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ADMINISTRATIVE RECORD

PHASE II
SAMPLING AND ANALYSIS PLAN
FOR OPERABLE UNIT 3
LIBBY ASBESTOS SUPERFUND SITE

Part A: Surface Water and Sediment

Prepared by
U.S. Environmental Protection Agency
Region 8
Denver, CO

With Technical Assistance from:

Syracuse Research Corporation
Denver, CO

and

NewFields Boulder LLC
Boulder, CO

APPROVAL PAGE

Part A of the Phase II Sampling and Analysis Plan for Operable Unit 3 of the Libby Asbestos Superfund Site has been prepared by the U.S. Environmental Protection Agency, Region 8, with technical support from Syracuse Research Corporation and NewFields Boulder LLC, and is approved without conditions.

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3/20/08
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LIST OF ATTACHMENTS

Attachment	Title
A	Detailed Phase I Data Summary
B	Standard Operating Procedures

LIST OF ACRONYMS

AOC	Administrative Order on Consent
CAR	Corrective Action Request
CCV	Continuing Calibration Verification
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
cfs	cubic feet per second
COC	Chain-of-Custody
CSM	Conceptual Site Model
DO	Dissolved Oxygen
DQO	Data Quality Objective
EDD	Electronic Data Deliverable
EDXA	Energy Dispersive X-Ray Analysis
EPA	U.S. Environmental Protection Agency
EPH	Extractable Petroleum Hydrocarbons
FS	Feasibility Study
FSDS	Field Sample Data Sheets
FSP	Field Sampling Plan
FTP	File Transfer Protocol
GC/MS	Gas chromatography/mass spectroscopy
GO	Grid opening
GPS	Global Positioning System
GSD	geometric standard deviation
HASP	Health and Safety Plan
HQ	Hazard Quotient
ICV	Initial Calibration Verification
ID	Identification
IL	Inter-laboratory
ISO	International Organization for Standardization
IS	Internal Standard
KDC	Kootenai Development Corporation
LA	Libby Amphibole
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
MCE	Mixed Cellulose Ester
MCL	Maximum Contaminant Level
MDEQ	Montana Department of Environmental Quality
MFL	Million fibers per liter
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NVLAP	National Voluntary Laboratory Accreditation Program
OU	Operable Unit
PAH	Polyaromatic Hydrocarbon
PCB	Polychlorinated Biphenyl
PDF	Portable Document Format

LIST OF ACRONYMS (cont.)

PE	Performance Evaluation
PLM	Polarized Light Microscopy
PLM-VE	Polarized Light Microscopy Visual Area Estimation Method
PLN	Poisson lognormal
PR	Percent Recovery
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QATS	Quality Assurance Technical Support
QC	Quality Control
RD	Recount Different
RF	Response Factors
RI	Remedial Investigation
RPD	Relative Percent Difference
RPM	Remedial Project Manager
RS	Recount Same
RSD	Relative Standard Deviation
SAED	Selective Area Electron Diffraction
SAP	Sampling and Analysis Plan
SOP	Standard Operating Procedure
SVOC	Semi-volatile Organic Compound
TAL	Target Analyte List
TCL	Target Compound List
TEH	Total Extractable Hydrocarbons
TEM	Transmission Electron Microscopy
TRV	Toxicity Reference Value
USGS	U.S. Geological Survey
VOC	Volatile Organic Compound
VPH	Volatile Petroleum Hydrocarbons

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PHASE II
SAMPLING AND ANALYSIS PLAN
FOR OPERABLE UNIT 3
LIBBY ASBESTOS SUPERFUND SITE

PART A: SURFACE WATER AND SEDIMENT

1.0 PROJECT OVERVIEW

1.1 Purpose of this Document

This document is Part A of the Phase II Sampling and Analysis Plan (SAP) for the collection and analysis of samples of environmental media to support a remedial investigation/feasibility study (RI/FS) within Operable Unit 3 (OU3) of the Libby Asbestos Superfund Site near Libby, Montana. OU3 includes the property in and around the former open pit vermiculite mine that is located northeast of the community of Libby, as well as the geographic area surrounding the former vermiculite mine that has been impacted by releases and subsequent migration of hazardous substances and/or pollutants or contaminants from the mine, including ponds, Rainy Creek, Carney Creek, Fleetwood Creek, and the Kootenai River. Rainy Creek Road is also included in OU3. The exact geographic area of OU3 has not yet been defined but will be based primarily upon the extent of contamination associated with releases from the former vermiculite mine as determined in the remedial investigation (RI) of OU3. The purpose of Part A of the Phase II SAP for OU3 is to guide the collection of data on mining-related contaminants in surface water and sediment in streams and ponds to assess the impact of releases from the mined area. The complete scope of Phase II is expected to include collection of data on other environmental media of potential concern in OU3. Requirements will be described in subsequent parts of the Phase II SAP for OU3. These data will be used to support an RI of OU3, the goal of which is to characterize the nature and extent of mining-related contamination in OU3, and to characterize the nature and level of risk posed by mining-related contamination to human and ecological receptors in OU3.

This SAP contains the elements required for both a field sampling plan (FSP) and quality assurance project plan (QAPP). This SAP has been developed in accordance with Environmental Protection Agency (EPA) Requirements for Quality Assurance Project Plans (EPA 2001) and the Guidance on Systematic Planning Using the Data Quality Objectives Process – EPA QA/G4 (EPA 2006). The SAP is organized as follows:

Section 1 – Project Overview
Section 2 – Background and Problem Definition

Section 3 – Summary of Phase I Data
Section 4 – Data Quality Objectives
Section 5 – Sampling Program
Section 6 – Laboratory Analysis Requirements
Section 7 –Site-Specific Toxicity Testing requirements
Section 8 – Quality Control
Section 9 – Data Management
Section 10 – Assessment and Oversight
Section 11 – Data Validation and Usability
Section 12 – References

1.2 Project Management and Organization

Project Management

EPA is the lead regulatory agency for Superfund activities within OU3. The EPA Remedial Project Manager (RPM) for OU3 is Bonita Lavelle, EPA Region 8. Ms. Lavelle is a principal data user and decision-maker for Superfund activities within OU3.

The Montana Department of Environmental Quality (MDEQ) is the support regulatory agency for Superfund activities within OU3. The MDEQ Project Manager for OU3 is Catherine LeCours. EPA will consult with MDEQ as provided for by the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), the National Contingency Plan, and applicable guidance in conducting Superfund activities within OU3.

EPA has entered into an Administrative Order on Consent (AOC) with Respondents W.R. Grace & Co.-Conn. and Kootenai Development Corporation (KDC). Under the terms of the AOC, W.R. Grace & Co.-Conn. and KDC will implement this SAP. The designated Project Coordinator for Respondents W.R. Grace & Co.-Conn. and KDC is Robert Medler of Remedium Group, Inc.

Technical Support

EPA will be supported in this project by a number of contractors, including:

- Syracuse Research Corporation (SRC) will assist in the development of sampling and analysis plans, in the evaluation and interpretation of the data, and preparation of the baseline risk assessments for OU3.
- NewFields Boulder LLC, working as a subcontractor to SRC, will provide support in development of sampling and analysis plans, evaluation and interpretation of data, mapping and other GIS applications, and design and evaluation of the feasibility study.

Field Sampling Activities

All field sampling activities described in this SAP will be performed by W.R. Grace & Co.-Conn. and KDC, in strict accord with the sampling plans developed by EPA. W.R. Grace & Co.-Conn. and KDC will be supported in this field work by MWH Americas, Inc. (MWH). Individuals responsible for implementation of field sampling activities are listed below:

- MWH Project Director: Michael DeDen
- MWH Project Manager: John D. Garr
- MWH Field Quality Control Officer: Jeremy S. Collyard
- MWH Quality Assurance Officer: Stephanie A. Boehnke

On-Site Field Coordinator

Access to the mine is currently restricted and is controlled by EPA. The on-site point of contact for access to the mine is Courtney Zamora of the U.S. Department of Transportation, John A. Volpe National Transportation Systems Center (Volpe).

Sample Preparation and Analysis

All samples collected as part of the Phase II investigation will be sent for preparation and/or analysis at laboratories selected and approved by EPA.

- All analyses of samples for asbestos will be performed by EMSL Analytical, Inc.
- All analyses of samples for non-asbestos analytes will be performed by Energy Laboratories, Inc. (ELI)
- All samples of soil or soil-like media to be analyzed for asbestos will be prepared for analysis by EPA's soil preparation facility in Denver, CO, operated by CDM.
- All validation and verification activities for asbestos and non-asbestos data will be performed by SRC or their subcontractors.

Data Management

Administration of the master OU3 database for OU3 will be performed by EPA contractors (SRC and NewFields). The primary database administrator will be Lynn Woodbury. She will be responsible for sample tracking, uploading new data, performing error checks to identify inconsistent or missing data, and ensuring that all questionable data are checked and corrected as needed.. When the OU3 database has been populated, checked and validated, relevant asbestos data will be transferred into the Libby2 database for final storage.

2.0 BACKGROUND AND PROBLEM DEFINITION

2.1 Site Description

Libby is a community in northwestern Montana that is located near a large open-pit vermiculite mine. Vermiculite from the mine at Libby is known to be contaminated with amphibole asbestos that includes several different mineralogical classifications, including richterite, winchite, actinolite and tremolite. For the purposes of EPA investigations at the Libby Superfund Site, this mixture is referred to as Libby Amphibole (LA).

Historic mining, milling, and processing of vermiculite at the site are known to have caused releases of vermiculite and LA to the environment. Inhalation of LA associated with the vermiculite is known to have caused a range of adverse health effects in exposed humans, including workers at the mine and processing facilities (Amandus and Wheeler 1987, McDonald et al. 1986, McDonald et al. 2004, Sullivan 2007, Rohs et al. 2007), as well as residents of Libby (Peipens et al. 2003). Based on these adverse effects, EPA listed the Libby Asbestos Site on the National Priorities List in October 2002.

Starting in 2000, EPA began taking a range of cleanup actions at the site to eliminate sources of LA exposure to area residents and workers using CERCLA (or Superfund) authority. Given the size and complexity of the Libby Asbestos Site, EPA designated a number of Operable Units (OUs). In the early stages, efforts were focused mainly on wastes remaining at former vermiculite processing areas including OU1 (the export plant) and OU2 (the screening plant). As work progressed, attention shifted to cleanup of current homes and workplaces in the main residential/commercial areas of Libby, designated by EPA as OU4. To date, Superfund investigation and cleanup activities have been conducted by EPA within OU4 and some of the historic processing areas in and around the town of Libby. Environmental investigations of the nearby town of Troy, designated as OU7, began in the summer of 2007. The Phase I RI for OU3 was implemented in September – October of 2007.

Figure 2-1 shows the location of the mine and a preliminary study area boundary for OU3. EPA established the preliminary study area boundary for the purpose of planning and developing the scope of the RI/FS for OU3. This study area boundary may be revised as data are obtained during the RI for OU3 on the nature and extent of environmental contamination associated with releases that may have occurred from the mine site.

2.2 Basis for Concern at OU3

EPA is concerned with environmental contamination in OU3 because the area is used by humans for logging and a variety of recreational activities, and also because the area is habitat for a wide

range of ecological receptors (both aquatic and terrestrial). Contaminants of potential concern to EPA in OU3 include not only LA, but any other mining-related contaminants that may have been released to the environment.

2.3 Scope and Strategy of the RI at OU3

As noted above, EPA is conducting an RI in OU3 in order to characterize the nature and extent of environmental contamination in OU3 and to evaluate risks to humans and ecological receptors from mining-related contaminants in the environment.

Respondents W.R. Grace & Co.-Conn. and KDC performed the first round of RI sampling (referred to as Phase I) in OU3 in the fall of 2007 in accord with the Phase I Sampling and Analysis Plan for Operable Unit 3 (USEPA 2007). The primary goal of the Phase I investigation was to obtain preliminary data on the levels and spatial distribution of asbestos and also other non-asbestos contaminants that might have been released to the environment in the past as a consequence of the mining and milling activities at the site. A more extensive sampling and analysis effort, referred to as Phase II, will be performed in the spring, summer, and fall of 2008.

One component of the RI at OU3 includes characterizing exposure and risk to aquatic receptors that reside in surface water bodies that may be impacted by releases from the mined area. This includes the waters of Fleetwood Creek, Carney Creek, Rainy Creek, the on-site tailings and Mill Ponds, and potentially (if data indicate), the Kootenai River. Typically, water flow in these surface water features varies seasonally, being highest during the spring snowmelt period. Data are not available for typical flow patterns in Rainy Creek, but are available for several other streams in the area (Figure 2-2). As seen, in these creek, flow usually begins to increase around day 80 (March 20) and peaks around day 140 (May 20), although this can vary widely from year to year. It is expected that flow patterns in Rainy Creek will be generally similar, but may be offset due to differences in elevation, gradient, and slope aspect.

Variation in water flow rate is potentially important because flow might have significant effects on the concentrations and amounts of asbestos and/or non-asbestos contaminants being carried by the water. For example, increases in both dissolved and total metal concentrations in surface water have been observed during spring runoff at other sites investigated by EPA in Region 8 (e.g., ISSI 2001, USEPA 2005). It is not known if asbestos or any other constituent will show similar patterns in the Rainy Creek watershed, but if such seasonal variations do occur, it is important to characterize the timing and magnitude of the variations. For this reason, the Phase II sampling and analysis plan for surface water and sediment is being prepared ahead of the other components of the Phase II SAP, in order to ensure that sample collection can include the spring runoff period. This portion of the Phase II SAP is referred to as Phase IIA. The remaining parts of the Phase II SAP will be provided in subsequent documents, referred to as Phase IIB and Phase IIC.

3.0 SUMMARY OF PHASE I SURFACE WATER AND SEDIMENT DATA

3.1 Sampling Stations

During Phase I, surface water and sediment samples were collected at a total of 24 locations, as shown in Figure 3-1. As seen, sampling stations include a number of locations along Carney Creek, Fleetwood Creek, and Rainy Creek, including ponds and impoundments on these streams, as well as seeps and springs that were located nearby.

3.2 Chemical Analyses

Surface Water

All surface water samples collected during Phase I were analyzed for asbestos, metals and metalloids, petroleum hydrocarbons, anions, and other water quality parameters. In addition, several selected surface water samples were analyzed for a broad suite of other chemicals, including VOCs, SVOCs, pesticides, PCBs, PAHs, nitrogen-containing compounds, and selected radionuclides. These locations were selected specifically to characterize waters generated by the confluence of flows from the upper and lower portions of the mined area. Table 3-1 lists the analytical methods that were employed, and Table 3-2 shows the analyses that were performed at each station.

Sediment

All sediment samples collected during Phase I were analyzed for asbestos, metals and metalloids, petroleum hydrocarbons, and several sediment quality parameters. In addition, several selected sediment samples were analyzed for a broad suite of other chemicals, including cyanide, pesticides, PCBs, VOCs, SVOCs, and PAHs. Table 3-3 lists the analytical methods that were employed, and Table 3-4 shows the analyses that were performed at each station.

3.3 Results for Phase I

Detailed data from the Phase I investigation for both asbestos and non-asbestos analytes are provided in Attachment A. The following sections summarize the results of the Phase I investigation. Data reported here include summary statistics on the detection frequency and observed levels of each analyte evaluated in each medium (surface water and sediment).

In considering these data, it is important to note that detection of a chemical in a site medium may not indicate that a release has occurred, since many of the detected chemicals occur naturally in the environment. In addition, concentration values may tend to vary over geographic area and time (e.g., concentrations may potentially be higher during spring runoff than during the

fall). Therefore, it is important to collect data that provide adequate spatial and temporal representativeness before comparing to benchmarks or using the data to assess potential risk to humans or environmental receptors.

Asbestos in Surface Water

Table 3-5 summarizes the results of the analysis of surface water and seeps for asbestos (LA). Results are expressed in terms of million fibers per liter (MFL). As seen, concentration values of total LA ranged widely (more than four orders of magnitude), from < 0.1 to 125 MFL.

Figure 3-2 is a map that displays the spatial pattern of results. The highest levels were observed in samples located in ponds or impoundments, including the tailings impoundment, the Mill Pond, and the pond on Fleetwood Creek, as well as from several seeps along the south side of the mined area. Levels in lower Rainy Creek (below the Mill Pond) tended to be relatively low. A sample collected just upstream of the confluence of Rainy Creek and the Kootenai River was non-detect.

Asbestos in Sediment

Sediment samples were divided into two fractions (coarse and fine) by sieving. Concentrations of LA in the coarse fraction were measured gravimetrically and expressed as a mass percent (grams of LA per 100 grams of coarse fraction) Concentrations in the fine fraction were measured using polarized light microscopy using a visual area estimation approach (PLM-VE). Results for PLM-VE are expressed as mass percent if the concentration is 1% or higher. If the estimated concentration is <1%, the results are expressed semi-quantitatively, according to the following scheme:

PLM-VE Result	Range of Mass Percent
Bin A (ND)	None detected (likely < 0.05%)
Bin B1 (Trace)	LA detected, > 0% but < 0.2%
Bin B2 (<1%)	LA detected, >0.2% but < 1%

Results that are >1% are categorized as Bin C. Table 3-6 summarize the results of the analysis of asbestos (LA) in sediment. As seen, nearly all (22 out of 24) of the sediment samples collected contain LA. In the fine fraction, values ranged from trace (<0.2%) up to 7%. In the coarse fraction, levels generally ranged from 0.1% to 0.5%.

Figure 3-3 shows the spatial pattern of LA in the fine fraction of sediment. As shown, LA was be detected in most samples, except those collected in the upper-most reaches of Rainy Creek and Fleetwood Creek. Concentrations of 1% or higher (Bin C) were detected in multiple locations. The highest levels observed were in samples collected from on-site seeps.

Results for Non-Asbestos Chemicals in Surface Water

Table 3-7 presents summary statistics on the frequency and level of analytes detected in surface water samples analyzed as part of the Phase I investigation. As seen, a number of inorganic constituents (metals, anions, and nitrogen compounds) were detected, as were several indicators of petroleum hydrocarbons, but no VOCs, SVOCs, PCBs, or PAHs were detected.

Results for Non-Asbestos Chemicals in Sediment

Table 3-8 summarizes the results for analytes detected in sediment samples analyzed as part of the Phase I investigation. As seen, a number of inorganic constituents were detected, as were several indicators of petroleum hydrocarbons. The laboratory noted that the composition of some of the petroleum hydrocarbons detected did not resemble the composition expected for man-made fuels, and might be natural in origin. In addition, methyl acetate was detected in two samples, and pyrene was detected in one sample. All other chemical analytes were never detected in any sample. As noted above, it is not appropriate to draw any strong conclusions regarding whether or a release has occurred or whether any of the values are of potential concern until additional data are collected to ensure adequate representativeness of the data.

Results of Flow Measurements

Flow measurements were performed at a number of stations in the Rainy Creek watershed. Results are presented in Table 3-9. As seen, flows were generally low, especially in Fleetwood Creek , Carney Creek, and the upper reaches of Rainy Creek (URC-1 and URC-2). Flows from the tailings impoundment and in lower Rainy Creek were in the range of 0.3 to 0.8 ft^3/sec.

4.0 DATA QUALITY OBJECTIVES

4.1 Overview of the DQO Process

Data Quality Objectives (DQOs) define the type, quality, quantity, purpose, and intended uses of data to be collected (EPA, 2006). The design of a study is closely tied to its DQOs, which serve as the basis for important decisions regarding key design features such as the number and location of samples to be collected and the analyses to be performed. In brief, the DQO process typically follows a seven-step procedure, as follows:

1. State the problem that the study is designed to address
2. Identify the decisions to be made with the data obtained
3. Identify the types of data inputs needed to make the decision
4. Define the bounds (in space and time) of the study
5. Define the decision rule which will be used to make decisions
6. Define the acceptable limits on decision errors
7. Optimize the design using information identified in Steps 1-6

Following these seven steps helps ensure that the project plan is carefully thought out and that the data collected will provide sufficient information to support the key decisions which must be made.

4.2 Conceptual Site Models

Figure 4-1 presents the conceptual site model for how humans may be exposed to contaminants in surface water and sediment within OU3. The maximally exposed human receptor is assumed to be an area resident who hikes or fishes along surface water features such as Rainy Creek or some of the ponds. Exposure is primarily *via* incidental ingestion of surface water or sediment. This would apply primarily to non-asbestos contaminants, but oral exposure to LA via incidental ingestion of water or sediment might also be of potential concern[1]. In some cases, airborne exposure may also occur by disturbance of dried sediments along stream banks. This would be of concern mainly for asbestos contamination, with only low concern for non-asbestos contaminants.

Figure 4-2 presents the conceptual site model for how ecological receptors may be exposed to contaminants in surface water and sediment at the site. The maximally exposed ecological

[1] Note: In most cases, inhalation exposure to asbestos is the main reason for human health concern, with only minor concern for ingestion exposures. This is true, for example, in Libby OU4. In OU3, because of the assumed intake of LA in water and sediment by some recreational visitors, it is possible that oral exposure might begin to contribute risks that are not minor compared to the inhalation pathway, so ingestion exposure to LA will be evaluated in this case.

receptors are fish or benthic invertebrates that live in the streams or ponds. Wildlife (birds, mammals) may also be exposed while feeding or drinking along the streams or ponds.

4.3. Data Quality Objectives for Phase IIA Surface Water and Sediment Sampling

4.3.1 State the Problem

Data from Phase I on the levels of LA and other analytes in surface water and sediment represent an observation at one point in time. Because concentrations of contaminants in surface water may vary over time, especially in cases where there are large fluctuations in flow (e.g., during spring runoff), additional data are needed to characterize the levels of site-related contaminants in surface water as a function of time (season) as well as space. Similarly, the degree of variation in sediment concentrations over time is not known, and additional data are needed to assess how the levels of asbestos and other contaminants in sediments vary over season in OU3 drainages.

4.3.2 Identify the Decision

Ultimately, the data collected during the OU3 RI is intended to help EPA decide if and what response actions, if any, are needed to protect human and/or ecological receptors from unacceptable risks from asbestos and any other mining-related contaminants in surface water and sediment in OU3.

4.3.3 Identify the Types of Data Needed

Contaminant Concentration Data

One type of data that is needed to evaluate risks from contaminants in surface water and sediment is reliable and representative measurements of the concentration of contaminants in surface water and sediment as a function of both time and space. This type of data is valuable both to support risk evaluations (when reliable toxicity values are available) as well as to identify sources of contaminant releases.

In Phase I, the target analyte lists for surface water and sediment included not only asbestos but also a wide variety of other chemical classes (see Tables 3-1 and 3-3) in order to seek information on the occurrence of a number of chemicals that might have been used at the site or that might have been released to the environment due to mining activities. However, because Phase I reflects concentrations at only one point in time, these data may not be representative of variations in concentration levels over time (season), and hence the Phase I data alone are not considered sufficient to allow reliable evaluation of either the nature and extent of contamination or of the level of risk to humans or ecological receptors. Therefore, all of the analytes assessed in surface water and sediment during Phase I are retained for further evaluation in Phase IIA.

Site-Specific Toxicity Tests

For ecological receptors, direct measurements of observed effects from exposing receptors (fish, benthic macroinvertebrates) to site media (surface water, sediment) are used to assess risks especially for contaminants for which reliable toxicity values are not available to use in the HQ approach for evaluating measured concentration values. In site-specific toxicity tests, ecological receptors are exposed to site media of known concentrations in order to observe whether the media causes adverse effects on growth, survival, and/or reproduction in laboratory test species. Data from toxicity tests are used to establish a reliable site-specific exposure response curve. Using this relationship, it may be possible to identify reference concentrations of contaminants in water or sediment that represent the boundary between acceptable and unacceptable effects on fish. If so, then these reference concentrations may be used in the evaluation of other site waters and sediments that have not been tested using aquatic receptors.

For the purposes of the Phase IIA sampling plan, the medium that is most time-critical for evaluation by site-specific toxicity testing is surface water. This is because it is expected that the concentration of LA (and possibly other site-related contaminants) may change during spring runoff. Site-specific sediment toxicity testing will also be a key component of the OU3 RI and ecological risk assessment. However, because it is not thought that sediment samples are likely to be as time-variable as water samples, collection of sediment samples for toxicity testing is not time critical and can be deferred for planning and implementation to Phase IIB.

Flow and Loading Data

If it is determined that releases of LA or any other contaminant from site pose an unacceptable risk to humans and/or environmental receptors, then EPA needs to identify the sources of those unacceptable releases in order to evaluate remedial alternatives. One of the most useful types of information for evaluating the relative significance of water-borne releases is loading (the amount of contaminant carried in water per unit time). Loading is calculated as the product of concentration and flow. Thus, data on surface water flow rates are needed to characterize the temporal variations in stream flow rates at numerous locations in the Rainy Creek watershed so that the load carried by each reach may be assessed.

4.3.4. Define the Bounds of the Study

Spatial Bounds

The primary focus of Part A of the Phase II investigation is the Rainy Creek watershed, including upper and lower Rainy Creek, Fleetwood Creek, and Carney Creek, as well as ponds and impoundments on these streams. In addition, Phase IIA will include an evaluation of surface waters in the Kootenai River in the vicinity of the confluence with Rainy Creek.

Temporal Bounds

Because surface water flow conditions are variable over time, the Phase IIA surface water investigations will be conducted during a typical range of annual flow conditions. The Phase IIA investigations will begin at close as feasible to the start of the rising hydrograph, and will continue through the high flow period and extend into the summer and fall. The purpose of this temporal sampling pattern is to characterize, at least within the year 2008, the pattern of temporal variability in concentration levels of contaminants of potential concern.

4.3.5. Define the Decision Rule

Consistent with EPA guidance (EPA 1991), if the cumulative carcinogenic risk to an individual human within OU3 based on the reasonable maximum exposure for both current and future land use does not exceed 1E-04 and the non-carcinogenic HQ does not exceed 1, remedial action generally is not warranted within OU3 unless there are adverse environmental impacts. EPA may also compare measured concentrations in OU3 to chemical-specific applicable or relevant and appropriate requirements (ARARs) to determine whether remedial action is warranted.

For ecological receptors, risk characterization will, to the extent that data allow, be based on a weight-of-evidence approach that utilizes one or more of the following strategies:

- Calculation of Hazard Quotient (HQ) values based on measured concentration values and available toxicity reference values (TRVs)
- Exposure of test organisms to samples of surface water and/or sediment collected from the site to evaluate the magnitude and frequency of any effects on growth or survival
- Direct surveys of receptor density and diversity in site streams in comparison to appropriate reference streams in the same area

The ecological decision rule will likely take the form that, if the weight-of-evidence indicates that adverse effects on fish and/or benthic organisms are occurring, and that these effects are likely to result in a meaningful decrease in the density and/or diversity of receptors compared to what would be expected in the absence of site-related contamination, then a response action will be appropriate.

4.3.6. Define the Acceptable Limits on Decision Errors

Two types of decision errors are possible when making risk management decisions:

- A false negative decision error occurs when it is decided that risk is acceptable when the true risk is actually above the level of concern

- A false positive decision error occurs when it is decided that risk is not acceptable when the true risk is actually below the level of concern

Of these two types of errors, EPA is primarily concerned with avoiding false negative errors, since an error of this type can leave human or ecological receptors exposed to unacceptable levels of contamination and risk. The EPA usually identifies 5% as the maximum acceptable probability of making a false negative decision.

A false positive decision error does not leave humans or ecological receptors at risk, but is also of concern to EPA because this type of error may result in the expenditure of resources (time, money) that might be better invested elsewhere. For the OU3 RI and risk assessment process, the goal is as follows: if the true level of risk is less than ½ the acceptable risk level, then there should be no more than a 20% chance that the risk will be declare to be unacceptable.

4.3.7. Optimize the Design

4.3.7.1 Optimizing the Design for Evaluation of Risks to Humans

The evaluation of risks to humans from exposure to asbestos and other site-related contaminants of potential concern will be based on a computational approach in which HQ values and lifetime excess cancer risks are calculated from available data. The probability of making either a false negative or a false positive decision error depends on the accuracy of all of the information used to make the calculations, including the concentration term, the exposure parameters, and the toxicity term. In general, EPA seeks to limit the risk of false negative decision errors by ensuring that all uncertain inputs into risk calculations are "conservative" (i.e., are more likely to overestimate than underestimate risk). Of the uncertain inputs, the only one that is amenable to control during field sampling and analysis is the uncertainty in the concentration term. For this reason, attention in the sampling plan is focused on optimizing the number of samples that will be available for estimating average exposure levels in each exposure area for each environmental medium.

The number of samples needed to limit uncertainty in concentration term depends mainly on the nature of the underlying distribution and the degree of between-sample variability. The degree of uncertainty that can be accepted depends mainly on how close the data are to a decision criterion. That is, greater uncertainty is acceptable when the values are far removed (either below or above) the decision criterion than when the values are near a decision criterion.

Non-Asbestos Analytes

For non-asbestos analytes, environmental data sets are generally observed to be right-skewed and are often reasonably well approximated by lognormal distributions. Based on this, Monte Carlo simulation can be used to characterize the relationship between the number of samples collected

and the probability of a false positive decision error based on the UCL of the mean of an observed lognormal data set. Figure 4-3 shows the results of several example situations. In Panel A, the geometric standard deviation of the lognormal distribution is assumed to be 2.0 (a typical value for environmental data sets). The blue curve shows the false positive error rate when the true mean of the lognormal distribution is assumed to be ½ the risk-based concentration (RBC). As seen, for this GSD, about 14 samples are needed to achieve the DQO (<20% false positives when the true mean is ½ the RBC). When the true mean is further below the RBC (ratio = 0.2), then only about 6 samples are needed to limit the false positive decision error to 20% (red line). Panel B shows the same relationships, except the GSD is assumed to be 3 (a value that may occur is some data sets). In this case, the number of samples needed to achieve the DQO when the mean is ½ the RBC is about 38 (blue line), and is about 12 when the true mean is 1/5 the RBC (red line).

LA in Water

For asbestos, uncertainty in the mean concentration of a data set arises not only from the authentic between-sample variability, but also from uncertainty that is inherent in the methods used to measure the asbestos concentration. For water samples, concentration values for each sample are derived based on the number of fibers observed during a microscopic inspection of an aliquot of the sample. The number of fibers observed is a random variable characterized by a Poisson distribution. Because of this Poisson variation in each measured value, the overall uncertainty is a combination of the sampling variability and the measurement error, which results in a Poisson-lognormal (PLN) distribution. At present, the EPA has not established a method for quantifying the uncertainty in the mean of such a data set. However, it is known that the magnitude of the uncertainty depends on the number of samples, the variability of the underlying lognormal distribution, and the average number of LA particles counted in the analysis of each sample. In general, if the number of particles counted is large (e.g., 50 or more), then the contribution of Poisson uncertainty tends to be small and the source of variation is mainly due to the lognormal sampling variability. If the number of LA particles counted is small (e.g., less than 10), then the contribution of the counting error becomes more important and uncertainty bounds widen substantially.

For this reason, analytical counting rules are set such that, if fibers are present at concentration that is near the level of concern, then the number of fibers observed during an analysis will be high (about 50 or more). Figure 4-4 shows the results of several Monte Carlo simulations based on this condition (average count = 50). Results are presented as the ratio of the observed sample mean divided by the true mean. As expected, the width of the uncertainty distribution (the distance between the 5th and the 95th percentile) tends to narrow as sample number increases, and tends to increase as the GSD increases.

In the absence of an approved method for computing the UCL of a PLN data set, it is not yet possible to perform a quantitative analysis of decision error rates as a function of sample size.

However, it is apparent from inspection of Panel A that at least 20 samples are needed to ensure that the uncertainty range does not exceed about ± 50-60% for a distribution whose assumed GSD is 2-3. Increasing the sample number to 40 would be expected to narrow the uncertainty interval to about ±30-40%. Thus, when concentrations are near a level of concern, the target is to collect 20-40 samples. If the concentration is not near a decision criterion, then a data set of 10-20 samples is likely to be acceptable.

LA in Sediment

For sediment, the best method currently available for asbestos yields mainly semi-quantitative results, and the uncertainty around each measurement can not be quantified. Thus, there is no statistically valid approach for deriving a quantitative estimate of the mean for a set of samples, or to quantify the uncertainty about the mean. In the absence of a valid statistical approach, based on general statistical principles, EPA has determined that a data set of about 10-20 samples per exposure unit is likely sufficient in order to have a semi-quantitative understanding of spatial and potentially temporal variability of sediment levels in the exposure unit.

Summary

In summary, reliable characterization of the nature and extent of mining-related contamination and the level of risk to humans associated with contaminants in surface water and sediment require that samples are representative in space and time, and that a sufficient number of samples are collected so that estimates of average concentration values may be calculated with acceptable confidence.

As described above, the sampling plan for surface water has been designed to ensure good spatial and temporal representativeness of the drainages in OU3. The exact number of samples needed to limit uncertainty in the mean in any specific location is a complex function of the between-sample variability and how close the values are to a decision criterion. For OU3, the following guidelines have been selected to guide the sampling strategy:

Analyte Class	Number of Samples per Exposure Unit	
	Values are not near a decision threshold	Values are near a decision threshold
Non-asbestos	6-12	14-38
Asbestos	10-20	20-40

Although work on the human health risk assessment has not begun, it is expected that the following exposure units will be used:

- Upper Rainy Creek
- Lower Rainy Creek

- Tailings Impoundment
- Mill Pond
- Fleetwood Creek
- Carney Creek

If so, the number of samples for each exposure unit at the completion of Phase IIA (including the data from Phase I) would be as shown in Tables 4-1 (surface water) and Table 4-2 (sediment). As seen, all onsite stations will have at least 3 samples, and most proposed Exposure Units will have a sufficient number of samples (15 to 40) to achieve the DQOs for evaluation of human health risk.

4.3.7.2 Optimizing the Design for Evaluation of Risks to Aquatic Receptors

As noted above, risks to ecological receptors, including fish and benthic invertebrates, will be based on a weight of evidence evaluation. Consequently, it is not possible to develop statistical rules that limit the likelihood of false positive or false negative decision errors. Rather, the degree of confidence in the decision is based on the quality of the data available, and the degree to which different lines of evidence yield consistent conclusions. If multiple lines of evidence support the same conclusion, then confidence in the decision is increased. Conversely, if different lines of evidence yield inconsistent conclusions, then confidence in the decision is decreased.

HQ Approach

In the case of aquatic receptors, it is common to begin by an assessment of risks from surface water using the HQ approach. Note, however, that this requires the availability of suitable toxicity reference values (TRV) for the contaminants of concern in surface water. Such TRVs do exist for most non-asbestos analytes, and the HQ approach will be used as the first line of evidence for this group of contaminants. If the HQ results suggest that risks are below a level of concern, then no further evaluation will be needed. If the HQ approach suggests that risks may be occurring, then other lines of evidence will be investigated.

In the case of asbestos, no TRV values are currently available. Even if such values were available, their relevance to OU3 would be uncertain because the toxicity of asbestos may depend on the mineral type (LA) and on the particle size distribution in site waters. For this reason, the first line of evidence evaluated will be site specific toxicity testing. This will provide direct data on the toxicity of site waters to an appropriate fish species (rainbow trout). Assuming that the site water produces toxicity, then a site-specific TRV can be developed by testing a series of dilutions of the site water, and the resultant site-specific TRV may then be used to predict, using the HQ approach, the expected toxicity of LA in other site waters that have not been tested using fish.

