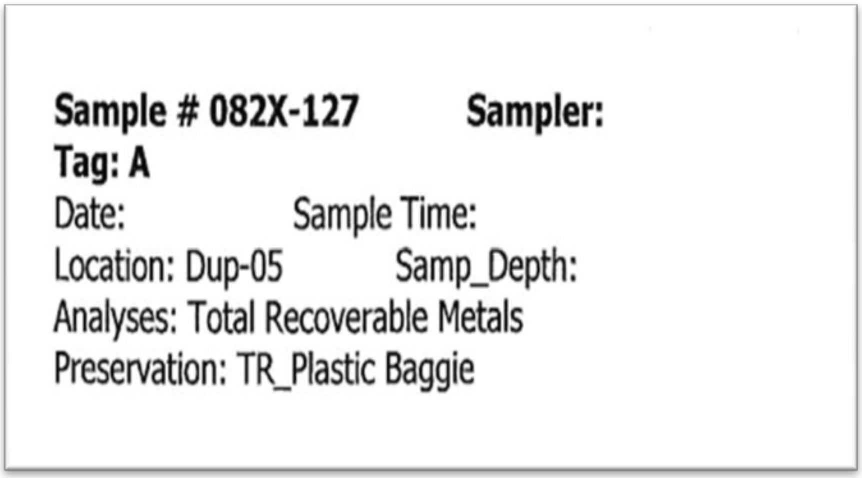
Relinquished By:

Received By (DATE/TIME):

Received By:

Cooler Temp:__________
ICE: Y N
pH: Y N
Cust. Seals: Y N
COC/Labels Agree: Y N
Containers Intact: Y N

Attachment B: Example Sample Label



Sample # 082X-127 **Sampler:**
Tag: A
Date: Sample Time:
Location: Dup-05 Samp_Depth:
Analyses: Total Recoverable Metals
Preservation: TR_Plastic Baggie

General Field Sampling Protocols

APPROVED:

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04/04/12
Date

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6/6/12
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This document has been prepared for the Environmental Protection Agency by the TechLaw, Inc. ESAT Region 8 Team and is intended to provide documentation of administrative, analytical and quality control procedures used in the daily performance of EPA and ESAT support services.

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

ANALYTICAL DETERMINATION OF TRACE METALS IN SOIL AND SEDIMENT BY FIELD PORTABLE X-RAY FLUORESCENCE SPECTROMETRY

APPROVED:

ESAT Region 8 QA Coordinator	07/16/13 Date
ESAT Region 8 Team Manager	07/11/13 Date
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ESAT Region 8 Task Lead	7/16/13 Date

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This document has been prepared for the Environmental Protection Agency by the TechLaw, Inc. ESAT Region 8 Team and is intended to provide documentation of administrative, analytical and quality control procedures used in the daily performance of EPA and ESAT support services.

TABLE OF CONTENTS

1.0 SCOPE AND APPLICABILITY

This standard operating procedure (SOP) provides procedures for the determination of elements in solid matrix samples. The X-Ray Fluorescence (XRF) provides analytical results for metals using a portable, hand-held instrument. The Innov-X Systems XRF Analyzer (XRFA) will be used as a field screening method to analyze in-situ (directly on the ground), bagged or prepared soil samples. The method is useful in that it incorporates a large number of screening-level measurements that provide better characterization than a small number of laboratory-based measurements. The method will be used as a screening tool along with confirmatory analysis using Environmental Protection Agency (EPA) approved methods.

The method will be used in a number of field exercises including identification of hot spots or contaminated sites, site investigation/assessment and confirmation of remediation applications. The method's strength is the ability to provide immediate results to allow quick decisions to be made in the field. The method's weakness is detection limits above the toxicity characteristic regulatory level of some Resource Conservation and Recovery Act (RCRA) analytes. The detection limits depend on the analyte of interest, the strength of the excitation source, count times used, matrix effects and inter-elemental spectral interferences.

2.0 SUMMARY OF METHOD

XRF is a nondestructive qualitative and quantitative analytical technique used to determine the chemical composition of a sample. An element is identified by exposing a substance to high energy photons emitted from an x-ray tube. The incident radiation causes electrons to be ejected from the atomic shells of any elements present in the sample and as a result energy is released (fluoresced). The fluorescent energy is detected by the XRF analyzer as a characteristic x-ray spectra. The x-ray spectra emitted is characteristic for each element and is the qualitative analysis. The number of counts at a characteristic energy level per unit time is representative of the element concentration in a sample and is the basis for the quantitative analysis.

The hand-held, battery operated field portable XRFA is factory calibrated and generally does not require recalibration. Certified standards are used as the continuing calibration standards to check the initial factory calibration. Discrete samples are collected in suitable sampling containers and are homogenized before on-site analysis. The samples are crushed and sieved and put in sample cups for XRFA analysis.

3.0 ACRONYMS

As	Arsenic
^{o}C	Degrees Celsius
Co	Cobalt
Cr	Chromium
Cu	Copper
CVC	Calibration Verification Check
DQO	Data Quality Objective
EPA	US Environmental Protection Agency
Fe	Iron
FP	Fundamental Parameter
ICP	Inductively Coupled Plasma
Hg	Mercury

MDL	Method Detection Limit
mR/hr	MilliRoentgen Per Hour
NIST	National Institute of Standards and Technology
PPE	Personal Protective Equipment
ppm	Parts Per Million
RCRA	Resource Conservation and Recovery Act
RSD	Relative Standard Deviation
SRM	Standard Reference Material
SSCS	Site Specific Calibration Standard
TBD	To Be Determined
V	Vanadium
XRF	X-Ray Fluorescence
XRFA	Innov-X Systems XRF Analyzer

4.0 HEALTH, SAFETY AND CAUTIONS

The XRFA is a safe instrument when used according to the manufacturer's recommended safety procedures as detailed in the user manual. According to the manufacturer, radiation levels during testing were <0.1 mR/hr on all surfaces of the analyzer except at or near the exit port for the radiation. Therefore, if the operator follows the SOP and XRFA user manual, they will not obtain any detectable radiation dose above naturally occurring background radiation.

Proper use of the instrument includes never pointing the instrument at another person, pointing the instrument into the air to perform a test and never holding a sample in one's hand to perform the test.

The XRFA should be stored in a locked case or locked cabinets when not in use. The location of storage and use should be of restricted access to limit potential exposure to ionizing radiation.

Operators should minimize the time around the energized instrument, maximize the distance from the instrument window and shoot into high density materials whenever possible. Keep one's hand away from the source-end of the XRFA when the instrument is ionizing the sample. Always be aware of the instrument's radiation source and direction of beam of x-rays.

Each analyst should wear appropriate personal protective equipment (PPE), including gloves and safety glasses.

The XRFA should be transported in the waterproof, drop proof carrying case, equipped with padlocks and a lead plate for the source window. There are no travel restrictions regarding bridges, tunnels or planes. There may be disclosure and/or licensing requirements if the XRFA is taken across state or national boundaries. Check with appropriate agencies for these details.

5.0 INTERFERENCES

5.1 Method Error

Total method error for XRF analysis is defined as the square root of the sum of squares of both instrument precision and user- or application-related error. Generally, instrument precision is the least significant source of error in XRF analysis. User- or application-related error is generally more significant and varies with each site and method used. Some sources of interference can be minimized or controlled by the instrument operator, but others cannot. Common sources of user- or application-related error are discussed below.

5.2 Physical Matrix Effects

This form of interference results from variations in the physical character of the sample.

These variations may include such parameters as particle size, uniformity, homogeneity, and surface condition.

Note: For example, if any analyte exists in the form of very fine particles in a coarser-grained matrix, the analyte's concentration measured by the XRF will vary depending on how fine particles are distributed within the coarser-grained matrix. If the fine particles "settle" to the bottom of the sample cup, the measurement will be higher than if the fine particles are mixed in well in the cup.

One way to reduce such error is to grind and sieve all soil samples to a uniform particle size, thus reducing sample to sample particle size variability.

Homogeneity is always a concern when dealing with soil samples. Every effort should be made to thoroughly mix and homogenize soil samples before analysis.

5.3 Moisture Content

Moisture in the sample may affect the accuracy of analysis of soil and sediment sample analyses.

When the moisture content is between 5 and 20 percent, the overall error from moisture may be minimal. However, moisture content may be a major source of error when analyzing samples of surface soil or sediment that are saturated with water.

This error can be minimized by drying the samples in a convection oven.

5.4 Positioning of Samples for Analysis

Inconsistent positioning of samples in front of the probe window is a potential source of error because the x-ray signal decreases as the distance from the radioactive source increases.

This error is minimized by maintaining the same distance between the window and each sample. For the best results, the window of the probe should be in direct contact with the sample, which means that the sample should be flat and smooth to provide a good contact surface.

5.5 Chemical Matrix Effects

This effect results from differences in the concentrations of interfering elements. These effects occur as either spectral interferences (peak overlaps) or as x-ray absorption and enhancement phenomena. Both effects are common in soils contaminated with heavy metals.

Note: As examples of absorption and enhancement effects; iron (Fe) tends to absorb copper (Cu) x-rays, reducing the intensity of the Cu measured by the detector, while chromium (Cr) will be enhanced at the expense of Fe because the absorption edge of Cr is slightly lower in energy than the fluorescent peak of Fe.

The effects can be corrected mathematically through the use of Fundamental Parameter (FP) coefficients.

The purpose of XRF analysis with Fundamental Parameters (FP) is to convert elemental peak intensities to elemental concentrations and/or film thicknesses.

This is achieved though a calibration step, where the XRF response function (related to parameters that are independent of the sample matrix) for each element is measured using a known standard of some kind. ESAT will never calibrate the XRF, as it will always be sent back to the manufacturer for such maintenance

The effects also can be compensated for using Site Specific Calibration Standards (SSCS), which contain all the elements present on site that can interfere with one another.

5.6 Spectrum Overlap

When present in a sample, certain x-ray lines from different elements can be very close in energy and, therefore, can cause interference by producing a severely overlapped spectrum.

The degree to which a detector can resolve the two different peaks depends on the energy resolution of the detector. If the energy difference between the two peaks in electron volts is less than the resolution of the detector in electron volts, then the detector will not be able to fully resolve the peaks.

The most common spectrum overlaps involve the K line of element Z-1 with the K line of element Z. This is called the Ka/Kb interference. For example, large amounts of vanadium (V) and Fe will interfere with the quantization of Cr or Cobalt (Co) respectively. The presence of Fe is a frequent problem because it is often found in soils at tens of thousands of parts per million (ppm).

5.7 Ambient Temperature Changes

Ambient temperature changes affect the gain of the amplifiers, producing instrument drift. The instrument should be recalibrated when large changes in ambient temperature are noted.

6.0 PERSONNEL QUALIFICATION

This test is restricted to use by or under the supervision of analysts experienced in the use of the XRFA. Each analyst must demonstrate the ability to generate acceptable results with this test method before approved to operate the instrument.

7.0 EQUIPMENT AND SUPPLIES

Innov-X-Systems XRF Analyzer, model Alpha 4000 S
Battery Charger
Extra Battery
Polyethylene sample cups, Chemplex Cat. No. 1330 30.7mm diameter x 23.5 mm, or equivalent
X-ray window film, Chemplex Cat. No. 425, polypropylene, or equivalent
#60 stainless steel sieve, top and bottom
Mortar and pestle
Grinders
Trowels for collecting and smoothing soil
Plastic baggies for collection and homogenization of soil samples
Drying oven
Kim wipes
Large squirt bottle with DI water
Permanent ink markers
Mallet or rock hammer
Beaker(s)
Logbooks, project and instrument

8.0 REAGENTS AND STANDARDS

Standards are specific for the site and the projects data quality objective's (DQOs). The XRFA is self-calibrating. However, standard reference materials (SRMs) should be incorporated into all projects to assure the self-calibration is operating properly. National Institute of Standards and Technology (NIST) SRM 2710, Montana Soil, is a good selection for an SRM and will be utilized for this method. SSCS may be used for better accuracy if required by the site DQOs.

8.1 Site-Specific Calibration Standards

The SSCS must be representative of the matrix to be analyzed by the XRFA. These samples must be well homogenized. A minimum of ten samples spanning the concentration ranges of the analytes of interest and of the interfering elements must be obtained from the site.

Each sample should be oven-dried for 2 to 4 hours at a temperature of less than 150°C depending on DQOs. If mercury (Hg) or arsenic (As) are analytes of interest, samples should be dried at 60°C for a minimum of 2 hours. When the sample is dry, all large, organic debris and non-representative material should be removed. The sample should be ground with a mortar and pestle and passed through a #60-mesh sieve.

The sample should be homogenized well. Approximately 5 grams of the sample should then be removed and placed in a sample cup for XRF analysis. The rest of the prepared sample is analyzed by Inductively Coupled Plasma (ICP). The method used for confirmatory analysis should meet the DQOs of the project.

Site Specific Calibration Standard (SSCSs) are used during a survey to predict the way the XRF will perform at a certain site. Data from the SSCSs are used to generate a statistical based curve in order to establish Action Levels and are not necessarily used during in situ analysis. Therefore, the calibration standard is not actually calibrating the XRF, but instead providing a statistical correlation or a regression analysis.

8.2 Method Blank

The method blank consists of silica sand or lithium carbonate and must be free of any analytes a concentrations above the method detection limits (MDLs).

This method blank undergoes the same preparation procedures as the samples, and is used to monitor for cross-contamination and laboratory-induced contaminants or interferences.

8.3 Instrument Blank Standard

The blank standard consists of silicon dioxide, polytetrafluroethylene, a quartz block, “clean” sand, or lithium carbonate and must be free of analytes at concentrations above the method detection limits.

The instrument blank verifies that no contaminant of interest exists in the spectrometer or in on the probe window.

8.4 Standard Reference Materials

SRMs are standards containing certified amounts of metals in soil or sediment.

These standards are used for accuracy and performance checks of XRF analyses.

SRMs can be obtained from the NIST. NIST SRM 2710, Montana Soil will be used to verify the performance of the XRFA. The certified values for NIST 2710 can be found in Attachment I. NIST 2709 is used to establish the MDLs of the instrument and in order to do that it must be run a minimum of 7 times.

8.5 Second Source Standard

Solid matrix standards of known concentration that may be used for second source verification.

RTC certified XRF-408 standard will be used for second source verification. The certified values for RTC XRF-408 can be found in Attachment II.

9.0 PROCEDURE

9.1 Instrument Setup and Calibration

1. Prior to beginning a test, be certain the battery pack has sufficient charge. Always carry a spare battery pack. Place battery into analyzer.
2. Power on the XRFA main power (on/off switch located on back of analyzer). The green LED light next to the button will illuminate when the analyzer is on.
3. Power on the iPAQ (button located in upper right hand corner of iPAQ).

4. Select Innov-X from start menu located in upper left corner of iPAQ screen.

5. Read radiation safety notice and press start.

6. Select soil mode (XRFA undergoes 60 second hardware initialization).

7. From the Bluetooth Browser screen, select ok and proceed with standardization sequence.

8. Attach standardization clip or coupon (for use with test stand) on the nose of the analyzer and select standardization button on the screen.

   8a. Once standardized, remove clip or coupon.

   8b. Standardization must be performed any time the analyzer is initiated or restarted and should be repeated if the instrument is operating more than 4 hours.

   8c. Standardization is initiated from the Analysis Screen of any mode.

   8d. Record the energy resolution into the XRFA log book and select ok.

9. Select lock icon in the lower right hand corner of iPAQ screen to release trigger lock.

   9a. Test the required calibration check standard as required for the site and the project DQO's. This will be the NIST SRM 2710 or the SSCS as discussed in Section 6.0. Elemental concentrations should be within 20% of the standard value for all required analytes. The quality control requirements for the standard are detailed in Section 8.2. The calibration check standard values are reported in Appendixes I and II.

   9b. Analyze instrument calibration check blank labeled silicon dioxide. The instrument check blank confirms there is no contamination associated with the analyzer. The quality control requirements for the instrument blank are discussed in Section 8.3

   9c. Analyze method blank. This is only required if laboratory samples are prepared. This is used to verify that cross contamination has not occurred during the sample preparation process. The quality control requirements for the method blank are discussed in Section 8.4.

9.2 In-situ Sample Analysis

1. The detailed procedure will vary according to site specific plans, so the following is a general guideline. Consult the specific project SAP/QAPP for specific sampling details.

2. Select surface to be analyzed. Selected spot should not be saturated with water.

3. Clear any surface debris or vegetation. Level and flatten the exposed surface.

    3a. Hold the XRFA in one hand or place in analyzer holder. Do not place hand on the end of the plate of the instrument while measuring.

    3b. Place the XRFA on the polypropylene film on the ground of the sampling site and pull the trigger. The red light in the instrument will flash as long as the instrument is testing. The instrument is pre-programmed for the length of the test, typically 1-2 minutes. Pulling the trigger a second time will stop the test. All data will be stored in a data base of the PDA. After each test, the information (sampling location, operator, sample ID, etc) should be input into the data base for that sample.

9.3 Prepared Sample Analysis

1. The detailed procedure will vary according to site-specific plans, so the following is a general guideline. Consult the specific project SAP/QAPP for specific sampling details.

2. Select location to be analyzed. The sample location should have no greater than 25% moisture. (Rule of thumb, if sample remains clumped together after being squeezed by hand, sample needs to be dried.)

    2a. Remove any debris and other foreign objects from the surface so that the natural surface is exposed.

    2b. Using a sampling spade or trowel, take a vertical slice and place in a clean pail. Mix the sample by stirring and rotating the pail at a 45º degree angle. Don’t shake the pail as this will stratify the sample by weight.

    2c. Collect a sample from a 4x4 inch square that is approximately 1/2 inch deep. Collect at least 100 grams of sample and place in a plastic baggie or plastic bucket and mix thoroughly. The sample will be homogenized, dried and ground before analysis. The sample can be homogenized before or after drying.

    2d. Dry the sample. The drying method will depend upon the project and may include drying for 2 to 4 hours in a convection oven at a temperature not greater than 150°C. If As or Hg are required, drying at a temperature of 60°C for a minimum of 2 hours is recommended (until dry).

    2e. Sieve the sample using a clean #60 mesh screen until the required fraction is passed. Grinding is not recommended because it can alter the sample and negatively affect the data. Instead, large clumps should be broken up and the entire collected sample should be sieved. The sieving will allow adequate homogenization.

2f. Fill the XRF sample cup. Place a circle of 0.24 mil polypropylene film on top of the XRF cup. The top is the end of the cup with the indented ring. Secure the film with the collar. The film window should be taught and smooth. Tamp the sample into the cup and fasten the cap.

2g. Document the sampling identification on the XRF sample cup using a permanent ink marker. Set-up the XRFA using the testing platform. Samples are now ready for analysis.

2h. Pull the trigger on the XRFA. The red light in the instrument will flash as long as the instrument is testing. The instrument is pre-programmed for the length of the test, typically 1-2 minutes. Pulling the trigger a second time will stop the test. All data will be stored in a data base of the PDA. After each test, the information (sampling location, operator, sample ID, etc) should be input into the data base for that sample.

9.4 Laboratory Preparation and Analysis

1. The detailed procedure will vary according to site specific plans, so the following is a general guideline. Consult the specific project SAP/QAPP for specific sampling details.

2. The sample should be homogenized, dried, and ground before analysis. The sample can be homogenized before or after drying.

3. If the sample is homogenized before drying, it should be thoroughly mixed in a beaker or similar container, or if the sample is moist and has a high clay content, it can be kneaded in a plastic bag.

4. If the sample is homogenized after drying, spread out sample in shallow drying pan, break up any clumps with stainless steel spatula, air dry overnight, or oven dry for 2 to 4 hours in a convection oven at a temperature not greater than 150 °C. If As or Hg are required, dry at 60 °C for a minimum of 2 to 4 hours (until dry).

5. Sub-sample, grind, and sieve through a #60 mesh screen until enough sample has been collected to fill a XRF sample cup.

5a. Fill the XRF sample cup. Place a circle of 0.24 mil polypropylene film on top of the XRF cup. The top is the end of the cup with the indented ring.