Sampling Design for Site-Specific Toxicity Testing

The objective of site-specific toxicity testing is to develop a site-specific exposure-response curve for toxicity in fish. This is best achieved by testing waters at regularly-spaced concentration intervals ranging from low to high. Serial dilution of one surface water sample collected from one sampling location within OU3 was selected as the sampling design to meet the Phase IIA objective

Since one site water will be used in Phase IIA, it is important to select this water such that the level of LA is at or near the high end of the range of concentrations that occur in on-site waters. This is because the data would have low utility if the water sample tested had relatively low levels of LA, and no toxicity was observed. In order to ensure that water sample selected for testing is at the high end of the range observed on-site, samples of site waters from six stations will be evaluated weekly for LA during the rising and peak phase of the hydrograph. Analytical results will be reported as quickly as possible by the analytical laboratory so that when a sample is observed that is judged to be at or near the maximum, it will be possible to quickly direct the collection of a large sample of that water for testing.

Inspection of the surface water results for LA from Phase I, it appears that the highest concentrations of LA tended to occur in the ponds and impoundments, and also in the influent waters to those ponds. On this basis, the 6 stations selected for rapid turn-around monitoring during the spring are as follows:

Fleetwood Creek Pond and FC-1
Tailing impoundment and URC-2
Mill Pond and TP-TOE2

Other lines of evidence (site-specific population surveys, examination of the frequency and severity of histological lesions in fish) will be included in Phase IIB of the OU3 SAP.

4.3.7.3 Optimizing the Design for Evaluation of Flow and Loading

The basic strategy for identifying sources and relative amounts of LA release into site waters is to collect flow and LA concentration data at multiple locations at the site. By comparing loading (concentration x flow) at different stations, the amount of LA that is added (or lost) in each segment of the Rainy Creek drainage can be computed. As noted above, because flow is variable over time, especially during spring runoff, the sampling strategy is to collect measures of flow and concentration at each station at a series of times during the rising and falling phases of the hydrograph, during storm events, and during more nearly baseflow conditions in the summer and fall.

5.0 SAMPLING PROGRAM

All sampling of environmental media within OU3 described in this SAP will be performed by personnel who are properly trained in the field collection methods summarized in the OU3 Standard Operating Procedures (SOPs) provided in Attachment B and the Phase IIA experimental sampling design details presented below. The field sampling teams will follow procedures in the Health and Safety Plan (HASP) prepared by MWH for the OU3 investigation.

Table 5-1 provides an overview of a number of data collection activities that will be performed under Phase IIA of the OU3 RI. The following sections present the experimental design, including sampling details and rationale, for the Phase IIA elements of surface water and sediment characterization.

5.1 Rainy Creek Watershed Monitoring – Experimental Design

A Phase I investigation within the Rainy Creek watershed was completed in the fall of 2007 to provide an initial characterization of conditions at and surrounding the Libby Mine site. Further characterization of surface water and sediment was anticipated as part of Phase II. This section describes the experimental design for Phase IIA data collection activities developed to meet data needs for surface water and sediments within the Rainy Creek watershed, as discussed above in Section 4.3.

5.1.1 Element 1: Seasonal Surface Water and Sediment Monitoring

As noted previously, it is expected that flow and contaminant concentration will vary in each portion of the Rainy Creek watershed as a function of time of year. The purpose of Element 1 is to measure stream flow and contaminant concentrations (LA as well as other non-asbestos contaminants) in surface water and sediment at each location previously sampled in Phase I to characterize levels during spring and summer flow conditions. These data may be combined with similar Phase I data collected during the fall of 2007 to develop an understanding of the seasonal variability in flow and concentration patterns across the site. These data, in turn, will form one part of the data set used to evaluate exposure and risk to human and ecological receptors, as well as an understanding of sources and loading during spring and summer flow regimes.

Surface Water Samples

Figure 5-1 identifies the locations where samples of surface water will be collected during Phase IIA. These are the same locations where samples were collected during Phase I, plus the following additional locations:

- Upper Tailings Pond (UTP). The tailings impoundment consists of two parts: the main tailing impoundment, which is relatively shallow, and an upper pond that is somewhat deeper. In Phase I, surface water was collected only from the shallow portion of the impoundment. However, the deeper portion of the impoundment is relevant habitat for ecological receptors, and also requires sampling. Because of its depth, samples will be collected both from the surface and from near the bottom.
- Tailings Pond overflow (TP-overflow). This location in the overflow channel for the tailing impoundment has been added to characterize the concentration and load of LA and other contaminants that may be released during periods of high flow that over-top the impoundment
- Rainy Creek upstream of the mining-disturbed area and about 100 yards north of Rainy Creek Road (URC-1A). This station has been added to help define the contribution of releases from the road to contaminant levels at URC-2
- Pond on Carney Creek (CC-Pond). This station has been added because the existence of the pond was not recognized during the Phase I sampling program and no data from this location have been collected.

If any significant new seeps, springs, or other water features are observed by the field sampling crews during Phase IIA that were not recognized during Phase I, the EPA RPM will be notified and these will also be selected as new sampling stations.

Table 5-2 identifies and describes all of the Phase IIA surface water monitoring locations.

Surface water samples will be collected from each station once in late spring following peak runoff and once in the summer. All surface water samples collected under Phase IIA Element 1 will be analyzed using the same approach as used previously in Phase I. In brief, all samples will be analyzed for asbestos, metals/metalloids, petroleum hydrocarbons, anions, and other water quality parameters. In addition, a broad suite of analyses will be performed for samples collected at the tailings impoundment toe drain (TP-TOE1) and Lower Rainy Creek downstream of the confluence with Carney Creek (LRC-2). These locations were selected because they appear to have the best potential of characterizing releases from the mine. The additional analyses for surface water include PCBs, pesticides, herbicides, gross alpha/gross beta, volatile and semi-volatile organic compounds, and cyanide. These analyses will provide a more comprehensive screen for potential contaminants associated with mine wastes and process chemicals used during mine operations. Details on the specific chemical analyses that will be performed for surface water samples under Element 1 are discussed in Section 6 (see Table 6-1).

Water quality data for springs will provide information on shallow groundwater quality. Seep water will provide information on whether contaminants are being released from mine waste piles and disposal areas. These data, along with any groundwater sampling data collected as part of Phase IIB, will allow for identification of mine-related contaminants and possibly an assessment of transport pathways.

At locations where flowing water is present, stream flow rate will be measured following the collection of surface water and sediment samples. Flows will be measured at locations on Fleetwood Creek, Rainy Creek, Carney Creek, at the TP-TOE1 drain and TP overflow (if running). Flow data will be used with contaminant concentration data to assess contaminant mass transport along surface water transport pathways.

Sediment Samples

Sediment samples will be collected once in late spring following peak runoff and once in late summer, from the same locations and at the same time as surface water samples (see Figure 5-1). The sediment data collected during Phase IIA will be used in conjunction with data collected during Phase I to evaluate sediment heterogeneity at each location and to assess seasonal variability in sediment concentrations, if any.

The Phase IIA sediment sampling plan differs from Phase I in that the tailings impoundment and each of the ponds (the Mill Pond and the ponds on Carney Creek and Fleetwood Creek) will each be sampled by collecting a series of grab samples rather than 1-2 composite samples. The purpose of collecting multiple grab samples from the tailings impoundment, the Mill Pond, and the ponds on Carney Creek and Fleetwood Creek is to provide information on the spatial variability within each feature. This is important mainly for evaluating risks to benthic organisms in the ecological risk assessment.

Figure 5-2 shows the approximate location of 17 sampling locations in the tailings impoundment. These sample locations are focused mainly in areas that are always or usually inundated with water, since these areas are most likely to serve as habitat for aquatic receptors. However, five samples are placed in areas that may be occasionally inundated. The exact sampling locations may be revised based on field conditions at the time of sampling.

At the three other ponds (the Mill Pond and the ponds on Carney and Fleetwood Creeks), a total of 5 sediment grab samples will be collected from each pond. These will consist of 3 samples from around the margins of the pond (at least 3 feet in from the edge), and two from near the center of the pond.

More detailed procedures for collection of sediment samples associated with this element of Phase IIA are given below in Section 5.3.4.

All sediment samples will be analyzed for asbestos, metals/metalloids, and petroleum hydrocarbons, and total organic carbon. Sediments from lower Rainy Creek and the tailings impoundment toe drain will also be analyzed for PCBs to assess the potential effects of use of oil for dust control along the adjacent road. Sediment collected from TP-TOE2 and LRC-2 will also be analyzed for pesticides, herbicides, volatile and semi-volatile organic compounds, and

cyanide. Details on the specific analyses that will be performed for sediment are discussed in Section 6.

5.1.2 Element 2: Spring Runoff Monitoring

The purpose of Element 2 is to monitor stream flow and surface water asbestos concentration values at selected stations within the Rainy Creek watershed during the rising and falling limbs of the spring-season snowmelt-runoff hydrograph. These data will be used as part of the data set used to evaluate exposure and risk to human and ecological receptors from LA, as well as to track changes in the asbestos loading and transport in surface water as stream flows first rise in response to snowmelt runoff and then decline as snowmelt ends.

Surface water samples will be collected once during winter base-flow conditions, and then weekly beginning at the onset of rising stream flows in response to snowmelt, continuing through the spring high-flow season, and ending approximately 4 weeks after the seasonal peak in flow is observed on Rainy Creek. Table 5-3 and Figure 5-3 identify the locations where samples of surface water will be collected under Element 2. Surface water samples will be collected weekly during the spring runoff season at the following locations:

- Tailings impoundment (TP), impoundment toe drain (TP-Toe1), and impoundment overflow (TP-overflow)
- Mill Pond (MP)
- Rainy Creek (URC-1A, URC-2) upstream of the mine-disturbed areas
- Fleetwood Creek (FC-2) and Carney Creek (CC-2) downstream of mine-disturbed areas
- Lower Rainy Creek below the Mill Pond (LRC-1) and below Carney Creek (LRC-2, LRC-6)
- Pond on Carney Creek (CC-Pond)

All of the locations listed were sampled during the Phase I investigation except for one new station on upper Rainy Creek (URC-1A) and one new station at a pond along lower Carney Creek (CC-Pond). All but two of the locations selected for Element 2 are downstream of potential primary sources of asbestos, including mine waste piles and the coarse and fine tailings disposal areas, and downstream of potential secondary sources, including sediments in the Mill Pond and sediments deposited along lower Rainy Creek.

All surface water samples will be analyzed for asbestos. At locations where flowing water is present, stream discharge will be measured following the collection of surface water samples. Flows will be measured at locations on Fleetwood Creek, Rainy Creek, Carney Creek, at the TP-TOE1 drain and TP overflow (if running). Stream flows will be measured above and below the tailings impoundment (TP), Mill Pond (MP), and Carney Creek pond (CC-Pond) to evaluate flow-through and water residence times in these surface impoundments.

5.1.3 Element 3: Summer and Fall Monitoring

Routine Monitoring

Element 3 is an extension of Element 2 into the summer and early fall that is designed to provide ongoing information on asbestos concentrations and stream flow rates downstream of asbestos sources within the Rainy Creek watershed. Locations sampled in Element 3 are the stations on lower Rainy Creek below Carney Creek (LRC-2) (this location receives flow from both the upper and lower portions of the mined area) and lower Rainy Creek near its discharge to the Kootenai River (LRC-6). Element 3 will start immediately upon completion of Element 2 (i.e., approximately 4 weeks after the seasonal peak in flow), and continue through September 30. Instead of weekly sample collection and flow measurement at each location as for Element 2, monitoring will be reduced to every other week for Element 3. The surface water samples will be analyzed for asbestos. Wherever flowing water is present, stream discharge will be measured following sample collection.

The two locations selected for Element 3 both represent points on the potential surface water transport pathway from mine-related sources of asbestos to lower Rainy Creek and the Kootenai River. LRC-2 is downstream of all potential mining-related sources of asbestos and downstream of two ponds representing potential secondary sources to surface water (Mill Pond and pond on Carney Creek), and LRC-6 is downstream of all potential primary and secondary asbestos sources, including sediments deposited in the lower Rainy Creek drainage downstream of LRC-2.

Storm Event Monitoring

Element 3 will also include sampling and flow measurement triggered by precipitation events that occur after the spring snowmelt-runoff season. The same two locations, LRC-2 and LRC-6, will be used for monitoring during storm events. Asbestos concentration values and flow measurements will be obtained at LRC-2 and LRC-6 to describe asbestos transport associated with at least three separate storm/rainfall events within the Rainy Creek drainage and to evaluate the effect of short-term, episodic surface runoff in the mine area, and related increases in local stream flow, on the asbestos content of surface water downstream of the known sources of asbestos.

The exact details of how storm-event monitoring will be implemented have not yet been established. The following sections provide a conceptual approach that may be modified based on a consideration of the recommendations of the field sampling team.

The most appropriate trigger for initiation of storm-even monitoring is not known, because no data exist regarding how much flow in rainy Creek increases when storm events occur. A value

of 100% (a doubling of average flow) will be used as an initial starting trigger. This value is at the low end of the possible increases in flow that are predicted by modeling. This value may be revised by EPA either upward or downward, as data are obtained on the actual changes in flow rate in the Rainy Creek drainage that result from storm events.

The amount of rain needed to cause a flow increase of this size in the Rainy Creek drainage is not known. Modeling conducted by Schafer (1992) indicates that a 10-year storm event of 2.4 inches over a 24-hour period causes increased flow in local drainage approximately 12 hours following the start of rainfall. The model used by Schafer to describe the storm hydrograph was developed for a basin of undisturbed, mature forest in good condition with moderately sloped topography. The model input parameters do not match conditions in the sparsely vegetated and bare areas of steeply sloping mine waste in the mining-disturbed portion of the basin. Therefore, storm hydrographs associated with runoff from the mine-disturbed areas may be larger and may occur sooner than predicted by the Schafer model.

A rain gage will be installed at the Libby Mine site meteorological monitoring station to provide the data needed to trigger storm-event monitoring. The rain gage will be equipped with a data logger to automatically track precipitation amounts and durations. When a rainfall event takes place, data collected at the meteorological station can be used to establish whether the event meets (or is likely to meet) the criteria given above for storm-event sampling. If so, surface water sample collection will be initiated after rising stream flow is observed in response to a qualifying storm event. Stream flow monitoring at LRC-2 may also be used to trigger storm-event-related monitoring.

The surface water sample collected at each location during each storm event will be a 24-hour, flow-weighted, composite sample. If the duration of rainfall is longer than 24 hours, additional 24-hour, flow-weighted composite samples will be collected to monitor water quality throughout the storm-related hydrograph (i.e., elevated stream flow associated with the rainfall event).

Collection of flow-weighted composite samples will require automated samplers with flow monitoring equipment. Automated samplers will be set up at LRC-2 and LRC-6 to collect the composite samples. Initially, a weir will be installed in the stream channel to allow for continuous flow monitoring. When conditions allow, the weirs will be replaced with installation of a flume. Each flow-weighted composite sample will be collected over a 24-hour period. The same equipment will also be utilized for Element 4, as described below. Operation and maintenance of these automated sampling stations will be in accordance with procedures described in Section 5.3.3 and SOP Nos. 14 and 15.

5.1.4 Element 4: Continuous Precipitation and Flow Monitoring

Element 4 consists of continuous precipitation monitoring at the meteorological monitoring station at the Libby Mine site and continuous stream flow monitoring on lower Carney Creek and lower Rainy Creek. Flow monitoring will be conducted on lower Carney Creek during

spring snowmelt runoff and on lower Rainy Creek during the spring snowmelt runoff, summer, and early fall. The purpose of collecting flow data on a continuous basis at these locations is to:

- characterize the spring snow-melt hydrograph while the other elements of the Phase IIA investigation are being implemented;
- track changes in flow in response to local precipitation events; and
- provide detailed flow measurements for use with asbestos concentration data to characterize asbestos mass loading to Rainy Creek from the mine site and from lower Rainy Creek to the Kootenai River.

Precipitation amounts and durations will be recorded using an 8-inch-diameter, heated, tipping-bucket rain gauge installed at the same location as the meteorological station that is currently in use at the Libby Mine site. The purpose of collecting continuous precipitation data is to determine the runoff coefficient as it relates to the precipitation intensity/duration curves for the Rainy Creek watershed. Installation and calibration of the precipitation station will be in accordance with the procedures described below in Section 5.3.5 and SOP No.17. This equipment will be installed in early spring to allow for developing relationships between precipitation and stream flow in Rainy Creek during the Phase IIA investigation.

Flumes/weirs (see Section 5.1.3) will be installed in the stream channels at three monitoring stations: LRC-2, LRC-6, and CC-2 (see Figure 5-3). Stream flow through the flumes will be monitored using water level sensors, and the continuous flow data will be recorded using a computerized data logger. Installation and calibration will be in accordance with the procedures described below in Section 5.3.6 and SOP No. 15. The automated flow monitoring equipment will be installed before the initial rise in stream flow associated with the spring snowmelt-runoff season and that equipment will be maintained for use through the summer and early fall.

5.1.5 Element 5: Collection of Water for Toxicity Testing

As discussed in Section 4.3.3, one of the most direct methods for evaluating toxicity of site media such as surface water and sediment to ecological receptors (fish, benthic invertebrates) is through site-specific toxicity testing. In this approach, test organisms are exposed to site media in the laboratory to determine if the site media causes adverse effects on survival, growth and/or reproduction.

In Phase IIA, one site water will be selected for use in site-specific toxicity testing. As discussed previously, the water to be used will be selected by monitoring the levels of LA as a function of space and time, and seeking to collect a sample at a time and place that is at or near the high end of the range of concentrations observed on-site. This sample will then be evaluated in the toxicity testing protocol in an undiluted form, and in a set of serial dilutions that generate lower concentration values. The purpose of this study design is to generate a site-specific exposure-response curve that can then be used to evaluate site waters that have not been tested using fish.

If no toxicity is observed in the most concentrated (undiluted) water, this will be taken to indicate that, at least for the water tested, toxicity to fish is unlikely to be of concern. However, because of the potential for temporal variation between years in concentration levels of LA and other contaminants, follow-up studies may be needed to assess whether concern might exist in other years, and whether those higher values might be toxic.

The volume of water collected from the selected station will be approximately 200 L (about 50 gallons), which is sufficient to support the serial dilution toxicity testing protocol as described in Section 7. This water will be promptly chilled and transmitted to the toxicity testing laboratory.

5.2 Kootenai River Monitoring – Experimental Design

5.2.1 Surface Water

Phase IIA includes two rounds of sampling of water from the Kootenai River in the vicinity of Rainy Creek to assess the effect of Rainy Creek on asbestos levels in the river water. The first sampling event will occur at approximately the time of maximum flow in Rainy Creek, and the second event will occur under summer baseflow conditions in Rainy Creek.

Sampling stations for each event will include one location (designated UKR) upstream of Rainy Creek, three stations (designated KR1, KR2 and KR3) parallel to the northern river bank downstream of the mouth of Rainy Creek, and 5 stations (designated KR4 to KR8) along a perpendicular transect downstream of Rainy Creek. The approximate locations of these stations are shown in Figure 5-4. These locations were selected to provide asbestos concentration values upstream and downstream of Rainy Creek and to include river locations with the greatest potential for elevated asbestos concentrations due to transport via Rainy Creek.

Each river-water sample will be collected from a discrete location using a depth-integrated sampler and in accordance with SOP No. 16 (Surface Water Sampling Using Depth-Integrated Samplers) and the instructions for surface water sampling in Section 5.3.1.

All samples of river water will be analyzed for asbestos.

5.2.2 Sediment

For the Kootenai River, sediment samples that will be collected include the following:

- One grab sample from a depositional area located along the north bank of the Kootenai upstream of Rainy Creek. This will serve as a frame of reference for evaluating downstream samples.
- Two or three grab samples from a depositional area located along the north bank of the Kootenai downstream of Rainy Creek, but within a distance of 1/2 mile.

- Two borings from the large sandbar located in the center of the river about 1/2 mile downstream. One boring will be from the highest location on the sandbar, since this may contain the oldest sediments. The other boring will be from a location near the downstream tip of the sandbar. Each boring will be to a depth that encounter the water level of the river, plus about 6 additional inches. Each boring will be subdivided into a total of four depths, or into 6-inch strata, whichever is smaller.

All samples of sediment collected from the Kootenai River will be analyzed for asbestos.

5.3 Field Procedures

5.3.1 Surface Water Sampling Methods and Procedures

The sampling procedures for collection of surface water grab samples are presented in OU3 SOP No. 3. During each monitoring event conducted for Rainy Creek watershed Elements 1 and 2, stream water samples will be collected from downstream to upstream locations to minimize the effect of sampling activities on the samples collected. To minimize the potential effect of time variability, all samples from a single stream drainage (i.e., Rainy Creek) will be collected on the same day. All samples will be grab samples, collected by pumping directly from the source into laboratory collection containers using a peristaltic pump. Samples will be collected from representative flowing water (usually the mid-channel).

Both filtered and unfiltered samples will be collected directly from the water bodies into sample bottles. At each station, the unfiltered sample will be collected before the filtered sample (or any other activities) to minimize the potential disruption of the sediment and resuspension of LA. For the filtered samples (to be analyzed for metals only), water from the source water body will be pumped through a 0.45 μm in-line, high-capacity filter using either a battery-operated peristaltic pump or hand-held manual pump. The in-line filter will be purged with approximately 200 mL of sample water before the laboratory container is filled. A new (0.45 μm) in-line filter and tubing will be used for each site to collect water for analyses of "dissolved" constituent concentrations. The filter will then be removed, and the sample for unfiltered metals and other water quality parameters will be collected.

The method for collection of water at springs, seeps, and ponds will be the same as above, except in locations of very shallow water. In such locations, water can be collected from a depression created to increase the depth of water and allow for sampling using a pump and tubing, as described in OU3 SOP No. 3.

5.3.2 Surface Water Field Measurements and Flow Monitoring

Whenever grab samples of surface water are collected, the in-stream temperature, pH, specific conductance, dissolved oxygen (DO), and turbidity will also be measured using portable field

meters. Field parameter measurement and calibration protocols will be performed according to manufacturer's specifications and OU3 SOP No. 10. These measurements will be recorded on field sampling forms.

At locations where flowing water is present, stream discharge will always be measured following the collection of surface water and sediment samples. The stream flow will be measured and recorded in accord with OU3 SOP No. 4. In brief, discharge will be measured using one of three portable methods, as dictated by flow or channel characteristics. Depending on the channel characteristics and flow, an area-velocity method, a portable flume, a volumetric method, or some combination of these methods, will be used to obtain the stream discharge measurements. Field personnel responsible for stream-discharge measurements must have prior experience using the methods and equipment described in OU3 SOP No. 4.

In cases where water depth is greater than 0.3 feet or the channel cross section is wide, flow generally will be measured using the area-velocity method of stream-flow gauging as described in the National Handbook of Recommended Methods for Water Data Acquisition (USGS 1977), and explained in detail in OU3 SOP No. 4. Using this method, the stream cross section is divided into a series of subsections where the average depth, average velocity, and width for the subsections are measured.

A portable cutthroat flume will be used to measure flow when low discharge and/or channel geometry preclude the use of a velocity meter. The flume will have a throat width adjustable from 2 to 8 inches, which can be used to measure flows from approximately 0.01 to 2.2 cfs. All water will be routed through the leveled flume, to the extent practicable, after which the height (to the nearest 0.01 foot), throat width, and leakage estimate as a percentage (if any) will be recorded. Discharge will be calculated using these data and an equation that is specific to the flume size.

In cases where flows are too small or stream gradients are too great to be measured using the area-velocity method or a cutthroat flume, measurements will be made volumetrically using a calibrated collection container and a stopwatch. Stream flow will be routed through a PVC pipe and the time to fill a collection container to a known volume will be measured. A minimum of five trials will be executed for each volumetric measurement, and discharge will be taken as an average of the five trials. An estimate of any leakage around the routing pipe will be recorded.

5.3.3 Automated Sampler Specifications and Procedures

The automated sampler chosen for this application must be capable of creating flow-derived composite samples. Therefore it is crucial that the pressure transducer in the flume and the automated sampler are compatible. The flow-derived composite sample can be created by varying the aliquot volume at a constant time interval or by varying the time interval and keeping

the aliquot volume constant. Either method should be adequate to determine contaminant loading rates during storm runoff.

The automated sampler will be located out of the floodplain, on relatively level ground, but not above the suction head capacity of the automated sampler pump. Additionally, the intake line to the sampler will be kept as short as possible to minimize cross-contamination of the samples and the intake installed upstream or within the approach to the flume. It may be necessary to place the sampler in a securable enclosure if extreme weather and/or vandalism are reasonably anticipated. If connection to a power source is available, the automated sampling station will operate on 120VAC.

Routine maintenance of the automated sampler will be completed during each visit. The unit will be checked for faults, errors, or alarms during the previous sampling interval and the associated issues will be resolved. It will be determined if adequate sample volumes were collected and reprogramming of the sampling interval/volume will be completed if necessary. After each sampling event, the pump tubing will be inspected for wear and replaced if necessary. The intake line will be cleaned and the intake foot will be inspected to verify that is not buried in newly deposited sediment or plugged by debris.

Additional detailed instructions for the operation and maintenance of automated sampling stations is provided in SOP No. 14.

Respondents W.R Grace & Co.-Conn, and KDC shall submit to EPA and MDEQ the details of the automated sampler chosen for Phase IIA including specifications, conceptual drawings of the installation and operations and maintenance instructions.

5.3.4 Sediment Sampling Methods and Procedures

At each sampling location, sediment will be collected in accord with OU3 SOP No. 5. In brief, a single sediment sample will be collected from each station. Each sample will consist of a grab sample collected from low-energy (i.e., depositional) portions of the stream channel that are inundated by creek water at the time of sampling (i.e., locations of sediment deposition to channel). Each grab sample will be collected using the "direct sampling" method and compositing instructions included in OU3 SOP No. 5. The mass of sediment collected may be estimated by visual assessment of sediment volume.

All sampling and field measurement equipment that is used at more than one sample station must be decontaminated following each use. Appropriate equipment decontamination procedures are provided in OU3 SOP No. 7.

5.3.5 Precipitation Monitoring

The precipitation monitoring station will be installed at the same location as the existing meteorological station at the Site. This monitoring station will be equipped with a wind shield for better accuracy as well as 2 heaters maintained at 40°F (one on the collector funnel and one on the drain tube) to allow for measurement of precipitation under freezing conditions. The gauge will be compatible with the power supply and the data-logger/data-transmission system that are currently used at the meteorological station. The gauge will have a resolution of 0.01 inches with a range of 0-10 inches per hour. The accuracy will be at least ± 0.02 inches or 4% of the hourly total (whichever is greater). The gauge will log or transmit the date and time for each tip (0.01 inches) so that duration/intensity curves can be derived from the data collected.

The rain gauge will be installed in an area that is representative of the Rainy Creek watershed within a reasonable distance (i.e., <100yds) from the existing meteorological station. The instrument will be calibrated after installation and the calibration will be checked at least once every three months thereafter. Installation, calibration, and maintenance procedures are provided in OU3 SOP No.17.

Respondents W.R Grace & Co.-Conn, and KDC shall submit to EPA and MDEQ the details of the precipitation monitoring station chosen for Phase IIA including specifications, conceptual drawings of the installation and operations and maintenance instructions.

5.3.6 Continuous Flow Monitoring with Data Logger

Each continuous flow monitoring station will be capable of measuring flows from 0.1 to 10 cfs (40-4,000 gpm). This flow range should be adequate to measure a typical spring snowmelt event as well as base-flow conditions, but it may not be large enough to measure large storm events such as the predicted 10 year, 24-hour storm which has a 150 cfs peak flow (Schaffer 1992).

The flow monitoring station design is dependent on the specific sampling location chosen in the drainage. At a minimum, the final design must be capable of measuring the flows described in the above paragraph (0.1 to 10 cfs) while achieving an accuracy of no worse that ±10% and a precision no worse than ±15%. The final design must also be capable of withstanding complete submergence without incurring permanent damage. If it is infeasible to install the final design prior to the 2008 spring runoff, a temporary monitoring station may be used until a permanent station can be constructed. Temporary monitoring stations must be capable of measuring the same flow range but with an accuracy of no worse than ±25% and a precision no worse than ± 50%. The accuracy and precision should be checked at least quarterly.

The data logger must be setup to record water level measurements on intervals no longer than 15 minutes. If possible, a shorter interval is preferred because it will allow for post-processing of the data to reduce the inherent noise of the instrument and measurement technique. The data

logger must be capable of storing at least one month's worth of measurements. A pressure transducer with internal memory, an internal battery, and capable of measuring up to 5 psi (11.5 ft of H_2O) is recommended for this application. It should also be capable of communicating with an autosampler to trigger flow-weighted composite sample collection.

Respondents W.R Grace & Co.-Conn, and KDC shall submit to EPA and MDEQ the details of the continuous flow monitoring stations including specifications, conceptual drawings of the installation and operations and maintenance instructions.

5.3.7 Field Documentation

Field documentation procedures are described in Section 5.5 and OU3 SOP No. 9. Field documentation associated with surface water and sediment sampling will also contain information of sufficient detail to fully describe:

- sample depth (sediment),
- sampling method, and
- associated field measurements, including stream discharge if measured, and field measurement methods.

Field measurement values are generally reported directly in the units of final use in the field notebook and data sheets without need for additional calculations (e.g., pH, temperature, and conductivity measurements). The field data will be reviewed daily by the field supervisor to identify anomalous data and transcriptional and/or computational errors. Corrective actions will be initiated as appropriate; these actions may consist of re-measuring a particular parameter, collecting a new sample, or other applicable corrective action measures.

5.4 Sample Handling Instructions

5.4.1 Sample Containers

All sample containers used for sample collection and analysis for this project will be prepared according to the procedures contained in the EPA document, *Specifications and Guidance for Obtaining Contaminant-Free Sample Containers,* dated December 1992. This document specifies the acceptable types of containers, the specific cleaning procedures to be used before samples are collected, and requirements relevant to the containers and cleaning procedures. The analytical laboratories will supply all sample containers utilized for this investigation, both for asbestos and non-asbestos analyses. If field personnel observe any cracked or dirty containers, or if the appropriate preservative is missing in the sample bottles, those containers will be discarded and the laboratory will be notified of the problem to prevent its re-occurrence.

Tables 5-4 and 5-5 identify the appropriate sample containers for the analysis methods used in Phase IIA for surface water and sediment samples, respectively.

5.4.2 Sample Preservation and Storage

Tables 5-4 and 5-5 describe the sample preservation and storage requirements for solid and aqueous media, respectively. Samples will be preserved using appropriate preservatives in order to prevent or minimize chemical changes that could occur during transit and storage. Solid samples (soil and sediment) typically do not require preservation other than temperature control during storage and transfer to the laboratory. The exception is solid samples collected for analyses of volatile organic compounds, including VPH and TCL VOCs. Soil and sediment samples collected for analysis of VPH and TCL VOCs will be preserved in the field with methanol based on EPA SW-846 method 5035.

5.4.3 Sample Holding Times

A holding time is defined as the allowable time between sample collection and analysis and/or extraction recommended to ensure accuracy and representativeness of analysis results, based on the nature of the analyte of interest and chemical stability factors. The holding time is calculated from the date and time of sample collection to the time of sample preparation and/or analysis. Sample holding times are established to minimize chemical changes in a sample prior to analysis and/or extraction. Samples will be shipped to the laboratory as soon as possible after collection or processing. There are currently no EPA guidelines for holding times for solid samples analyzed for metals/metalloids and most other inorganic constituents, but a six-month holding time is recommended. There is no holding time requirement for asbestos.

Tables 5-4 and 5-5 define method-specific analytical holding times for solid and aqueous media, respectively.

5.4.4 Sample Archival and Final Disposition

Unused samples and containers of environmental media will be maintained in storage at the laboratory for a minimum of 90 days following completion of the analysis, unless otherwise directed by EPA. Except as noted below, after 90 days or approval from EPA for disposal, the laboratory will be responsible for proper disposal of any remaining samples, sample containers, shipping containers, and packing materials in accordance with sound environmental practice, based on the sample analytical results. The laboratory will maintain proper records of waste disposal methods, and will have disposal company contracts on file for inspection.

Materials that shall not be disposed of but held in archive include:

- unanalyzed portions of filters and grids that have been prepared for asbestos analysis. These shall be held in archive at the asbestos analytical laboratory.
- the archive portion and three fine-ground aliquots of sediment samples will be shipped from the soil preparation laboratory to the analytical laboratory, where these materials will be held in archive until otherwise directed by EPA.

All data generated during the analysis of project samples must be stored by the laboratory for a period of ten years. Revised copies of the applicable SOPs and QAPPs must also be maintained and available should the data be required.

5.5 Sample Documentation and Identification

Data regarding each sample collected will be documented in accord with OU3 SOP No. 9 using Libby-specific field sample data sheets (FSDS). Any special circumstances that influence sample collection or result in deviations from sampling SOPs will be documented in a field log book.

At the time of collection, each sample will be labeled with a unique 5-digit sequential identification (ID) number. The sample ID for all samples collected as part of Phase II (including both Phase IIA and IIB) sampling activities will have a prefix of "P2" (e.g., P2-12345). Information on whether the sample is representative of a field sample or a field-based quality control (QC) sample (e.g., field blank, field split) will be documented on the FSDS, but this information will not be included on the chain-of-custody to make certain that the sample type is unknown to the analytical laboratory.

Each field sampling team will maintain a field log book. The log book shall record all potentially relevant information on sampling activities and conditions that are not otherwise captured on the FSDS forms. Examples of the type of information to be captured in the filed log include:

- Names of team members
- Current and previous weather conditions
- Field sketches
- Physical description of the location relative to permanent landmarks
- Number and type of samples collected
- Any special circumstances that influenced sample collection

As necessary for sample collection and location documentation, photographs will be taken using a digital camera. GPS coordinates will be recorded for all sampling locations on the FSDS form.

A stake or pole identifying the sampling station will be placed at or near the sampling station for future identification of the location.

5.6 Sample Chain of Custody and Shipment

Field sample custody and documentation will follow the requirements described in OU3 SOP No. 9. Sample packaging and shipping will follow the requirements described in OU3 SOP No. 8.

A chain-of-custody form specific to the Phase IIA OU3 sampling shall accompany every shipment of samples to the analytical laboratory. The purposes of the chain-of-custody form are: a) to establish the documentation necessary to trace possession from the time of collection to final disposal, and b) to identify the type of analysis requested. All corrections to the chain-of-custody record will be initialed and dated by the person making the corrections. Each chain-of-custody form will include signatures of the appropriate individuals indicated on the form. The originals will accompany the samples to the laboratory and copies documenting each custody change will be recorded and kept on file. One copy of the chain-of-custody will be kept by field personnel.

All required paper work, including sample container labels, chain-of-custody forms, custody seals and shipping forms will be fully completed in ink (or printed from a computer) prior to shipping of the samples to the laboratory. Shipping to the appropriate laboratory from the field or sample storage will occur through overnight delivery.

All samples that may require special handling by laboratory personnel to prevent potential exposure to LA or other hazardous substances will be clearly labeled.

Upon receipt, the samples will be given to the laboratory sample custodian. The shipping containers will be opened and the contents inspected. Chain-of custody forms will be reviewed for completeness and samples will be logged and assigned a unique laboratory sample number. Any discrepancies or abnormalities in samples will be noted and the Laboratory Manger and the EPA Remedial Project Manager will be promptly notified.

Chain-of-custody will be maintained until final disposition of the samples by the laboratory and acceptance of analytical results.

6.0 LABORATORY ANALYSIS REQUIREMENTS

6.1 Analytical Methods for Asbestos

All laboratories that analyze samples of surface water or sediment for asbestos as part of this project must participate in and have satisfied the certification requirements in the last two proficiency examinations from the National Institute of Standards and Technology/National Voluntary Laboratory Accreditation Program (NVLAP). Laboratories must also have demonstrated proficiency by successful analysis of Libby-specific performance evaluation samples and/or standard reference materials, and must participate in the on-going laboratory training program developed by the Libby laboratory team.