5b. Secure the film with the collar. The film window should be taught and smooth.

5c. Tap the sample into the cup and fasten the cap.

5d. Document the sampling identification on the XRF sample cup using a permanent ink marker.

5e. Set-up the XRFA using the testing platform. Samples are now ready for analysis.

5f. Pull the trigger on the XRFA. The red light in the instrument will flash as long as the instrument is testing. The instrument is pre-programmed for the length of the test, typically 1-2 minutes. Pulling the trigger a second time will stop the test.

6. All data will be stored in a data base of the PDA. After each test, the information (sampling location, operator, sample ID, etc) should be input into the data base for that sample.

9.5 Instrument and iPAQ Shutdown

1. Following the last analytical sequence, select file located at the bottom left hand corner of the
   iPAQ and select exit.
2. Select file and exit again until the start screen appears then turn off the iPAQ by selecting the button in the upper right corner.
3. Turn off the XRFA main power (on/off switch located on back of analyzer).
4. Remove the iPAQ from the XRFA and place both units in the water proof case for safe storage.

9.6 XRF Interference Calculation

The below procedure corrects for specific interferences in soils, or for matrixes other than soil, such as water, where analyses from the XRF has already been compared to laboratory (ICP) data. For example, if measurements for a specific element from the XRF are consistently elevated by a factor of 2, then the element rate within the XRF should be decreased by 2. Then the measurements from the XRF will match the laboratory data.

1. In soil mode, at the Standardization Screen, choose Options
2. Next, choose Element Rates
3. Enter the administrator password – lower case z
4. Change the Factor Number of the element by the percentage the XRF measurements are consistently different from the laboratory measurements. For example, if arsenic readings using the XRF are always twice the amount of the laboratory analysis, and the Factor Number for arsenic is 1500, then the new Factor Number for arsenic should be 750. This function should not be adjusted. If the values are that far off then the instrument needs to be sent back to the

manufacturer for maintenance

10.0 DATA RECORDS AND MANAGEMENT

All XRFA printouts, worksheets, logbook entries, and associated documentation must be filed in the project folder. Copies of the documentation may be required in submitted data reports. Any maintenance performed should be recorded in the XRFA Instrument Logbook.

11.0 QUALITY CONTROL

11.1 Internal Calibration Check

The energy calibration check is performed automatically during the initial standardization procedure when the instrument is started.

The software does not allow the XRFA to be used if the energy calibration check procedure fails. The energy resolution and source strength information is recorded into the XRFA log book.

11.2 Calibration Verification Check (CVC)

A calibration verification sample is used to check the accuracy of the instrument and to assess the stability and consistency of results for the analytes of interest.

A sample control check is analyzed following the initial standardization procedure of each working day, during sample analysis, and at the end of the day.

The frequency of the checks during analysis and which standards will be used for the check is DQO dependent. It is recommended to check every 20 samples or after four hours of continual use, whichever occurs first.

The measured value for each target analyte should be ± 20% of the value considered accurate in order for the CVC to pass. Refer to Appendix I for a list of analytes and their true values.

If the %D falls outside of this acceptance range, the analyzer must be restandardized followed by an acceptable CVC before analysis can continue. The batch of samples analyzed before the unacceptable CVC must be reanalyzed.

11.3 Instrument Blank

An instrument blank verifies that no contamination exists in the spectrometer or on the probe window. The instrument blank should be analyzed daily, following the standardization and CVC, every twenty samples or four hours of continual use, whichever occurs first, and at the end of the day.

All target analyte concentrations should be less than its MDL. The detection limits of the elements detected by the XRFA are given in Attachment III. If concentrations exceed these limits, then the probe window should be checked for contamination.
Clean the probe window and reanalyze the instrument blank. If contamination is not the problem,

zero the XRFA as detailed in the user manual.

Once the window is cleaned or the instrument is zeroed, the XRFA should be re-standardized and verified with acceptable CVC and instrument blank check before analyzing samples. If acceptable, re-analyze the samples prior to the last acceptable blank check.

11.4 Method Blank

The method blank is used to monitor for contaminates during preparations of samples and undergoes the same preparation procedure as the samples. The concentration of all required analytes should be less than its MDL and should be analyzed as required by site-specific DQOs.

A method blank is only required if samples are prepared according to section 9.3. If the concentration exceeds its MDL, identify the problem and all samples associated with the method blank must be reanalyzed.

11.5 Method Precision

A minimum of one sample (NIST 2709) per project should be analyzed at least seven times in replicate unless instrument drift or maintenance has occurred, with each measurement made for the same analysis time as for the field samples.

For the XRF data to be considered adequately precise, the relative standard deviation (RSD) for each analyte should not be greater than 20% except for Cr. The RSD value for Cr should not be greater than 30%. Precision requirements are defined in the site-specific SAP/QAPP.

11.6 Confirmation Samples

To verify XRF data, laboratory analysis is performed on a portion of the XRF analyzed samples as required by the site-specific SAP/QAPP.

The confirmation samples are splits of the homogenous sample material analyzed by the XRFA. Submit a minimum of 10% of the total samples for laboratory confirmation analysis.

12.0 DATA ANALYSIS AND CALCULATIONS

The XRFA instrument has software capable of storing all analytical results and spectra. The instrument software automatically performs all calculations including minimum detection levels and 95% confidence intervals.

Calculations:

Method Precision RSD = (SD/Mean Concentration) x 100

Where:
RSD = Relative standard deviation for the precision measurement of the analyte
SD = Standard deviation of the concentration for the analyte
Mean Concentration = Mean concentration for the analyte

13.0 REFERENCES

Method 6200, Field Portable X-Ray Fluorescence Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment, Revision 0, January 1998.

Innov-X Systems Alpha Series X-Ray Fluorescence Spectrometers Version 2.1, August 2005.

14.0 APPENDIX

Appendix I: NIST 2710 Reference Material (Certified)
Appendix II: NIST 2710 Reference Material (Non-Certified)
Appendix III: RTC XRF-408 Certificate of Analysis
Appendix IV: Target Analyte List

Appendix I: NIST 2710 Reference Material (certified)

Table 1. Certified Value

Element	Mass Fraction (%)	Element	Mass Fraction (mg/kg)
Aluminum	6.44 ± 0.08	Antimony	38.4 ± 3
Calcium	1.25 ± 0.03	Arsenic	626 ± 38
Iron	3.38 ± 0.10	Barium	707 ± 51
Magnesium	0.853 ± 0.042	Cadmium	21.8 ± 0.2
Manganese	1.01 ± 0.04	Copper	2950 ± 130
Phosphorus	0.106 ± 0.015	Lead	5532 ± 80
Potassium	2.11 ± 0.11	Mercury	32.6 ± 1.8
Silicon	28.97 ± 0.18	Nickel	14.3 ± 1.0
Sodium	1.14 ± 0.06	Silver	35.3 ± 1.5
Sulfur	0.240 ± 0.006	Vanadium	76.6 ± 2.3
Titanium	0.283 ± 0.010	Zinc	6952 ± 91

Noncertified Values: Noncertified values shown below are provided for information only. An element concentration value is not certified if a bias is suspected in one or more of the methods used for certification, or if two independent methods are not available.

Appendix II: NIST 2710 Reference Material (noncertified)

Table 2. Noncertified Values

Element	Mass Fraction (%)	Element	Mass Fraction (mg/kg)
Carbon	3	Bromine	6
		Cerium	57
		Cesium	107
		Chromium	39
		Cobalt	10
		Dysprosium	5.4
		Europium	1
		Gallium	34
		Gold	0.6
		Hafnium	3.2
		Holmium	0.6
		Indium	5.1
		Lanthanum	34
		Molybdenum	19
		Neodymium	23
		Rubidium	120
		Samarium	7.8
		Scandium	8.7
		Strontium	330
		Thallium	1.3
		Thorium	13
		Tungsten	93
		Uranium	25
		Ytterbium	1.3
		Yttrium	23

Appendix III: RTC XRF-408 Certificate of Analysis

Analyte: **Metals in Soil**
Solvent: **Soil**

Product Number: **XRF-408**
Hazard: **Irritant**

Lot: **010291**

	Certified Value	Verification Value	Units	Uncertainty	Verified to NIST SRM
Arsenic, As	500	501	mg/Kg	15.2	3103a
Barium, Ba	500	500	mg/Kg	15.2	3104a
Cadmuim, Cd	500	500	mg/Kg	15.1	3108
Chromium, Cr	500	500	mg/Kg	15.1	3112a
Lead, Pb	500	500	mg/Kg	15.1	3128
Mercury, Hg	500	501	mg/Kg	15.1	3133
Selenium, Se	500	500	mg/Kg	15.1	3149
Silver, Ag	500	500	mg/Kg	15.1	3151

The certified concentration is based upon gravimetric procedures. The uncertainty associated with the certified value is +/- 0.3% relative, which is the sum of the estimated errors due to the purity of the raw material, the gravimetric preparation of the solution, and losses due to packaging.

Traceability: The standard was manufactured under an ISO 9001 registered quality system. The balance used to weigh raw materials is accurate to +/- 0.0001g and calibrated regularly using mass standards, which are traceable to NST. All dilutions are performed gravimetrically. The standard is directly traceable to NIST SRM’s as listed above.