6.1.1 Surface Water

All surface water samples collected during Phase IIA sampling will be submitted for asbestos analysis using transmission electron microscopy (TEM) in accord with the International Organization for Standardization (ISO) 10312 method (ISO 1995) counting protocols, with all applicable Libby site-specific laboratory modifications, including the most recent versions of modifications LB-000016, LB-000019, LB-000028, LB-000029, LB-000030, LB-000053, and LB-000066. An aliquot of water (generally about 100 mL) will be filtered through a 47 mm mixed cellulose acetate (MCE) filter with pore size of 0.2 um, using a backing filter with pore size of 5 um. All amphibole structures (including not only LA but all other amphibole asbestos types as well) that have appropriate Selective Area Electron Diffraction (SAED) patterns and Energy Dispersive X-Ray Analysis (EDXA) spectra, and having length greater than or equal to 0.5 um and an aspect ratio (length:width) > 3:1, will be recorded on the Libby site-specific laboratory bench sheets and electronic data deliverable (EDD) spreadsheets ("TEM Water EDD.xls"). Data recording for chrysotile, if observed, is not required.

The target analytical sensitivity for asbestos in water is 50,000 f/L (0.05 million fibers per liter, abbreviated as MFL). The human health maximum contaminant level (MCL) for asbestos in drinking water is 7,000,000 f/L and is based on fibers longer than 10 um in length. Upon review of available ecological toxicity data in the literature, it appears that effects thresholds range from about 10,000-1,000,000 f/L for aquatic receptors and wildlife. Therefore, a target analytical sensitivity of 50,000 f/L should be adequate to provide screening level risk estimates for humans and most ecological receptors of interest. This sensitivity can be achieved by filtering 100 mL of water and counting about 20 grid openings (GOs), assuming that filter overloading does not occur.

Stopping rules for these analyses are as follows:

1. Calculate the number of GOs needed to achieve the target sensitivity.

2. If the target sensitivity can be achieved by counting 50 or fewer GOs, count until the target sensitivity is achieved, or until 50 LA structures are observed. If 50 LA structures are observed, finish counting the GO containing the 50th structure, then stop.
3. If the target sensitivity requires more than 50 GOs, count until 50 GOs are counted, or until 50 LA structures are observed. If 50 LA structures are observed, finish counting the GO containing the 50th structure, then stop.

6.1.2 Sediment

Sample Preparation

All sediment samples collected for asbestos analysis will be transmitted to the CDM soil preparation laboratory in Denver, Colorado. Samples will be prepared in accordance with ISSI-LIBBY-01 Revision 10. In brief, the raw sediment sample is dried and then split into two aliquots. One aliquot is placed into archive, and the other aliquot is sieved into coarse (> ¼ inch) and fine fractions. The fine fraction is ground to reduce particles to a diameter of 250 um or less and this fine-ground portion is split into 4 aliquots.

Sample Analysis

Each sediment sample will be analyzed for LA in accordance with Libby site-specific SOPs. The coarse fraction (if any) will be examined using stereomicroscopy, and any particles of LA will be removed and weighed in accordance with SRC-LIBBY-01 Revision 2. One of the fine ground fraction aliquots will be analyzed by polarized light microscopy (PLM) using the visual area estimation method (PLM-VE) in accordance with SRC-LIBBY-03 Revision 2. Mass fraction estimates and optical property details will be recorded on the Libby site-specific laboratory bench sheets and EDD spreadsheets.

6.2 Analytical Methods for Other (Non-Asbestos) Analytes

This section describes the laboratory analysis methods selected to provide non-asbestos chemical data to support the Phase IIA data quality objectives. Methods employed are derived from the following sources:

- *Test Methods for Evaluating Solid Waste Physical/Chemical Methods* (EPA, 1986)
- *Methods for Chemical Analysis of Water and Wastes* (EPA, 1994b)
- Montana Department of Environmental Quality method specifications for petroleum hydrocarbons (MDEQ, 2003)

Detailed calibration procedures and quality control practices associated with each referenced method are described later in Section 8.

The laboratories performing chemical analyses will be required to follow procedures for each referenced method in accordance with the method protocols in the original source documents. All method-specific quality control measures, such as external and internal standard calibration procedures, instrument performance verifications, and quantitation using method of standard additions, specified within any referenced EPA method number will be performed.

6.2.1 Water

Non-asbestos analyses required for surface water samples are listed in Table 6-1. Analytes included under each method are identified in Table 3-1.

6.2.2 Sediment

Non-asbestos analyses required for surface water samples are listed in Table 6-2. Analytes included under each method are identified in Table 3-3.

6.3 Instrument Calibration and Frequency

All laboratory instruments used in the analysis of samples generated during this project must be calibrated by the laboratory in accordance with the requirements of the instrument manufacturer and the requirements specified in the relevant analytical method. Calibration records will be kept in logbooks for all instruments. It is the responsibility of the Laboratory Quality Assurance (QA) Officer to assure that calibration data is properly logged in the logbooks for each analysis.

6.4 Laboratory Custody Procedures and Documentation

The laboratories will implement the following procedures:

- A sample custodian will be designated.
- Upon receipt at the laboratory, each sample shipment will be inspected to assess the condition of the shipping container and the individual samples.
- Enclosed chain-of-custody records will be cross-referenced with all the samples in the shipment. These records will be signed by the sample custodian and placed in the project file.
- Sample storage will be secured (in the appropriate environment, i.e., refrigerated, dry, etc.), sample storage records and intra-laboratory sample custody records will be maintained, and sample disposal and disposal date will be properly documented.
- Internal chain-of-custody procedures will be followed by assigning a unique laboratory number to each sample on receipt; this number identifies the sample through all further handling;
- Internal logbooks and records will maintain the chain-of-custody throughout sample preparation and analysis, and data reporting will be kept in the project files.

- The original chain-of-custody record will be returned to the Project QA Officer with the resulting data report from the laboratory.

It is the laboratory's responsibility to maintain internal logbooks and records throughout sample preparation, analysis, and data reporting.

6.5 Laboratory Health and Safety

All laboratories analyzing samples from OU3 must be properly trained in the safe handling, storage and disposal of samples that may contain LA and other potentially hazardous materials.

6.6 Documentation and Records

Data reports will be submitted to the Project Manager and include a case narrative that briefly describes the number of samples, the analyses, and any analytical difficulties or QA/QC issues associated with the submitted samples. The data report will also include signed chain-of-custody (COC) forms, analytical data summary report pages, and a summary of laboratory QC sample results and raw data, where applicable. Raw data are to consist of instrument preparation and calibration logs, instrument printouts of field sample results, laboratory QC sample results, calibration and maintenance records, COC check in and tracking, raw data count sheets, spectra, micrographic photos, and diffraction patterns.

6.7 Data Deliverables

Asbestos data generated during this project will be entered into Libby-specific EDD spreadsheets by appropriately trained data entry staff. The data to be captured will include all relevant field information regarding each environmental sample collected, as well as the analytical results provided by the laboratory. Analytical results will include the structure-specific data for all TEM analyses and optical properties data for all PLM analyses. All data entry will be reviewed and validated for accuracy by the laboratory data entry manager or appointed delegate.

Non-asbestos data generated for this project will be transmitted via an EDD spreadsheet. The specific structure and format of this spreadsheet will be specified by the project data manager and will be provided to the laboratory for data submittal. All data entry will be reviewed and validated for accuracy by the laboratory data entry manager or appointed delegate.

All asbestos and non-asbestos EDDs will be submitted to EPA technical contractors (SRC) electronically. Whenever possible, data files should be transmitted by e-mail to the following address:

LibbyOU3@syrres.com

When files are too large to transmit by e-mail, they should be provided on compact disk to the following address:

Lynn Woodbury
Syracuse Research Corporation
999 18th Street, Suite 1975
Denver CO 80202

All original data records (both hard copy and electronic) will be cataloged and stored in their original form until otherwise directed by the EPA Remedial Project Manager. At the termination of Phase I, all original data records will be provided to the EPA Remedial Project Manager for incorporation into the OU3 project files.

7.0 TOXICITY TESTING REQUIREMENTS

This section provides a general description of the surface water toxicity testing protocol that will be implemented during Phase IIA. Some details of this general design may be revised based on information and suggestions from the toxicity testing laboratory.

Test Waters

One large volume (about 200 L) of site water containing LA will be provided to the toxicity testing laboratory. This water will be stored in the dark at 1-4°C prior to use in the tests in order to minimize the growth of algae or any other biological organisms.

This water will be tested for toxicity to fish at a series of dilutions. Assuming the starting concentration of LA in the site water is about 100 MFL total LA, the following dilutions will be used:

100% (undiluted)
10%
1%
0.1%
0.01%
0.001%
0%

These dilutions are selected based on the findings of Belanger (1985) that concentrations as low as 0.01 MFL of chrysotile caused observable effects in fish. In addition, it is important that the test waters include at least one concentration that represents a no-effect concentration (NOEC), since this is the most likely basis for a site-specific TRV.

Dilutions of the site water shall be prepared using laboratory water that is prepared to have a hardness and pH that is similar to the site water (see Section 7.2.3 of EPA 2002 for methods of producing site waters). As indicated above, laboratory water with no added site water will also be tested to serve as a reference water.

Water Chemistry

The concentration of LA, other contaminants of potential concern (e.g., metals) and selected water quality parameters (e.g., hardness, pH) is this water will be measured before testing. Representative aliquots of each serial dilution will be removed for confirmation analysis by TEM of the concentration achieved. (Note: due to the limitations imposed by Poisson counting uncertainty, the nominal concentrations will be used instead of the measured values for dose-

response modeling, unless a measured value is statistically outside the confidence limits of the nominal concentration).

All test waters (including reference water) will be monitored in the laboratory for the following parameters:

Parameter	Frequency
Temperature	Daily
pH	Daily
Dissolved oxygen	Once per 5 days before swimup, and then at the start and end of each static renewal (every 3 days)
Ammonia	

Test Species

The test species will be rainbow trout (*Oncorhynchus mykiss*)

Life Stage

The life stage will be newly hatched larvae.

Exposure Conditions

Exposure will be performed using a static renewal protocol in 4-Liter aquaria. The renewal water must be thoroughly mixed each time before drawing samples for replacement of aquaria waters.

There will be 15 larvae per aquarium, with three aquaria per test water (a total of 45 larvae per test water).

Water temperature of test aquaria will be maintained at 12 ±1°C.

Exposure duration will be 6 weeks (42 days).

During the larval stage, water will be changed once every 10 days.

Swim-up is expected to occur on or about day 20 (after about 240 degree-days). After swim-up occurs, water will be changed once every three days.

In order to ensure that fiber settling does not occur, each aquarium will be equipped with an air bubbler placed in the bottom and run continuously. In addition, each aquarium will be equipped with a circulating filter, used without any filter media.

PRELIMINARY TEST OF MIXING

In order to confirm that aeration and circulation are sufficient to keep LA fibers suspended in the aquaria, the following procedure will be performed before the tests with fish are begun:

1. Place a site test water containing LA into one 4-L aquarium and establish aeration and circulation conditions exactly as will be done when fish are present.
2. After 3 days, remove approximately 100 mL of water from the tank at a depth of about 1-2 cm below the surface of the water. Place the sample into a clean plastic bottle. Label this bottle "LA Fiber Suspension Test: Sample A".
3. Remove approximately 100 mL from a depth of 0-2 cm above the bottom of the tank and place this sample into a second clean plastic bottle labeled "LA Fiber Suspension Test: Sample B".
4. Promptly transmit both bottles to the laboratory for analysis of LA. Both samples will be counted using TEM until a minimum of 100 LA structures have been enumerated in each sample. This will allow more than a 90% probability of detecting a relative percent difference (RPD) of 50%between the top and the bottom of the tank.
5. If no statistically significant difference is detected, tests with fish may begin. If a significant difference is detected, then alternative methods for ensuring that LA fibers are well mixed throughout the tank will be investigated before testing begins.

Feeding

No feeding will occur during the larval stage.

After swim-up occurs, fish will be fed freshly-hatched brine shrimp (about 12 hours post hatch) daily at a rate of 0.05 grams of brine shrimp per gram of fish in the aquariums. The average mass of each fish as a function of time may be estimated from measurements on fish grown in parallel tanks, or from historical growth curves.

Endpoints

Behavior

All aquaria will be observed daily for indications of differences in behavior between control fish and fish exposed to site waters. This may include, for example, differences in the frequency and duration of swimming events of the larvae, swimming and feeding behavior of the fry, etc. These observations will be recorded using the behavioral observation log sheet provided as Table 7-1.

Mortality

Observations on mortality will be recorded twice daily at approximately 8:30 AM and 4:30 PM. Table 7-2 is a form that will be used for assigning a unique identifies to each fish and for recording date and time of death of each fish.

Histopathology

All fish that die during the study and all fish alive at the end of the study will be preserved by being placed into fixative solution for subsequent histopathological evaluation. Detailed SOPs for sample preservation, slide preparation, and histological examination will be provided by the histological laboratory. Based on the work of Belanger (1985), it is expected that relevant endpoints may include dermal thickening and abrasion, abrasion or lesions of the gill, as well as lesions of the kidneys and GI tract. However, the histologist should seek to identify any potentially meaningful changes that appear to be treatment-related.

Growth

No measures of growth will be performed during the larval stage. After swim-up, measures of growth will include length and mass of the fish at the time of death or at the end of the study. Data on growth will be recorded using the form provided as Table 7-2.

Data Reporting

For each water sample tested, the laboratory shall record data using the data sheets provided in Tables 7-1 and 7-2. (EPA will provide the laboratory with electronic copies of these tables to facility data entry and transmittal). The laboratory shall also provide a text report in which the conditions of the test and any deviations from the study protocol or any other issues are described and evaluated.

8.0 QUALITY CONTROL

Quality Control (QC) is a component of the QAPP, and consists of the collection of data that allow a quantitative evaluation of the accuracy and precision of the field data collected during the project. QC samples that will be collected during this project include both field-based and laboratory-based QC samples.

8.1 Field-Based Quality Control Samples

Field-based QC samples are those samples which are prepared in the field and submitted to the laboratory in a blind fashion. That is, the laboratory is not aware the sample is a QC sample, and should treat the sample in the same way as a field sample. In general, there are three types of field QC sample: blanks, field splits/duplicates, and performance evaluation (PE) samples. Table 8-1 summarizes the types and frequency of field QC samples which will be collected during Phase IIA.

8.1.1 Blanks

Field Blanks

A field blank is a sample of the same medium as field samples, but which does not contain any contaminant. Field blanks are collected for water samples, but not for sediment.

A field blank for water shall be prepared by placing an appropriate volume of analyte-free reagent water (e.g., ASTM Type II) into a sample collection container. Field blanks for water will be collected at a rate of at least 10% (1 field blank per 10 field samples, or 1 per sample batch, whichever is greater).

Trip Blanks

The trip blank is used to indicate potential contamination of field samples by VOCs during sample shipping and handling. A trip blank consists of analyte-free laboratory reagent water which accompanies the empty sample bottles to the field and is placed in each cooler containing samples scheduled for VOC analysis. The trip blank is not opened until analysis in the laboratory with the corresponding site samples.

During Phase IIA sampling, one trip blank per cooler will be prepared to accompany aqueous samples when they are shipped to the laboratory for VOC analysis. One trip blank per cooler will also be prepared to accompany solid samples shipped for analysis of EPH and VPH.

Equipment Rinsate Blanks

Equipment rinsate blanks determine if decontamination procedures of field equipment are adequate to prevent cross-contamination of samples during sample collection. An equipment rinsate blank is prepared by rinsing decontaminated field equipment with analyte-free reagent water. Equipment rinsate blanks will be collected at a rate of 1 per sampling team per day. If field equipment is not re-used between sampling locations (i.e., dedicated equipment is used or equipment is disposable and decontamination is not necessary), equipment rinsate blanks will not be collected.

8.1.2 Field Splits/Duplicates

A field split is a sample that is prepared by thoroughly homogenizing a field sample, dividing the homogenized sample into two parts, and analyzing each independently. A comparison of field split samples is a measure of the precision of the sample preparation and analysis methods.

A field duplicate is a field sample that is collected at the same place and time as an original field sample. However, because of potential variation in field duplicate samples (even those from similar locations, especially for media such as sediment), it is not appropriate to assume that field duplicate pairs must necessarily have the same or similar concentration values. Rather, field duplicates help to evaluate variability due to small-scale media heterogeneity, along with analytical precision.

Table 8-1 summarizes the frequency that field splits and duplicates will be collected for each media. In general, field splits/duplicates will be prepared at a rate of approximately 10% (1 field split/replicate per 10 field samples). The specific stations at which field splits/duplicates will be collected will be determined in the field based on sampling conditions.

8.1.3 Performance Evaluation (PE) Samples

Performance Evaluation (PE) samples are samples of a matrix that contain a known and certified level of a contaminant. The results of PE sample analysis help evaluate analytical accuracy. PE samples for water and soil are available through the EPA Quality Assurance Technical Support (QATS) program. A total of 4 water PE samples and 3 soil PE samples containing a range of inorganic and organic analytes will be added in random order to the field samples by the field collection teams.

PE samples for LA in soil are available from USGS. These PE samples were prepared by mixing uncontaminated soil samples from Libby with known amounts of LA collected from the mine, so the true mass fraction of LA is known. A total of 4 PE samples representing a range of LA levels will be added to the sediment sample preparation and analysis train in random order at the time of sediment sample preparation by the preparation laboratory.

Equipment Rinsate Blanks

Equipment rinsate blanks determine if decontamination procedures of field equipment are adequate to prevent cross-contamination of samples during sample collection. An equipment rinsate blank is prepared by rinsing decontaminated field equipment with analyte-free reagent water. Equipment rinsate blanks will be collected at a rate of 1 per sampling team per day. If field equipment is not re-used between sampling locations (i.e., dedicated equipment is used or equipment is disposable and decontamination is not necessary), equipment rinsate blanks will not be collected.

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Table 8-1 summarizes the frequency that field splits and duplicates will be collected for each media. In general, field splits/duplicates will be prepared at a rate of approximately 10% (1 field split/replicate per 10 field samples). The specific stations at which field splits/duplicates will be collected will be determined in the field based on sampling conditions.

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8.2 Laboratory-Based Quality Control Samples for Asbestos Analysis by TEM

The QC requirements for TEM analyses of air samples at the Libby site are patterned after the requirements set forth by NVLAP. There are three types of laboratory-based QC analyses that are performed for TEM. Each of these is described in more detail below.

Lab Blank - This is an analysis of a TEM grid that is prepared from a new, unused filter by the laboratory and is analyzed using the same procedure as used for field samples.

Recounts - A recount is an analysis where TEM grid openings are re-examined after the initial examination. The type of recount depends upon who is performing the re-examination. A *Recount Same* (RS) describes a re-examination by the same microscopist who performed the initial examination. A *Recount Different* (RD) describes a re-examination by a different microscopist within the same laboratory than who performed the initial examination. An *Interlab* (IL) describes a re-examination by a different microscopist from a different laboratory.

Repreparation - A repreparation is an analysis of a TEM grid that is prepared from a new aliquot of the same field sample as was used to prepare the original grid. Typically, this is done within the same lab as did the original analysis, but a different lab may also prepare grids from a new piece of filter.

As described the most recent Libby-specific Laboratory Modification #29 (LB-000029), laboratory blanks will be performed at a frequency of 4%, recounts will be performed at a frequency of 5%, and repreparations will be performed at a frequency of 1%. LB-000029 summarizes the project-specific acceptance criteria for TEM QC analyses for all participating laboratories.

8.3 Laboratory-Based Quality Control Samples for Asbestos Analysis by PLM

8.3.1 Preparation Laboratory QC Samples

Sediment preparation QC samples are collected to ensure proper sample handling and decontamination of sediment preparation equipment. Preparation QC samples are assigned unique field identifiers and are submitted blind to the analytical laboratory along with the field samples. Thus, the analytical laboratories cannot distinguish field samples from preparation QC samples. Two types of preparation QC samples are included for PLM analysis. Each of these is described in more detail below.

Preparation Blank – A preparation blank consists of asbestos-free quartz sand which is processed with each batch of field samples. A batch of samples is defined as a group of

samples that have been prepared together for analysis at the same time (approximately 125). Preparation blanks determine if cross-contamination is occurring during sample preparation processing (i.e., drying, sieving, grinding, and splitting). The target number of preparation blanks is 1 per batch. All preparation blanks shall be PLM-VE Bin A (non-detect). If a preparation blank is ranked as a detect, the procedures for equipment decontamination between samples will be revised and revised as needed.

Preparation Splits – Preparation splits are prepared by dividing a sample into two parts after drying but prior to sieving and grinding. One preparation duplicate is included for every 20 field samples prepared. Because preparation splits may be authentically different due to within-sample heterogeneity, there are no acceptance criteria for preparation splits. Comparison of the results for preparation splits with the paired original field samples helps to evaluate the variability that arises during the preparation and analysis steps.

8.3.2 Analytical Laboratory QC Samples

As part of PLM-VE analysis, laboratory duplicate analyses will be prepared at a frequency of 10% (1 per 10 analyses). A *laboratory duplicate* is a re-preparation of a soil sample slide by a different analyst than who performed the initial analysis. Laboratory duplicates are performed to evaluate potential analytical differences between analysts. The acceptance criterion for laboratory duplicate analyses is that no more than 10% of all samples shall be discordant (assigned different PLM-VE bins). If the discordance rate is greater than 10%, laboratory procedures for sample examination and bin-assignment shall be reviewed and staff re-trained, as needed.

8.4 Laboratory-Based Quality Control Samples for Non-Asbestos Analyses

The following subsections describe laboratory-based quality control measures used to assess and document the quality of analytical results for non-asbestos parameters. Laboratory QC sample analysis frequencies and control limits used by contracted laboratories will be in accordance with referenced analytical method protocols, and the QC analyses and results will be documented and reported to EPA by the selected laboratory.

Table 8-2 summarizes all laboratory quality control measures, control limits, and corrective actions for this project, by analysis method. All laboratory QC data will be reported with results of associated sample analyses to allow for comparison of QC results to the QC criteria specified for this project.

8.4.1 Method Blank

Method blanks are designed to measure laboratory-introduced contamination of environmental samples. Method blanks verify that method interferences caused by airborne contaminants, solvents, reagents, glassware, or other sample processing hardware are known and minimized. The blank will be ASTM Type II water (or equivalent) for water samples. The method/reagent blank is processed through all procedures, materials, and lab-ware used for sample preparation and analysis.

The frequency for method blank preparation and analysis is a minimum of one per twenty field samples or per analytical batch, whichever is most frequent. An analytical batch is defined as samples which are analyzed together with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods. Samples in each batch are to be of similar composition or matrix.

Acceptance criteria and corrective action for out-of-control method blanks are provided in Table 8-2.

8.4.2 Laboratory Control Samples

Laboratory control samples (LCSs) are designed to check the accuracy of the analytical procedure by measuring a known concentration of an analyte of interest. LCS samples are prepared by spiking clean, laboratory-simulated matrices (reagent-free water or purified solid matrix) with representative analytes at known concentrations that are approximately 10 times greater than the method's quantitation limits. These spiked samples are then subjected to the same preparation and analytical procedures as associated environmental samples. A LCS will be analyzed with every analytical batch, and the measured concentrations will be compared to the known, or spiked, concentrations of the LCS to compute a percent recovery value.

LCSs will be analyzed at a minimum frequency of one per every 20 samples or one per analytical batch of no more than 20 samples. Control limits for laboratory control samples are listed on Table 8-2. Failure of the LCS to meet recovery criteria requires corrective action before any further analyses can continue.

For some methods, a duplicate of the LCS is also analyzed with each analytical batch and the difference between the LCS and the LCS Duplicate (LCSD) indicates the precision of laboratory sample preparation and analysis methods at a known concentration level. Control limits for precision measured by the RPD of LCS/LCSD results are listed in Table 8-2. When LCSD samples are analyzed, the minimum frequency of analysis is one per every 20 samples.

8.4.3 Matrix Spikes/Matrix Spike Duplicates

Matrix spike/matrix spike duplicate (MS/MSD) samples are designed to evaluate the effect of the sample matrix on analytical data, by measuring precision and accuracy from a known concentration of a target analyte that has been added to a particular sample matrix. MS/MSD samples are prepared by spiking environmental field samples with a standard solution containing known concentrations of representative target analytes. The MS/MSD sample pair is prepared from three volumes of an environmental sample. Two portions of the sample (the MS and the MSD) are spiked with the standard solution. The remaining volume is not spiked. The spiked samples are analyzed, and the percent recovery (PR) and relative percent difference (RPD) between the results of the MS analysis and the MSD analysis are calculated. The unaltered sample volume is analyzed as an ordinary environmental sample.

Sampling personnel will identify for the laboratory which samples are to be used for MS/MSD preparation. Field blanks and field duplicates are not used as MS/MSDs. Typically, additional sample volume will be required to prepare the MS and MSD, especially for analyses of water samples for organic compounds. MS/MSDs will be analyzed at a minimum frequency of one per every 20 samples.

Background and interferences that have an effect on the actual sample analyte will have a similar effect on the spike. The calculated percent recovery of the matrix spike is considered to be a measure of the relative accuracy of the total analytical method, i.e., sample preparation and analysis. The matrix spike is also a measure of the effect of the sample matrix on the ability of the methodology to detect specific analytes. Acceptance criteria and corrective action procedures for out-of-control matrix spike results are listed in Table 8-2.

8.4.4 Surrogate Spike Analyses

Surrogate spike analyses are used to determine the efficiency of target analyte recovery during sample preparation and analysis. A surrogate spike is prepared by adding a known amount of surrogate compound to an environmental sample before extraction. The surrogate compound is selected to exhibit an analytical response that is similar to the response displayed by a target compound during sample analysis. The accuracy of the analytical method is measured using the calculated percent recovery of the spiking compound. Poor reproducibility and percent recovery during surrogate spike analyses may indicate sample matrix effects.

Surrogate compounds are not added to inorganic analyses; however, surrogates are required for most organic analyses. Both environmental and QC samples are spiked with surrogate compounds. Surrogate spike recoveries are acceptable if the results of a surrogate spike fall within the control limits established by laboratory QC protocol. Acceptance criteria and corrective action procedures for out-of-control surrogate spike results are listed in Table 8-2.

Frequencies for surrogate spike analyses will be consistent with the referenced method protocols.

8.4.5 Internal Standards

Internal Standards (ISs) are compounds of known concentrations used to quantitate the concentrations of target detections in field and QC samples. ISs are added to all samples after sample extraction or preparation. Because of this, ISs provide for the accurate quantitation of target detections by allowing for the effects of sample loss through extraction, purging, and/or matrix effects. ISs are used for any method requiring an IS calibration. Corrective action is required when ISs are out of control. Acceptance criteria and corrective action procedures for out-of-control internal standard spike results are listed in Table 8-2.

8.4.6 Instrument Calibration and Frequency

Analytical instruments will be calibrated in accordance with the referenced analytical methods. All target analytes that are reported to EPA will be present in the initial and continuing calibrations, and these calibrations must meet the acceptance criteria specified in referenced methods. Records of standard preparation and instrument calibration will be maintained by the contract laboratory. Records will unambiguously trace the preparation of standards and their use in calibration and quantitation of sample results. Calibration standards will be traceable to standard materials.

Analyte concentrations are determined with either calibration curves (linear regression) or response factors (RFs). All correlation coefficients for linear regression calibration curves or relative standard deviation (RSD) of RFs to determine linearity must meet the acceptability criteria specified within the method. For GC/MS methods, the average RF from the initial five-point calibration will be used to determine analyte concentrations. The continuing calibration curve will not be used to update the RFs from the initial five-point calibration. GC/MS methods also will meet all instrument performance and/or tuning criteria as specified by the methods.

Initial Calibration Verification

Initial calibration curves must be verified using a standard made from a source independent of the one used to make the initial calibration standards. All target compounds must be included within the initial calibration verification (ICV), typically at a concentration around the midpoint of the calibration curve. Control limits and corrective action procedures for out-of-control initial calibration verification results are listed in Table 8-2.

Continuing Calibration and Verification

Initial calibration curves must be verified daily prior to sample analysis. All target compounds must be included, typically at a concentration around the midpoint of the calibration curve.

Continuing calibration verifications (CCVs) are check samples required at frequencies specified in each analytical method, typically at the beginning and end of each analytical sequence and after every ten samples analyzed (as specified in each analytical method). Control limits and corrective action procedures for out-of-control CCV results are listed Table 8-2.

Calibration procedures for a specific laboratory instrument will consist of initial calibration (3- or 5-points), initial calibration verification (ICV) and continuing calibration verification (CCV). Calibration protocols included in method references, including calibration frequencies, conditions, and acceptance criteria, will be followed.

8.5 Quality Assurance Objectives For Measurement Data

This section identifies specific objectives for precision, accuracy, representativeness, completeness, and comparability of measurement data collected to support the Phase I data quality objectives.

8.5.1 Precision

Precision is defined as the agreement between a set of replicate measurements without assumption or knowledge of the true value. Agreement is expressed as either the relative percent difference (RPD) for duplicate measurements, or the range and standard deviation for larger numbers of replicates. Precision will be assessed through the calculation of the relative percent difference (RPD) for two replicate samples. RPD is calculated according to the following formula:

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

where: S = Original sample value
D = Duplicate sample value

Field precision is assessed through the collection and measurement of field duplicates. The variability between field duplicates reflect the combined variation in concentration between nearby samples and the variation due to measurement error. Because the variability between field duplicates is random and may be either small or large, no quantitative requirement for the agreement of field duplicates is established for this project.

Precision in the laboratory is assessed through calculation of RPDs for duplicate analyses or relative standard deviations (RSDs) for three or more replicate analyses of the same sample. Results from mine waste, soil, and sediment duplicate samples are expected to be more variable than results from duplicate water samples due to the physical and chemical heterogeneity of the solid matrices. Based on this, an RPDs of 50% for mine waste, soil, sediment field duplicate

samples and RPDs of 25% for water field duplicates will be used as advisory limits for analytes detected in both the original sample and its field duplicate at concentrations greater than 5 times the reported quantitation limit.

Differences greater than these advisory limits will be noted for data users through the data validation process.

8.5.2 Accuracy

Accuracy is a measure of the agreement between a measurement and the "true" value. The accuracy of a measurement may be affected by errors introduced by field contamination, sample preparation and handling, and sample analysis. The accuracy of an analytical method is generally assessed by analyses of samples with known concentration levels, including field calibration standards (for field based measurements), laboratory control samples, MS/MSD samples, and PE samples.

The accuracy required for data usability depends on a number of factors. In general, good accuracy is most important for samples whose concentration values are close to the level of concern, and a somewhat lesser level of accuracy may be acceptable for samples whose concentrations are either well below or well above a level of concern. Based on this, the goal is to achieve an analytical accuracy of ±25% for analytes that are within a factor of 10 of initial estimates of the level of concern, and ±50% for samples either 10-fold above or 10-fold below initial estimates of the level of concern.

8.5.3 Representativeness

Representativeness is the degree to which data accurately and precisely represent characteristics of a population, parameter variations at a sampling point, or an environmental condition. Representativeness of field measurements is dependent upon the proper design of the sampling program and will be satisfied by ensuring that the SAP and SOPs are followed. The sampling activities in this plan are designed to provide data that are representative of conditions at specific locations and times of sample collection.

8.5.4 Completeness

Data are considered complete when a prescribed percentage of the total intended measurements and samples are obtained. Analytical completeness is defined as the percentage of valid analytical results requested.

Field completeness is a measure of the amount of valid measurement data collected for the project. The target completeness objective for field measurements collected for this sampling program is 95 percent or more.

Laboratory completeness is a measure of the amount of valid laboratory-measurement data obtained for the project. For this sampling program, a minimum of 90% percent of the planned collection of individual samples for quantification must be obtained to achieve a satisfactory level of data completeness.

8.5.5 Comparability

Data are comparable if collection techniques, measurement procedures, methods, and reporting units are equivalent for the samples within a sample set. These criteria allow comparison of data from different sources. Comparable data will be obtained by specifying standard units for physical measurements and standard procedures for sample collection, processing, and analysis.

The criteria for field comparability will be to ensure and document that the sampling designs are properly implemented and the sampling procedures are consistently followed for the duration of the data collection program. Each sampling task will utilize standardized procedures for sample collection and field measurements, as specified in Section 5 of this plan.

The criteria for laboratory data comparability will be to ensure that the laboratory results generated during this phase of investigation will be comparable to laboratory data collected for all other environmental investigations at OU3 and comparable to the asbestos data already collected by EPA in the vicinity of OU3. This goal will be achieved through utilization of standard EPA Test Methods and site-specific asbestos analysis methods for sample analyses and adherence to quality assurance/quality control and analytical procedures specified for the OU3 RI.

9.0 DATA MANAGEMENT

9.1 Data Applications

All data generated as part of the Phase IIA sampling event will be maintained in an OU3-specific Microsoft Access® database. This will be a relational database with tables designed to store information on station location, sample collection details, preparation and analysis details, and analytical results. Results will include asbestos data (including detailed structure attributes for TEM analyses and optical properties for PLM analyses) and non-asbestos chemical data (e.g., metals.

9.2 Roles and Responsibilities for Data Flow

9.2.1 Field Personnel

W.R. Grace contractors will perform all Phase IIA sample collection in accordance with the project-specific sampling plan and SOPs presented above. In the field, sample details will be documented on hard copy media-specific FSDS forms and in field log books (see Section 5.5). COC information will be documented on hard copy forms (see Section 5.6). FSDS and COC information will be manually entered into a field-specific[2] OU3 database using electronic data entry forms. Use of electronic data entry forms ensures the accuracy of data entry and helps maintain data integrity. For example, data entry forms utilize drop-down menus and check boxes whenever possible. These features allow the data entry personnel to select from a set of standard inputs, thereby preventing duplication and transcription errors and limiting the number of available selections (e.g., media types). In addition, entry into a database allows for the incorporation of data entry checks. For example, the database will allow a unique sample ID to only be entered once, thus ensuring that duplicate records cannot be created.

Entry of FSDS forms and COC information will be completed weekly, or more frequently as conditions permit. Copies of all FSDS forms, COC forms, and field log books will be scanned and posted in portable document format (PDF) to a project-specific file transfer protocol (FTP) site weekly. This FTP site will have controlled access (i.e., user name and password are required) to ensure data access is limited to appropriate project-related personnel. File names for scanned FSDS forms, COC forms, and field log books will include the sample date in the format YYYYMMDD to facilitate document organization (e.g., FSDS_20070831.pdf). Electronic copies of all digital photographs will also be posted weekly to the project-specific FTP site. File names for digital photographs will include the station identifier, the sample date, and photograph identifier (e.g., ST-1_20070831_12459.tif).

[2] The field-specific OU3 database is a simplified version of the master OU3 database. This simplified database includes only the station and sample recording and tracking tables, as well as the FSDS and COC data entry forms.

After FSDS data entry is completed, a copy of the field-specific OU3 database will be posted by the field data manager to the project-specific FTP weekly, or more frequently as conditions permit. The field-specific OU3 database posted to the FTP site will include the post date in the file name (e.g., FieldOU3DB_20070831.mdb).

9.2.2 Laboratory Personnel

Each of the laboratories performing asbestos analyses for the Phase IIA sampling event are required to utilize all applicable Libby-specific Microsoft® Excel spreadsheets for asbestos data recording and electronic submittals (see Section 6.7). Upon completion of the appropriate analyses, EDDs will be transmitted via email to a designated email distribution list within the appropriate turn around time. Hard copies of all analytical laboratory data packages will be scanned and posted as a PDF to the project-specific FTP site. File names for scanned analytical laboratory data packages will include the laboratory name and the job number to facilitate document organization (e.g., LabX_12365-A.pdf).

9.2.3 Database Administrators

Day-to-day operations of the master OU3 database will be under the control of EPA contractors. The primary database administrator will be responsible for sample tracking, uploading new data, performing error checks, and making any necessary data corrections. New records will be added to the master OU3 database within an appropriate time period of FSDS and/or EDD receipt.

Incremental backups of the master OU3 database will be performed daily Monday through Thursday, and a full backup will be performed each Friday. The full backup tapes will be stored off-site for 30 days. After 30 days, the tape will be placed back into the tape library to be overwritten by another full backup.

9.3 Data Storage

All original data records (both hard copy and electronic) will be cataloged and stored in their original form until otherwise directed by the EPA Remedial Project Manager. At the termination of this project, all original data records will be provided to the EPA Remedial Project Manager for incorporation into the site project files.

10.0 ASSESSMENT AND OVERSIGHT

Assessments and oversight reports to management are necessary to ensure that procedures are followed as required and that deviations from procedures are documented. These reports also serve to keep management current on field activities. Assessment, oversight reports, and response actions are discussed below.

10.1 Assessments

10.1.1 Field Oversight

All individuals who collect samples during field activities will be provided a copy of this SAP and will be required to participate in a pre-sampling readiness review meeting to ensure that methods and procedures called for in this SAP and associated SOPs are understood and that all necessary equipment is on hand. EPA may perform random and unannounced field audits of field sampling collection activities, as may be deemed necessary.

10.1.2 Laboratory Oversight

All laboratories selected for analysis of samples for asbestos will be part of the Libby analytical team. These laboratories have all demonstrated experience and expertise in analysis of LA in environmental media, and all are part of an on-going site-specific quality assurance program designed to ensure accuracy and consistency between laboratories. These laboratories are audited by EPA and NVLAP on a regular basis. Additional laboratory audits may be conducted upon request from the EPA, as may be needed.

10.2 Response Actions

If any inconsistencies or errors in field or laboratory methods and procedures are identified, response actions will be implemented on a case-by-case basis to correct quality problems. All response actions will be documented in a memo to the EPA RPM for OU3 at the following address:

Bonita Lavelle
U.S. EPA Region 8
1595 Wynkoop Street
Denver, CO 80202-1129
E-mail: lavelle.bonita@epa.gov

Any problems that cannot be corrected quickly through routine procedures may require implementation of a corrective action request (CAR) form.

10.3 Reports to Management

Field and analytical staff will promptly communicate any difficulties or problems in implementation of the SAP to EPA, and may recommend changes as needed. If any revisions to this SAP are needed, the EPA RPM will approve these revisions before implementation by field or analytical staff.

11.0 DATA VALIDATION AND USABILITY

11.1 Data Validation and Verification Requirements

Data validation, review, and verifications must be performed on sample results before distribution to the public for review.

Validation of Non-Asbestos Data

For non-asbestos analytical data, data validation will be performed in accord with the most current versions of EPA's National Functional Guidelines. In brief, the validation process consists of examining the sample data package(s) in order to determine if the data comply with the requirements specified in the National Functional Guidelines. The validator may examine, as appropriate, the reported results, QC summaries, case narratives, COC information, raw data, initial and continuing instrument calibration, and other reported information to evaluate the accuracy and completeness of the data package. During this process, the validator will determine if analytical methodologies were followed and QC requirements were met. The validator may recalculate selected analytical results to verify the accuracy of the reported information, as appropriate, and will assign qualifiers to the data as needed.

Verification of Asbestos Data

For asbestos analytical data, data verification includes checking that all required data have been entered on the laboratory bench sheets and field sample data sheets, and that results have been transferred correctly to the EDD. Some of the data verification checks are performed as a function of built-in quality control checks in the Libby-specific data entry spreadsheets. Additional verifications of field and analytical results will be performed manually by independent review of the bench sheets and FSDS. The initial frequency of manual review will be 10% of all samples. This initial rate may be revised either upward or downward depending on the frequency and nature of errors that are identified by the verification process.

11.2 Reconciliation with Data Quality Objectives

Once all samples have been collected and the analytical data have been reported and validated, the data will be reviewed by data users to determine if DQOs were achieved. A report of the data quality evaluation will be posted on the Libby OU3 site web page, when completed.

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Table 3-1. Phase I Analytical Methods for Surface Water

Category	Method	Analytes			
Metals	SW6020 & SW 6010B	Aluminum	Beryllium	Copper	Selenium
		Antimony	Cadmium	Lead	Silver
		Arsenic	Chromium	Manganese	Thallium
		Barium	Cobalt	Nickel	Vanadium
		Boron	Iron	Potassium	Zinc
		Calcium	Magnesium	Sodium	
	SW7470A	Mercury			
Pesticides	SW8081A	4,4'-DDD	beta-BHC	Endosulfan sulfate	Heptachlor
		4,4'-DDE	Chlordane	Endrin	Heptachlor epoxide
		4,4'-DDT	delta-BHC	Endrin aldehyde	Isodrin
		Aldrin	Dieldrin	Endrin ketone	Methoxychlor
		alpha-BHC	Endosulfan I	gamma-BHC (Lindane)	Toxaphene
		alpha-Chlordane	Endosulfan II	gamma-Chlordane	
	SW8151A	2,4,5-T	Dalapon	MCPA	
		2,4,5-TP (Silvex)	Dicamba	MCPP	
		2,4-D	Dichlorprop	Pentachlorophenol	
Organophosphorus Pesticides	8141A	Dichlorvos	Diazinon	Chlorpyrifos	Stirophos (Tetrachlorovinphos)
		Mevinphos	Disulfoton	Trichloronate	Bolstar (Sulprofos)
		Demeton-O,S	Dimethoate	Methyl Parathion	Fensulfothion
		Ethoprop (Prophos)	Ronnel	Mathion	EPN
		Phorate	Merphos	Tokuthion (Prothiofos)	Azinphos-methyl (Guthion)
		Sulfotep	Fenthion	Ethyl Parathion	Coumaphos
PCBs	SW8082	Aroclor 1016	Aroclor 1242	Aroclor 1260	
		Aroclor 1221	Aroclor 1248	Aroclor 1262	
		Aroclor 1232	Aroclor 1254	Aroclor 1268	
VOCs	SW8260B	1,1,1-Trichloroethane	1,3-Dichlorobenzene	Chlorodibromomethane	Methyl isobutyl ketone
		1,1,2,2-Tetrachloroethane	1,4-Dichlorobenzene	Chloroethane	Methyl tert-butyl ether (MTBE)
		1,1,2-Trichloro-1,2,2-trifluoroethane	1,4-Dioxane	Chloroform	Methylcyclohexane
		1,1,2-Trichloroethane	2-Hexanone	Chloromethane	Methylene chloride
		1,1-Dichloroethane	Acetone	cis-1,2-Dichloroethene	o-Xylene
		1,1-Dichloroethene	Benzene	cis-1,3-Dichloropropene	Styrene
		1,2,3-Trichlorobenzene	Bromochloromethane	Cyclohexane	Tetrachloroethene
		1,2,4-Trichlorobenzene	Bromodichloromethane	Dichlorodifluoromethane	Toluene
		1,2-Dibromo-3-chloropropane	Bromoform	Ethylbenzene	trans-1,2-Dichloroethene
		1,2-Dibromoethane	Bromomethane	Isopropylbenzene	trans-1,3-Dichloropropene
		1,2-Dichlorobenzene	Carbon disulfide	m+p-Xylenes	Trichloroethene
		1,2-Dichloroethane	Carbon tetrachloride	Methyl acetate	Trichlorofluoromethane
		1,2-Dichloropropane	Chlorobenzene	Methyl ethyl ketone	Vinyl chloride
SVOCs	SW8270C	1,2,4,5-Tetrachlorobenzene	3,3'-Dichlorobenzidine	bis(-2-chloroethyl)Ether	Hexachlorocyclopentadiene
		2,3,4,6-Tetrachlorophenol	3-Nitroaniline	bis(2-chloroisopropyl)Ether	Hexachloroethane
		2,4,5-Trichlorophenol	4,6-Dinitro-2-methylphenol	bis(2-ethylhexyl)Phthalate	m+p-Cresols
		2,4,6-Trichlorophenol	4-Bromophenyl phenyl ether	Butylbenzylphthalate	Nitrobenzene
		2,4-Dichlorophenol	4-Chloro-3-methylphenol	Caprolactam	n-Nitroso-di-n-propylamine
		2,4-Dimethylphenol	4-Chlorophenyl phenyl ether	Carbazole	n-Nitrosodiphenylamine
		2,4-Dinitrophenol	4-Nitroaniline	Dibenzofuran	o-Cresol
		2,4-Dinitrotoluene	4-Nitrophenol	Diethyl phthalate	p-Chloroaniline
		2,6-Dinitrotoluene	Acetophenone	Dimethyl phthalate	Pentachlorophenol
		2-Chloronaphthalene	Atrazine	Di-n-butyl phthalate	Phenol
		2-Chlorophenol	Benzaldehyde	Di-n-octyl phthalate	
		2-Nitroaniline	Biphenyl	Hexachlorobenzene	
		2-Nitrophenol	bis(-2-chloroethoxy)Methane	Hexachlorobutadiene	
PAHs	SW8270C	2-Methylnaphthalene	Benzo(a)pyrene	Dibenzo(a,h)anthracene	Naphthalene
		Acenaphthene	Benzo(b)fluoranthene	Fluoranthene	Phenanthrene
		Acenaphthylene	Benzo(g,h,i)perylene	Fluorene	Pyrene
		Anthracene	Benzo(k)fluoranthene	Indeno(1,2,3-cd)pyrene	
		Benzo(a)anthracene	Chrysene	Isophorone	
Extractable hydrocarbons	MA-EPH	C11 to C22 Aromatics	C9 to C18 Aliphatics		
		C19 to C36 Aliphatics	Total Extractable Hydrocarbons		
	SW8015M	Total Extractable Hydrocarbons			
Volatile hydrocarbons	MA-VPH	C5 to C8 Aliphatics	Benzene	Methyl tert-butyl ether (MTBE)	
		C9 to C10 Aromatics	Ethylbenzene	Naphthalene	
		C9 to C12 Aliphatics	Toluene	m+p-Xylenes	
		Total Purgeable Hydrocarbons	Xylenes, Total	o-Xylene	
Nitrogen cmpds	E350.1	Nitrogen, Ammonia as N			
	E351.2	Nitrogen, Kjeldahl, Total as N			
	E353.2	Nitrogen, Nitrate+Nitrite as N			
	E353.2	Nitrogen, Nitrite as N	Nitrogen, Nitrate as N		
Radionuclides	E900.0	Gross Alpha			
	E903.0	Radium 226			
	RA-05	Radium 228			
	A7500-RA	Radium 226 + Radium 228			
Anions	E300.0	Chloride	Fluoride	Sulfate	
	E365.1	Orthophosphate as P			
	Kelada mod	Cyanide, Total			
Water quality parameters	A2320 B	Alkalinity, Total as CaCO3			
		Bicarbonate as HCO3			
		Carbonate as CO3			
		Hardness as CaCO3			
	A2540 C,D	Solids, Total Dissolved TDS			
	A5310 C	Organic Carbon, Dissolved (DOC)			

Table 3-2. List of Phase I Surface Water Stations and Analyses

			Asbestos (LA)	Cations			Pesticides			PCBs	VOCs	SVOCs	PAHs	Pertroleum Hydrocarbons			Nitrogen Compunds				Radionuclides				Anions			Water quality parameters		
				TAL Metals		Hg								Extractable HC		Volatile HC	NH4	Total N	N02+NO3	NO2	Gross α	Ra226	Ra228	Ra226+228	Cl, F, SO4	PO4	CN	HCO3,CO3	TDS	DOC
Sample	Reach	Station	EPA 100 2	SW6020	SW6010B	SW7470A	SW8081A	SW8151A	8141A	SW8082	SW8260B	SW8270C	SW8270C	MA-EPH	SW8015M	MA-VPH	E350 1	E351.2	E353 2	E353 2	E900 0	E903 0	RA-05	A7500-RA	E300 0	E365 1	Kelada	A2320 B	A2540C,D	A5310 C
1	Upper Rainy Creek	URC-1	X	X	X	X									X	X	X	X	X	X					X	X		X	X	X
2		URC-2	X	X	X	X									X	X	X	X	X	X					X	X		X	X	X
3	Tailings impoundment	TP	X	X	X	X									X	X	X	X	X	X					X	X		X	X	X
4		TP-TOE1	X	X	X	X	X	X	X		X	X	X		X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
5		TP-TOE2	X	X	X	X									X	X	X	X	X	X					X	X		X	X	X
6	Mill pond	MP	X	X	X	X									X	X				X					X	X		X	X	X
7	Lower Rainy Creek	LRC-1	X	X	X	X									X	X	X	X	X	X					X	X		X	X	X
8		LRC-2	X	X	X	X	X	X	X	X	X	X	X		X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
9		LRC-3	X	X	X	X									X	X	X	X	X	X					X	X		X	X	X
10		LRC-4	X	X	X	X									X	X	X	X	X	X					X	X		X	X	X
11		LRC-5	X	X	X	X									X	X	X	X	X	X					X	X		X	X	X
12		LRC-6	X	X	X	X									X	X	X	X	X	X					X	X		X	X	
13	Fleetwood Creek	FC-1	X	X	X	X									X	X	X	X	X	X					X	X		X	X	X
14		FC-Pond	X	X	X	X							X	X	X	X	X	X	X	X					X	X		X	X	X
15		FC-2	X	X	X	X									X	X	X	X	X	X					X	X		X	X	X
16	Carney Creek	CC-1	X	X	X	X									X	X				X					X	X		X	X	X
17		CC-2	X	X	X	X									X	X				X					X	X		X	X	X
18	Seeps	CCS-1	X	X	X										X	X	X	X	X	X					X	X		X	X	X
19		CCS-6	X	X	X	X									X	X				X					X	X		X	X	X
20		CCS-8	X	X	X	X									X	X				X					X	X		X	X	X
21		CCS-9	X	X	X	X									X	X				X					X	X		X	X	X
22		CCS-11	X	X	X	X									X	X				X					X	X		X	X	X
23		CCS-14	X	X	X	X							X	X	X	X				X					X	X		X	X	X
24		CCS-16	X	X	X	X									X	X				X					X	X		X	X	X

X= Sample analyzed

Table 3-3. Phase I Analytical Methods for Sediment

Category	Method	Analytes			
Metals	SW6020 & SW6010B	Aluminum	Chromium	Selenium	
		Antimony	Cobalt	Silver	
		Arsenic	Copper	Thallium	
		Barium	Iron	Vanadium	
		Beryllium	Lead	Zinc	
		Boron	Manganese		
		Cadmium	Nickel		
	SW7471A	Mercury			
Cyanide	SW9012	Total cyanide			
Pesticides	SW8081A	4,4'-DDD	beta-BHC	Endosulfan sulfate	Heptachlor
		4,4'-DDE	Chlordane	Endrin	Heptachlor epoxide
		4,4'-DDT	delta-BHC	Endrin aldehyde	Isodrin
		Aldrin	Dieldrin	Endrin ketone	Methoxychlor
		alpha-BHC	Endosulfan I	gamma-BHC (Lindane)	Toxaphene
		alpha-Chlordane	Endosulfan II	gamma-Chlordane	
	SW8151A	2,4,5-T	Dalapon	MCPA	
		2,4,5-TP (Silvex)	Dicamba	MCPP	
		2,4-D	Dichlorprop	Pentachlorophenol	
Organophosphorus Pesticides	8141A	Dichlorvos	Diazinon	Chlorpyrifos	Stirophos (Tetrachlorovinphos)
		Mevinphos	Disulfoton	Trichloronate	Bolstar (Sulprofos)
		Demeton-O,S	Dimethoate	Methyl Parathion	Fensulfothion
		Ethoprop (Prophos)	Ronnel	Mathion	EPN
		Phorate	Merphos	Tokuthion (Prothiofos)	Azinphos-methyl (Guthion)
		Sulfotep	Fenthion	Ethyl Parathion	Coumaphos
PCBs	SW8082	Aroclor 1016	Aroclor 1242	Aroclor 1260	
		Aroclor 1221	Aroclor 1248	Aroclor 1262	
		Aroclor 1232	Aroclor 1254	Aroclor 1268	
VOCs	SW8260B	1,1,1-Trichloroethane	1,3-Dichlorobenzene	Chlorodibromomethane	Methyl isobutyl ketone
		1,1,2,2-Tetrachloroethane	1,4-Dichlorobenzene	Chloroethane	Methyl tert-butyl ether (MTBE)
		1,1,2-Trichloro-1,2,2-trifluoroethane	1,4-Dioxane	Chloroform	Methylcyclohexane
		1,1,2-Trichloroethane	2-Hexanone	Chloromethane	Methylene chloride
		1,1-Dichloroethane	Acetone	cis-1,2-Dichloroethene	o-Xylene
		1,1-Dichloroethene	Benzene	cis-1,3-Dichloropropene	Styrene
		1,2,3-Trichlorobenzene	Bromochloromethane	Cyclohexane	Tetrachloroethene
		1,2,4-Trichlorobenzene	Bromodichloromethane	Dichlorodifluoromethane	Toluene
		1,2-Dibromo-3-chloropropane	Bromoform	Ethylbenzene	trans-1,2-Dichloroethene
		1,2-Dibromoethane	Bromomethane	Isopropylbenzene	trans-1,3-Dichloropropene
		1,2-Dichlorobenzene	Carbon disulfide	m+p-Xylenes	Trichloroethene
		1,2-Dichloroethane	Carbon tetrachloride	Methyl acetate	Trichlorofluoromethane
		1,2-Dichloropropane	Chlorobenzene	Methyl ethyl ketone	Vinyl chloride
SVOCs	SW8270C	1,2,4,5-Tetrachlorobenzene	3,3'-Dichlorobenzidine	bis(-2-chloroethyl)Ether	Hexachlorocyclopentadiene
		2,3,4,6-Tetrachlorophenol	3-Nitroaniline	bis(2-chloroisopropyl)Ether	Hexachloroethane
		2,4,5-Trichlorophenol	4,6-Dinitro-2-methylphenol	bis(2-ethylhexyl)Phthalate	m+p-Cresols
		2,4,6-Trichlorophenol	4-Bromophenyl phenyl ether	Butylbenzylphthalate	Nitrobenzene
		2,4-Dichlorophenol	4-Chloro-3-methylphenol	Caprolactam	n-Nitroso-di-n-propylamine
		2,4-Dimethylphenol	4-Chlorophenyl phenyl ether	Carbazole	n-Nitrosodiphenylamine
		2,4-Dinitrophenol	4-Nitroaniline	Dibenzofuran	o-Cresol
		2,4-Dinitrotoluene	4-Nitrophenol	Diethyl phthalate	p-Chloroaniline
		2,6-Dinitrotoluene	Acetophenone	Dimethyl phthalate	Pentachlorophenol
		2-Chloronaphthalene	Atrazine	Di-n-butyl phthalate	Phenol
		2-Chlorophenol	Benzaldehyde	Di-n-octyl phthalate	
		2-Nitroaniline	Biphenyl	Hexachlorobenzene	
		2-Nitrophenol	bis(-2-chloroethoxy)Methane	Hexachlorobutadiene	
PAHs	SW8270C	2-Methylnaphthalene	Benzo(a)pyrene	Dibenzo(a,h)anthracene	Naphthalene
		Acenaphthene	Benzo(b)fluoranthene	Fluoranthene	Phenanthrene
		Acenaphthylene	Benzo(g,h,i)perylene	Fluorene	Pyrene
		Anthracene	Benzo(k)fluoranthene	Indeno(1,2,3-cd)pyrene	
		Benzo(a)anthracene	Chrysene	Isophorone	
Extractable hydrocarbons	MA-EPH	C11 to C22 Aromatics	C9 to C18 Aliphatics		
		C19 to C36 Aliphatics	Total Extractable Hydrocarbons		
	SW8015M	Total Extractable Hydrocarbons			
Volatile hydrocarbons	MA-VPH	C5 to C8 Aliphatics	Benzene	Methyl tert-butyl ether (MTBE)	
		C9 to C10 Aromatics	Ethylbenzene	Naphthalene	
		C9 to C12 Aliphatics	Toluene	m+p-Xylenes	
		Total Purgeable Hydrocarbons	Xylenes, Total	o-Xylene	
Anions	E300.0	Fluoride			
	E365.1	Total Phosphorus			
Sediment quality parameters	ASAM10-3.2	pH, sat. paste			
	SW3550A	Moisture			
	Leco	Carbon, Organic			

Table 3-4. List of Phase I Sediment Stations and Analyses

			Asbestos (LA)		Cations			Cyanide	Pesticides			PCBs	VOCs	SVOCs	PAHs	Petroleum Hydrocarbons			Anions		Sediment quality parameters		
					TAL Metals		Hg									Extractable HC		Volatile HC	Fluoride	Phosphorus	pH	Moisture	OC
Sample	Reach	Station	PLM-VE	PLM-GRAV	SW6020	SW6010B	SW7471A	SW9012	SW8081A	SW8151A	8141A	SW8082	SW8260B	SW8270C	SW8270C	MA-EPH	SW8015M	MA-VPH	E300.0	E365.1	ASAM10-3.2	SW3550A	Leco
1	Upper Rainy Creek	URC-1	X		X	X	X								X	X	X	X	X	X	X	X	X
2		URC-2	X	X	X	X	X								X	X	X	X	X	X	X	X	X
3	Tailings impoundment	TP	X	X	X	X	X								X	X	X	X	X	X	X	X	X
4		TP-TOE1	X	X	X	X	X										X	X	X	X	X	X	X
5		TP-TOE2	X	X	X	X	X	X	X	X	X	X	X	X			X	X	X	X	X	X	X
6	Mill pond	MP	X		X	X	X								X	X	X	X	X	X	X	X	X
7	Lower Rainy Creek	LRC-1	X	X	X	X	X		X	X		X					X	X	X	X	X	X	X
8		LRC-2	X	X	X	X	X	X	X	X	X	X	X	X			X	X	X	X	X	X	X
9		LRC-3	X		X	X	X		X	X		X			X	X	X	X	X	X	X	X	X
10		LRC-4	X		X	X	X		X	X		X					X	X	X	X	X	X	X
11		LRC-5	X	X	X	X	X		X	X		X					X	X	X	X	X	X	X
12		LRC-6	X		X	X	X		X	X		X			X	X	X	X	X	X	X	X	X
13	Fleetwood Creek	FC-1	X	X	X	X	X										X	X	X	X	X	X	X
		FC-Pond	X		X	X	X								X	X	X	X	X	X	X	X	X
14		FC-2	X	X	X	X	X								X	X	X	X	X	X	X	X	X
16	Carney Creek	CC-1	X	X	X	X	X								X	X	X	X	X	X	X	X	X
17		CC-2	X	X	X	X	X										X	X	X	X	X	X	X
18	Seeps	CCS-1	X		X	X	X										X	X	X	X	X	X	X
19		CCS-6	X	X	X	X	X										X	X	X	X	X	X	X
20		CCS-8	X	X	X	X	X								X	X	X	X	X	X	X	X	X
21		CCS-9	X	X	X	X	X										X	X	X	X	X	X	X
22		CCS-11	X	X	X	X	X								X	X	X	X	X	X	X	X	X
23		CCS-14	X	X	X	X	X										X	X	X	X	X	X	X
24		CCS-16	X		X	X	X								X	X	X	X	X	X	X	X	X

X = Sample analyzed

Table 3-5. Phase I Asbestos Results for Surface Water

Reach	Station	Sensitivity 1E 06/L	Total LA (MFL)				LA > 10 um in Length (MFL)			
			LA Count	Best Estimate	95% Conf. Bounds		LA Count	Best Estimate	95% Conf. Bounds	
Upper Rainy Creek	URC-1	0.05	0	<0	0.0	0.1	0	<0.05	0.0	0.1
	URC-2	0.11	52	5.8	4.3	7.5	1	0.1	0.0	0.5
Tailings Impoundment	TP	1.99	57	114	86.9	146.0	19	38	23.6	57.9
	TP-TOE1	0.05	0	<0.1	0.0	0.1	0	<0.05	0.0	0.1
	TP-TOE2	0.20	10	2.0	1.0	3.5	6	1.2	0.5	2.5
Mill Pond	MP	0.50	54	27	20.4	34.8	20	10	6.3	15.1
Lower Rainy Creek	LRC-1	0.05	4	0.2	0.1	0.5	0	<0.05	0.0	0.1
	LRC-2	0.05	2	0.1	0.0	0.3	1	0.05	0.0	0.2
	LRC-3	0.05	4	0.2	0.1	0.5	0	<0.05	0.0	0.1
	LRC-4	0.05	21	1.0	0.7	1.6	3	0.2	0.0	0.4
	LRC-5	0.05	25	1.2	0.8	1.8	2	0.1	0.0	0.3
	LRC-6	0.05	0	<0.1	0.0	0.1	0	<0.05	0.0	0.1
Fleetwood Creek	FC-1	0.08	51	3.9	2.9	5.1	12	0.9	0.5	1.6
	FC-Pond	2.49	50	125	93.5	162.7	3	7.5	2.1	19.9
	FC-2	0.05	4	0.2	0.1	0.5	1	0.05	0.0	0.2
Carney Creek	CC-1	0.05	20	0.9	0.6	1.4	7	0.3	0.1	0.7
	CC-2	0.05	1	0.00	0.0	0.2	1	0.05	0.0	0.2
Seeps	CCS-9	0.05	0	<0.1	0.0	0.1	0	<0.05	0.0	0.1
	CCS-8	0.05	0	<0.1	0.0	0.1	0	<0.05	0.0	0.1
	CCS-6	1.99	50	100	74.8	130.2	2	4.0	0.8	12.8
	CCS-1	0.14	53	7.5	5.7	9.8	3	0.4	0.1	1.1
	CCS-11	0.33	50	17	12.5	21.7	10	3.3	1.7	5.9
	CCS-14	0.20	55	11	8.3	14.2	0	<0.2	0.0	0.5
	CCS-16	0.08	0	<0.1	0.0	0.2	0	<0.08	0.0	0.2

Table 3-6. Phase I Asbestos Results for Sediment

Reach	Station	Index ID	MASS (grams)		RESULTS	
			Fine Fraction	Coarse Fraction	$MF_{LA\%}$ fine	$MF_{LA\%}$ coarse
Upper Rainy Creek	URC-1	P1-00409	137.7	0	ND	--
	URC-2	P1-00408	123.1	47.9	<1%	Tr
Tailings Impoundment	TP	P1-00407	100.2	6.6	<1%	Tr
	TP-TOE1	P1-00326	142.2	30.6	2%	0.38%
	TP-TOE2	P1-00325	183.2	29	3%	0.03%
Mill Pond	MP	P1-00348	166.7	0	<1%	--
Lower Rainy Creek	LRC-1	P1-00338	210.9	44.7	<1%	0.13%
	LRC-2	P1-00336	256.9	36.2	<1%	Tr
	LRC-3	P1-00335	98.86	0	2%	--
	LRC-4	P1-00329	137.8	0	<1%	--
	LRC-5	P1-00328	129.8	35	<1%	Tr
	LRC-6	P1-00327	183.5	0	<1%	--
Fleetwood Creek	FC-2	P1-00406	203.7	14.3	Tr	ND
	FC-Pond	P1-00405	89.2	0	<1%	--
	FC-1	P1-00404	200.9	31.2	ND	ND
Carney Creek	CC-2	P1-00399	153.9	37.4	<1%	0.20%
	CC-1	P1-00395	126.1	28.6	4%	0.52%
Seeps	CCS-9	P1-00400	111.9	8.7	7%	Tr
	CCS-8	P1-00398	75.6	33.6	6%	0.41%
	CCS-6	P1-00397	163.9	21.8	2%	Tr
	CCS-1	P1-00396	170.2	53.3	2%	Tr
	CCS-11	P1-00402	183.3	26.4	<1%	0.20%
	CCS-14	P1-00403	129.6	4.1	<1%	Tr
	CCS-16	P1-00289	119	0	4%	--

TABLE 3-7. PHASE I NON-ASBESTOS RESULTS FOR SURFACE WATER

Category	Detected Analytes	Units	Detection Frequency (DF)	Mean Detection Limit (DL)	Concentration	
					Mean[1]	Max
Metals†	Barium	mg/L	24 / 24 100%	na	0.47	1.00
	Copper	mg/L	1 / 24 4%	0.002	0.0011	0.004
	Iron	mg/L	3 / 24 13%	0.03	0.071	1.34
	Manganese	mg/L	5 / 24 21%	0.02	0.045	0.66
	Vanadium	mg/L	1 / 24 4%	0.01	0.0052	0.01
	Calcium	mg/L	24 / 24 100%	na	82	131
	Magnesium	mg/L	24 / 24 100%	na	24	49
	Potassium	mg/L	24 / 24 100%	na	13	33
	Sodium	mg/L	24 / 24 100%	na	8	15
Volatile Hydrocarbons	Benzene	ug/L	1 / 24 4%	0.5	0.27	0.65
	C5 to C8 Aliphatics	ug/L	3 / 24 13%	20	13.6	62
	TPH	ug/L	3 / 24 13%	20	13.0	53
Extractable Hydrocarbons	TEH	mg/L	2 / 24 8%	0.30	0.17	0.47
Nitrogen Compounds	Nitrate	mg/L	10 / 15 67%	0.01	0.1	1.2
	Nitrite	mg/L	1 / 24 4%	0.01	0.0	0.01
Radionuclides	Gross Alpha	pCi/L	2 / 2 100%	na	2.1	2.5
Anions	Chloride	mg/L	22 / 24 92%	1	4.5	10
	Fluoride	mg/L	24 / 24 100%	na	0.4	0.9
	Sulfate	mg/L	24 / 24 100%	na	19.9	58
	PO4	mg/L	24 / 24 100%	na	0.2	1.16
Water Quality Parameters	Hardness as CaCO3	mg/L	20 / 20 100%	na	307	464
	Carbonate as CO3	mg/L	2 / 24 8%	4	2.5	11
	TDS	mg/L	24 / 24 100%	na	371	549
	TSS	mg/L	4 / 24 17%	10	7.8	36
	DOC	mg/L	23 / 23 100%	na	4.1	15

na = not applicable, all samples detected
TPH = Total Purgeable Hydrocarbons
TEH = Total Extractable Hydrocarbons
†Data presented in this table are based on the dissolved fraction for metals
[1] Mean calculated assuming 1/2 DL for NDs

TABLE 3-8. PHASE I NON-ASBESTOS RESULTS FOR SEDIMENT

Category	Detected Analytes	Detection Frequency (DF)	Mean Detection Limit (DL) (mg/kg)	Concentration (mg/kg)	
				Mean[a]	Max
Metals	Aluminum	24 / 24 100%	na	12,419	33,800
	Arsenic	10 / 24 42%	2.00	2.1	7
	Barium	24 / 24 100%	na	844	4,930
	Chromium	24 / 24 100%	na	149	988
	Cobalt	23 / 24 96%	5.00	18	75
	Copper	24 / 24 100%	na	31	66
	Iron	24 / 24 100%	na	21,817	54,600
	Lead	23 / 24 96%	5.00	27	100
	Manganese	24 / 24 100%	na	1,240	12,700
	Mercury	2 / 24 8%	0.10	0.1	0.1
	Nickel	23 / 24 96%	5.00	37	226
	Selenium	4 / 24 17%	0.50	0.4	1.4
	Thallium	3 / 24 13%	0.60	0.5	4.3
	Vanadium	24 / 24 100%	na	45	105
	Zinc	24 / 24 100%	na	27	54
PAH	Pyrene	1 / 14 7%	0.87	0.4	1.2
VOC	Methyl acetate	2 / 2 100%	na	0.3	0.4
Extractable Hydrocarbons	C11 to C22 Aromatics	4 / 12 33%	24.41	63	436
	C19 to C36 Aliphatics	4 / 12 33%	25.63	71	350
	C9 to C18 Aliphatics	2 / 12 17%	26.40	28	162
	Total Extractable Hydrocarbons (MA-EPH)	4 / 12 33%	25.13	188	1,240
	Total Extractable Hydrocarbons (SW8015M)	23 / 24 96%	9.80	176	928
Volatile Hydrocarbons	C9 to C10 Aromatics	1 / 24 4%	3.86	2.3	10
	C9 to C12 Aliphatics	1 / 24 4%	3.95	2.0	10
	Total Purgeable Hydrocarbons	3 / 24 13%	3.65	2.9	17
Anions	Fluoride	5 / 24 21%	1.00	0.9	4.1
	Total Phosphorus	24 / 24 100%	na	2,564	10,200
Sediment Quality Parameters	pH, sat. paste	24 / 24 100%	na	7.2	8
	Moisture	24 / 24 100%	na	39.9	86
	Carbon, Organic	24 / 24 100%	na	2.5	15

na = not applicable

[a] Mean calculated assuming 1/2 DL for NDs

Table 3-9. Phase I Surface Water Flow Data

Station ID	Date	Time	Flow (ft^3/sec)	Flow (gal/min)
URC-1	10/18/2007	12:00	0.09	39
URC-2	10/18/2007	11:30	0.04*	20*
TP-TOE1	10/18/2007	12:20	0.29	132
TP-TOE2	10/18/2007	12:35	0.58	259
LRC-1	10/18/2007	12:15	0.41	184
LRC-2	10/18/2007	11:55	0.50	224
LRC-3	10/18/2007	11:33	0.76	341
LRC-4	10/18/2007	11:12	0.34	153
LRC-5	10/18/2007	10:50	0.63	283
LRC-6	10/18/2007	10:44	0.41	184
FC-1	10/18/2007	10:45	0.14	65
FC-2	10/18/2007	11:10	0	0
CC-1	10/18/2007	10:15	0.07	30
CC-2	10/18/2007	10:00	0.19	84

*Flow was observed at less than 19 gallons per minute with 5% leakage.
After adjusting for leakage a value of 20 gallons per minute was estimated.

TABLE 4-1. PROJECTED NUMBER OF SURFACE WATER SAMPLES

EXPOSURE UNIT	STATION	ASBESTOS (LA)			NON-ASBESTOS					
					Analytical Suite 1			Analytical Suite 2		
		P1	P2A[a]	Total	P1	P2A[a]	Total	P1	P2A[a]	Total
Upper Rainy Creek	URC-1	1	2	30	1	2	8			
	URC-1A		13			2				
	URC-2	1	13		1	2				
Tailings Impoundment	TP-Overflow		13	44		2	11			3
	TP+UTP	1	13		1	2				
	TP-TOE1	1	13		1	2		1	2	
	TP-TOE2	1	2		1	2				
Mill Pond	MP	1	13	14	1	2	3			
Lower Rainy Creek	LRC-1	1	13	65	1	2	18			3
	LRC-2	1	20		1	2		1	2	
	LRC-3	1	2		1	2				
	LRC-4	1	2		1	2				
	LRC-5	1	2		1	2				
	LRC-6	1	20		1	2				
Fleetwood Creek	FC-1	1	2	20	1	2	9			
	FC-Pond	1	2		1	2				
	FC-2	1	13		1	2				
Carney Creek	CC-1	1	2	30	1	2	8			
	CC-2	1	13		1	2				
	CC-Pond		13			2				
Seeps	CCS-9	1	2	21	1	2	21			
	CCS-8	1	2		1	2				
	CCS-6	1	2		1	2				
	CCS-1	1	2		1	2				
	CCS-11	1	2		1	2				
	CCS-14	1	2		1	2				
	CCS-16	1	2		1	2				
Kootenai River	UKR		2	18						
	KR-1		2							
	KR-2		2							
	KR-3		2							
	KR-4		2							
	KR-5		2							
	KR-6		2							
	KR-7		2							
	KR-8		2							

[a] Approximate number of samples to be collected (may vary based on field conditions at the time of sampling).