Appendix IV: Target Analyte List

OLYMPUS

Your Vision, Our Future

HANDHELD XRF ANALYZERS

Limits of Detection

Low-Density Sample Types - (soils, powders, liquids)

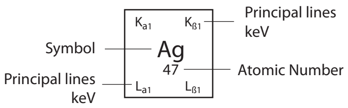
▨ Not Measured	🟧 <1 %	🟢 <0.5 %	🟥 <200 ppm
🟪 <50 ppm	🟦 <20 ppm	🟨 <10 ppm	🟩 <5 ppm

	IIA	IIIB	IVB	VB	VIB	VIIB	Group VIII			IB	IIB	IIIA	IVA	VA	VIA	VIIA	
▨ H 1																	▨ He 2
▨ 0.05 Li 3	▨ 0.11 Be 4											▨ 0.18 B 5	▨ 0.28 C 6	▨ 0.39 N 7	▨ 0.52 O 8	▨ 0.68 F 9	▨ 0.85 Ne 10
▨ 1.04 1.07 Na 11	🟧 1.25 1.3 Mg 12											🟧 1.49 1.56 Al 13	🟧 1.74 1.84 Si 14	🟢 2.01 2.14 P 15	🟥 2.31 2.46 S 16	🟥 2.62 2.82 Cl 17	▨ 2.96 3.19 Ar 18
🟪 3.31 3.59 K 19	🟪 3.69 4.01 Ca 20 0.34 0.34	🟪 4.09 4.46 Sc 21 0.4 0.4	🟨 4.51 4.93 Ti 22 0.45 0.46	🟨 4.95 5.43 V 23 0.51 0.52	🟨 5.41 5.95 Cr 24 0.57 0.58	🟨 5.9 6.49 Mn 25 0.64 0.65	🟨 6.4 7.06 Fe 26 0.71 0.72	🟨 6.93 7.65 Co 27 0.78 0.79	🟨 7.48 8.26 Ni 28 0.85 0.87	🟨 8.05 8.91 Cu 29 0.93 0.95	🟩 8.64 9.57 Zn 30 1.01 1.03	🟩 9.25 10.26 Ga 31 1.1 1.12	🟩 9.89 10.98 Ge 32 1.19 1.22	🟩 10.54 11.73 As 33 1.28 1.32	🟩 11.22 12.5 Se 34 1.38 1.42	🟩 11.92 13.29 Br 35 1.48 1.53	▨ 12.65 14.11 Kr 36 1.59 1.64
🟩 13.4 14.96 Rb 37 1.69 1.75	🟩 14.17 15.84 Sr 38 1.81 1.87	🟩 14.96 16.74 Y 39 1.92 2	🟩 15.78 17.67 Zr 40 2.04 2.12	🟩 16.62 18.62 Nb 41 2.17 2.26	🟩 17.48 19.61 Mo 42 2.29 2.39	🟩 18.37 20.62 Tc 43 2.42 2.54	🟩 19.28 21.66 Ru 44 2.56 2.68	🟨 20.22 22.72 Rh 45 2.83	🟨 21.18 23.82 Pd 46 2.84 2.99	🟨 22.16 24.94 Ag 47 2.98 3.15	🟨 23.17 26.1 Cd 48 3.13 3.32	🟨 24.21 27.28 In 49 3.29 3.49	🟦 25.27 28.49 Sn 50 3.44 3.66	🟦 26.36 29.73 Sb 51 3.6 3.84	🟦 27.47 31 Te 52 3.77 4.03	🟦 28.61 32.29 I 53 3.94 4.22	▨ 29.78 33.62 Xe 54 4.11 4.42
🟦 30.97 34.99 Cs 55 4.29 4.62	🟦 32.19 36.38 Ba 56 4.47 4.83		🟨 55.79 63.23 Hf 72 7.9 9.02	🟨 57.53 65.22 Ta 73 8.15 9.34	🟨 59.32 67.24 W 74 8.4 9.67	🟨 61.14 69.31 Re 75 8.65 10.01	🟨 63 71.41 Os 76 8.91 10.36	🟨 64.9 73.56 Ir 77 9.18 10.71	🟨 66.83 75.75 Pt 78 9.44 11.07	🟨 68.8 77.98 Au 79 9.71 11.44	🟩 70.82 80.25 Hg 80 9.99 11.82	🟩 72.87 82.58 Tl 81 10.27 12.21	🟩 74.97 84.94 Pb 82 10.55 12.61	🟩 77.11 87.34 Bi 83 10.84 13.02	🟩 79.29 89.8 Po 84 11.13 13.45	🟩 81.52 92.3 At 85 11.43 13.88	▨ 83.78 94.87 Rn 86 11.73 14.32
🟩 86.1 97.47 Fr 87 12.03 14.77	🟩 88.47 100.13 Ra 88 12.34 15.24																
Lanthanides 57-71		🟦 33.44 37.8 La 57 4.65 5.04	🟦 34.72 39.26 Ce 58 4.84 5.26	🟦 36.03 40.75 Pr 59 5.03 5.49	🟦 37.36 42.27 Nd 60 5.23 5.72	🟦 38.72 43.83 Pm 61 5.43 5.96	🟦 40.12 45.41 Sm 62 5.64 6.21	🟦 41.54 47.04 Eu 63 5.85 6.46	🟦 43 48.7 Gd 64 6.06 6.71	🟦 44.48 50.38 Tb 65 6.27 6.98	🟦 46 52.12 Dy 66 6.5 7.25	🟦 47.55 53.88 Ho 67 6.72 7.53	🟦 49.13 55.68 Er 68 6.95 7.81	🟦 50.74 57.52 Tm 69 7.18 8.1	🟦 52.39 59.37 Yb 70 7.42 8.4	🟦 54.07 61.28 Lu 71 7.66 8.71	
Actinides 89-103		🟩 90.88 102.85 Ac 89 12.65 15.71	🟩 93.35 105.61 Th 90 12.97 16.2	🟩 95.87 108.43 Pa 91 13.29 16.7	🟩 98.44 111.3 U 92 13.61 17.22	🟩 101.00 114.18 Np 93 13.95 17.74	🟩 103.65 117.15 Pu 94 14.28 18.28	▨ 106.35 120.16 Am 95 14.62 18.83	▨ 109.10 123.24 Cm 96 14.96 19.39	▨ 111.90 126.36 Bk 97 15.31 19.97	▨ 114.75 129.54 Cf 98 15.66 20.56	▨ 117.65 132.78 Es 99 16.02 21.17	▨ 120.60 136.08 Fm 100 16.38 21.79	▨ Md 101	▨ No 102	▨ Lr 103	

Alloy Analysis:

Elements detected: Magnesium (Mg, Z=12) through Sulfur (S, Z=16) and Titanium (Ti, Z=22) through Plutonium (Pu, Z=94).

Please see separate Alloy Analysis LOD Specifications.



INNOV-X

Detection limits are a function of testing time, sample matrix and presence of interfering elements.
Detection limits are estimates based on 1-2 minutes test times and detection confidence of 3σ (99.7% confidence).
Interference-free detection limits are intended as guidelines: please contact Olympus Innov-X to discuss your specific application.

PHOTON ENERGIES, IN ELECTRON VOLTS, OF PRINCIPAL K- AND L-SHELL EMISSION LINES

Element	Symbol	Atomic #	$K_{\alpha1}$	$K_{\beta1}$	$L_{\alpha1}$	$L_{\beta1}$
Actinium	Ac	89	90.88	102.85	12.65	15.71
Aluminum	Al	13	1.49	1.56	0	0
Antimony	Sb	51	26.36	29.73	3.6	3.84
Argon	Ar	18	2.96	3.19	0	0
Arsenic	As	33	10.54	11.73	1.28	1.32
Astatine	At	85	81.52	92.3	11.43	13.88
Barium	Ba	56	32.19	36.38	4.47	4.83
Beryllium	Be	4	0.11	0	0	0
Bismuth	Bi	83	77.11	87.34	10.84	13.02
Boron	B	5	0.18	0	0	0
Bromine	Br	35	11.92	13.29	1.48	1.53
Cadmium	Cd	48	23.17	26.1	3.13	3.32
Calcium	Ca	20	3.69	4.01	0.34	0.34
Carbon	C	6	0.28	0	0	0
Cerium	Ce	58	34.72	39.26	4.84	5.26
Cesium	Cs	55	30.97	34.99	4.29	4.62
Chlorine	Cl	17	2.62	2.82	0	0
Chromium	Cr	24	5.41	5.95	0.57	0.58
Cobalt	Co	27	6.93	7.65	0.78	0.79
Copper	Cu	29	8.05	8.91	0.93	0.95
Dysprosium	Dy	66	46	52.12	6.5	7.25
Erbium	Er	68	49.13	55.68	6.95	7.81
Europium	Eu	63	41.54	47.04	5.85	6.46
Fluorine	F	9	0.68	0	0	0
Francium	Fr	87	86.1	97.47	12.03	14.77
Gadolinium	Gd	64	43	48.7	6.06	6.71
Gallium	Ga	31	9.25	10.26	1.1	1.12
Germanium	Ge	32	9.89	10.98	1.19	1.22
Gold	Au	79	68.8	77.98	9.71	11.44
Hafnium	Hf	72	55.79	63.23	7.9	9.02
Holmium	Ho	67	47.55	53.88	6.72	7.53
Indium	In	49	24.21	27.28	3.29	3.49
Iodine	I	53	28.61	32.29	3.94	4.22
Iridium	Ir	77	64.9	73.56	9.18	10.71
Iron	Fe	26	6.4	7.06	0.71	0.72
Krypton	Kr	36	12.65	14.11	1.59	1.64
Lanthanum	La	57	33.44	37.8	4.65	5.04
Lead	Pb	82	74.97	84.94	10.55	12.61
Lithium	Li	3	0.05	0	0	0
Lutetium	Lu	71	54.07	61.28	7.66	8.71
Magnesium	Mg	12	1.25	1.3	0	0
Manganese	Mn	25	5.9	6.49	0.64	0.65
Mercury	Hg	80	70.82	80.25	9.99	11.82
Molybdenum	Mo	42	17.48	19.61	2.29	2.39
Neodymium	Nd	60	37.36	42.27	5.23	5.72
Neon	Ne	10	0.85	0	0	0
Nickel	Ni	28	7.48	8.26	0.85	0.87
Niobium	Nb	41	16.62	18.62	2.17	2.26
Nitrogen	N	7	0.39	0	0	0
Osmium	Os	76	63	71.41	8.91	10.36
Oxygen	O	8	0.52	0	0	0
Palladium	Pd	46	21.18	23.82	2.84	2.99
Phosphorus	P	15	2.01	2.14	0	0
Platinum	Pt	78	66.83	75.75	9.44	11.07
Polonium	Po	84	79.29	89.8	11.13	13.45
Potassium	K	19	3.31	3.59	0	0
Praseodymium	Pr	59	36.03	40.75	5.03	5.49
Promethium	Pm	61	38.72	43.83	5.43	5.96
Protactinium	Pa	91	95.87	108.43	13.29	16.7
Radium	Ra	88	88.47	100.13	12.34	15.24
Radon	Rn	86	83.78	94.87	11.73	14.32
Rhenium	Re	75	61.14	69.31	8.65	10.01
Rhodium	Rh	45	20.22	22.72	2.7	2.83
Rubidium	Rb	37	13.4	14.96	1.69	1.75
Ruthenium	Ru	44	19.28	21.66	2.56	2.68
Samarium	Sm	62	40.12	45.41	5.64	6.21
Scandium	Sc	21	4.09	4.46	0.4	0.4
Selenium	Se	34	11.22	12.5	1.38	1.42
Silicon	Si	14	1.74	1.84	0	0
Silver	Ag	47	22.16	24.94	2.98	3.15
Sodium	Na	11	1.04	1.07	0	0
Strontium	Sr	38	14.17	15.84	1.81	1.87
Sulfur	S	16	2.31	2.46	0	0
Tantalum	Ta	73	57.53	65.22	8.15	9.34
Technetium	Tc	43	18.37	20.62	2.42	2.54
Tellurium	Te	52	27.47	31	3.77	4.03
Terbium	Tb	65	44.48	50.38	6.27	6.98
Thallium	Tl	81	72.87	82.58	10.27	12.21
Thorium	Th	90	93.35	105.61	12.97	16.2
Thulium	Tm	69	50.74	57.52	7.18	8.1
Tin	Sn	50	25.27	28.49	3.44	3.66
Titanium	Ti	22	4.51	4.93	0.45	0.46
Tungsten	W	74	59.32	67.24	8.4	9.67
Uranium	U	92	98.44	111.3	13.61	17.22
Vanadium	V	23	4.95	5.43	0.51	0.52
Xenon	Xe	54	29.78	33.62	4.11	4.42
Ytterbium	Yb	70	52.39	59.37	7.42	8.4
Yttrium	Y	39	14.96	16.74	1.92	2
Zinc	Zn	30	8.64	9.57	1.01	1.03
Zirconium	Zr	40	15.78	17.67	2.04	2.12