LA = Libby amphibole
P1 = Phase I
P2A = Phase IIA
Suite 1 = Partial analytical suite (may include cations, petroleum hydrocarbons, nitrogen compounds, anions, and water quality parameters).

Suite 2 = Full analytical suite (may include cations, pesticides, PCBs, VOCs, SVOCs, PAHs, petroleum hydrocarbons, nitrogen compounds, radionuclides, anions, and water quality parameters).

TABLE 4-2. PROJECTED NUMBER OF SEDIMENT SAMPLES

EXPOSURE UNIT	STATION	ASBESTOS			NON-ASBESTOS					
		LA			Analytical Suite 1			Analytical Suite 2		
		P1	P2A[a]	Total	P1	P2A[a]	Total	P1	P2A[a]	Total
Upper Rainy Creek	URC-1	1	2	8	1	2	8			
	URC-1A		2			2				
	URC-2	1	2		1	2				
Tailings Impoundment	TP+UTP	1	17	24	1	17	24			3
	TP-TOE1	1	2		1	2				
	TP-TOE2	1	2		1	2		1	2	
Mill Pond	MP	1	5	6	1	5	6			
Lower Rainy Creek	LRC-1	1	2	18	1	2	18		2	13
	LRC-2	1	2		1	2		1	2	
	LRC-3	1	2		1	2			2	
	LRC-4	1	2		1	2			2	
	LRC-5	1	2		1	2			2	
	LRC-6	1	2		1	2			2	
Fleetwood Creek	FC-1	1	2	12	1	2	12			
	FC-Pond	1	5		1	5				
	FC-2	1	2		1	2				
Carney Creek	CC-1	1	2	11	1	2	11			
	CC-2	1	2		1	2				
	CC-Pond		5			5				
Seeps	CCS-9	1	2	21	1	2	21			
	CCS-8	1	2		1	2				
	CCS-6	1	2		1	2				
	CCS-1	1	2		1	2				
	CCS-11	1	2		1	2				
	CCS-14	1	2		1	2				
	CCS-16	1	2		1	2				
Kootenai River	UKR		1	6						
	North Bank		3							
	Sand Bar		2							

[a] Approximate number of samples to be collected (may vary based on field conditions at the time of sampling).

LA = Libby amphibole
P1 = Phase I
P2A = Phase IIA
Suite 1 = Partial analytical suite (may include cations, PAHs, petroleum hydrocarbons, anions, and sediment quality parameters).
Suite 2 = Full analytical suite (may include cations, pesticides, PCBs, VOCs, SVOCs, petroleum hydrocarbons, anions, and sediment quality parameters).

Table 5-1. Libby OU3 Phase IIA Surface Water and Sediment Sampling Program Elements

Part A. Rainy Creek Watershed

Program Element		Goal	Medium	Sampling Locations	Sampling Medium/ Frequency/Duration	Sample Type	Chemical Analytes	Field Measures
1	Seasonal Monitoring	Characterize conditions at multiple stations, as a function of season	Surface water	Same as Phase I plus URC-1A, UTP (2 depths), and Carney Ck Pond	Spring (one sample) Summer (one sample)	Grab	Same as Phase I	Flow
			Sediment	Same as Phase I plus URC-1A and multiple samples from Carney Ck pond and Mill Pond	Late spring after peak runoff (one sample) Summer (one sample)	Grab	Same as Phase I	---
2	Spring Runoff Monitoring	Evaluate changes in water quality during rising and falling limbs of Spring snowmelt-runoff hydrograph	Surface water	FC-2 URC-2 URC-1A TP TP-TOE1 TP-overflow (if flowing) CC-2 CC-Pond LRC-1 LRC-2 LRC-6 MP	Once per week One sample before rise in hydrograph to establish baseline. Once rise begins, weekly until 4 weeks past peak (may be adjusted based on field observation)	Grab	Asbestos	Flow
3	Summer-fall monitoring	Continue measurements from element 2, but reduce stations and frequency	Surface water	LRC-2 LRC-6	beginning with cessation of element 2, once per two weeks plus 3 storm events until September 30	24-hour flow-weighted composite with auto sampler	Asbestos	Flow
4	Continuous Flow Monitoring	Characterize spring-snowmelt hydrograph. Use flow data with asbestos data to characterize asbestos loads.	Surface water	LRC-2 LRC-6 CC-2	LRC-2 and LRC-6 Begin approx. March 1 and end September 30 CC-2 During spring runoff period only (see element 2)	Continuous data logger (install flume and automated monitoring equipment).		Flow
5	Water collection for toxicity testing	Establish site-specific TRV for asbestos	Surface water	URC-1 LRC-2 TP LRC-4 MP LRC-6	Once (max concentration)	Approx 150 L	Asbestos TAL metals	

Table 5-1. Libby OU3 Phase IIA Surface Water and Sediment Sampling Program Elements
(continued)

II. Part B: Kootenai River

Sampling Program Element		Goal	Medium	Sampling Locations	Sampling Medium Frequency/Duration	Sample Type	Analytes
1	Kootenai River Sampling	Evaluate asbestos levels in river water (and sediment) near mine area.	Water	Mid-river upstream (1 location); Transect across river downstream of Rainy Creek (5 locations just downstream of sandbar); Along north bank downstream of Rainy Ck. (3 locations)	Once at time of Rainy Ck peak snowmelt-runoff flow One time in summer	Grab	Asbestos
			Sediment	One sample in depositional area along north bank, upstream of Rainy Creek 2-3 samples from depositional areas along north bank, downstream of Rainy Creek, within 1/2 mile Two samples from the large sandbar mid-channel, downstream of Rainy Creek	One time in summer	Grab Grab Boring to water	Asbestos

Table 5-2
Phase IIA Surface Water/Sediment Sampling Locations in the Rainy Creek Watershed

Station ID	Description
URC-1	Upper Rainy Creek above Mine Area
URC-1A	Upper Rainy Creek above Mine Area 100 yards north of Rainy Creek Rd.
URC-2	Upper Rainy Creek above Mine Area
LRC-1	Lower Rainy Creek above confluence with Carney Creek
LRC-2	Lower Rainy Creek below confluence with Carney Creek
LRC-3	Lower Rainy Creek
LRC-4	Lower Rainy Creek
LRC-5	Lower Rainy Creek
LRC-6	Lower Rainy Creek just above confluence with the Kootenai River
FC-1	Fleetwood Creek above Mine Area
FC-2	Fleetwood Creek above Tailings Impoundment
UTP	Upper Tailings Impoundment
TP	Tailings Impoundment
TP-TOE1	Toe drain of impoundment
TP-TOE2	Toe drain flow to Rainy Creek below diversion
TP-overflow*	In the overflow ditch from tailings impoundment
MP	Mill Pond
CC-1	Carney Creek
CC-2	Carney Creek just above confluence with Rainy Creek
CC-Pond	Pond on lower Carney Creek
CCS-1	Spring from base of west waste rock pile
CCS-6	Spring below west waste rock pile
CCS-8	Spring below west waste rock pile
CCS-9	Spring discharging to lower Carney Creek
CCS-11	Spring below central waste rock pile
CCS-14	Spring between central and east waste rock piles
CCS-16	Spring below east waste rock pile

* Sample will be collected if there is overflow from the impoundment during scheduled monitoring activities

Table 5-3
Phase IIA - Rainy Creek Watershed Surface Water Monitoring Summary

Station ID	Element 1	Element 2	Element 3	Element 4	Element 5
URC-1	X				
URC-1A	X	X			
URC-2	X	X			
LRC-1	X	X			
LRC-2	X	X	X	X	X
LRC-3	X				
LRC-4	X				X
LRC-5	X				
LRC-6	X	X	X	X	X
FC-1	X				
FC-2	X	X			
TP	X	X			X
UTP	X*				
TP-TOE1	X	X			
TP-TOE2	X				
TP-overflow	X**	X**			
MP	X	X			X
CC-1	X				
CC-2	X	X		X (spring)	
CC-Pond	X	X			
CCS-1	X				
CCS-6	X				
CCS-8	X				
CCS-9	X				
CCS-11	X				
CCS-14	X				
CCS-16	X				

* Samples will be collected at two depths
** Sample will be collected if there is overflow from the impoundment during scheduled monitoring activities

Table 5-4. Sample Containers, Preservation and Handling Requirements, and Holding Times for Sediment Samples

Container Description	Analyses	Method	Preservation and Handling	Extraction/Analysis Holding Times
8-oz glass jar	TAL Metals + Boron	EPA 6010/6020	Cool 4°C	180 days
	Mercury	EPA 7471A	Cool 4°C	28 days
	Total organic carbon (TOC)	EPA 9060/415.1	Cool 4°C protect from sunlight and atmospheric oxygen	28 days
	Paste pH	EPA 9045D/ ASAM10-3.2	Cool 4°C	14 days
	Fluoride and total phosphorus	EPA 300.0/ SM4500-F-C EPA 365.1	Cool 4°C	14 days (F) 28 days (P)
	Cyanide	EPA 9012	Cool 4°C	14 days
10-mL glass vial with Teflon-lined crew cap (pre-preserved with methanol)	Volatile Petroleum Hydrocarbons	MADEP-VPH-04-1.1	Cool 4°C	28 days
4-oz wide-mouth amber glass jar with Teflon-lined screw cap	Extractable Petroleum Hydrocarbons	MADEP-EPH-04-1	Cool 4°C	14 days
4-oz glass jar	Organophosphate Pesticides	EPA 8141	Cool 4°C	14 days/40 days
8-oz glass jar	Chlorinated Pesticides	EPA 8081	Cool 4°C	14 days/40 days
	Herbicides	EPA 8151	Cool 4°C	14 days/40 days
	Polychlorinated Biphenyls (PCBs)	EPA 8082	Cool 4°C	14 days/40 days
10-mL glass vial with Teflon-lined crew cap (pre-preserved with methanol)	Volatile Organic Compounds (VOCs) (a)	EPA 8260B	Cool 4°C	14 days
4-oz amber glass jar with Teflon-lined screw cap	Semi-volatile Organic Compounds (SVOCs) (a)	EPA 8270C	Cool 4°C	14 days/40 days
500 g in Ziploc bag (soil) or plastic jar (sediment)	Asbestos	PLM-Grav: SRC-LIBBY-01 (Rev. 2) PLM-VE: SRC-LIBBY-03 (Rev. 2)	None	None
8-oz glass jar	*[Archive sample]*		Cool 4°C	–

(a) CLP analyte list

(b) with Libby-specific modifications

Table 5-5. Sample Containers, Preservation and Handling Requirements, and Holding Times for Aqueous Sample Matrices

Container Description	Analyses	Method	Preservation and Handling	Extraction/ Analysis Holding Times
250-mL plastic (pre-preserved with HNO_3)	TAL Metals+Boron (Total)	6010/6020 and EPA 200 series methods (a)	Cool 4°C; HNO_3, pH<2	180 days
	Mercury	7470A/ EPA 245.1	Cool 4°C	28 days
250-mL plastic filtration container	TAL Metals+Boron (Dissolved), Hardness	6010/6020 and EPA 200 series methods (a)	Cool 4°C; HNO_3 (preserve sample in field after filtering)	180 days
1-L amber glass	Dissolved Organic Carbon (DOC)	9060/ EPA 415.1	Cool 4°C; H_3PO_4 (preserve sample in field after filtering)	28 days
500-mL plastic (pre-preserved with H_2SO_4)	Nitrate, Ammonia, Total Kjeldahl Nitrogen (TKN)	EPA 353.2, 350.1/350.2, 351.2	Cool 4°C; H_2SO_4, pH<2	28 days
	Orthophosphate	EPA 365.2	Cool 4°C; H_2SO_4, pH<2	28 days
3 x 40-mL amber glass vial with Teflon-lined screw cap (pre-preserved with HCl)	Volatile Petroleum Hydrocarbons (VPH)	MA-DEP VPH modified	HCl to pH <2 Cool 4°C	14 days
2 x 1-L amber glass bottle with Teflon-lined screw cap (pre-preserved with H_2SO_4)	Extractable Petroleum Hydrocarbons (EPH)	MA-DEP EPH modified	H_2SO_4 to pH <2, Cool 4°C	14 days/40 days
1-L plastic	Fluoride/Chloride/Sulfate	EPA 300.0	Cool 4°C	28 days
	Total Suspended Solids (TSS)	Standard Methods 2540D	Cool 4°C	7 days
	Nitrite	EPA 353.2	Cool 4°C	48 hours
	Total Dissolved Solids (TDS)	Standard Methods 2540C	Cool 4°C	7 days
	Alkalinity	Standard Methods 2320B	Cool 4°C	14 days
2 x 1-L amber glass	Organophosphate Pesticides	EPA 8141	Cool 4°C	7 days/40 days
2 x 1-L amber glass	Chlorinated Pesticides	EPA 8081	Cool 4°C	7 days/40 days
	Polychlorinated Biphenyls (PCBs)	EPA 8082	Cool 4°C	7 days/40 days
2 x 1-L amber glass	Herbicides	EPA 8151	Cool 4°C	7 days/40 days
2 x 1-L amber glass (b)	Semi-volatile Organic Compounds (SVOCs) (c)	EPA 8270C	Cool 4°C	7 days/40 days
3 x 40 mL vials; no headspace (pre-preserved with HCl)	Volatile Organic Compounds (VOCs) (c)	EPA 8260B	Cool 4°C; HCl pH<2	14 days
1-L plastic (pre-preserved with HNO_3)	Radiochemistry (gross alpha and gross beta)	EPA 900.0	Cool 4°C; HNO_3	None
	Radium, Uranium	EPA 900.3, EPA 200 series	Cool 4°C; HNO_3	180 days
500-mL plastic(pre-preserved with NaOH)	Cyanide	EPA 335.4	Cool 4°C; NaOH, pH>12	14 days
1 L HDPE container	Asbestos	ISO 10312 (d)	Cool 4°C	Filtered within 48 hours

(a) 200 series methods: 200.7, 200.8
(b) 2 additional 1-L amber glass containers will be needed for MS/MSD
(c) CLP analyte list
(d) with Libby-specific modifications

Table 6-1. *List of Non-Asbestos Analyses Required for Surface Water for Element 1 (Spring, Summer)*

			Cations			Pesticides			PCBs	VOCs	SVOCs	PAHs	Petroleum Hydrocarbons			Nitrogen Compunds				Radionuclides				Anions			Water quality parameters		
			TAL Metals		Hg								Extractable HC		Volatile HC	NH4	Total N	N02+NO3	NO2	Gross α	Ra226	Ra228	Ra226+228	Cl, F, SO4	PO4	CN	HCO3,CO3	TDS	DOC
Index	Reach	Station	SW6020	SW6010B	SW7470A	SW8081A	SW8151A	8141A	SW8082	SW8260B	SW8270C	SW8270C	MA-EPH	SW8015M	MA-VPH	E350 1	E351.2	E353 2	E353.2	E900 0	E903.0	RA-05	A7500-RA	E300 0	E365.1	Kelada	A2320 B	A2540C,D	A5310 C
1	Upper Rainy Creek	URC-1	X	X	X								X	X	X	X	X	X	X					X	X		X	X	X
2		URC-1A	X	X	X								X	X	X	X	X	X	X					X	X		X	X	X
3		URC-2	X	X	X								X	X	X	X	X	X	X					X	X		X	X	X
4	Tailings impoundment	TP	X	X	X								X	X	X	X	X	X	X					X	X		X	X	X
		UTP	X	X	X								X	X	X	X	X	X	X					X	X		X	X	X
5		TP-TOE1	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
		TP-TOE2	X	X	X								X	X	X	X	X	X	X					X	X		X	X	X
6		TP-Overflow	X	X	X								X	X	X	X	X	X	X					X	X		X	X	X
7	Mill pond	MP	X	X	X								X	X	X	X	X	X	X					X	X		X	X	X
8	Lower Rainy Creek	LRC-1	X	X	X								X	X	X	X	X	X	X					X	X		X	X	X
9		LRC-2	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
10		LRC-3	X	X	X								X	X	X	X	X	X	X					X	X		X	X	X
11		LRC-4	X	X	X								X	X	X	X	X	X	X					X	X		X	X	X
12		LRC-5	X	X	X								X	X	X	X	X	X	X					X	X		X	X	X
13		LRC-6	X	X	X								X	X	X	X	X	X	X					X	X		X	X	X
14	Fleetwood Creek	FC-1	X	X	X								X	X	X	X	X	X	X					X	X		X	X	X
15		FC-Pond	X	X	X								X	X	X	X	X	X	X					X	X		X	X	X
16		FC-2	X	X	X								X	X	X	X	X	X	X					X	X		X	X	X
17	Carney Creek	CC-1	X	X	X								X	X	X	X	X	X	X					X	X		X	X	X
18		CC-Pond	X	X	X								X	X	X	X	X	X	X					X	X		X	X	X
19		CC-2	X	X	X								X	X	X	X	X	X	X					X	X		X	X	X
20	Seeps	CCS-1	X	X	X								X	X	X	X	X	X	X					X	X		X	X	X
21		CCS-6	X	X	X								X	X	X	X	X	X	X					X	X		X	X	X
22		CCS-8	X	X	X								X	X	X	X	X	X	X					X	X		X	X	X
23		CCS-9	X	X	X								X	X	X	X	X	X	X					X	X		X	X	X
24		CCS-11	X	X	X								X	X	X	X	X	X	X					X	X		X	X	X
25		CCS-14	X	X	X								X	X	X	X	X	X	X					X	X		X	X	X
26		CCS-16	X	X	X								X	X	X	X	X	X	X					X	X		X	X	X

x = Sample analyzed

Table 6-2. List of Non-Asbestos Analyses Required for Sediment in Element 1 (Spring and Summer)

			Cations			Pesticides			PCBs	VOCs	SVOCs	PAHs	Pertroleum Hydrocarbons			Anions		Sediment quality parameters		
			TAL Metals		Hg								Extractable HC		Volatile HC	Fluoride	Phosphorus	pH	Moisture	OC
Sample	Reach	Station	SW6020	SW6010B	SW7470A	SW8081A	SW8151A	8141A	SW8082	SW8260B	SW8270C	SW8270C	MA-EPH	SW8015M	MA-VPH	E300.0	E365.1	ASAM10-3.2	SW3550A	Leco
1	Upper Rainy Creek	URC-1	X	X	X								X	X	X	X	X	X	X	X
.		URC-1A	X	X	X								X	X	X	X	X	X	X	X
2		URC-2	X	X	X								X	X	X	X	X	X	X	X
3	Tailings impoundment	TP	X	X	X								X	X	X	X	X	X	X	X
		UTP	X	X	X								X	X	X	X	X	X	X	X
4		TP-TOE1	X	X	X								X	X	X	X	X	X	X	X
5		TP-TOE2	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
6	Mill pond	MP	X	X	X								X	X	X	X	X	X	X	X
7	Lower Rainy Creek	LRC-1	X	X	X	X	X		X				X	X	X	X	X	X	X	X
8		LRC-2	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
9		LRC-3	X	X	X	X	X		X				X	X	X	X	X	X	X	X
10		LRC-4	X	X	X	X	X		X				X	X	X	X	X	X	X	X
11		LRC-5	X	X	X	X	X		X				X	X	X	X	X	X	X	X
12		LRC-6	X	X	X	X	X		X				X	X	X	X	X	X	X	X
13	Fleetwood Creek	FC-1	X	X	X								X	X	X	X	X	X	X	X
		FC-Pond	X	X	X								X	X	X	X	X	X	X	X
14		FC-2	X	X	X								X	X	X	X	X	X	X	X
16	Carney Creek	CC-1	X	X	X								X	X	X	X	X	X	X	X
		CC-Pond	X	X	X								X	X	X	X	X	X	X	X
17		CC-2	X	X	X								X	X	X	X	X	X	X	X
18	Seeps	CCS-1	X	X	X								X	X	X	X	X	X	X	X
19		CCS-6	X	X	X								X	X	X	X	X	X	X	X
20		CCS-8	X	X	X								X	X	X	X	X	X	X	X
21		CCS-9	X	X	X								X	X	X	X	X	X	X	X
22		CCS-11	X	X	X								X	X	X	X	X	X	X	X
23		CCS-14	X	X	X								X	X	X	X	X	X	X	X
24		CCS-16	X	X	X								X	X	X	X	X	X	X	X

x = Sample analyzed

TABLE 7-1. BEHAVIORAL LOG

Water Sample = _____ Study Start Date = _____

Day	Observations
1	
2	
3	
4	
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TABLE 7-2. MORTALITY AND GROWTH LOG

Test water description = Mill Pond
Test water code = MP

Aquarium	Fish	Label Assigned	Day of death	Time death was noted	Length (mm)	Weight (mg)	Notes
A	1	MP-A1					
	2	MP-A2					
	3	MP-A3					
	4	MP-A4					
	5	MP-A5					
	6	MP-A6					
	7	MP-A7					
	8	MP-A8					
	9	MP-A9					
	10	MP-A10					
	11	MP-A11					
	12	MP-A12					
	13	MP-A13					
	14	MP-A14					
	15	MP-A15					
B	1	MP-B1					
	2	MP-B2					
	3	MP-B3					
	4	MP-B4					
	5	MP-B5					
	6	MP-B6					
	7	MP-B7					
	8	MP-B8					
	9	MP-B9					
	10	MP-B10					
	11	MP-B11					
	12	MP-B12					
	13	MP-B13					
	14	MP-B14					
	15	MP-B15					
C	1	MP-C1					
	2	MP-C2					
	3	MP-C3					
	4	MP-C4					
	5	MP-C5					
	6	MP-C6					
	7	MP-C7					
	8	MP-C8					
	9	MP-C9					
	10	MP-C10					
	11	MP-C11					
	12	MP-C12					
	13	MP-C13					
	14	MP-C14					
	15	MP-C15					

Table 8-1. Summary of Field Quality Control Samples

Field QC Sample Type	Applicable Sample Media	Minimum Collection Frequency	Analyses to be Performed	Acceptance Criteria	Corrective Action
Field Blank	Water	1 per 10 field samples (10%)	TEM	No LA structures detected	Assign qualifier to analyte(s) in field samples associated with field blank (same day, same team)
			Metals, Anions, VPH, EPH, DOC	< ½ PQL for all target analytes	
Trip Blank	Water	1 per cooler of samples for VOC and VPH analyses	SW 8260 or MA-DEP-VPH (a)	< ½ PQL for all target analytes	Assign qualifier to analyte(s) in field samples associated with trip blank (same cooler)
	Solid Media		SW 8260 or MA-DEP-VPH (a)	< ½ PQL for all target analytes	
Equipment Rinsate Blank	Water	1 per sampling team per day	TEM	No LA structures detected	Assign qualifier to analyte(s) in field samples associated with field blank (same day, same team)
			Metals, Anions, VPH, EPH, DOC (SW only)	< ½ PQL for all target analytes	
	Solid Media		TEM	No LA structures detected	
			Metals, Fluoride, Phosphorus, VPH, EPH, PCBs	< ½ PQL for all target analytes	
Field Duplicate	Water	1 per 10 field samples (10%)	TEM	< 5% statistically different	Assign qualifier to analyte(s) in parent field sample
			Same analyte list as original sample	20% RPD for target analytes	
	Sediment	1 per 10 field samples (10%)	PLM-VE	*[Not applicable for field duplicates]*	*[Not applicable for field duplicates]*
			Same analyte list as original sample		
Performance Evaluation (PE)	Water	4 PE samples	Inorganic and organic analytes	(b)	Assign qualifier to field samples for analyte(s) outside of acceptance criteria
	Solid Media	4 PE samples	PLM-VE	80% concordance	
		3 PE samples	Inorganic and organic analytes	(b)	

(a) depending on analyses requested with associated samples
(b) meet analyte-specific criteria specified by QATS certification program

Table 8-2. Summary of Laboratory Quality Control Measures, by Analysis

Analytical Method[a]	QC Element	Frequency	Acceptance Criteria	Corrective Action
ICP Metals SW-846 6010B (and EPA 200.7 for aqueous samples)	Initial calibration (1 point + blank minimum)	Daily prior to analysis	Correlation coefficient (r) ≥0.995	• Recalibrate
	Interference check standard (ICS)	Beginning and end of each analytical run	Results +/- 20% of true value	• Terminate analysis • Recalibrate instrument • Reanalyze all samples back to last acceptable ICS
	Initial calibration verification (ICV)	After calibration, prior to sample analysis	Results <10% from calibration standard	• Reanalyze ICV • Recalibrate, if ICV still out
	Continuing calibration verification (CCV)	Every 10 samples and end of analytical sequence	Results < 10% from calibration standard	• Reanalyze affected samples back to the last acceptable CCV
	Calibration blank - Initial calibration blank (ICB), Continuing calibration blank (CCB)	After initial calibration verification, each subsequent calibration verification, and at the end of the run	<3x the Method detection limit (MDL)	• Reanalyze blank • Clean system • Reanalyze all samples back to last acceptable blank
	Method blank	1 per preparation batch (≤20 samples)	< ½ x Practical quantitation limit (PQL)	• Reanalyze method blank. • If fails, analyze a calibration blank • Reprep/reanalyze analytical batch as appropriate
	Matrix spike (MS)	1 per preparation batch (≤20 samples)	% Recovery +/-25% of actual value	• Assess data (4 x rule) • If LCS recoveries are within acceptance criteria, then matrix interference may be suspected • Reanalyze reprep once if matrix is not a factor • Narrate all outliers
	Matrix spike duplicate (MSD)	1 per preparation batch (≤20 samples)	RPD <20%	• Same as MS
	Laboratory Control Sample (LCS)	1 per preparation batch (≤20 samples)	% Recovery +/- 20% of actual value	• Reanalyze LCS • Reprep/reanalyze LCS and affected samples • Narrate all outliers
ICP-MS Metals SW-846 6020 (and EPA 200.8 for aqueous samples)	Mass calibration and resolution check (4 replicates)	Daily prior to analysis	Mass calibration < 0.1 amu; resolution <0.9 amu at 10% peak height; RSD <5%	• Recalibrate
	Initial multipoint calibration (1 point + blank minimum); average of 3 integrations	Daily prior to analysis	None	• None
	Initial calibration verification (ICV); mid-level standard second source	After calibration, prior to sample analysis	± 10% from true value	• Reanalyze ICV • Recalibrate, if ICV still out
	Continuing calibration verification (CCV)	Every 10 samples and end of run sequence	± 10% from true value	• Reanalyze affected samples back to the last acceptable CCV

Analytical Method[a]	QC Element	Frequency	Acceptance Criteria	Corrective Action
ICP-MS Metals SW-846 6020 (and EPA 200.8 for aqueous samples)	Interference check solution	At beginning of analytical sequence or once every 12 hours, whichever is more frequent	Recoveries +/- 20% of theoretical value	• Internal QC review only; flag data to indicate interference
	Internal Standards	Every CCV, ICB/CCB	Recoveries +/- 20% of initial calibration	• Recalibrate and verify calibration • Reanalyze affected samples
		Every sample	Recoveries 30-120% for samples	• Dilute sample 5x and reanalyze • Repeat until within limits
	Calibration blank Initial calibration blank (ICB) Continuing calibration blank (CCB)	After initial calibration and each subsequent calibration verification	< 3 x Method detection limit (MDL)	• Reanalyze blank • Clean system if still out • Reanalyze affected samples back to the last acceptable CCB
	Method blank	1 per preparation batch (≤ 20 samples)	< ½ x PQL	• Reanalyze method blank. • If fails, analyze a calibration blank • Reprep/reanalyze analytical batch as appropriate
	Matrix spike (MS)	1 per preparation batch (≤ 20 samples)	% Recovery +/- 25% of true value	• Assess data • Reanalyze MS if matrix is not a factor
	Matrix spike duplicate (MSD) or Matrix duplicate (MD)	1 per preparation batch (≤ 20 samples)	RPD < 20% (for values > 100 x MDL)	• Same as MS
	Post-digestion spike addition	As necessary to assess matrix interference	% Recovery +/- 25% of actual value	• Perform dilution test • Or, perform method of standard addition
	Dilution test	1 per 20 samples	% Recovery +/- 10% of true value	• Use method of standards addition
	Laboratory control sample (LCS)	1 per preparation batch (≤ 0 samples)	%Recovery within +/- 20% of true value	• Reanalyze LCS • Reprep/reanalyze LCS and affected samples • Narrate all outliers
Mercury SW-846 7470A/7471A	Initial multipoint calibration (3 point + blank minimum)	Daily, prior to analysis	Correlation coefficient (r) ≤0.995	• Recalibrate
	Initial calibration verification (ICV); mid-level standard	After calibration, prior to sample analysis	± 20% of true value	• Reanalyze ICV • Rerun initial calibration
	Continuing calibration verification (CCV); mid-level standard	Every 10 samples and at end of analytical sequence	± 20% of true value	• Reanalyze affected samples back to last acceptable CCV
	Calibration blank (ICB/CCB)	After calibration, and after each subsequent calibration verification	< ½ x PQL	• Reanalyze blank • Clean system if still out • Reanalyze affected samples back to last acceptable CCB
	Method blank	1 per preparation batch (≤20 samples)	< ½ x PQL	• Reanalyze method blank. • If fails, analyze a calibration blank • Reprep/reanalyze analytical batch as appropriate

Analytical Method[a]	QC Element	Frequency	Acceptance Criteria	Corrective Action
Mercury SW-846 7470A/7471A	Matrix spike (MS)	1 per preparation batch (≤20 samples)	% Recovery +/- 25% of true value	• If LCS recoveries are within acceptance criteria, matrix interference may be suspected • Reprep/reanalyze once if problem cannot be attributed to matrix • Narrate all outliers
	Matrix spike duplicate (MSD)	1 per preparation batch (≤20 samples)	RPD < 20%	• Same as MS
	Laboratory control samples (LCS)	1 per preparation batch (≤20 samples)	%Recovery within +/- 20% of true value	• Reanalyze LCS • Reprep/reanalyze LCS and affected samples • Narrate all outliers
SW-846, 8260B Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)	Tune instrument with a 4-bromofluorobenzene standard (BFB)	Every 12 hours	Must meet key ions and ion abundance criteria established by method.	
	Initial multi-point calibration; 5 point minimum. Lowest point at or below PQL. Includes calibration check compounds (CCC) and system performance check compounds (SPCC), and Internal Standards Compounds (IS).	Prior to analysis, and as required	RSD< 30 % for CCC; Average RF ≥ 0.1 for SPCC (≥0.3 for chlorobenzene, 1,1,2,2-Tetrachloroethane) If % RSD < 15% average RF may be used; linear calibration required	• Evaluate system • Repeat calibration
	Continuing calibration verification (CCV): CCC, SPCC, and IS	Every 12 hours	Percent difference <20% for CCC; RF ≥0.1 for SPCC (≥0.3 for chlorobenzene and 1,1,2,2-Tetrachloroethane).	• Evaluate system/standard • Reanalyze calibration check standard • Repeat initial calibration •
	IS	Every sample, method blank, LCS, MS/MSD	Retention time for each internal standard must be within 30 seconds of most recent CCV and the EICP area for all internal standards must be within -50% to +100% of the most recent CCV.	• Evaluate system • Reanalyze sample once • Re-extract/reanalyze sample once • If due to media interference report both sets of data • Narrate all outliers
	Method blank	1 per preparation batch (≤20 samples)	< ½ x PQL	• Reanalyze method blank • Reanalyze batch
	Internal standards	Every sample, method blank, LCS, and MS/MSD	Retention time for each internal standard must be within 30 seconds of most recent CCV and the EICP area for all internal standards must be within -50% to +100% of the most recent CCV	• Evaluate system/standard • Reanalyze samples • If still out, report both sets of data • Narrate all outliers

Analytical Method[a]	QC Element	Frequency	Acceptance Criteria	Corrective Action
SW-846, 8260B Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)	Surrogate spike	Every sample, method blank, LCS, MS/MSD	No more than one surrogate outside QC acceptance criteria. No surrogate below 10% recovery.	• Reanalyze sample once • Re-extract and reanalyze if >1 surrogate outside QC acceptance limits • If still out, report both sets of data • Narrate all outliers
	Matrix spike (MS)	1 per preparation batch (≤20 samples)	Percent recovery within QC acceptance criteria	• Assess data (4x rule) • If LCS and surrogate recoveries are within acceptance criteria matrix interferences may be suspected • Reprep/reanalyze once if matrix is not a factor • Narrate all outliers
	Matrix spike duplicate (MSD) or Matrix Duplicate (MD)	1 per preparation batch (≤20 samples)	% Recovery and/or RPD within QC acceptance criteria	• Same as MS
	Laboratory control sample (LCS)	1 per preparation batch (≤20 samples)	% Recovery within QC acceptance criteria	• Reanalyze LCS • Reprep/reanalyze LCS and all associated samples • Narrate all outliers
SW-846 8270C Semi-Volatiles by GC/MS	Tune the instrument using a decafluorotriphenylphosine (DFTPP) standard	Every 12 hours	Must meet the ion abundance criteria specified in the Degradation of DDT ≤ 20% Benzidine and PCP present at normal response without excessive tailing	• Retune instrument • Repeat standard analysis • Perform injection port, column maintenance as necessary
	Initial calibration (5 point minimum); includes Calibration Check Compounds (CCC), System Performance Calibration Check (SPCC), and Internal Standard Compounds (IS)	Prior to analysis and as required	% RSD for CCC ≤30%; average RF ≥0.05 for SPCC *If % RSD ≤15 % average RF may be used; linear calibration required*	• Evaluate the system • Repeat calibration
	Continuing calibration verification (CCV); includes CCC, SPCC, and IS	Every 12 hours	CCV percent difference for CCC ≤30%; RF ≥0.05 for SPCC EICP area of each internal standard - 50% to +100% of all IS areas in most recent CCV. Retention time for each internal standard must be within 30 seconds of most recent CCV	• Evaluate system/standard • Reanalyze calibration check standard • Repeat the initial calibration as necessary
	Method blank	1 per preparation batch (≤20 samples)	< ½ x PQL	• Reanalyze blank • Reprep/reanalyze blank and all associated samples

Analytical Method[a]	QC Element	Frequency	Acceptance Criteria	Corrective Action
SW-846 8270C Semi-Volatiles by GC/MS	Internal Standard	Every sample, method blank, LCS and MS/MSD	The EICP area for all internal standards must be within -50% and +100% of most recent CCV Retention time for each internal standard must be within 30 seconds of most recent CCV	• Evaluate system/standard • Reanalyze the sample • If still out, report both sets of data
	Surrogate spike	Every sample, method blank, LCS and MS/MSD	No more than one surrogate per fraction outside of acceptance criteria (Refer to Table B1-a) No surrogate below 10% recovery	• Reanalyze sample once • Re-extract and reanalyze if >1 surrogate per fraction outside acceptance limits • Narrate all outliers
	Matrix spike (MS)	1 per preparation batch (≤20 samples)	% Recovery within QC acceptance criteria	• Assess data (4x rule) • Reanalyze once; if matrix is not a factor • If LCS and surrogate recoveries are within acceptance criteria matrix interference maybe suspected • Narrate all outliers
	Matrix spike duplicate (MSD) or Matrix Duplicate (MD)	1 per preparation batch (≤20 samples)	% Recovery and/or RPD within QC acceptance criteria	• Same as MS
	Laboratory control sample	1 per preparation batch (≤20 samples)	% Recovery within project QC acceptance criteria for all spiked analytes	• Reanalyze LCS • Re-prep/reanalyze LCS and all associated samples • Narrate all outliers
SW-846 8082 Polychlorinated biphenyls (PCBs) by Gas Chromatography	Initial calibration (5 point minimum) Lowest standard at or below PQL; Expected Aroclors or Aroclor 1016/1260 five-point if unknown with single-point mid-level standards for other Aroclors for pattern recognition and retention times, or	Prior to analysis and as required	RSD <20%, average calibration factor or response factor(a) may be used; linear calibration required	• Evaluate the system • Repeat initial calibration
	Initial calibration verification (ICV) Mid level standard Expected Aroclors or Aroclor 1016/1260 if unknown	Prior to each 12 hour shift	% Difference ≤15% of expected concentration compared to response from ICAL	• Evaluate system/standard • Reanalyze ICV standard • Repeat initial calibration
	Continuing calibration verification (CCV) Mid level standard Expected Aroclors or Aroclor 1016/1260 if unknown	After every 20 samples and at the end of the analytical sequence	% Difference ≤15% of expected concentration compared to response from ICAL for each bracketing standard	• Evaluate system/standard • Reanalyze CCV and samples back to last acceptable CCV
	Retention time windows	Established with each new column installation Updated with each daily initial calibration standard	Retention times must be within retention time window established by the daily initial calibration standard Every CCV and every sample	• Evaluate system/standard; pattern recognition may be sufficient • Reanalyze CCV/affected samples

Analytical Method[a]	QC Element	Frequency	Acceptance Criteria	Corrective Action
SW-846 8082 Polychlorinated biphenyls (PCBs) by Gas Chromatography	Method Blank	1 per preparation batch (≤20 samples)	< ½ x PQL	• Reanalyze blank • Re-prep/reanalyze blank and associated samples
	Surrogate spike DCB (for Aroclors) TCMX (for PCB congeners)	Every sample, method blank, LCS and MS/MSD	% Recovery within QC acceptance criteria	• Re-extract/reanalyze once • If still out, report both sets of data Narrate all outliers
	Matrix spike (MS)	1 per preparation batch (≤20 samples)	% Recovery within QC acceptance criteria	• Assess data (4x rule) • If LCS and surrogate recoveries are within acceptance criteria matrix interference maybe suspected • Re-extract/reanalyze if matrix is not a factor • Narrate all outliers
	Matrix spike duplicate(MSD) or Matrix duplicate (MD)	1 per preparation batch (≤20 samples)	% Recovery and/or RPD within QC acceptance criteria	• Same as MS
	Laboratory control sample(LCS)	1 per preparation batch (≤20 samples)	% Recovery within project QC acceptance criteria	• Reanalyze LCS • Re-prep/reanalyze LCS and all associated samples • Narrate all outliers
SW-846 8081A Organochlorine Pesticides by Gas Chromatography	Column Evaluation Mix	Prior to analysis. both initial and daily	Degradation of DDT and Endrin < 15%	• Evaluate the system • Repeat standard
	Initial calibration (5 point minimum) Lowest at or below PQL Mid level multi-component standards for pattern recognition and retention times	Prior to analysis and as required	RSD < 20%, average CF may be used; linear calibration required	• Average RSD <20% across all analytes may be used if any analyte fails • Evaluate the system • Repeat initial calibration
	Initial calibration verification (ICV) Mid level standard Expected multi-component compounds	Prior to each 12 hour shift	% Difference ≤15% of expected concentration compared to response from ICAL	• Average % difference ≤15% across all analytes may be used if any analyte fails • Evaluate system/standard • Reanalyze ICV standard • Repeat initial calibration
	Continuing calibration verification (CCV) Mid level standard Expected multi-component compounds	After every 20 samples and at the end of the analytical sequence	% Difference ≤15% of expected concentration compared to response from ICAL for each bracketing standard	• Average % difference ≤15% across all analytes may be used if any analyte fails • Evaluate system/standard • Reanalyze CCV and affected samples • For CCV with response > initial calibration response and % difference >15%, samples need not be reanalyzed if no target compounds are detected

Analytical Method[a]	QC Element	Frequency	Acceptance Criteria	Corrective Action
SW-846 8081 Organochlorine Pesticides by Gas Chromatography	Retention time windows	Established with each new column installation Updated with each daily initial calibration standard	Retention times must be within retention time window established by the daily initial calibration standard Every CCV and every sample	• Evaluate system/standard; pattern recognition may be sufficient for multi-component compounds only • Reanalyze CCV/affected samples
	Method Blank	1 per preparation batch (≤ 20 samples)	< ½ x PQL	• Reanalyze blank • Re-prep/reanalyze blank and associated samples
	Surrogate spike DCB and TCMX	Every sample, method blank, LCS and MS/MSD	% Recovery within QC acceptance criteria. One surrogate must fall within established control limits	• Re-extract/reanalyze once • If still out, report both sets of data • Narrate all outliers
	Matrix spike (MS)	1 per preparation batch (≤20 samples)	% Recovery within QC acceptance criteria	• Assess data (4 x rule) • If LCS and surrogate recoveries are within acceptance criteria, matrix interference maybe suspected • Re-extract/reanalyze once if matrix is not a factor • Narrate all outliers
	Matrix spike duplicate(MSD) or Matrix Duplicate (MD)	1 per preparation batch (≤20 samples)	% Recovery and/or RPD within QC acceptance criteria.	• Same as MS
	Laboratory control sample (LCS)	1 per preparation batch (≤ 20 samples)	% Recovery within QC acceptance criteria	• Reanalyze LCS • Re-prep/reanalyze LCS and all associated samples • Narrate all outliers
SW-846 8141A Organphosphorus Pesticides by Gas Chromatography	Initial calibration (5 point minimum) Lowest at or below ½ x PQL	Prior to analysis and as required	If %RSD < 20% average RF may be used If linear regression used r > 0.995 or R2 >0.990 Alternate evaluation: Mean % RSD for all target analytes <20% with no individual compound >40%	• Average RSD <20% across all analytes may be used if any analyte fails • Evaluate the system • Repeat initial calibration
	Initial calibration verification (ICV), second source Mid level standard	Prior to every analytical sequence	% Difference ≤15% of expected concentration compared to response from ICAL	• Evaluate system/standard • Reanalyze ICV standard • Repeat initial calibration
	Continuing verification standard (CVS) Mid level standard	After every 10 samples and at the end of the analytical sequence	%D or % Drift >15%	• Evaluate system/standard • Repeat sample analysis to last acceptable CVS
	Retention time windows	Established with each new column installation Updated with each daily initial calibration standard	Retention times must be within retention time window established by the daily initial calibration standard Every CVS and every sample	• Evaluate system/standard; pattern recognition may be sufficient for multi-component compounds only • Reanalyze CVS/affected samples
	Target analyte confirmation	All detected analytes	RPD < 40%	• If greater than 40% qualify data as estimated

Analytical Method[a]	QC Element	Frequency	Acceptance Criteria	Corrective Action
SW-846 8141A Organphosphorus Pesticides by Gas	Method Blank	1 per preparation batch (≤ 20 samples)	< ½ x PQL	• Reanalyze blank • Reprep/reanalyze blank and associated samples
	Surrogate spike	Every sample, method blank, LCS and MS/MSD	% Recovery within QC acceptance criteria	• Reanalyze • Reprep/reanalyze once • If still out, report both sets of data • Narrate all outliers
	Matrix spike (MS)	1 per preparation batch (≤20 samples)	% Recovery within QC acceptance criteria	• Reanalyze • Reprep/reanalyze once • If still out, report both sets of data • Narrate all outliers
	Matrix spike duplicate(MSD)	1 per preparation batch (≤20 samples)	% Recovery and/or RPD within QC acceptance criteria	• Same as MS
	Laboratory control sample (LCS)	1 per preparation batch (≤ 20 samples)	% Recovery within QC acceptance criteria	• Reanalyze LCS • Reprep/reanalyze LCS and all associated samples • Narrate all outliers
SW-846 8151A Organochlorine Herbicides and Pentachlorophenol by Gas Chromatography	Initial calibration (5 point minimum) Lowest point at or below PQL	Prior to analysis and as required	%RSD <20%, average CF may be used; linear calibration required	• Average RSD <20% across all analytes may be used if any analytes fail • Evaluate the system • Repeat initial calibration
	Initial calibration verification (ICV) second source Mid level standard	Prior to each daily analytical sequence	% Difference ≤15% of expected concentration compared to response from ICAL	• Average %D ≤15% across all analytes may be used if any analytes fail • Evaluate system/standard • Reanalyze ICV standard • Repeat initial calibration
	Continuing calibration verification (CCV) Mid level standard	After every 20 samples and at the end of the analytical sequence	% Difference ≤15% of expected concentration compared to response from ICAL for each bracketing standard	• Evaluate system/standard • Reanalyze CCV and all samples back to last acceptable CCV
	Retention time windows	Established with each new column installation Updated with each daily initial calibration standard	Retention times must be within retention time window established by the daily initial calibration standard Every CCV and every sample	• Evaluate system/standard; • Reanalyze CCV and affected samples
	Method blank	1 per preparation batch (≤20 samples)	< ½ x PQL	• Reanalyze blank • Re-prep/reanalyze blank and all associated samples
	Surrogate spike DCAA	Every sample, method blank, LCS and MS/MSD	% Recovery within project QC acceptance criteria	• Re-extract/reanalyze once • If still out, report both sets of data • Narrate all outliers

Analytical Method[a]	QC Element	Frequency	Acceptance Criteria	Corrective Action
SW-846 8151A Organochlorine Herbicides and Pentachlorophenol by Gas Chromatography	Matrix spike (MS)	1 per preparation batch (≤20 samples)	% Recovery within QC acceptance criteria	• Assess data (4x rule) • If LCS and surrogate recoveries are within acceptance criteria, matrix interference maybe suspected • Re-exact/reanalyze once if matrix is not a factor • Narrate all outliers
	Matrix spike duplicate (MSD) or Matrix duplicate (MD)	1 per preparation batch (≤20 samples)	% Recovery and/or RPD within QC acceptance criteria	• Same as MS
	Laboratory control sample (LCS)	1 per preparation batch (≤20 samples)	% Recovery within QC acceptance criteria	• Reanalyze LCS • Re-prep/reanalyze LCS and all associated samples • Narrate all outliers
Total Cyanide SW-846 9012B	Initial calibration curve (six standards and a calibration blank)	Initial daily calibration prior to sample analysis	Correlation coefficient ≥0.995 for linear regression	• Correct problem then repeat initial calibration
	Distilled standards (one high and one low)	Once per initial calibration	Cyanide within ±10% of true value	• Correct problem then repeat distilled standards
	Second-source calibration verification	One per preparation batch (<20 samples)	Cyanide within ±15% of expected value	• Correct problem then repeat initial calibration
	Method blank	One per analytical batch	< ½ x PQL	• Correct problem then reprep and analyze method blank and all samples processed with the contaminated blank
	LCS for all analytes	One per preparation batch (<20 samples)	QC acceptance criteria	• Correct problem then reanalyze the affected batch • If still out, reprep and reanalyze the LCS and all samples in the affected batch
	MS/MSD	One per preparation batch (<20 samples)	QC acceptance criteria	• None
Gross Alpha and Gross Beta SW-846-9310	Initial calibration with standard reference materials	Daily before sample analysis	Analytical method control limits	• Correct problem and repeat calibration
	Method Blank	One per analytical batch	< ½ x PQL	• Identify and reduce contamination then reanalyze
	Analytical Duplicate	One per analytical batch	RPD < 20	• Evaluated problem and correct the reanalyze
	Spiked Sample or standard reference material	One per analytical batch	80-120% recovery	• Evaluated problem and correct the reanalyze

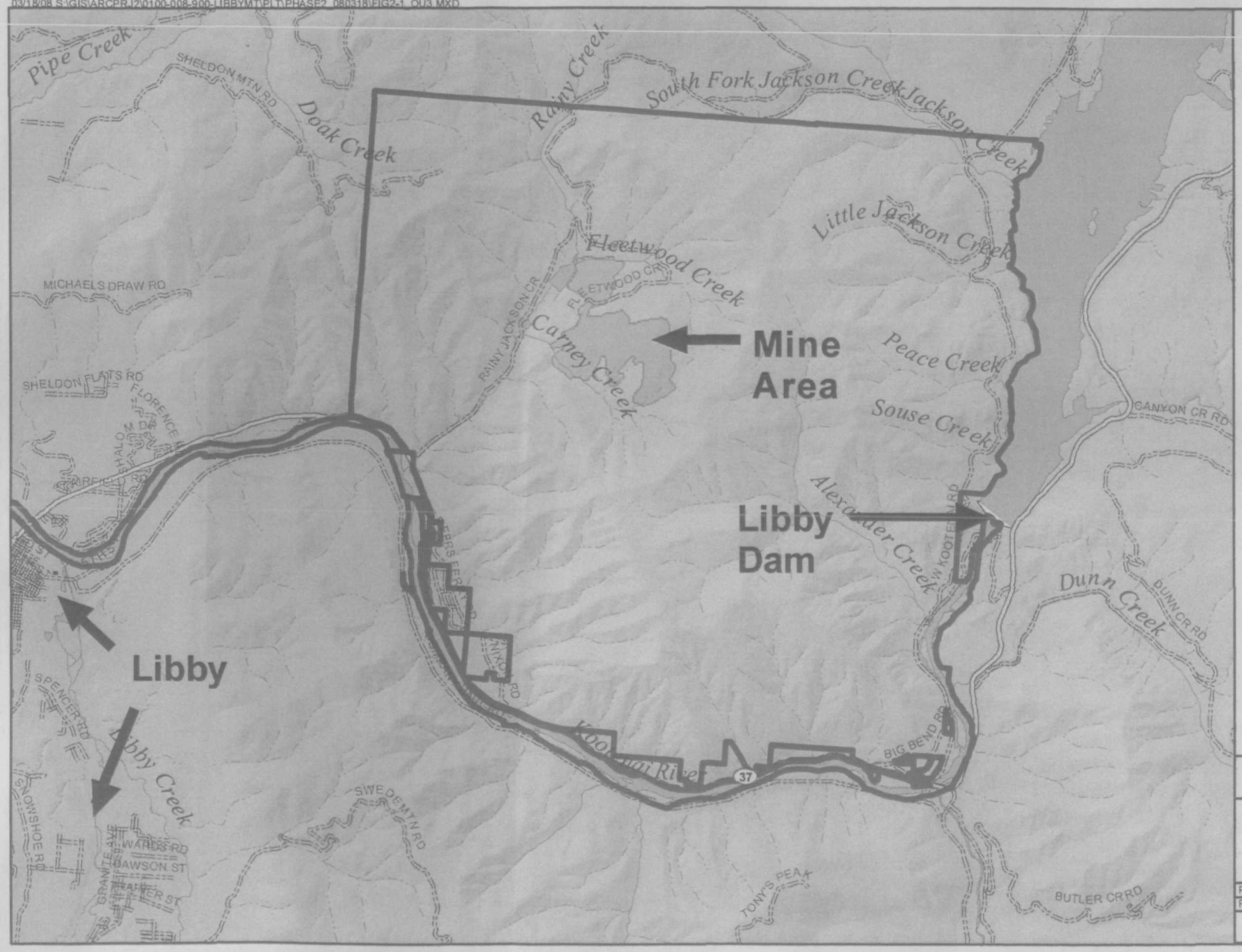
EICP Extracted ion current profile
QC Quality control
RF Response factor
RSD Relative standard deviation

Color Map(s)

The following pages contain color that does not appear in the scanned images.

To view the actual images, contact the Region VIII Records Center at (303) 312-6473.

03/18/08 S:\GIS\ARCPRJ2\0100-008-900-LIBBYMT\PLT\PHASE2_080318\FIG2-1_OU3.MXD



Legend

- Operable Unit 3
- County Road
- Primary Road
- Open Water
- Perennial Stream
- Intermittent Stream
- Mined Area

N

0 1 2

Miles

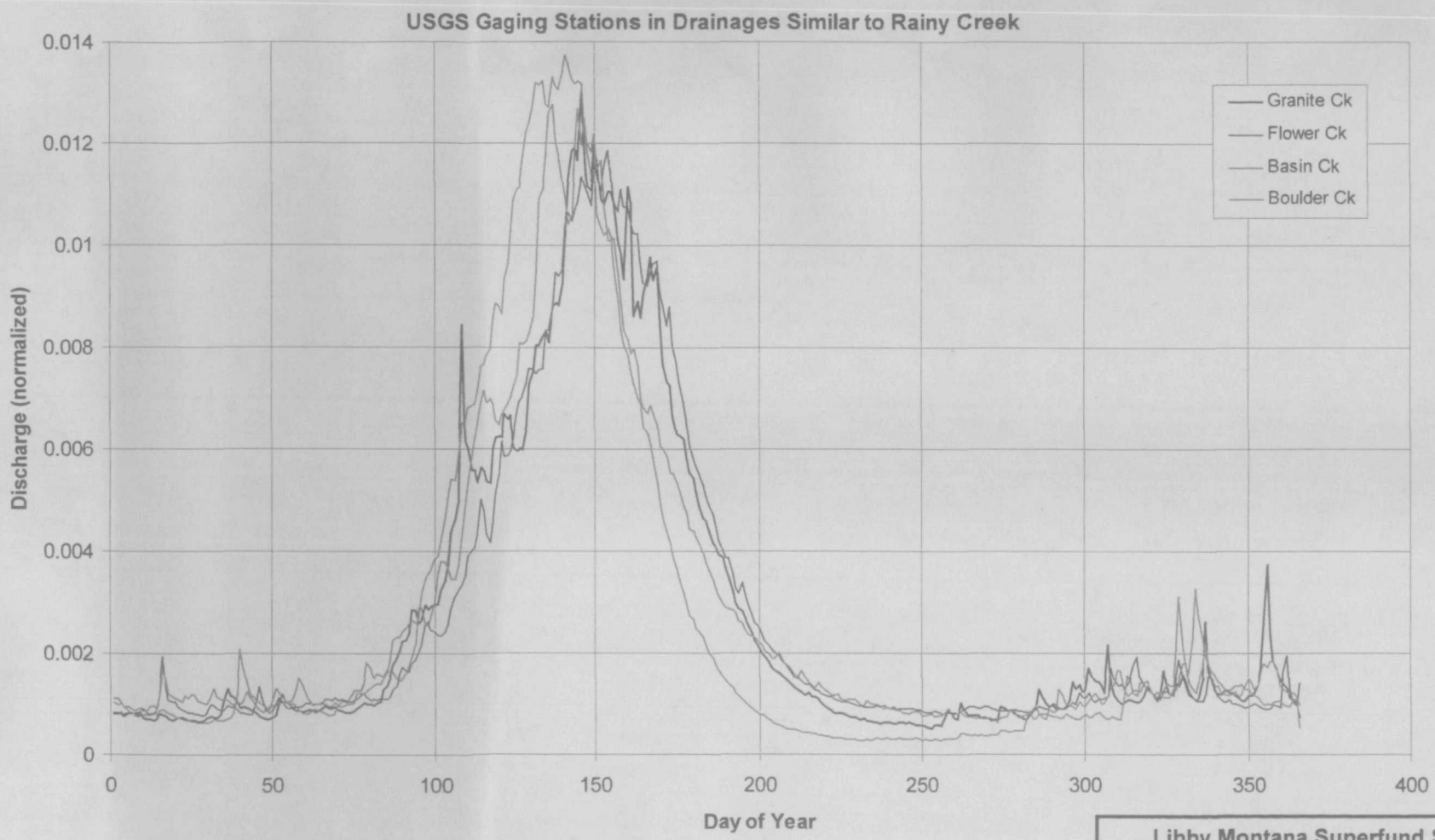
Libby Montana Superfund Site
Operable Unit 3

FIGURE 2-1

Operable Unit 3

PROJECT: 0100-008-900	MAR 18, 2008	
REV: 0	BY: VFS	CHECKED: ACK

S:\Jobs\0100-008-900-USEPA-LibbySFSOU3\Phase 2 Surface Water Sampling\Watersheds\CalDay.ppt



Notes:
Normalized discharge represents the average daily discharge divided by the average annual discharge.

12305500 – Boulder Creek (POR: 1928 – 1977)
12303100 – Flower Creek (POR: 1960 – 1992)
12302500 – Granite Creek (POR: 1933 – 1969)
12304040 – Basin Creek (POR: 1990 – 2000)

Libby Montana Superfund Site
OPERABLE UNIT 3

FIGURE 2-2
SPRING RUNOFF HYDROGRAPHS

PRJ: 0100-008-900	DATE: February 20, 2008	
REV: 1	BY: JPJ	CHK: KJT

NEWFIELDS

02/21/08 S:\GIS\ARCPRJ2\0100-008-900-LIBBYMT\PLT\PHASE2_080124\FIG3-1_SURFWATSED.MXD

Legend

- County Road
- Primary Road
- Open Water
- Perennial Stream
- Intermittent Stream
- Surface Water/Sediment Sampling Location

N

0 2,000 4,000

Feet

LIBBY MONTANA SUPERFUND SITE

OPERABLE UNIT 3

FIGURE 3-1

SURFACE WATER / SEDIMENT SAMPLING LOCATIONS

PROJECT: 0100-008-900	FEB 21, 2008	
REV: 0	BY: CRL	CHECKED: ACK

NEW FIELDS

02/21/08 S:\GIS\ARCPRJ2\0100-008-900-LIBBYMT\PLT\PHASE2_080124\FIG3-2_ASBESTOS_SW.MXD

Legend

- County Road
- Primary Road
- Open Water
- Perennial Stream
- Intermittent Stream

Asbestos in Surface Water (Total LA)

- Non Detect
- > ND - 1 MFL
- 1 - 10 MFL
- 10 - 100 MFL
- > 100 MFL

N

0 2,000 4,000

Feet

LIBBY MONTANA SUPERFUND SITE

OPERABLE UNIT 3

FIGURE 3-2

ASBESTOS LEVELS IN SURFACE WATER

PROJECT: 0100-008-900	FEB 21, 2008	
REV: 0	BY: VFS	CHECKED: KJT

NEW FIELDS

02/14/08 F:\0100-008-900-LIBBYMT\PLT\PHASE2_080124\FIG3-3_ASBESTOS_SED.MXD

Legend

- County Road
- Primary Road
- Open Water
- Perennial Stream
- Intermittent Stream

Asbestos Levels in Sediment (PLM-VE)

- ND
- B1 (TR)
- B2 (<1%)
- C (2 - 6%)

N

0 2,000 4,000

Feet

LIBBY MONTANA SUPERFUND SITE

OPERABLE UNIT 3

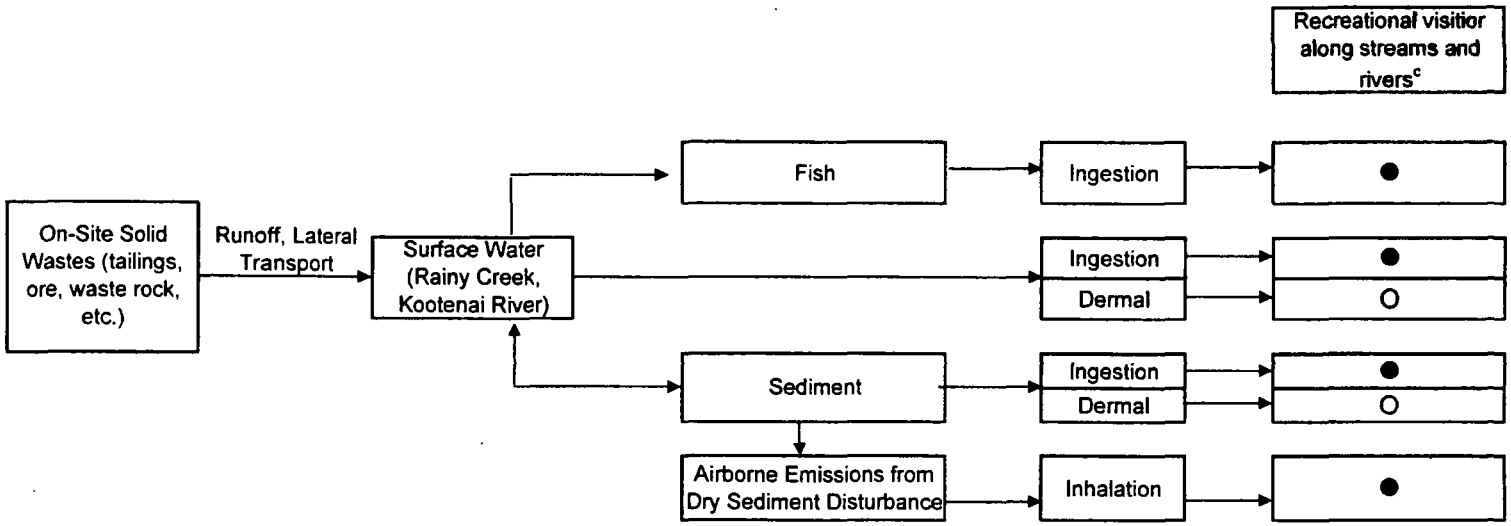
FIGURE 3-3

ASBESTOS LEVELS IN SEDIMENT

PROJECT: 0100-008-900	FEB 14, 2008	
REV: 0	BY: VFS	CHECKED: KJT

NEWFIELDS

Figure 4-1. Site Conceptual Model for Human Exposure to Surface Water and Sediment



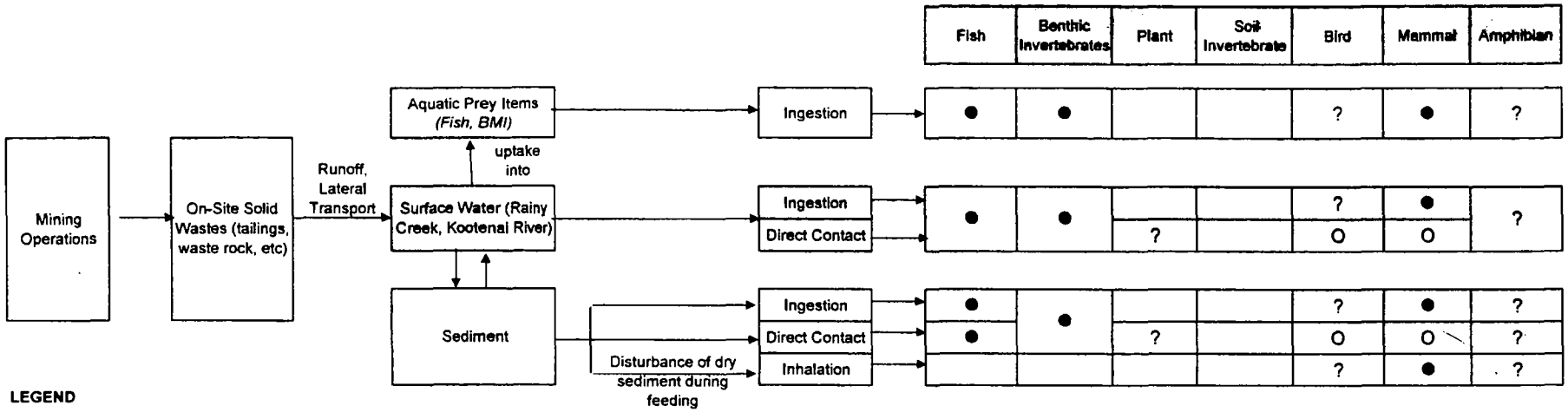
LEGEND

●	Pathway is complete and exposure may be significant; quantitative evaluation is warranted
O	Pathway is complete but is believed to be minor in comparison to other pathways; qualitative evaluation is warranted
	Pathway is incomplete or believed to be negligible; further evaluation is not warranted

NOTES:

a. Recreational visitors in forrest areas may include a range of activities, such as camping, hiking, dirt bike or ATV riding, hunting, etc.
b. Woodcutting may include exposures of area residents gathering wood for personal use as well as commercial logging activities
c. Recreational visitors along streams and rivers may include a range of activities such as hiking, fishing and wading/swimming

Figure 4-2. Conceptual Site Model for Ecological Exposure to Surface Water and Sediment

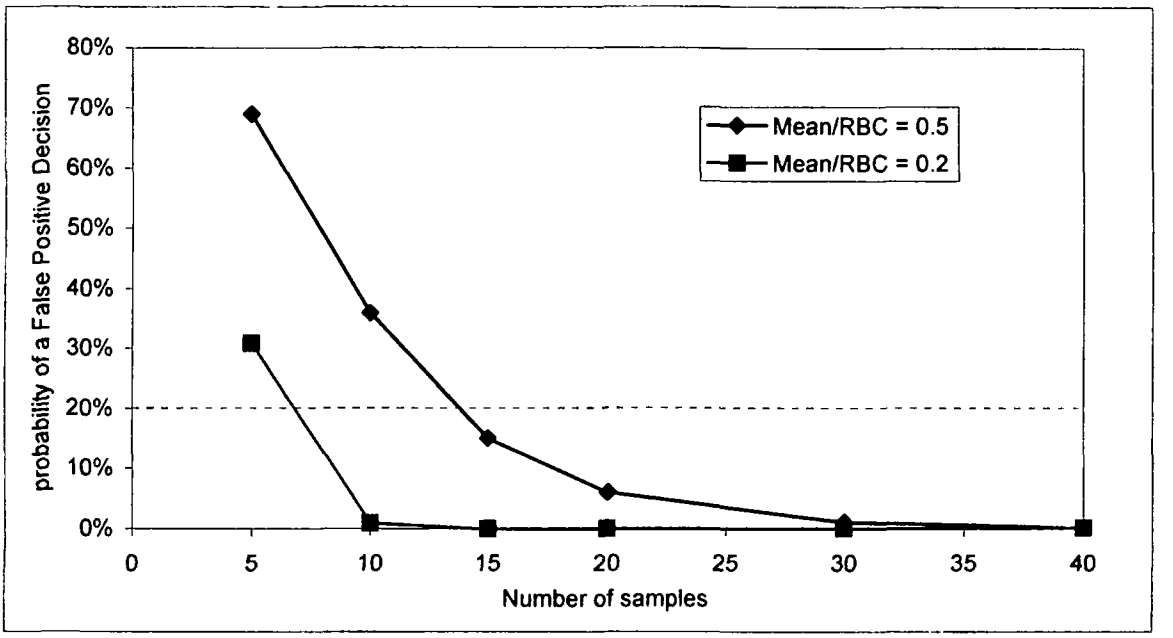


LEGEND

●	Pathway is believed to be complete, and which may provide an important contribution to the total risk to the receptor. This pathway will be evaluated using one or more lines of evidence.
O	Pathway is believed to be complete but is unlikely to be a major contributor to the total risk to the receptor (in comparison to one or more other pathways that are evaluated). This pathway will not be evaluated quantitatively.
	Pathway is incomplete or believed to be negligible; further evaluation is not warranted
?	Pathway that is or might be complete, but current data are not adequate to decide if it is a significant contributor to the total risk of the receptor.