COLLECTION, ANALYSIS AND DISPOSAL OF ESAT LABORATORY WASTE

APPROVAL PAGE

Written By: David B Roberts Jr — 1-18-07
ESAT Senior Analytical Chemist — Date

ESAT Review: — 1/17/2007
ESAT QA/QC Coordinator — Date

ESAT Approval: — 1/17/2007
ESAT Regional Manager — Date

EPA Review:
Gregory Saunders — Date
EPA Region 8 Laboratory Health and Safety

TABLE OF CONTENTS

1.0 SCOPE AND APPLICATION

1.1 This Standard Operating Procedure describes the procedures and practices for safely collecting, analyzing and storing aqueous corrosive wastes.

1.2 This procedure is applicable to the following aqueous waste streams; acidic instrument waste, acidic reagents (except standards), sample digestates and samples preserved for analysis with acid.

1.3 This procedure is applicable to solid wastes such as soils, vegetation and biota samples.

2.0 SUMMARY OF PROCEDURE

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

2.2 Solid wastes may contain metal concentrations which exceed disposal standards.

2.2 This waste must be properly labeled, contained and stored in accordance with all state, federal and ESAT regulations.
- 2.2.1 This procedure includes the initiation of satellite waste containers, documentation accompanying the waste and procedures for placing the waste in the designated waste storage area.

3.0 DEFINITIONS / ACRONYMS

3.1 Aqueous Corrosive Waste - An aqueous solution with a pH<2.

3.2 Evidentiary Materials – Samples, sample containers and sample residuals.

3.3 F Waste: Hazardous waste from nonspecific sources (See 40 CFR 261.31.).

3.4 Satellite Waste Container - A container used to collect waste during generation.

3.5 Secondary Containment
- 3.5.1 A second level of containment ensuring no release if the initial containment fails.

3.6 Waste Control Officer (WCO)
- 3.6.1 Person responsible for maintaining control of all documentation of waste disposal.

4.0 HEALTH AND SAFETY

4.1 All pertinent procedures outlined in the EPA Region 8 Chemical Hygiene Plan (CHP) will be followed in performance of the handling of wastes.
  4.1.1 The use of laboratory equipment and chemicals exposes the analyst to several potential hazards.
  4.1.2 Good laboratory technique and safety practices should be followed at all times.

4.2 Solutions classified as aqueous corrosive wastes normally contain percentage levels of mineral acids and can contain certain inorganic elements known to be hazardous.
  4.2.1 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.

4.3 Satellite waste containers must always be tightly capped when not in use.

4.4 Satellite waste containers can weigh in excess of 50 lbs and should be lifted carefully.

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

5.0 CAUTIONS

5.1 Prior to starting work that involves handling wastes, personnel should review the project plan safety requirements, analytical procedure safety requirements, and this waste management procedure.
  5.1.1 When in doubt as to the proper procedure to follow, contact the Health and Safety Officer for guidance.
  5.1.2 Personnel should minimize exposure to potential health hazards through the use of engineering and administrative controls, work practice procedures and practices, and proper protective equipment.

6.0 PERSONNEL QUALIFICATIONS

6.1 Personnel Responsibilities
  6.1.1 Personnel responsibilities for hazardous waste management at 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).

6.2 Personnel Training
  6.2.1 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.
  6.2.2 This training is designed to keep employees familiar with waste handling procedures in place at EPA, along with applicable regulations.

  6.2.3 Training completion will be enforced by the supervisor and documented for each individual by the ESAT Health and Safety Officer.

7.0 EQUIPMENT AND SUPPLIES

7.1 The WCO (Waste Control Officer) will assure that a supply of appropriate waste containers and labels are available for use.

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

8.0 SATELLITE WASTE CONTAINER PREPARATION

8.1 The waste container will be properly labeled as appropriate.
  8.1.1 A Waste Container ID is assigned to the container by the person who initiates the container's designation.
    8.1.1.1 The container ID will be written on the container with a Sharpie in such a location as to be easily seen.
  8.1.2 The Container ID will follow the YYMMDD-XXXX-# format where "XXXX" describes the laboratory room number and the "#" will be the number of the container generated on that particular day.
  8.1.3 Affix an appropriate red and white hazardous label for containers used for suspected or known hazardous materials.
  8.1.4 Affix a black and white label warning of the presence of corrosives (pH<2) when the contents to be added to the container are known to be acidic.

8.2 A waste container inventory log will be initiated and placed next to the container.
  8.2.1 An attached (or located in the near vicinity) hazardous waste container inventory log, listing the accumulated waste maintained by the generator(s).
  8.2.2 The waste inventory sheet should 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 sheet serves two important purposes. It guards against addition of incompatible chemicals to the container mix and allows packers to determine the correct classification of the waste for transport and disposal.*

9.0 WASTE COLLECTION AND ANALYSIS

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

9.2 Containers must be kept closed except when waste is being added.

9.3 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.

9.4 Full aqueous waste containers will be sub-sampled, the waste inventory sheet filled out and transported to the waste storage area within 24 hours.
  9.4.1 The waste inventory sheet must be clearly labeled as "Awaiting analysis".

9.5 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: D00I, ignitable liquids; D002, corrosive materials; D003, reactive materials; and D004 to D043, toxic materials.
  9.5.1 Each unit will be labeled with the start of accumulation date and, as appropriate, a "hazardous waste" or "non-hazardous waste" label.

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

9.7 Solid wastes designated for disposal are collected in an approved container.
  9.7.1 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.
  9.7.2 In general, segregating the solids into LIMS workorder specific groups will make calculating the metals concentration less complicated.

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

10.0 DATA AND RECORDS MANAGEMENT

10.1 At EPA Region 8 laboratory, 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.

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

11.0 WASTE MINIMIZATION

11.1 Metals laden waste volumes are minimized by the use of a dedicated waste receptacle in which no other laboratory waste is placed.
  11.1.1 Waste concentrations are minimized by judicious use of metals standard solutions and materials.
  11.1.2 In addition, ESAT chemists are working with field personnel to reduce the amount of excess sample collected.

12.0 REFERENCES

12.1 EPA Region 8, Chemical Hygiene Plan, current version.

12.2 EPA Region 8, Health and Safety Plan, current version.

12.3 EPA Region 8, Waste Management SOP, current version.

16-LAB-05.03
SAMPLE RECEIPT, CUSTODY, STORAGE, AND LIMS DATA ENTRY

Technical Approval: Scott C. Walker
PRINT NAME

SIGNATURE **Date:** 8/6/2014

QA Management Approval: Nikki Thomsen
PRINT NAME

SIGNATURE **Date:** 08/06/14

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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

°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.

6.1.12 Dip a non-bleeding pH indicator strip into sample container 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 version

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

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

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

ESAT Region 8 Health and Safety Plan, current version

Document Change History			
Revision No.	**Status[1] (I, R, C)**	**Effective Date**	**Changes Made**
0	I	01/17/07	Not applicable
2	R	05/06/09	Entire document reviewed and updated (note that Revision 1 of this SOP was never finalized or approved, and therefore, not included in the document change history)
3	R	08/06/14	Entire document reviewed and updated

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

TECHLAW STANDARD OPERATING PROCEDURES

FIELD PROCEDURES — ANALYTICAL SUPPORT AND LABORATORY SELECTION

SOP Number: 02-06-05
Effective Date: 04/27/12

Technical Approval: ____Date:____________

QA Management Approval: ___________________________________Date:__________

SOP Description

This Standard Operating Procedure (SOP) describes the process to be followed by TechLaw 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 project manager 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 forms to the LAT and forwarding invoices to the project files. A LAT member checklist is provided in Attachment D and a TechLaw 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

Procedures for Submitting and Processing the Form

The ASRF (see Attachment B) 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. If supplies are not required from the laboratory, the ASRF may be submitted at least 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. Faxed, hand-written requests or electronic requests are acceptable as long as they are legible and complete.

The assigned LAT member will ensure that all necessary information is included in the ASRF, and make 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 SharePoint. All samples must be sent to a TechLaw-approved laboratory. The laboratory is selected on the ability to perform the requested analysis, availability of laboratory space, and analysis cost. 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 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.

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; and
- 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

Chain-of-Custody (COC) Forms

Within one week of shipment of samples (within 24 hours ideally), the project manager must ensure that legible copies of all COC forms have been sent to the assigned LAT member. Faxes or scanned e-mail copies are acceptable.

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 project manager, unless the project manager 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 Accounts Payable and send a copy to the TechLaw Project Manager. A copy of the approved invoice is uploaded by the LAT member to the company intranet site, under LAT > Project Files.

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 Quality Assurance Program Plan 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-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 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 plan (must include a description and number of instruments, staff resumes and a summary of quality assurance/quality control procedures), SOPs, method detection limits (MDLs), quantitation limits, PE sample results and pricing.

A laboratory audit may also be performed by the TechLaw LAT. Audits will be performed for any laboratory that will be utilized on an ongoing basis. A laboratory audit may not be required if so directed by the client, or if only a specialty analysis is requested and the laboratory will not be used on a routine basis. The laboratory audit checklist is included as Attachment F to this SOP.

After a laboratory has been approved by the LAT, a Laboratory Agreement shall be arranged by the TechLaw 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 annually, as necessary. LAT laboratories are contacted to obtain any updated laboratory documentation and pricing. Audits will be conducted at least every other year for laboratories performing routine analyses in the program.

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 chain-of-custody). Facility initials, TechLaw project number or other identifier should be used that will not reveal facility information to the laboratory, but will be evident to TechLaw employees involved with the project. If the laboratory becomes aware of the site name, the LAT member should inform the TechLaw 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 Project Manager Checklist

Attachment F — Laboratory Audit Checklist

References

TechLaw, Corporate Quality Management Plan, most current revision.

TechLaw, Health and Safety Program, most current version

ATTACHMENT A [Revised 04/27/12]
SOP Number: 02-06-05

TechLaw, Inc. Approved LAT Members

LAT Coordinator: Ms. Kim Whitlock
205 West Wacker Drive, Suite 1622
Chicago, IL 60606
(312) 345-8930
(217) 721-5483 (mobile)

LAT Members:

Ms. Rachel Ireland 7 Technology Drive, Unit 202 North Chelmsford, MA 01863 (978) 275-9749 (617) 283-1332 (mobile)	Ms. Zara Brown 14500 Avion Parkway, Suite 300 Chantilly, VA 20151 (703) 818-3213 (865) 898-3815 (mobile)
Mr. Gene Nance 5455 County Road 2 Chesapeake, OH 45619 (740) 867-0968 (304) 830-1442 (mobile)	Ms. Amy Dahl 1325 4th Ave, Suite 555 Seattle, WA 98101 (206) 826-5375 (206) 818-8891 (mobile)
Mr. Scott Walker 16194 W. 45th Dr. Golden, CO 80403 (303) 312-7726 (303) 453-9018 (mobile)	

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

Ms. Jana Dawson
14500 Avion Parkway, Suite 300
Chantilly, VA 20151
(703) 818-3254
(703) 627-0821 (mobile)

Mr. Terry Zdon
1299 Washington Ave, Suite 270
Golden, CO 80401
(303) 552-5807
(773) 343-8974 (mobile)

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

ATTACHMENT B [Revised 04/27/12]
SOP Number: 02-06-05

ANALYTICAL SUPPORT REQUEST FORM

Project Number (Bill Code): ___________________ TechLaw 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 Project Manager Signature: ____________________ Date: ________________

LAT Signature:________________________ Date: ____________________

ATTACHMENT C [Revised 04/27/12]
SOP Number: 02-06-05

Date:

To: Contact
Laboratory
Address
Address

From: Kim Whitlock, LAT Coordinator
205 West Wacker Drive, Suite 1622
Chicago, IL 60606
(312) 345-8930
(312) 345-8979 (fax)

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

This document authorizes work on the subject Task Order as outlined in the attached Scope of Work and Pricing quotation (Attachment A). The expenditure limits on the Task Order are XXXXX. 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 the form, faxing a signed copy of this Task Order Authorization Form to Brenda Smith at (703) 818-8813, and returning the original within 5 days of your receipt to [LAT member name]. 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, Inc. 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, Inc.	**Laboratory Name**
Name:	Name:
Title:	Title:
Date:	Date:

ATTACHMENT D [Revised 04/27/121]
SOP Number: 02-06-05

LAT Member Checklist

1. If a TechLaw project manager contacts you regarding laboratory procurement, please ask them to fill out the Analytical Support Request Form (ASRF) in Attachment B of SOP 02-06-05, 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 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 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. Fill out the Work Authorization Form in Appendix C of SOP 02-06-05, PDF file, and attach the analytical quote to end of the PDF. Submit the form to the selected laboratory via e-mail (copy Ms. Brenda Smith and Ms. Judy Manley). Ms. Smith will email the LAT member the PO associated with the analytical request.
5. 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.
6. Create a folder for your project under LAT > Project Files.
7. Update the Project Tracking spreadsheet under LAT > Project Tracking and ensure justification for laboratory selection is included in the Comments column.
8. Upon submission to the laboratory, upload a copy of the Work Authorization Form (unsigned by lab) to LAT > Project Files > Project Name.
9. Once a signed copy of the Work Authorization Form is received from laboratory, upload to LAT > Project Files > Project Name.
10. Confirm with laboratory when/where to send bottleware.
11. Inform the laboratory when to expect samples (it is also a good idea to remind them one day before they will receive samples).
12. Ensure the TechLaw field team submits a copy of the COC to you for verification of invoicing/sample data.
13. 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.
14. If the items above are acceptable, approve invoice by signing, dating, and adding the proper PO number (including line item number) and forward to Patti Pinkard.
15. Upload a copy of the approved invoice to the project folder under LAT > Project Files.
16. Email a copy of the approved invoice to the TechLaw project manager.
17. Ask the TechLaw Project Manager where the data should be sent, and send out ASAP. If electronic data is available, upload a copy to the project folder under LAT > Project Files

ATTACHMENT E [Revised 04/27/12]
SOP Number: 02-06-05

TechLaw Project Manager Checklist for LAT

1. Fill out the Analytical Support Request Form in Attachment B of SOP 02-06-05.
2. Notify LAT member of any changes in schedule/requirements as soon as possible.
3. Ensure Field Team provides a copy of completed COCs to LAT member.
4. 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 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 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)

LABORATORY AUDIT CHECKLIST

Laboratory On-Site Visits

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 Program – includes all aspects of EPA's "Good Automated Laboratory Practices" (GALP) Guidance.
    - Appropriate evidentiary procedures, Chain-of-Custody 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 exists, 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 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 at a minimum.

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 Technical and Evidentiary on site visits (audits) are attached. They are designed to be used together 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.

If items (e.g., SOPs) are addressed in the evidentiary section, then they are not addressed in the technical section. Since there is so much in common for organics and inorganics, the checklist is combined, with items unique to one or the other clearly identified.

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.

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Laboratory On-site Visit Evaluation

Laboratory name:

Address:

Telephone number:

Date of in-site visit:

Type of evaluation 1) organics 2) inorganics 3) evidentiary

Evaluation Team Members

Name	Title	Evaluated 1, 2 or 3 above?

Personnel interviewed/contacted

Name	Title	Interviewed for 1, 2 or 3 above?

Evidentiary Procedures Evaluation Checklist

I. Sample Receipt

	Check one Yes	No
1. Is there a designated sample custodian and alternate for each shift?	_______	_______

Names:

Sample custodians	Alternates
________________________	____________________________
________________________	____________________________

	Yes	No
2. Are standard operating procedures (SOPs) for sample receipt in place and readily available?	_______	_______
3. Are SOPs for sample receipt followed by laboratory personnel?	_______	_______
4. Is the sample receipt area secured against non-authorized personnel?	_______	_______
5. Does sample custodian verify 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 chain-of-custody record(s)	_______	_______
f. Presence or absence of airbill stickers	_______	_______
g. Airbill or airbill sticker number	_______	_______
h. Presence or absence of sample tags	_______	_______
i. Sample tag numbers (if applicable)	_______	_______
j. Condition of sample containers	_______	_______
k. Discrepancies in any information recorded on chain-of-custody records, client requests, airbills, sample containers, etc.	_______	_______

			Yes	No
		l. Documentation of hand deliveries	_______	_______
		m. Problems encountered	_______	_______
	6.	Obtain examples of all forms used during sample receipt	_______	_______
II.	Sample Identification			
	1.	Does the laboratory have a unique sample identification system (i.e., vs. using client sample identification numbers)?	_______	_______
		a. Is the number assigned upon receipt? If no when?____________________________	_______	_______
		b. If "yes", are numbers cross referenced to client numbers in a log?	_______	_______
	2.	Does the system clearly apply to samples, extracts, digestates, etc.?	_______	_______
	3.	Are SOPs readily available for sample identification?	_______	_______
	4.	Are SOPs for sample identification followed by laboratory personnel?	_______	_______
	5.	Obtain example of laboratory's sample identification system (e.g., example sample number with cross reference)	_______	_______
III.	Sample Storage and Tracking			
	1.	Are sample (and extracts, etc.) storage areas secured and access to samples (extracts, etc.) available only to authorized personnel?	_______	_______
	2.	Are samples (extracts, etc.) logged in/out of storage area(s) when accessed?	_______	_______
	3.	Are samples (extracts, etc.) tracked throughout analytical process (e.g., a traveler sheet)?	_______	_______
	4.	Are SOPs for storage and tracking of samples (extracts, etc.) readily available?	_______	_______

		Yes	No
5.	Are SOPs for storage and tracking followed by laboratory personnel?	_______	_______
6.	Obtain examples of all forms/documents used for storage and tracking records	_______	_______