FIGURE 4-3. EFFECT OF SAMPLE SIZE FOR NON-ASBESTOS ANALYTES ON FALSE-POSITIVE DECISION ERROR RATE

Panel A: GSD = 2



Panel B: GSD = 3

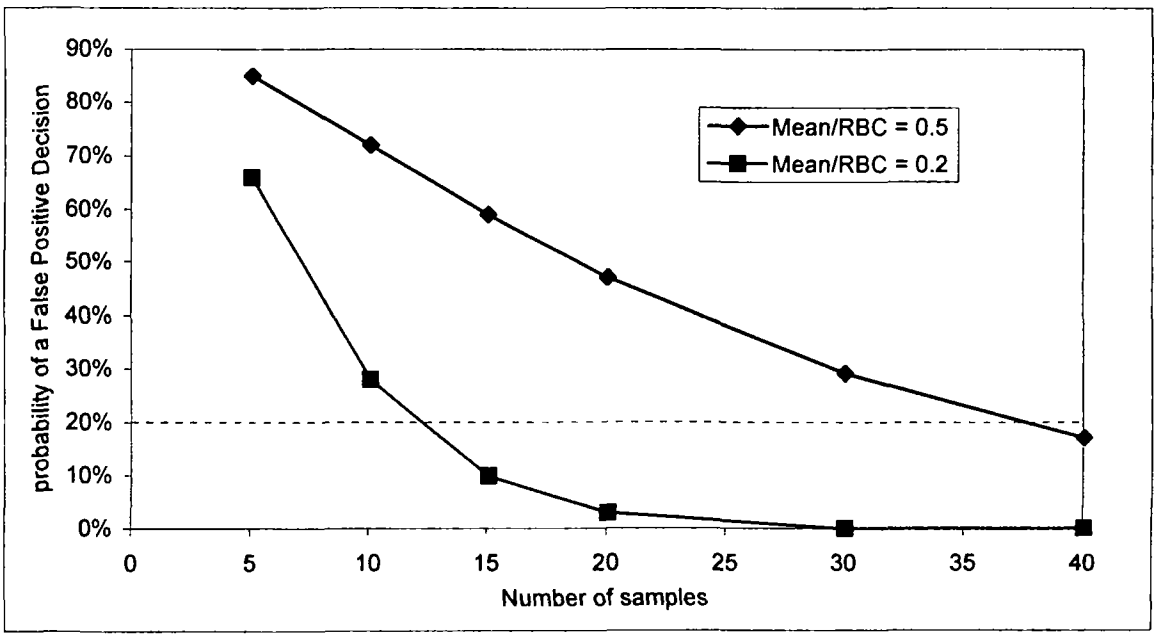


Fig 4-3.xls

FIGURE 4-4. EFFECT OF SAMPLE SIZE ON
VARIABILITY IN OBSERVED MEAN OF ASBESTOS CONCENTRATION VALUES

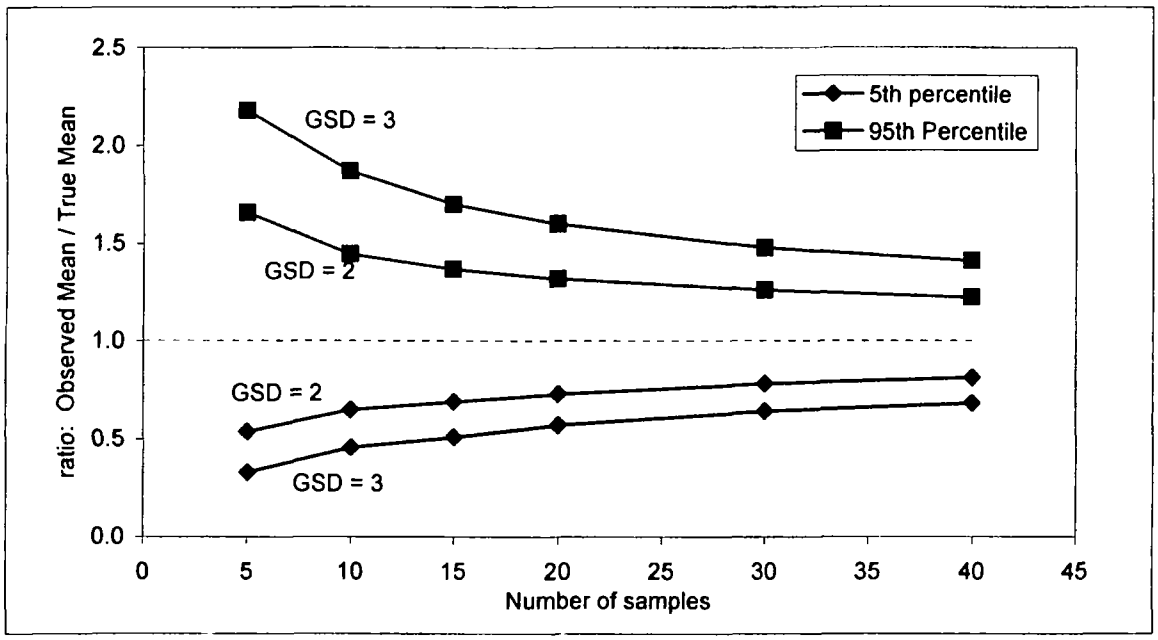
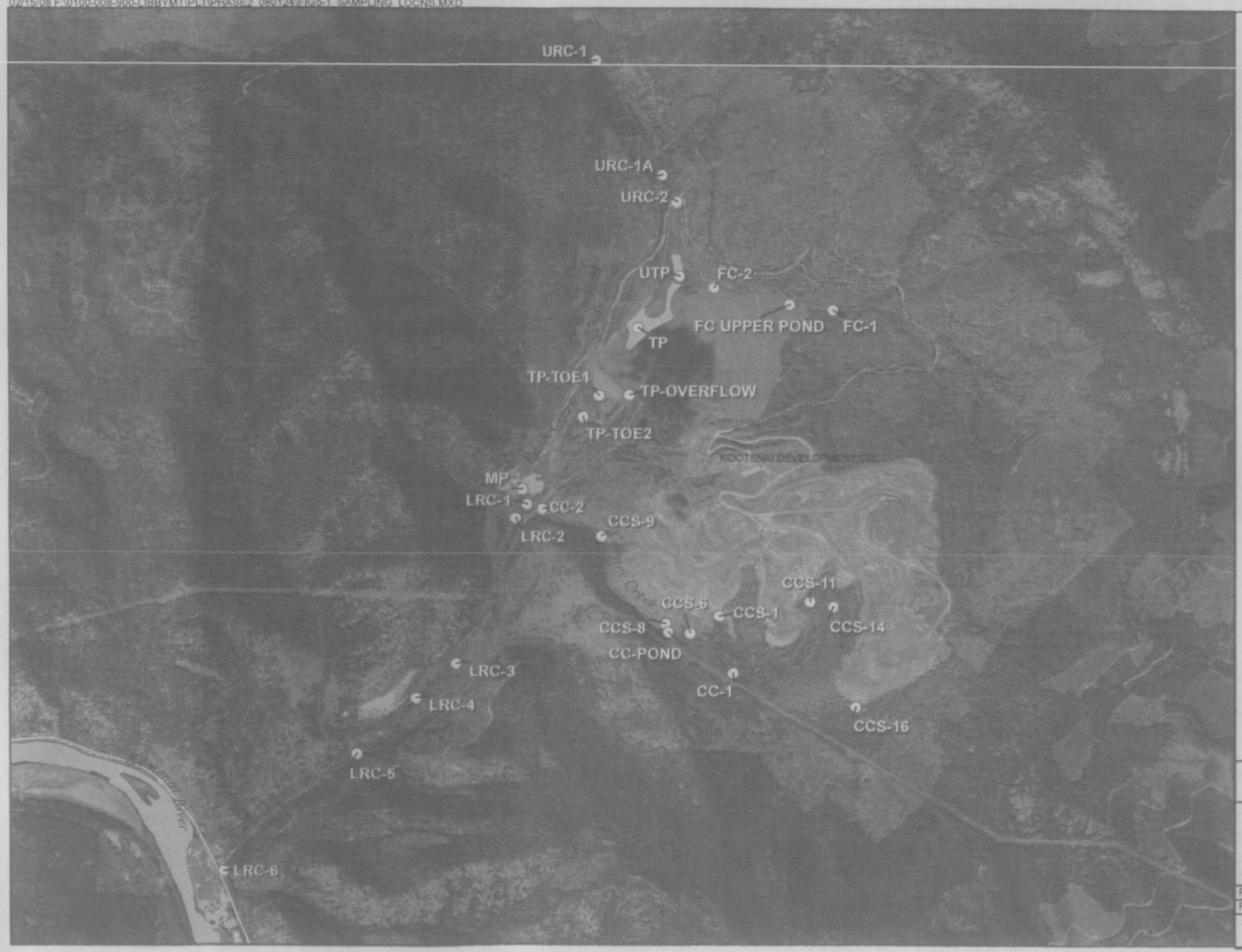


Fig 4-4.xls

02/15/08 F:\0100-008-900-LIBBYMT\PLT\PHASE2_080124\FIG5-1_SAMPLING_LOCNS.MXD



Legend

- Surface Water/Sediment Sampling Location
- County Road
- Primary Road
- Open Water
- Perennial Stream
- Intermittent Stream

N

0 2,000 4,000

Feet

LIBBY MONTANA SUPERFUND SITE
OPERABLE UNIT 3

FIGURE 5-1

PHASE IIA: SAMPLING LOCATIONS IN RAINY CREEK WATERSHED ELEMENT 1

PROJECT: 0100-008-900	JAN. 29, 2008	
REV: 0	BY: VFS	CHECKED: ACK

NEWFIELDS

02/21/08 S:\GIS\arcprj2\0100-008-900-LibbyMT\plt\Phase2_080124\Fig5-2_TailPondSampPlan.mxd

Legend

- Open Water
- Water Diversion
- Perennial Stream
- Intermittent Stream
- Mined Area
- Tailings Impoundment
- Sample Location

N

Feet

0 800 1,600

LIBBY MONTANA SUPERFUND SITE

OPERABLE UNIT 3

FIGURE 5-2

SEDIMENT SAMPLING PLAN FOR IMPOUNDMENT, RAINY CREEK WATERSHED - ELEMENT 1

PRJ: 0100-008-900	Feb 21, 2008	
REV: 0	BY: CRL	CHECKED: KJT

NEWFIELDS

29-JAN-2008 S:\GIS\ARCPRJ2\0100-008-900-LIBBYMT\PLT\PHASE2_080124\FIG5-3_SURF_WAT_MON_LOC.MXD

Legend

- County Road
- Primary Road
- Open Water
- Perennial Stream
- Intermittent Stream
- Surface Water Monitoring Location

N

0 0.25 0.5

Miles

LIBBY MONTANA SUPERFUND SITE

OPERABLE UNIT 3

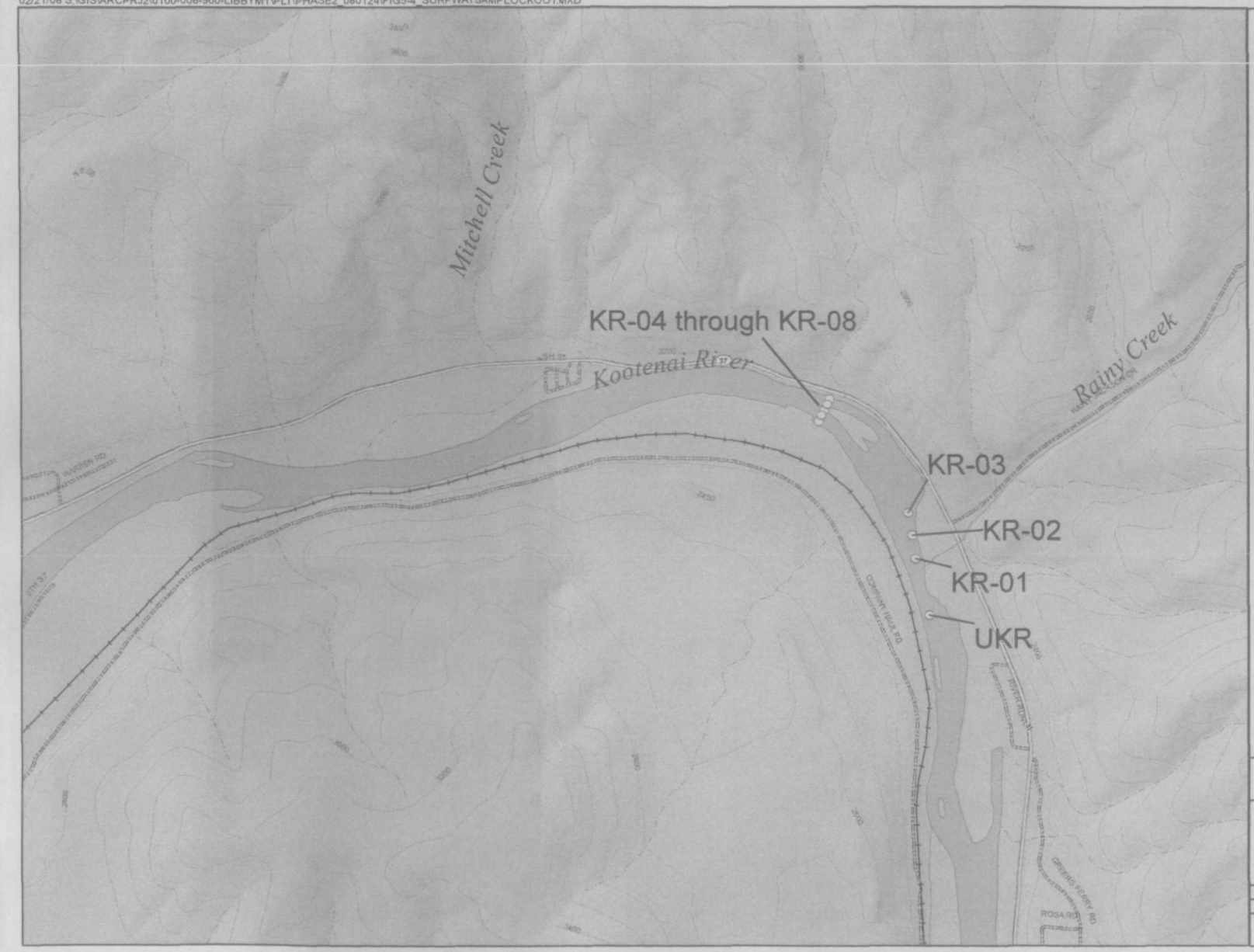
FIG. 5-3 PHASE IIA

SURFACE WATER MONITORING LOCATIONS - ELEMENT 2 RAINY CREEK WATERSHED

PROJECT: 0100-008-900	DATE: JAN. 29, 2008	
REV: 0	BY: CRL	CHECKED: ACK

NEWFIELDS

02/21/08 S:\GIS\ARCPRJ2\0100-008-900-LIBBYMT\PLT\PHASE2_080124\FIG5-4_SURFWATSAMPLOCKOOT.MXD



Legend

- Open Water
- Perennial Stream
- Intermittent Stream
- Primary Road
- County Road
- 200ft Index Contour
- Surface Water Monitoring Locations

N

Feet

0 1,500 3,000

LIBBY MONTANA SUPERFUND SITE

OPERABLE UNIT 3

FIGURE 5-4

SURFACE WATER SAMPLING LOCATIONS - KOOTENAI RIVER

PROJECT: 0100-008-900	FEB 21, 2008	
REV: 0	BY: VFS	CHECKED: ACK

NEWFIELDS

FINAL

ATTACHMENT A

DETAILED PHASE I DATA SUMMARY

TARGET SHEET

EPA REGION VIII

SUPERFUND DOCUMENT MANAGEMENT SYSTEM

DOCUMENT NUMBER: 1067268

SITE NAME: LIBBY ASBESTOS

DOCUMENT DATE: 03/20/2008

DOCUMENT NOT SCANNED

Due to one of the following reasons:

- ☐ PHOTOGRAPHS
- ☐ 3-DIMENSIONAL
- ☐ OVERSIZED
- ☑ AUDIO/VISUAL
- ☐ PERMANENTLY BOUND DOCUMENTS
- ☐ POOR LEGIBILITY
- ☐ OTHER
- ☐ NOT AVAILABLE
- ☐ TYPES OF DOCUMENTS NOT TO BE SCANNED
  (Data Packages, Data Validation, Sampling Data, CBI, Chain of Custody)

DOCUMENT DESCRIPTION:

ATTACHMENT A: FINAL PHASE II SAMPLING & ANALYSIS PLAN FOR LIBBY ASBESTOS SITE OU 3; PART A: SURFACE WATER & SEDIMENT

Contact the Superfund Records Center to view available document.
(303) 312-6473

FINAL

ATTACHMENT B

STANDARD OPERATING PROCEDURES

SOP Description	SOP ID
Surface Water Sampling	No. 3 (Rev. 0)
Surface Water Discharge Measurement	No. 4 (Rev. 0)
Sediment Sampling for Chemical Analysis	No. 5 (Rev. 0)
Equipment Decontamination	No. 7 (Rev. 0)
Sample Handling and Shipping	No. 8 (Rev. 0)
Field Documentation	No. 9 (Rev. 2)
Field Equipment Calibration	No. 10 (Rev. 1)
GPS Data Collection	No. 11 (Rev. 1)
Investigation Derived Waste (IDW) Management	No. 12 (Rev. 0)
Automated Water Sampling (a)	No. 14 (Rev. 0)
Installation, Operation, and Maintenance of Permanent Flumes	No. 15 (Rev. 0)
Surface Water Sampling Using Depth-Integrated Samplers	No. 16 (Rev 0)
Precipitation Monitoring (a)	No. 17 (Rev. 0)
Soil Sample Preparation	ISSI-LIBBY-01 (Rev. 10)
Qualitative Estimation of Asbestos in Coarse Soil by Visual Examination Using Stereomicroscopy and Polarized Light Microscopy (PLM)	SRC-LIBBY-01 (Rev. 2)
Analysis of Asbestos Fibers in Soil By Polarized Light Microscopy (PLM)	SRC-LIBBY-03 (Rev. 2)

(a) SOP to be prepared by MWH

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Libby Superfund Site Operable Unit 3 Standard Operating Procedure

Date: September 26, 2007 OU3 SOP 3 (Rev. 0)

Title: SURFACE WATER SAMPLING

APPROVALS:

TEAM MEMBER	SIGNATURE/TITLE	DATE
EPA Remedial Project Manager		9/26/07
SOP Author		9/26/07

Revision Number	Date	Reason for Revision
0	09/26/2007	--

1.0 INTRODUCTION

This standard operating procedure (SOP) is based on MWH SOP-12, Surface Water Sampling, Revision 1.0, March 2004, modified for use at the Libby Asbestos Superfund Site OU3. This SOP describes methods and equipment commonly used for collecting environmental samples of surface water for either on-site examination and chemical testing or for laboratory analysis. The information presented in this SOP is generally applicable to all environmental sampling of surface waters except where the analyte(s) may interact with the sampling equipment. The collection of concentrated sludges or hazardous waste samples from disposal or process lagoons often requires methods, precautions, and equipment different from those described herein.

This document focuses on methods and equipment that are readily available and typically applied in collecting surface water samples. It is not intended to provide an all-inclusive discussion of sample collection methods. Specific sampling problems may require the adaptation of existing equipment or design of new equipment. Such innovations shall be clearly described in the project-specific sampling plan and approved by the Project Manager and the Quality Manager.

2.0 HEALTH AND SAFETY WARNING

All personnel engaged in surface water sampling must follow health and safety protocols described in the health and safety plan. Asbestos fibers are thin and long fibers so small that they cannot be seen by the naked eye. Asbestos fibers are easily inhaled when disturbed and when embedded in the lung tissue can cause health problems. Significant exposure to asbestos increases the risk of lung cancer, mesothelioma, asbestosis (non-cancerous lung disease), and other respiratory diseases (ATSDR 2006).

3.0 DEFINITIONS

Bailer: A long, narrow, tubular device with an open top and a check valve at the bottom. Bailers may be made of Teflon®, Polyethylene, or stainless steel.

Specific Conductance: How well water can conduct an electrical current.

Dip Sampler: A sample collection container that may be held directly or attached to a pole, used to collect surface water samples from the surface or just beneath the surface of a water body.

Dissolved Oxygen (DO): A measure of the quantity of oxygen dissolved in water. DO data is collected in the field using direct measure probes.

Environmental Sample: A liquid sample collected for chemical analysis. These samples are used to support remedial investigation, feasibility studies, treatability studies, remediation design and performance assessment, waste characterization, etc.

Oxidation-Reduction Potential (ORP): A measurement of the reducing or oxidation potential of a given system or medium. ORP data is collected in the field using direct-measure probes.

Peristaltic Pump: A low volume pump that operates by suction lift.

pH: A measure of the acidity or alkalinity of a solution, numerically equal to 7 for neutral solutions, increasing with increasing alkalinity to a maximum value of 14, and decreasing with increasing acidity to a minimum value of 1.

Temperature: A measure of the thermal energy contained in a given system. Units are commonly in degrees Celsius (°C).

Turbidity: Cloudiness in water due to suspended and colloidal organic and inorganic material. Units are commonly in Nephelometric Turbidity Units (NTUs).

4.0 RESPONSIBILITIES

This section presents a brief definition of field roles, and the responsibilities generally associated with them. This list is not intended to be comprehensive and often, additional personnel may be

involved. Project team member information will be included in project-specific plans (e.g., work plan, field sampling plan, quality assurance plan, etc.), and field personnel will always consult the appropriate documents to determine project-specific roles and responsibilities. In addition, one person may serve in more than one role on any given project.

Project Manager: Selects site-specific field sampling program with input from other key project staff, and applicable oversight agencies.

Quality Control Manager: Overall management and responsibility for quality assurance and quality control (QA/QC). Selects QA/QC procedures for the sampling and analytical methods, performs project audits, and ensures that data quality objectives are fulfilled.

Field Team Leader (FTL) and/or Geologist, Hydrogeologist, or Engineer: Implements the sampling program, supervises other sampling personnel, and ensures compliance with SOPs and QA/QC requirements. Prepares daily logs of field activities.

Sampling Technician (or other designated personnel): Assists the FTL and/or geologist, hydrogeologist, or engineer in the implementation of tasks. Performs the actual sample collection, packaging, and documentation (e.g., sample label and log sheet, chain-of-custody record, etc).

5.0 SURFACE WATER SAMPLING PROCEDURES

5.1 Background

The methods and procedures described in this SOP were developed from these sources:

U.S. Geological Survey, variously dated. *National field manual for the collection of water-quality data: U.S. Geological Survey Techniques of Water-Resources Investigations, Book 9, Chapters A1-A9.* Available online at http://pubs.water.usgs.gov/twri9A.

- U.S. Environmental Protection Agency, Region 8, 2003. *SOP EH-#1 Technical Standards Operating Procedure – Surface Water Sampling East Helena Site, Montana.* Available online at

http://www.epa.gov/region8/r8risk/pdf/r8-src_eh-01.pdf

5.2 Surface Water Sample Collection

Rivers, Streams and Ponds: Where multiple sampling stations exist along a moving water source (i.e., a creek or drainage channel), water samples generally will be collected from downstream to upstream locations, to minimize the effect of sampling activities on the samples collected. The samples will be grab samples collected from representative flowing water (usually the mid-channel), that is the portion of the water with the maximum flow at any given sampling station, unless otherwise specified.

A surface water sample will be collected according to one of the following, or similar, techniques.

1. Direct Method -- Sample bottle is uncapped and inverted, submerged to the specified depth, turned upright pointing upstream, removed from the water, and then capped. Add preservative, if any, after sample collection.

2. Dipper Method -- Sample bottle or container attached to a pole is dipped in the water, raised above the water, and then capped (if actual sample bottle used).

3. Bailer Method -- A appropriate sampling bailer with a ball check valve is submerged to the desired sample depth, either directly or by suspending the bailer on a rope from a pole.

4. Peristaltic Pump Method -- The sample is collected through a section of new, clean, flexible Tygon (polyvinylchloride) tubing. The tubing intake will be secured manually or by attaching weights. This procedure may be modified to collect the sample through a Teflon tube into a sample flask by running the pump on a vacuum.

5. Kemmerer Bottle – Use a properly decontaminated Kemmerer bottle. Set the sampling device so that the upper and lower stoppers are pulled away from the body, allowing water to enter the tube. Lower the pre-set sampling device to the predetermined depth. Avoid disturbing the bottom. Once at the required depth, send the weighted messenger down the suspension line, closing the device. Retrieve the sampler and discharge the first 10-20 mL from the drain to clear water that may not be representative of the sample. Repeat as needed to fill collect the needed volume.

6. Van Dorn Sampler – Set the device so that the end stoppers are pulled away from the body allowing surface water to enter the tube. Lower the sampler to the

predetermined depth. Once at the required depth send the weighted messenger down the suspension line, closing the sampling device. Retrieve the sampler and decant the first 10-20 mL from the drain to clear water that may not be representative of the sample from the valve. Repeat as needed until the required volume to fill sample bottles is collected.

7. Bacon Bomb Sampler -- Lower the bacon bomb sampler carefully to the desired depth, allowing for the trigger to remain slack at all times. When the desired depth is reached, pull the trigger line until taught. This will allow the sampler to fill. Release the trigger line and retrieve the sampler. Decant the first 10-20 mL from the drain to clear water that may not be representative of the sample from the valve. Repeat as needed until the required volume to fill sample bottles is collected.

For very shallow waters, a syringe method can be used, where a disposable plastic filtering syringe may be used to collect a sample without disturbing the sediment. Alternatively, at low flowing seeps and springs a small depression may be created to capture water. If a depression is made, the disturbance-related turbidity should be allowed to clear (settle) before the sample is collected.

5.3 Sampling Equipment and Techniques

The selection of sampling equipment listed above depends on the site conditions and sample type required. In addition, the following equipment is needed to collect surface water samples:

- Field notebook, indelible marker
- Global Positioning System (GPS) unit
- Marking stakes
- Digital Camera
- Compass
- 100 m measuring tapes
- Detergent solution (0.1-0.3 % Alconox)
- Distilled water
- Latex gloves
- Ziploc bags
- Paper Towel

- Chain of custody and sample labels
- Coolers
- Sample bottles
- Plastic sheeting

For collecting surface water samples, the procedures outlined below shall be followed.

1. Don appropriate health and safety equipment
2. Setup clean plastic sheeting in area for processing samples
3. Collect a surface water sample beginning from the most downstream location using one of the methods specified above
4. Place a stake or pole at or near the sampling location for future ease of identification
5. The first collected water will be used to rinse the sampling equipment. Sample bottles that do not contain preservative should be rinsed with the sample water prior to filling
6. Pour the sample from the sampling equipment down the side of the sample container in such a manner as to minimize turbulence during the transfer, or alternatively, collect the sample directly into the sample container. However, a primary concern with sample collection directly into sample containers is the loss of sample preservative from the sample container as it is dipped directly into the surface water.
7. Collect samples for Volatile Organic Compounds (VOC) first. Do not collect samples for VOC analysis using a peristaltic pump.
8. Label sample containers with the sample location and sample analysis information in accordance with the procedures in SOP-9.

9. Measure and record water quality parameters pH, DO, temperature, specific conductance, ORP, and turbidity using equipment calibrated according to manufacturer's specifications

10. Measure stream discharge according to SOP-4 and locate the sample using a site map or GPS according to SOP-11.

11. Pack samples containers to avoid leakage or breakage during shipment.

12. Store and ship samples on ice at 4 degrees Celsius. For further details on shipping and handling refer to SOP-8.

5.4 Sample Filtration: When required, a field-filtered water sample will be collected using a disposable, in-line 0.45 µm filter. The water sample will be pumped through the filter using a peristaltic pump and a section of Tygon (polyvinylchloride) or non-reactive (Teflon®) tubing or other appropriate method. An aliquot of approximately 100 ml of sample will be run through the tubing and filter prior to collection into the sampling containers. Both the filter and tubing will be disposed of between samples.

5.5 Sample Containers and Volumes: Certified clean sample containers appropriate to the analytical method will be obtained from the water analysis laboratory or other approved source. Different containers will be required for specific groups of analytes in accordance with U. S. EPA Methods, project specific requirements, and/or other local jurisdictional guidance. The sampler will confirm with the laboratory performing the analyses that the appropriate bottleware and preservatives are used and ensured that sufficient volume of the sample is collected.

Sample bottles and bottle caps will be protected from dust or other contamination between time of receipt by the sampling personnel and time of actual usage at the sampling site. Sampling equipment that will be used at multiple sampling locations will be cleaned after sampling at each location is completed. Decontamination of equipment will be completed in accordance with SOP-7.

5.6 Sample Preservation and Storage: If required by the project or analytical method, water samples submitted for chemical analysis will be stored at 4°C in ice-cooled, insulated containers immediately after collection. Preservation and storage methods depend on the chemical constituents to be analyzed and should be discussed with the water analysis laboratory prior to sample collection. EPA and/or other local jurisdictional requirements and/or the requirements of a project-specific plan (e.g., sampling and analysis plan, work plan, quality assurance project plan, etc.) shall be adhered to in preservation and storage of water samples.

5.7 Documentation: At each surface water station, sample details will be recorded on a Surface Water field sample data sheet (FSDS) form (see SOP-9, Attachment 1). Sampling conditions, including any deviations from this SOP in field logbook according to SOP-9. The sampling conditions that should be noted in the field log book will include:

A. Project identification;

B. Location identification (sampling station);

C. Detailed description of sampling location;

D. Sampling methods and equipment;

E. Condition of water (standing or moving);

F. Instrument calibration and cleaning record; and

G. Sketch map showing location of sampling station and permanent landmarks, and locate using a global positioning system according to SOP-11.

When the sampling activity is completed, the record will be checked by the Project Manager or his/her designee, and the original record will be placed in the project file.

6.0 QUALITY ASSURANCE AND QUALITY CONTROL

Field splits, field blanks, equipment rinsates, and matrix spike samples will be collected at the frequencies documented in the field sampling plan. Calibration checks will be performed at least once prior to and at least once following each day of instrument use in the field and the results documented in the field log book. All sampling data must be documented in the field logbooks and/or field forms, including rationales deviations from this SOP. The Field Team Leader or designated QA reviewer will check and verify that field documentation has been completed per this procedure and other procedures referenced herein. All equipment must be operated according to the manufacturer's specifications, including calibration and maintenance.

7.0 DECONTAMINATION

All equipment used in the sampling process shall be decontaminated prior to field use and between sample locations. Decontamination procedures are presented in SOP-7. Personnel shall don appropriate personal protective equipment as specified in the health and safety plan. Any investigation-derived waste generated in the sampling process shall be managed in accordance with the procedures outlined in SOP-12.

8.0 REFERENCES

Agency for Toxic Substances and Disease Registry. 2006. Asbestos Exposure and Your Health.

Feltz, H.R., 1980. Significance of Bottom Material Data in Evaluating Water Quality in Contaminants and Sediments. Ann Arbor, Michigan, Ann Arbor Science Publishers, Inc., Vol. 1, pp. 271-287.

Kittrell, F.W., 1969. A Practical Guide to Water Quality Studies of Streams. U.S. Federal Water Pollution Control Administration, Washington, D.C., 135 pp.

U.S. Environmental Protection Agency (USEPA), 1980. Standard Operating Procedures and Quality Assurance Manual. Water Surveillance Branch, USEPA Surveillance and Analytical Division, Athens, Georgia.

U.S. Environmental Protection Agency. 2007. Guidance for Preparing Standard Operating Procedures (SOPs). EPA QA/G-6. U.S. Environmental Protection Agency, Office of Environmental Information. EPA/600/B-07/001. April 2007.

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

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Libby Superfund Site Operable Unit 3 Standard Operating Procedure

Date: September 26, 2007 OU3 SOP 4 (Rev. 0)

Title: SURFACE WATER DISCHARGE MEASURMENT

APPROVALS:

TEAM MEMBER	SIGNATURE/TITLE	DATE
EPA Remedial Project Manager		9/26/07
SOP Author		9/26/07

Revision Number	Date	Reason for Revision
0	09/26/2007	--

1.0 INTRODUCTION

This Standard Operating Procedure (SOP) describes the protocol for collecting discharge measurements in streams and ditches. Discharge is defined as the volume rate of flow of water, including any substances suspended or dissolved in the water. Discharge will be expressed in cubic feet per second (cfs) or gallons per minute (gpm). This SOP provides a method for describing a current and three methods for measuring discharge: 1) volumetric method, 2) area-velocity method, and 3) flume method. Note that the protocol for collection of surface water samples is included in Libby OU3 SOP-3, Surface Water Sampling.

The procedures presented herein are intended to be general in nature and are applicable when referenced in a monitoring or field sampling plan. As the work progresses, and if warranted, appropriate revisions may be made when approved in writing by the Project Manager. All discharge measurements must be performed following health and safety protocols described in the health and safety plan.

2.0 HEALTH AND SAFETY WARNING

All personnel engaged in surface water discharge measurements must follow health and safety protocols described in the health and safety plan. Asbestos fibers are thin and long fibers so small that they cannot be seen by the naked eye. Asbestos fibers are easily inhaled when disturbed and when embedded in the lung tissue can cause health problems. Significant exposure to asbestos increases the risk of lung cancer, mesothelioma, asbestosis (non-cancerous lung disease), and other respiratory diseases (ATSDR 2006).

3.0 DEFINITIONS

Discharge: The volume of water transported in a certain amount of time through a cross-section of a stream. Discharge is expressed in cfs or gpm.

4.0 RESPONSIBILITIES

This section presents a brief definition of field roles, and the responsibilities generally associated with them. This list is not intended to be comprehensive and often additional personnel may be involved. Project team member information shall be included in project-specific plans (e.g., work plan, field sampling plan, quality assurance plan, etc.), and field personnel shall always consult the appropriate documents to determine project-specific roles and responsibilities. In addition, one person may serve in more than one role on any given project.

Project Manager: Selects site-specific sampling methods, sample locations, and constituents to be analyzed with input from other key project staff.

Quality Control Manager: Overall management and responsibility for quality assurance and quality control (QA/QC). Selects QA/QC procedures for the sampling and analytical methods, performs project audits, and ensures that data quality objectives are fulfilled.

Field Team Leader (FTL) and/or Field Geologist, Hydrogeologist, or Engineer: Implements the sampling program, supervises other sampling personnel, and ensures compliance with SOPs and QA/QC requirements. Prepares daily logs of field activities.

Sampling Technician (or other designated personnel): Assists the FTL, geologist, hydrogeologist, or engineer in the implementation of tasks. Performs the actual sample collection, packaging, and documentation (e.g., sample label and log sheet, chain-of-custody record, etc).

5.0 DISCHARGE MEASUREMENT PROCEDURES

5.1 Background

The methods and procedures described in this SOP were developed from these sources:

- Rantz, S.E. et al., 1982. *Measurement and Computation of Streamflow: Volume 1, Measurement of Stage and Discharge*, U.S. Geological Survey Water Supply Paper 2175. U.S. Government Printing Office, Washington, D.C.

- U.S. Department of the Interior, 1977. *National Handbook of Recommended Methods for Water-Data Acquisition*. Office of Water Data Coordination, U.S. Geological Survey, Reston, VA.

- U.S. Environmental Protection Agency, 1986. *Engineering Support Branch Standard Operating Procedures and Quality Assurance Manual*. Environmental Services Division, Region IV, Athens, GA.

The selection of an appropriate method for discharge measurement depends on the flow conditions. In some conditions, the flow measurement methods described here may be impossible to implement (e.g., extreme high-flow conditions). If flow conditions cannot be measured at a specific location, then field personnel will attempt to measure flow at a point upstream or downstream of the sample site. The field personnel will also note the conditions that inhibited more accurate measurement at the designated measurement location. All samples should be collected prior to performing the discharge measurements.

5.2 Qualitative Current Description

In cases where a discharge measurement is not required but a description of the direction and relative rate of flow is useful, qualitative measurements are made. Qualitative measurement of current is made by using a strip of soft tape or cloth attached to the end of a pole. The strip will indicate the presence of water flow and direction of flow at the location and depth. The diameter of the pole should be sufficiently small to prevent directional error.

5.3 Discharge Measurement

Quantitative measurements may be made using a current meter, a volumetric method, or flume method. The selection of discharge measurement method depends on streamflow rate and/or specific channel characteristics. For pipes, drain system outfalls and cases where flows are too small or stream gradients are too high, the volumetric method is appropriate. In cases where water depth is greater than 0.3 feet or the channel cross section is wide, discharge should be measured using the

area-velocity method. Where flows are below the practical limit that can be measured with the area-velocity method, the flume method is best. Where the total discharge is conveyed through two channels or differing types, a combination of these methods may be appropriate.

5.3.1 Volumetric Method

The volumetric method is a simple and accurate method for measuring flow from small discharges such as gravity flow discharged from pipe outlets, and is particularly useful for the measurement of small flows. This method involves observing the time required to fill or partly fill a calibrated container to a known volume. Alternatively, in the case of measuring discharge remotely in a sump or standpipe setting, the volumetric method may be performed by capturing flow in a container for a set period of time, no less than 10 seconds. This volume of water is then measured and discharge is determined.

Field personnel will observe and use judgment in approximating the flow volume and will select an appropriately sized volumetric container to use the volumetric method of flow measurement. Equipment required to make this measurement is a calibrated container and a stopwatch. Calibrated containers of varying sizes include: 5-gallon bucket, 2-liter graduated cylinder, 1-liter graduated cylinder, 1-liter bucket, etc. The incremental volume of a 5-gallon bucket can be determined by adding known volumes of water and recording the depth after each addition.

To measure flow, a technician will measure the time required to fill a volumetric container. The technician will time flow into the container for a minimum of 10 seconds. Five consecutive measurements will be made and noted, and the results will be averaged to determine the discharge. If remote measurement is necessary, a container will be attached to an extension rod. The technician will time flow for a minimum of 10 seconds. The volume of water will then be poured into a calibrated container, measured, and recorded. Five such measurements will be made, noted, and the results averaged to determine the discharge.
Calculations will be performed as follows:

- Record each of the five measurements in terms of gallons per second or milliliters per second, depending on the volumetric container.
- If one of the five measurements is 50 percent or more different from the other measurements, then this value will not be used. Instead, five additional measurements will be taken and, provided that none of these measurements differs by greater than 50 percent from the other measurements, these values will be used.
- Average the five values.
- Leakage around the discharge pipe, if any, will also be estimated and noted.
- Convert the averaged value to cfs as follows:
  - to convert ml/s to cfs, multiply by 3.53 x 10-5
  - to convert gal/s to cfs, multiply by 0.134
- Record discharge in cfs.

5.3.2 Velocity-Area Method

The vertical axis current meter may be used to perform velocity-area method discharge measurements. Common types of vertical axis current meters are the Price meter, type AA, and the Marsh-McBirney. A current meter is an instrument used to measure the velocity of flowing water. Operation of the Price Meter, type AA, is based on the proportionality between the velocity of the water and the resulting angular velocity of the meter rotor. By placing a current meter at a point in a stream and counting the number of revolutions of the rotor during a measured interval of time, the velocity of water at that point is determined. The number of revolutions of the rotor is obtained by an electrical circuit through the contact chamber. Contact points in the chamber are designed to complete an electrical circuit at selected frequencies of revolution. The electrical impulse produces an audible click in a headphone. The intervals during which meter revolutions are counted are timed with a stopwatch. The Marsh-McBirney uses electromagnetic induction to determine the velocity of water. As water flows over a sensor it changes the voltage within the sensor. The changing voltage is processed by the instrument which presents the output as a linear measurement of velocity.

A Price pygmy meter may be used in shallow depths and low-velocity waters. The pygmy meter is scaled two-fifths as large as the type AA meter. The pygmy meter makes one contact (click) per revolution and the type AA meter can make one click per revolution or one click per five revolutions. The Marsh-McBirney type meter can be used at any depth greater than 0.15 feet.

The current meter measures velocity at a point. The velocity-area method requires measurement of the mean velocity in selected subsections of the stream cross-section. By dividing the stream width into subsections, discharge becomes the total of discharges measured in each subsection. Velocity (V) is measured at each subsection, and discharge becomes the sum of the products of each velocity point and the cross-sectional area of each subsection:

$$Q = \sum(A_i * V_i)$$

where: Q = Streamflow in cfs,
A = Area of stream subsection in square feet, and
V = velocity in feet per second.

A cross section is defined by the depths at vertical points (i = 1, 2, 3, ...n) where the average velocity is measured.