IV. Document Review

		Yes	No
1.	Evaluate documents with the following questions in mind.	_______	_______
a.	Are activities (e.g., GC/MS-VOA, ICP-metals) identified on all analysis documents?	_______	_______
b.	Are titles on all documents?	_______	_______
c.	Are columns labeled with headers?	_______	_______
d.	Are reviewers’ signatures identified when applicable?	_______	_______
e.	Is the laboratory’s name on all documents?	_______	_______
f.	Are all entries fully dated (day, month, year)?	_______	_______
g.	Are entries signed by the responsible person for performing and recording activities?	_______	_______
h.	Are all logbook and other document entries in ink?	_______	_______
i.	Is error correction protocol followed? (single line through area to be corrected and corrector’s initials – no “white out”)	_______	_______
j.	Are pages in bound and unbound logbooks sequentially numbered?	_______	_______
k.	Are log-book entries in chronological order?	_______	_______
l.	Is inserted information taped into logbooks signed and dated when activity is performed?	_______	_______
m.	Are unused portions of documents lined out?	_______	_______

	Yes	No
2. Do documents provide a complete record of activities observed by the evaluator?	_______	_______
3. Are instrument run logs maintained to enable a reconstruction of the run sequence on an instrument?	_______	_______
4. Are records of failed runs maintained?	_______	_______
5. Is disposal/depletion of samples (extracts, etc.) documented?	_______	_______
6. Is data transferred electronically within the laboratory? If “yes”, is a hard copy printed and retained in a client/case file?	_______	_______
a. If data is transferred electronically, is the following information recorded?	_______	_______
1) Person responsible for electronic data transfer?	_______	_______
2) Date of electronic transfer?	_______	_______
3) Person to whom data was electronically transferred?	_______	_______
4) Status of electronically transferred data (e.g., draft final, etc.)?	_______	_______
5) Numerical identifier assigned to electronic data transfer?	_______	_______
b. Are SOPs readily available for electronic data transfer?	_______	_______
c. Are SOPs followed by laboratory personnel for electronic data transfer?	_______	_______

V. <u>Confidential Information</u>

1. If laboratory receives confidential information/documents, 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

Name:_______________ ____________________
Document Control Officer Alternate

		Yes	No
1.	Are case documents maintained in a secure area?	_______	_______
2.	Is shipment of deliverables to clients documented?	_______	_______
	a. Is recipient identified?	_______	_______
3.	Are deliverables sealed with custody seals?	_______	_______
	a. Are custody seals signed?	_______	_______
	b. Are custody seals dated?	_______	_______
4.	Does document control officer assemble and cross check information to assure that data on each case file is consistent and complete?	_______	_______

VII. Security of the Facility

		Yes	No
1.	Are visitors required to sign in?	_______	_______
2.	Are visitors required to display distinct badges/ID?	_______	_______
3.	Are all doors to outside locked except to reception area?	_______	_______
4.	Is access to laboratory and data reduction/report preparation areas limited to authorized personnel?	_______	_______

Observations/Comments by Evaluators

I Sample receipt –

II Sample identification –

III Sample storage/tracking –

IV Document review –

V Confidential information –

VI Case file organization and assembly –

VII Security of facility –

Technical Procedures Evaluation Checklist
(Organics and Inorganics)

		Yes	No
I.	Sample Receipt and Storage Area (Evaluate with I-III of Evidentiary Audit)		
1.	Are sample shipping coolers opened in a contamination-free area (e.g., fume hood or vented area)?	_______	_______
2.	Are adequate facilities provided for the cold storage of samples and unused samples for 60 days after data submission?	_______	_______
a.	Is the temperature of the cold storage recorded daily in a logbook?	_______	_______
b.	Are temperature excursions noted and appropriate actions taken when required? (Check SOP)	_______	_______
3.	For inorganic only: Is the pH of the samples recorded and available for the data review?	_______	_______
4.	For organic only: Are volatile samples stored separately from semi-volatile samples and extracts?	_______	_______
5.	For organic only: Are VOA holding blanks present in the volatile sample storage facility? (One per case)	_______	_______
6.	For organic only: Are sample extracts properly stored (2-6oC, separate) and easy to locate by reference to a logbook?	_______	_______
II.	Sample Preparation Area		
1.	Is the laboratory maintained in a clean and organized manner appropriate for trace level analyses (contamination free)?	_______	_______
2.	Does the laboratory appear to have adequate work space (6 linear feet or unencumbered bench top/analyst)?	_______	_______

			Yes	No
3.	Are laboratory benches made of suitable chemically resistant materials?		_______	_______
4.	Are sufficient functional hoods available?		_______	_______
5.	Is documented organic free water (for organics standards, blanks, dilutions) or distilled/ demineralized water (for inorganics) available?		_______	_______
6.	Are analytical balances located away from drafts and areas subject to rapid temperature changes?		_______	_______
	a.	Are the balances checked routinely (e.g., before each weighing session) with the appropriate range of weights and results recorded in a permanent notebook?	_______	_______
	b.	Are routine weights checked against class S weights at least once a month and Results recorded in a permanent notebook?	_______	_______
	c.	Have the balances been calibrated within one year by a certified technician?	_______	_______
	d.	Are the data generated from balances electronically transferred or manually entered into LIMS?	_______	_______
7.	Are sample preparation SOPs readily available?		_______	_______
	-	Are sample preparation SOPs followed by laboratory personnel?	_______	_______
8.	Are glassware preparation/cleaning SOPs readily available?		_______	_______
	-	Are they followed by laboratory personnel?	_______	_______
9.	Is all required sample preparation equipment available?		_______	_______
	<u>For organics only</u>:			
	a.	Sonicator	_______	_______

Make______Model________Backup_______(Y/N)

Yes No

b. GPC _______ _______

Make______Model________Backup_______ (Y/N)

c. GPC UV Detector _______ _______

Make______Model________

d. Do GPC logs indicate corrective actions are taken when there is a problem with calibration? _______ _______

e. Continuous liquid/liquid extractors?
Number_______ _______ _______

For inorganics only:

f. If microwave digestion is to be used, is an adequate microwave ovens (programmable power setting up to 600 watts) available? _______ _______

10. Do analysts record bench data in a neat and accurate manner? _______ _______

11. Do analysts record lot number of solvents, spiking solutions, etc., on bench sheets? _______ _______

12. Is there evidence of a secondary review of all documents and logbooks by someone other than the person generating the documents? _______ _______

13. Review the following procedures for oven drying:

a. Are the temperatures in the drying ovens verified against NIST traceable thermometers? _______ _______

b. Do ovens have temperature logbooks? _______ _______

c. Are “in” & “out” drying times recorded? _______ _______

III. Standards Preparation and Storage

1. Are SOPs for standards preparation readily available? _______ _______

		Yes	No
2.	Are SOPs followed by laboratory personnel?	_______	_______
3.	Are reagent grade or higher purity chemicals used to prepare standards?	_______	_______
4.	Are standards properly labeled with concentrations, date of preparation, expiration date, and/or a traceable reference code number?	_______	_______
5.	Are spiking/calibration standards preparation and tracking logbooks maintained for:	_______	_______
	Inorganics?	_______	_______
	Semi-volatiles?	_______	_______
	Pesticides?	_______	_______
	Volatiles?	_______	_______
	- Are logbook numbers and series of stock Solutions and reagents recorded?	_______	_______
6.	If the laboratory purchases commercially prepared standard mixes, is appropriate documentation available? (Manufacturer's Certificate of Analysis)	_______	_______
7.	<u>Inorganic only</u>: If the laboratory uses automatic pipets for preparing standards, are they routinely calibrated?	_______	_______

IV. <u>Analytical Instrumentation and Analyses-Specific Items</u>

<u>ORGANICS</u>

		Yes	No
A.	<u>GC/M's (for up 200 samples/month)</u>	_______	_______
1.	Instrument needs	_______	_______
	a. 1 VOA GC/MS/DS with purge and trap device	_______	_______
	b. 2 semi-VOA GC/MS	_______	_______
	c. 1 Backup GC/MS/DS and purge and trap device	_______	_______

GC/MS/DS			Purge and Trap	
Instr ID	Manuf./model	Software rev.	InstrID	Manuf/model

		Yes	No
2.	Are manufacturers' operating manuals readily available?	_______	_______
3.	Does laboratory have service contracts?	_______	_______
	a. Does lab have extensive replacement parts available?	_______	_______
4.	Is a permanent service record maintained for each instrument?	_______	_______
5.	Does the laboratory use a recent mass spectral library?	_______	_______
6.	Magnetic tape storage of GC/MS electronic data:	_______	_______
	a. Are raw data, including quantitative output files and libraries, archived on magnetic tape?	_______	_______
	b. Is a log of raw data contents of tapes maintained?	_______	_______
7.	VOA analyses:	_______	_______
	a. Is equipment available for heated purge and trap for low level soil analysis?	_______	_______
	b. Are VOA holding blanks results available?	_______	_______
8.	Can instrument operator show from the run log that corrective actions have been taken for:	_______	_______

			Yes	No
	a.	Re-analyses when internal standard areas are out of control?	_______	_______
	b.	Dilutions when calibration range is exceeded?	_______	_______
	c.	Blanks when previous sample showed saturation?	_______	_______
9.		Are SOPs readily available for GC/MS analyses and logbook completion?	_______	_______
		Are they followed by the laboratory?	_______	_______
10.		Is there evidence of a secondary review of all documents and logbooks by someone other than the person generating the documents?	_______	_______
B.		<u>GC/EC's (for up to 200 samples/month)</u>	_______	_______
1.		Instrument needs	_______	_______
	a.	2 GC/EC/DS with dual columns	_______	_______
	b.	1 backup GC/EC	_______	_______

<u>GC/EC</u> Instr ID	Manuf./Model	Detector	<u>Data System</u> Manuf./Model	Revision #

			Yes	No
2.		Are manufacturers' operating manuals readily available?	_______	_______
3.		Does the laboratory have service contracts?	_______	_______
	a.	Does the lab have extensive replacement parts available?	_______	_______
4.		Is a permanent service record maintained for each instrument?	_______	_______

		Yes	No
5.	Are SOPs readily available for GC/EC analyses and logbook completion?	_______	_______
	a. Are they followed by laboratory personnel?	_______	_______
6.	Is there any evidence of a secondary review of all documents by someone other than the person generating the document?	_______	_______

INORGANICS

A. ICPs (for up to 300 samples/month)

1.	Instrument needs	_______	_______
	a. 1 ICP	_______	_______

Instrument ID	Manuf./Model	Seq./Sim

		Yes	No
2.	Are manufacturers' operating manuals readily available?	_______	_______
3.	Does laboratory have service contracts?	_______	_______
4.	Are SOPs readily available for ICP analyses and logbook completion?	_______	_______
	Are they followed?	_______	_______
5.	Are stock standards current?	_______	_______
6.	Are calibration standards made from a ready made stock standard? Manufacturer?_________	_______	_______
	Are they prepared at least monthly?	_______	_______

Yes No

7. Have any of the instruments been modified? _______ _______

If "yes", which one and how?__________________
____
____

8. How will calibration intensity and gains be kept?____
____

9. Is a mass flow controller used? _______ _______

10. Is interference correction done automatically and are interelement correction factors determined on at least an annual basis? _______ _______

11. Is a permanent service record maintained for each instrument? _______ _______

B. ICP-MS (for up to 300 samples/month)

1. Instrument needs _______ _______

a. 1 ICP-MS _______ _______

Instrument ID Manuf./Model Seq./Sim
____
____
____
____

2. Are manufacturers' operating manuals readily available? _______ _______

3. Does laboratory have service contracts? _______ _______

4. Are SOPs readily available for ICP-MS analyses and logbook completion? _______ _______

Are they followed? _______ _______

5. Are stock standards current? _______ _______

6. Are calibration standards made from a ready made stock standard? Manufacturer?_________ _______ _______
Are they prepared at least monthly? _______ _______

		Yes	No
7.	Have any of the instruments been modified?	_______	_______

If "yes", which one and how?__________________
____
____

8. How will calibration intensity and gains be kept?____
____

9.	Are reanalysis performed when internal standards are out of control?	_______	_______
10.	Is a mass flow controller used?	_______	_______
11.	Is interference correction done automatically and are interelement correction factors determined on at least an annual basis?	_______	_______
12.	Is a permanent service record maintained for each instrument?	_______	_______

C. Atomic Absorption (AA) Spectrometer

1.	Instrument needs		
	a. 2 GFAA for up to 200 samples/month	_______	_______
	b. 4 GFAA for up to 400 samples/month	_______	_______

GFAAs

Instrument ID Manuf./Model
____
____
____
____

2.	Are element specific SOPs which list instrument conditions, background corrections, and required instrument sensitivity readily available?	_______	_______
	Are they followed?	_______	_______
3.	Are calibration results (i.e., sensitivity) kept in a permanent record to track instrument performance?	_______	_______

		Yes	No
4.	Are calibration standards prepared daily?	_______	_______
5.	Have any of the instruments been modified?	_______	_______

If "yes", which ones and how?_________________
____

		Yes	No
6.	Is an autosampler used?	_______	_______
7.	Are EPA or instrument manufacturers matrix modifiers used?	_______	_______

As: ______________________
Pb: ______________________
Se: ______________________
Tl: ______________________

		Yes	No
8.	Is a permanent service record maintained for each instrument?	_______	_______

D. Mercury analyzer – Cold Vapor AAs

		Yes	No
1.	Instrument needs.	_______	_______
a.	2 mercury cold vapor AAs for up 300 samples/month.	_______	_______

Cold Vapor AA
Instrument ID's Manuf./Model
____
____
____

		Yes	No
2.	Are SOPs readily available for analyses and logbook completion?	_______	_______
	Are they followed?	_______	_______
3.	Are calibration standards prepared daily?	_______	_______
	With the samples?	_______	_______
4.	Is an absorbance record kept to monitor sensitivity?	_______	_______
5.	Is a permanent service record maintained for each instrument?	_______	_______

E. Cyanide Distillation Apparatus _______ _______

1. Needs – 12 distillation apparatuses plus one photometer for up to 300 samples/month _______ _______

Photometer

Instrument ID's Manuf./Model

____
____
____

		Yes	No
2.	Are SOPs readily available for cyanide analysis?	_______	_______
	Are they followed?	_______	_______
3.	Is there a stock cyanide standard from a commercial source?	_______	_______
	If "no", made from KCN salt?	_______	_______
	Is standard titrated?	_______	_______
4.	Is the titrimetric manual or semi-automated colorimetric method used? Method:______________________	_______	_______
5.	Is the pH of the samples recorded and available for review?	_______	_______
6.	Are samples checked for the presence of sulfide and chlorine?	_______	_______
7.	Is a service log maintained for the photometer?	_______	_______
V.	Data Handling and Review (GALP)	_______	_______
1.	Are data calculations spot checked by a second person?	_______	_______
2.	Do records indicate appropriate corrective action when QC criteria are not met?	_______	_______
3.	Do supervisory personnel review the data and QC results prior to submission?	_______	_______
4.	Are SOPs for data handling/review readily available?	_______	_______
	Are they followed?	_______	_______

		Yes	No
5.	Are data and file access user ID of file password protected?	_______	_______
6.	Are deliverables checked for completeness and accuracy? (Hard copy and electronic)	_______	_______
Resubmittals?		_______	_______
7.	Is the monthly data entry error rate determined and recorded?	_______	_______
8.	When changes to deliverables are required, are the changes properly documented? (Rationale, review, initials.)	_______	_______
9.	Are user manuals and operations/systems manuals available?	_______	_______
10.	Is a written software test and acceptance plan available for installation of system changes?	_______	_______
VI.	<u>Quality Assurance Internal Inspections</u>		
1.	Is there an internal QA inspection procedure?	_______	_______
2.	Does the QA officer report to senior management?	_______	_______
3.	Are corrective actions documented?	_______	_______
4.	What kinds of internal audits are performed?	_______	_______
	a. Blind PE sample?	_______	_______
	b. Other:____________________ __________________________	_______	_______
5.	What kinds of QA records are kept?		
	a. PE sample results?	_______	_______
	b. Records of recoveries (extractions, etc.)	_______	_______
	c. Training/experience records of personnel?	_______	_______
	d. Method sensitivities?	_______	_______

			Yes	No
	e.	Control charts for QC purposes?	_______	_______
	f.	Other____________________		
VII.	Quality Assurance Plan (QAP)			
1.	Is a QAP available?		_______	_______
2.	Does it address 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	_______	_______
VIII.	Standard Operating Procedures			
1.	Are SOPs available for the following (many already addressed earlier during the audit)?		_______	_______
	a.	Evidentiary	_______	_______
	b.	Sample Receipt and storage	_______	_______
	c.	Sample preparation	_______	_______
	d.	Glassware cleaning	_______	_______
	e.	Calibration (balance)	_______	_______
	f.	Calibration (instruments)	_______	_______
	g.	Analytical procedures (for each system)	_______	_______
	h.	Maintenance activities (for each system)	_______	_______
	i.	Analytical standards	_______	_______
	j.	Data reduction procedures	_______	_______
	k.	Documentation policy/procedures	_______	_______
	l.	Data validation/self inspection procedures	_______	_______
	m.	Data management and handling	_______	_______

		Yes	No
IX.	Organization and Personnel Summary (See “Key Personnel” list – attached)	_______	_______
1.	Do personnel assigned to this project have the appropriate educational background to accomplish the objectives of the program?	_______	_______
2.	Is the organization adequately staffed to meet project commitments in a timely manner?	_______	_______
3.	Were all key personnel available? List those not present.	_______	_______

Name Position

____

____

____

		Yes	No
X.	Laboratory Capacity		
1.	Does the laboratory have sufficient analytical instrumentation to analyze the needed number of samples?	_______	_______
2.	Does the laboratory have sufficient technical administrative personnel to deliver the number of needed analyses?	_______	_______
3.	Does the laboratory have an adequate sample and data tracking system to handle the needed number of analyses?	_______	_______

XI. Summary

Provide an overall evaluation of the laboratory’s apparent technical capability to perform the needed work.

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KEY PERSONNEL LIST

List the personnel assigned to the following functional positions, and put a check next to any unqualified personnel.

Laboratory Operations Personnel

Project Manager

Name:______________________________________
Responsible for overall aspects of contract/project: primary contract

Quality Assurance Officer

Name:______________________________________
Generally requires a Bachelor’s degree in chemistry/science/engineering + 3 years’ laboratory experience, including 1 year applied experience with QA principles and practices in an analytical laboratory.

Laboratory Manager

Name:______________________________________
Responsible for overall management of the laboratory.

Sample Custodian

Name:______________________________________

Laboratory Personnel – Organics

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’ laboratory experience including 1 year supervisory experience. Three additional years experience may substitute for education requirement.

GC/MS Operator
Name:______________________________________
Name:______________________________________
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:______________________________________
Name:______________________________________
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:______________________________________
Name:______________________________________
Generally requires High School diploma and college level course in general chemistry + 1 year experience in extraction/concentration.

Backup Chemists (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:

Laboratory Personnel – Inorganics

Inorganic Laboratory Supervisor

Name:______________________________________
Generally requires a BS or BA in science. 1 year related experience, including 1 year as a supervisor.

ICP/ICP-MS Operator

Name:______________________________________
Name:______________________________________
Generally requires a BS or BA in science, 1 year ICP experience, 3 years additional experience in lieu of the education requirement.

Lachat Operator

Name:_____________________________________
Name:_____________________________________
Generally requires a BS or BA in science, 1 year Lachat experience, 3 years additional experience in lieu of the education requirement.

AA/Mercury Operator

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

Inorganic Sample Preparation Specialist

Name:_____________________________________
Name:_____________________________________
High school diploma and college level course in chemistry and 1 year related experience: 6 months experience plus 6 months experience with microwave digestion, if microwave technique is used.

Wet Chemistry Analyst

Name:_____________________________________
Name:_____________________________________
Generally requires a BS or BA in science and 1 year experience; 3 additional years experience may substitute for education requirement.

Attachment A
Blank 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

EVENT: eCOC Template

Sample #	Tag	Location	Sub Location	Sample Type	Collection	Matrix	Analyses	Preservation	Sample Date	Sample Time	Remarks	Sampler

Relinquished By (DATE/TIME):

Received By (DATE/TIME):

Sampling/Analysis Notes:

Cooler Temp:__________
ICE: Y N
pH: Y N
Cust. Seals: Y N
COC/Labels Agree: Y N
Containers Intact: Y N

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