In general, the person(s) measuring discharge should strive to measure no more than 5 percent of the flow in any one subsection. However, for small streams this is often impossible. Therefore, the person(s) should divide the channel cross-section into as many subsections as possible and make two complete discharge measurements using different sections for each. Subsections do not need to be identical in width. Velocities near banks are generally lower than velocities near the center of streams; therefore, these subsections may be wider than subsections near the center. Subsections will also be more closely spaced if a stream has an unusually deep portion in the cross-section. Typically, velocities will be measured by current meter for a 40- to 70-second period. It is recognized that 40 to 70 seconds is not long enough to ensure the accuracy of a single-point observation of velocity. However, because pulsations caused by turbulent and eddying effects are random and because velocity observations during a discharge measurement are made at several

verticals, there is little likelihood that the pulsations will bias the total measure discharge of a stream.

To make an area-velocity discharge measurement, the following conditions are required:

1. The stream must be channelized or contain relatively straight sections upstream of the measurement location.

2. Depth must be greater than 0.2 foot across most of the cross-section being measured.

The ideal channel cross-section is trapezoidal in shape, completely smooth in boundary materials, and possesses a uniform velocity distribution. Such an ideal condition is rarely observed. Therefore, minor modifications to the stream channels will be used to optimize measurement conditions. These modifications may include removal of aquatic vegetation, ice, and moving small stones that impact velocity upstream or downstream of the cross-section. However, no modifications should be made while measurements are being taken.

If flow conditions permit, current meter measurements will be made by wading. The type AA or pygmy meter and the Marsh-McBirney are used for wading measurements. The table below lists the type of meter and velocity method to be used for wading measurements at various depths. The persons(s) should stand at arm's length to the side of the meter.

Velocity Measurement Point Selection

Stream Depth (ft)	Type of Meter	Velocity Measuring Point(s) (Fraction of Depth)
2.5 or more	Type AA/Marsh-McBirney	0.2 and 0.8
1.5 - 2.5	Optional/Marsh-McBirney	0.6
0.3 - 1.5	Optional/Marsh-McBirney	0.6
< 0.3	Pygmy	0.5

Some departure from these specifications will be permitted. A Marsh-McBirney meter is appropriate for all depths deeper than 0.15 feet. Do not switch from one meter to another in the middle of a discharge measurement.

Under open channel laminar flow conditions, the effect of fluid contact with the bed of a stream channel and the air is a vertical distribution of velocities. Consistent with this velocity distribution, actual observation and mathematical theory has demonstrated that a single measurement of velocity taken at 0.6-depth or the average of two point velocities taken at 0.2 and 0.8 of the depth below the surface accurately results in mean velocity in the vertical (U.S.G.S. Water-Supply Paper 2175, 133-134 pp).

If the stream is generally less than 2.5 feet deep, the six-tenths (0.6) method will be used. If the stream is generally greater than 2.5 feet, the two- and eight-tenths (0.2 and 0.8) method, also known as the two-point method, will be used. A complete discussion concerning how to set the wading rod to place the current meter at proper depths is contained in Section 2.3.5, Field Procedures.

In the 0.6-depth method, an observation of velocity made in the vertical at 0.6 of the depth below the surface is used as the mean velocity in the vertical. In the two-point method of measuring velocities, observations are made in each vertical at 0.2 and 0.8 of the depth below the surface. The average of the two observations is taken as the mean velocity in the vertical.

A depth of 1.25 feet will accommodate the 0.6-depth method without causing the meter to be set closer than 0.5 feet from the stream bed; if the meter is set any closer to the stream bed, it will under-register the velocity. If the technician is at a measurement section that has only a few verticals shallower than 1.25 feet, the technician should use the type AA or Marsh-McBirney meter rather than the pygmy meter.

Mechanical vertical axis current meters do not register velocities accurately when placed close to a vertical wall. A Price meter held close to a right-bank vertical wall will under-register because the slower water velocity near the wall strikes the effective (concave) face of the cups. The converse is true at a left-bank vertical wall. (The terms "left bank" and "right bank" designate direction from the center of a stream for an observer facing downstream.) The Price meter also under-registers when positioned close to the water surface or close to the streambed.

5.3.2.1 Equipment And Techniques

Current meters, timers, depth and width measuring devices, and a means of counting meter revolutions are needed for measurement of discharge. The equipment includes:

- Field notebook, indelible marker
- Global Positioning System (GPS) unit;
- Digital Camera;
- Top-setting wading rod and current meter;
- Width-measuring devices, either engineer's tape or tagline;
- Digital counter or headset and stopwatch;
- Current meter rating tables;
- Stakes for width-measuring devices; and
- Calculator.

Top-Setting Wading Rod. The depth-measuring device that will be used is the wading rod. The current meter is attached to the wading rod. The top-setting wading rod has a 2-inch hexagonal main rod for measuring depth and a d-inch diameter round rod for setting the position of the current meter.

Current Meter. Vertical axis current meter, Marsh-McBirney, type AA meter, or pygmy meter.

Engineer's Tape or Tagline. Tape measures or premarked taglines are used for stream width measurements. Orientation normal to the flow patterns of the stream and elimination of most of the sag, through support or tension, are recommended for improved accuracy.

Digital Revolution Counter or Headset. The digital revolution counter attaches to an electronic connection at the top of the wading rod. The digital display shows the number of seconds of elapsed time. The person(s) stops the counter after 40 or more seconds, and the counter automatically displays the velocity.

If the digital counters are unavailable, the headset will be used as a means for determining the number of revolutions. A headset attaches to an electronic connection at the upper end of the wading rod. The person(s) wears this headset to listen to the audible clicking sounds produced by current meter revolutions. The number of rotations are counted and timed. Velocities as a function of time are listed on a current meter rating chart provided by the manufacturer, which is kept in the current-meter carrying case. When using the Marsh-McBirney meter a revolution counter or a headset will not be needed. This type of meter computes velocity directly. A velocity reading should be taken only after a suitable time has passed allowing the readings to stabilize.

Stopwatch. A stopwatch is used to measure time during which velocity is measured at each point in the cross-section.

5.3.2.2 Maintenance And Calibration

Prior to and following the use of the current meter, spin tests will be conducted to ensure that the unit performs acceptably. The spin test will be performed in an enclosed area, such as in the cab or in the enclosed rear of the trunk, to prevent wind interference. The test is to be performed prior to attaching the current meter to the wading rod. While holding the meter steady in an area sheltered

from breezes, the technicians will spin the rotor and then press the start button on the stopwatch. The technician will observe the meter until the rotor ceases to spin.

The duration of the spin for the pygmy meter will be more than 40 seconds, and for the Price AA meter, it will be more than 90 seconds. If the meter fails to meet the time-of-spin criteria, the meter will be cleaned and oiled before use. If the meter continues to spin well beyond these time limits, the record will indicate that the meter spun for 40+ seconds in the case of the pygmy meter, or for 90+ seconds in the case of the Price AA meter.

The Marsh-McBirney will have a zero check performed on the sensor to ensure accurate readings are obtained. First clean the sensor. Then place the sensor in a five gallon bucket of water. Keep it at least three inches away from the sides and bottom of the bucket. To make sure the water is not moving, wait 10 to 15 minutes after you have positioned the sensor and before taking any zero readings. If needed follow the manufactures recommendations to zero adjust the sensor.

To ensure reliable observations of velocity, it is necessary that the current meter be kept in good condition. Before and after each discharge measurement, the meter cups or vanes, pivot and bearing, and shaft will be examined for damage, wear, or faulty alignment. During measurements, the meter will be observed periodically when it is out of the water to be sure that the rotor spins freely and/or the sensor is free of debris.

Meters will be cleaned and oiled daily when in use. If measurements are made in sediment-laden water, the meter will be cleaned immediately after each measurement. After oiling, wipe away any excess oil and spin the rotor to ensure that it operates freely. If the rotor stops abruptly, the cause of the trouble will be determined and corrected before using the meter. The Marsh-McBirney should not be oiled in any way.

In addition to meter maintenance, the entire unit consisting of current meter, wading rod, and digital counter or headset will be checked before departure to the field each day as follows:

- Attach the current meter and digital counter/headset to the wading rod.
- Check the digital counter by ensuring that the readout is visible when the unit is turned on.
- If a headset is being used:
  - Spin the current meter to ensure that audible clicks occur.
  - If audible clicks do not occur, the following steps should be taken:
  - Check that electronic connections are tight.
  - Check that the cat's whisker lightly contacts the upper part of the shaft.
  - Spin again. If audible clicks still do not occur, check that the battery in the headset is properly aligned. Replace the battery, if necessary.

5.3.2.3 Procedures

Upon arrival at the site, the field technicians will evaluate the flow conditions to determine which measurement method is appropriate. Based on flow conditions, the Price AA meter, the pygmy meter, or the Marsh-McBirney meter will be selected to perform an area-velocity measurement.

At each measurement point (or section) across the stream cross-section, depth is measured prior to measurement of velocity. Place the wading rod about 0.5 feet downstream from the tagline. Place the wading rod in the stream so the base plate rests on the streambed. The depth of water is read from the graduated main rod. The main rod is graduated into 0.1-foot increments. These increments are indicated by a single score in the metal. Half-foot increments are marked by two scores in the metal, and each foot is marked by three scores in the metal. A vernier scale on the upper handle of the rod corresponds to 0.1-foot increments, and has 1 through 9 in raised numbers next to raised marks. A sliding, adjustable rod, known as the setting rod, to which the meter is attached, has single scored marks that are aligned with values on the vernier scale.

In high-velocity areas, it is recommended that depth be read as the value between depth on the upstream side of the rod and depth on the downstream side of the rod. Depth is measured to the nearest 0.2 foot. This depth is used to set the vertical location of the current meter.

The setting rod is then adjusted downward so that the scored mark of the setting rod that corresponds to the range of depth in feet (e.g., if depth = 0.46, range in feet = 0; or if depth = 1.72, range in feet = 1) is aligned with the stream depth value transposed to the vernier scale. This automatically positions the meter for use in the 0.6 method as the meter is then six-tenths of the total depth from the surface of the water.

For using the two-point method of velocity measurement, the depth of water is divided by 2. This value is set so that the meter will be at the 0.8-depth position from the water surface. The depth of water is then multiplied by two, and this value is set. The meter will then be at the 0.2-depth position measured down from the water surface. These two positions represent the conventional 0.2- and 0.8-depth positions. If depths are less than 0.30 foot, the 0.5 method may be used. The observation depth recorded will then be 0.5 of the total depth.

If water quality or sediments are sampled in conjunction with discharge measurement, samples will be collected prior to making discharge measurements. The following steps are to be followed in discharge measurement:

- Evaluate the measurement location. Choose a location where flow is least turbulent. If the prescribed location is in a stream reach with highly turbulent flow conditions, try to select a location immediately upstream or downstream. Flow should be visible from bank to bank. Eddies and slack water must not be present. Neither the type AA meter nor the pygmy meter will be used for measuring velocities slower than 0.1 ft/s unless absolutely necessary.

- Remove aquatic vegetation, ice, or other minor flow impediments. When such modifications are made, exercise great care to avoid unnecessary movement of sediments allow flow to stabilize before the current meter measurement begins.

- Position a tape about 1 foot above the surface of the water. Secure the tape so that it remains taut and perpendicular to the channel.

- Select a starting point at either the left bank (left edge of water, LEW) or the right bank (right edge of water, REW). LEW and REW are determined when facing downstream.

- Note the distance in feet, and the stream direction, that this cross-section lies from the prescribed location. For example, the note may read "25 feet downstream" or "15 feet upstream."

- Measure the width of the stream. After selecting the appropriate meter, select the number of subsections in which to measure velocity attempting to measure no more than 10 percent of the total flow in any one section, if possible.

- After determining the distance desired between measuring points, commonly referred to as sections, measurement can begin. Record the time and bank at which measurements start on the discharge measurement notes as "REW Start 0000", using REW or LEW depending upon whether starting at the right or the left edge of the water.

- Note the distance to the beginning edge of water from the initial point. The initial point is an arbitrary point on the tape, preferably zero, which lies on the shoreside of the stream. All station locations are recorded as distances from the initial point.

- Proceed to the first station beyond the edge of water. Record the distance from the initial point on the discharge measurement notes. Place the wading rod into the stream so the base plate rests on the stream bed.

- Stand downstream of the tagline or tape and face upstream. Do not stand behind or close to the meter. Raise the current meter on the wading rod so that it is well above the surface of the water.

- Measure stream depth at the measurement point as indicated on the wading rod. Record the stream depth to the nearest 0.2 foot (for example 0.32 feet or 1.54 feet).

- Lower the meter to the required depth and record the observation depth. The observation depth as a fraction of total depth is 0.6, 0.2, 0.8 or occasionally 0.5.

- The technician will stand in a position that least affects the velocity of the water passing the current meter. That position is usually obtained by facing upstream with the arm fully extended. The technician will stand at about a 45-degree angle downstream from the wading rod. The wading rod is held in a vertical position with the meter parallel to the direction of flow. Avoid standing in the water when possible.

- Start the digital counter. After 40 seconds, stop the counter. Note that the counter reports velocity.

- If using the headset rather than the digital counter, start the stopwatch on the first click and begin counting clicks. The first click counted after starting the stopwatch is counted as one.

- After at least 40 seconds have passed, stop the stopwatch on a click. Record the number of seconds and the number of revolutions (clicks) on the same line of the notes as the recorded depth.

- Determine velocity as a function of elapsed time and number of revolutions from the velocity chart specific to the flow meter. The manufacturer provides these charts with the instrument. Record velocity in the appropriate column. The flow meter must be aligned parallel to the direction of flow.

- Proceed to the next station. Record the distance from the initial point to the station. Repeat measurements of depth and velocity. Continue in this manner across the stream.

- After recording the distance measurement at the last station, record the time at which the ending edge of water is reached (e.g., LEW [or REW] FINISH 1330).

- Note velocity and depth at the edge of water as zero.

- Evaluate and record flow characteristics, weather conditions, air temperature, water temperature, observer(s), type of meter, and remarks.

- If less than 20 subsections have been used for the measurement, repeat the measurement steps. Begin from the opposite bank from where the previous measurement began.

5.3.2.4 Discharge Calculation

Calculate discharge on the discharge notes as follows:

- Use the distances from initial point to compute width for each subsection. The first width is computed by subtracting the first distance (edge of water) from the second distance and dividing this quantity by two. The second width will be the difference between the third distance and the first distance divided by two. For each subsequent width, subtract the previous station distance from the following station distance and divide this quantity by two. The final width is calculated as the difference between

the final distance and the second-to-last distance divided by two. Sum the width column and check to ensure that the calculated width equals the distance between the REW and LEW.

- Multiply the width by the depth for each station to determine the area of each subsection. Sum the areas to determine total area.
- Multiply the velocity by the area for each station to obtain the discharge for each subsection.
- Sum the discharges for each subsection to determine total discharge and record the value.
- If two sets of discharge measurements beginning at opposite banks were taken, repeat the discharge calculations for the second set of data. Average the total discharges for the two measurements. Record the average value and report it for input into the database.

5.3.3 Control Structures

Control structure such as flumes can be used to determine discharge. These structures have regular dimensions that allow for a consistent relationship between water level and discharge. A calibrated constriction placed in a stream channel changes the level of the water in or near the constriction. Flumes are constructed so that a restriction in the channel causes the water to accelerate, producing a corresponding change (drop) in the water level. When the physical dimensions of the flume constriction are known, discharge through constriction may be determined from measurement of depth. See below for a description of discharge measurement for Parshall flumes.

Typical flumes consist of three sections:

- A converging section to accelerate the approaching flow.
- A throat section, whose width is used to designate flume size.
- A diverging section, designed to ensure that the level downstream is lower than the level in the converging section.

The stage of a stream is the height of the water surface above an established elevation. Stage is usually expressed in feet. The Parshall flume consists of a converging section with a level floor, a throat section with a downward sloping floor, and a diverging section with an upward sloping floor. The principal feature of the Parshall flume (developed by R. Parshall in 1922) is an approach reach having converging sidewalls and a level floor, the downstream end of which is a critical depth cross-section. The primary stage measurement is made in the approach reach at some standard distance upstream from the critical-depth cross-section.

The flumes are designated by the width (w) of the throat. Flumes having throat widths from 3 inches (in.) to 8 feet (ft.) have a rounded entrance whose floor slope is 25 percent. Smaller and larger flumes do not have that feature. Ideally, flow rate through a flume may be determined by measurements at a single point some distance downstream from the inlet and above the throat.

5.3.3.1 Equipment

The following equipment will be needed:

- Field notebook, indelible marker
- GPS unit;
- Marking stakes;
- Digital Camera;
- Calculator;
- Current meter;
- Carpenter's level;
- Framing square;
- Measuring tapes; and
- Staff gauge.

5.3.3.2 Maintenance And Calibration

All flumes will be inspected to determine that entrance conditions provide a uniform influent flow distribution, the converging throat section is level, and that the throat section walls are vertical. The

flume will be closely examined to determine that it is discharging freely. Any problems observed during the inspection will be noted and reported to the field manager.

5.3.3.3 Procedures

Steps to be followed in measuring discharge.

- Remove any material that may have accumulated in the flume or on the weir;
- If the station includes a chart recorder, inspect the strip chart on the recorder to verify that it is operating;
- Note any deterioration of the station; report these conditions to the field manager at the conclusion of daily data collection activities;
- Measure and record the throat width (W) to the nearest 1/10 of an inch;
- Use the staff gauge to measure and record the gauge height (H) to the nearest 0.2 foot;
- Calculate discharge as described in Subsection 2.5.6; and
- Record the calculated discharge and the time and date of the site visit.

5.3.3.4 Discharge Calculation

A set of flume tables is necessary for calculating flows. The flume tables are specific to the type and size of flume and are usually supplied by the flume manufacturer. Based on the gage height (head, H, in feet) and the throat width of the flume (size of flume, W), the discharge is read directly from the table provided from the manufacturer. Note that the approximate values of discharge for heads other than those shown may be found by direct interpolation in the table. The following equation and table gives calculation coefficients for discharge calculations with Parshall flumes.

The free-flow discharge equations for the standard Parshall flume sizes are of the form:

$$Q = Ch_a^{\,n}$$

where:

h_a = measuring head (ft)
Q = discharge (ft^3/s)
C and n = coefficients specific to flume size

Coefficients (C) and exponents (n) for Parshall flume discharge calculations are listed in the following table.

Throat width	Coefficient (C)	Exponent (n)
1 in	0.338	1.55
2 in	0.676	1.55
3 in	0.992	1.55
6 in	2.06	1.58
9 in	3.07	1.53
1 ft	3.95	1.55
2 ft	8.00	1.55
3 ft	12.00	1.57
4 ft	16.00	1.58
5 ft	20.00	1.59
6 ft	24.00	1.59
7 ft	28.00	1.60
8 ft	32.00	1.61
10 ft	39.38	1.60
12 ft	46.75	1.60
15 ft	57.81	1.60
20 ft	76.25	1.60
25 ft	94.69	1.60
30 ft	113.13	1.60
40 ft	150.00	1.60
50 ft	186.88	1.60

6.0 DOCUMENTATION

Information required by this SOP, including rationale for changes, will be documented in detail in a bound field notebook and on appropriate field forms (see attached). This information includes the calibration data for flow measurement devices and field discharge measurement data.

Documentation will also include the type of flow measurement device, including a model number and instrument serial number; a detailed description of measurement location and weather conditions during the measurement; and calculations.

7.0 QUALITY ASSURANCE AND QUALITY CONTROL

All data must be documented in the field logbooks and/or field forms, including rationales deviations from this SOP. The Field Team Leader or designated QA reviewer will check and verify that field documentation has been completed per this procedure and other procedures referenced herein. All equipment must be operated according to the manufacturer's specifications, including calibration and maintenance.

7.1 Calculation Check

All calculations will be reviewed for accuracy and conformance with these procedures. The calculation review will be performed by a technically qualified individual before results are reported or interpreted. The calculation check shall be documented by the reviewer's initials and date of review. A copy of the reviewed calculations should be included in the project file.

7.2 Records Review and Management

The project manager or designated reviewer will verify that documentation has been completed and filed per this procedure.

8.0 DECONTAMINATION

All equipment used in the discharge measurement process shall be decontaminated prior to field use and between locations. Decontamination procedures are presented in SOP-7. Personnel shall don appropriate personal protective equipment as specified in the health and safety plan. Any investigation-derived waste generated in the sampling process shall be managed in accordance with the procedures outlined in SOP-12.

9.0 REFERENCES

Agency for Toxic Substances and Disease Registry. 2006. Asbestos Exposure and Your Health.

Rantz, S.E. et al., 1982. *Measurement and Computation of Streamflow: Volume 1, Measurement of Stage and Discharge*, U.S. Geological Survey Water Supply Paper 2175. U.S. Government Printing Office, Washington, D.C.

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U.S. Environmental Protection Agency, 1986. *Engineering Support Branch Standard Operating Procedures and Quality Assurance Manual.* Environmental Services Division, Region IV, Athens, GA.

U.S. Environmental Protection Agency. 2007. Guidance for Preparing Standard Operating Procedures (SOPs). EPA QA/G-6. U.S. Environmental Protection Agency, Office of Environmental Information. EPA/600/B-07/001. April 2007.

SURFACE WATER FLOW RECORD | STATION ID:

Date: ____________ Time: ________ Weather: ______________________________

______________________________ Page ____ of ____

Weather Past 48 hours: ______________________ Personnel: ______________________

Measuring Point Description: ____

UTM (NAD83):

Measuring Device:

VOLUMETRIC METHOD:

Trial #	Volume	Time	Discharge
1			
2			
3			
4			
5			

Average Discharge (gpm) =

FLUME METHOD:

Circle Flume: 4" Baski Cutthroat 8" Baski Cutthroat

Ha (ft) = ______________________

Flow from table (gpm) = ______________________

Estimated Leakage (%) = ______________________

Adjusted Flow for Leakage (gpm) = ______________________

Comments: ______________________

AREA - VELOCITY METHOD

Circle Method: 0.6 Times Depth 0.2 and 0.8 Times Depth

Bank Looking Downstream (Left/Right)	Tape Reading (ft)	Distance From Bank (ft)	Total Depth (ft)	Velocity (ft/sec)	Section Width (ft)	Section Area (ft^2)	Section Discharge (cfs)	Comments
							TOTAL DISCHARGE (cfs) =	

MAP / COMMENTS:

SIGNATURE: ______________________

LIBBY OU3
PHASE I

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Libby Superfund Site Operable Unit 3 Standard Operating Procedure

Date: September 26, 2007 OU3 SOP 5 (Rev. 0)

Title: SEDIMENT SAMPLING

APPROVALS:

TEAM MEMBER	SIGNATURE/TITLE	DATE
EPA Remedial Project Manager		9/26/07
SOP Author		9/26/07

Revision Number	Date	Reason for Revision
0	09/26/2007	–

1.0 INTRODUCTION

This Standard Operating Procedure (SOP) describes the protocols to be followed when sediment samples are collected for physical or chemical analysis. The procedures presented herein apply to sediment sampling from surface waters, wetlands, ponds, drainage structures, etc.

This document focuses on methods and equipment that are readily available and typically applied in collecting sediment samples. It is not intended to provide an all-inclusive discussion of sample collection methods. Specific sampling problems may require the adaptation of existing equipment or design of new equipment. Such innovations shall be clearly described in the project-specific sampling plan and approved by the Project Manager and the Quality Manager.

2.0 HEALTH AND SAFETY WARNING

All personnel engaged in sediment sampling must follow health and safety protocols described in the health and safety plan. Asbestos fibers are thin and long fibers so small that they cannot be seen by the naked eye. Asbestos fibers are easily inhaled when disturbed and when embedded in the lung tissue can cause health problems. Significant exposure to asbestos increases the risk of lung cancer, mesothelioma, asbestosis (non-cancerous lung disease), and other respiratory diseases (ATSDR 2006).

3.0 DEFINITIONS

Environmental Sample: A solid sample collected for chemical or geotechnical analysis. These samples are used to support remedial investigation, feasibility studies, treatability studies, remediation design and performance assessment, waste characterization, etc.

Hand Auger: A sampling tool consisting of a stainless steel tube with two sharpened spiral wings at the tip.

Shovel/Trowel: A sampling device consisting of a stainless steel spade attached to a handle.

Core Sampler: A variable diameter stainless steel tube that can be attached to a hammer for

driving into sediment. The tube can also be fitted with retaining liners.

4.0 RESPONSIBILITIES

This section presents a brief definition of field roles, and the responsibilities generally associated with them. This list is not intended to be comprehensive and often additional personnel may be involved. Project team member information shall be included in project-specific plans (e.g., work plan, field sampling plan (FSP), quality assurance plan, etc.), and field personnel shall always consult the appropriate documents to determine project-specific roles and responsibilities. In addition, one person may serve in more than one role on any given project.

Project Manager: Selects site-specific sampling methods, sample locations, and constituents to be analyzed with input from other key project staff.

Quality Control Manager: Overall management and responsibility for quality assurance and quality control (QA/QC). Selects QA/QC procedures for the sampling and analytical methods, performs project audits, and ensures that data quality objectives are fulfilled.

Field Team Leader (FTL) and/or Field Geologist, Hydrogeologist, or Engineer: Implements the sampling program, supervises other sampling personnel, and ensures compliance with SOPs and QA/QC requirements. Prepares daily logs of field activities.

Sampling Technician (or other designated personnel): Assists the FTL, geologist, hydrogeologist, or engineer in the implementation of tasks. Performs the actual sample collection, packaging, and documentation (e.g., sample label and log sheet, chain-of-custody record, etc).

5.0 SEDIMENT SAMPLING PROCEDURES

This section describes sediment sampling from the bottom of a surface water drainage course or pond. The collected samples will be placed in appropriate sample containers, as designated by the FSP or Quality Assurance Project Plan (QAPP), for transfer to a laboratory for the analyses identified in the FSP. Details of sample collection will be described on the attached sediment sampling form.

5.1 Sampling Equipment and Methods

All sediments, including sediment submerged under water, may be sampled by the following methods:

A. Direct Sampling Method -- Fluid sediment may be collected directly using the sample container. If sampled under water, the container will be capped in place to avoid disturbance while surfacing.

B. Manual Core Sampler Method -- A slide-hammer core sampler with stainless steel liners may be used to recover a relatively undisturbed core sample of the sediment. An extension section may be added to reach sediment intervals in deeper waters.

C. Remote Scoop Method -- A sampling cup or container attached to a pole may be used to collect a sediment sample in deeper water or where a longer reach is needed.

D. Bottom Sampling Dredge Method -- A sampling dredge attached to a cable also may be used to recover sediment samples in deeper waters.

The selection of sampling equipment listed above depends on the site conditions and sample type required. In addition, the following equipment is needed to collect sediment samples:

- Field notebook, indelible marker
- Global Positioning System (GPS) unit
- Marking stakes
- Digital Camera
- Compass
- 100 m measuring tapes
- Detergent solution (0.1-0.3 % Alconox)
- Distilled water
- Latex gloves
- Ziploc bags
- Paper Towel
- Chain of custody and sample labels
- Coolers
- Sample bottles
- Plastic sheeting

5.1.1 Stream Sediment Sample Collection

Collect surface water samples according to SOP-3 prior to collecting sediment samples. Stream sediment samples will be composite samples comprised of five subsamples collected from the surface to a depth of 4 inches, which is the most biologically active sediment zone. The five subsamples will be collected from random locations along the creek channel within 200 feet of the specified location. No sediments will be collected from overbank areas, unless specified in the FSP.

For collecting sediment samples, the procedures outlined below shall be followed.

1. Don appropriate health and safety equipment.
2. Setup clean plastic sheeting in area for processing samples.
3. Collect sediment samples using decontaminated equipment from inundated areas beginning at the most downstream location (i.e., no sediments will be collected from overbank areas).
4. Collect approximately 200 grams of sediment from each subsampling location.
5. Composite the five subsamples in a water-tight plastic bag (e.g., Ziploc© bags) and homogenize by hand kneading.
6. Carefully remove twigs, rocks, leaves and other undesirable debris not considered part of the sample.
7. Carefully decant excess water from the sample bag, if necessary, taking care to avoid decanting fine sediment.
8. Double bag the sample. Label outer bag with the Index ID, sample location, and sample analysis information in accordance with the procedures in SOP No. 9. Place in cooler on ice for storage and shipment (refer to SOP-8 for sample handling and shipping information).

9. Complete the appropriate sediment Field Sample Data Sheet (FSDS) form to document the station and sample details (see SOP No. 9, Attachment 1). Document sediment characteristics, sample location as well as any changes to this SOP in the field logbook.

10. Locate the sample using a site map or GPS according to SOP-11.

For duplicates, a second sediment composite sample will be collected (Steps 3-9 above) from the sampling reach and placed into a new sample bag.

5.1.2 Storm Water Detention Pond Sediment Sample Collection

A single composite sediment sample will be collected at each of the storm water detention ponds designated in the FSP according to the steps presented in section 5.1.1. Sediment will be collected from the bottom surface, not from the side slopes, of the pond in areas that would most frequently contain storm water.

If water is present in the detention pond at the time of sampling, then procedures for collecting subaqueous sediment cores from a lake or a pond will be adopted. When cores are collected by wading, the sampler will proceed in a circular direction around the pond to collect the five subsamples and avoid disturbing sediments at the subsampling locations prior to use of the coring device. Core samples will be collected using a Wildco® K-B Corer or similar device with clear plastic liners. The core barrel will be advanced a minimum of 6 inches or to the depth of refusal, but only the shallowest 4 inches will be processed as a sample according to the steps presented in section 5.1.0. Once collected, each sediment core will be capped (with top and bottom marked).

6.0 QUALITY ASSURANCE AND QUALITY CONTROL

Duplicate, equipment rinsate, and matrix spike samples will be collected at the frequencies documented in the field sampling plan. All sampling data must be documented in the field logbooks and/or field forms, including rationales deviations from this SOP. The Field Team Leader or designated QA reviewer will check and verify that field documentation has been completed per this procedure and the other procedures referenced herein. All equipment must be

operated according to the manufacturer's specifications, including calibration and maintenance.

7.0 DECONTAMINATION

All equipment used in the sampling process shall be decontaminated prior to field use and between sample locations. Decontamination procedures are presented in SOP-7. Personnel shall don appropriate personal protective equipment as specified in the health and safety plan. Any investigation-derived waste generated in the sampling process shall be managed in accordance with the procedures outlined in SOP-12.

8.0 REFERENCES

Agency for Toxic Substances and Disease Registry (ATSDR). 2006. Asbestos Exposure and Your Health.

U.S. Environmental Protection Agency. 2007. Guidance for Preparing Standard Operating Procedures (SOPs). EPA QA/G-6. U.S. Environmental Protection Agency, Office of Environmental Information. EPA/600/B-07/001. April 2007.

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Libby Superfund Site Operable Unit 3 Standard Operating Procedure

Date: September 26, 2007 OU3 SOP 7 (Rev. 0)

Title: EQUIPMENT DECONTAMINATION

APPROVALS:

TEAM MEMBER	SIGNATURE/TITLE	DATE
EPA Remedial Project Manager		9/26/07
SOP Author		9/26/07

Revision Number	Date	Reason for Revision
0	09/26/2007	--

1.0 INTRODUCTION

This Standard Operating Procedure (SOP) is based on MWH SOP-02, Equipment Decontamination, Revision 1.0, March 2004, modified for use at the Libby Asbestos Superfund Site OU3. Decontamination of drilling, sampling, and monitoring equipment is a necessary and critical aspect of environmental field investigations. Proper decontamination is a key element in reducing the potential for cross-contamination between samples from different locations, as well as ensuring that samples are representative of the sampled materials. Improper decontamination may result in costly re-collection and re-analysis of samples. All equipment used in the sampling process will be properly decontaminated prior to the collection of each sample and after completion of sampling activities.

The procedures outlined in this SOP will be followed during decontamination of field equipment used in the sampling process, including drilling, soil/water sample collection, and monitoring activities. Any deviations from these procedures will be noted in the field notebooks and approved by the appropriate oversight agency, if significant. Three major categories of field equipment, along with applicable decontamination methods for each, are discussed below.

2.0 HEALTH AND SAFETY WARNING

All personnel engaged in equipment decontamination must follow health and safety protocols described in the health and safety plan. Asbestos fibers are thin and long fibers so small that they cannot be seen by the naked eye. Asbestos fibers are easily inhaled when disturbed and when embedded in the lung tissue can cause health problems. Significant exposure to asbestos increases the risk of lung cancer, mesothelioma, asbestosis (non-cancerous lung disease), and other respiratory diseases (ATSDR 2006).

3.0 DEFINITIONS

Bailer: A cylindrical tool designed to remove material from a well. A valve at the bottom of the bailer retains the contents in the bailer.

Bladder Pump: Groundwater sampling equipment consisting of a flexible bladder, usually made of Teflon®, contained within a rigid cylindrical body (commonly made of stainless steel). The lower end of the bladder is connected to the intake port through a check valve, while the upper end is connected through a second check valve to a sampling line that leads to the ground surface.

Brass Sleeve: Hollow, cylindrical sleeves made of brass and used as liners in split-spoon samplers for collection of undisturbed samples.

Auger Flight: An individual auger section, usually 5 feet in length.

Continuous Core Barrel: 3-5 foot long steel barrels that can be joined together to allow continuous cores to be collected during a single run.

Drill Pipe: Hollow metal pipe used for drilling, through which soil and groundwater sampling devices can be advanced for sample collection.

Peristaltic Pump: A low-volume suction pump. The compression of a flexible tube by a rotor results in the development of suction.

Source Water: A drilling quality water source identified to be used for steam cleaning. This source should be sampled at the beginning of each field program to set baseline concentrations.

Distilled Water: Commercially available water that has been distilled. Each batch of distilled water should be analyzed to set baseline concentrations.

Hand Auger: A sampling tool consisting of a metal tube with two sharpened spiral wings at the tip.

Split-Spoon Sampler: A sampling tool consisting of a thick-walled steel tube with a removable head and drive shoe. The steel tube splits open lengthwise when the head and drive shoe are removed.

Scoop: A sampling hand tool consisting of a small shovel- or trowel-shaped blade.

Submersible Pump: Groundwater sampling pump that consists of a rotor contained within a chamber and driven by an electric motor.

4.0 RESPONSIBILITIES

This section presents a brief definition of field roles, and the responsibilities generally associated with them. This list is not intended to be comprehensive and often, additional personnel may be involved. Project team member information will be included in project-specific plans (e.g., work plan, field sampling plan, quality assurance plan, etc.), and field personnel will always consult the appropriate documents to determine project-specific roles and responsibilities. In addition, one person may serve in more than one role on any given project.

Project Manager: Responsible for project implementation and coordination, selects project-specific drilling and sampling methods, and associated decontamination procedures with input from other key project staff, and appropriate oversight agencies.

Quality Control Manager: Overall management and responsibility for quality assurance and quality control (QA/QC). Selects QA/QC procedures for the sampling and analytical methods, performs project audits, and ensures that data quality objectives are fulfilled.

Field Team Leader (FTL) and/or Geologist, Hydrogeologist, or Engineer: Implements the field program and supervises other sampling personnel, and ensures that SOPs are properly followed. Prepares daily logs cf field activities.

Field Sampling Technician (or other designated personnel): Assists the FTL, geologist, hydrogeologist, or engineer in the implementation of tasks and is responsible for the decontamination of sampling equipment.

5.0 DECONTAMINATION PROCEDURES

Drilling and sampling procedures require that decontaminated tools be employed in order to prevent cross-contamination. The decontamination procedures described below will be followed to ensure that only uncontaminated materials will be introduced to the subsurface during drilling and sampling. The equipment decontamination process will be undertaken before and after each use of the equipment and include either steam cleaning or washing. Steam cleaning of equipment, if used, will be performed at a temporary decontamination site. The flooring of the temporary decontamination site will be impermeable to water and large enough to contain the equipment and the rinsate produced.

If the quantity of water in the pad area exceeds its holding capacity, the water will be drummed temporarily until analytical results are obtained and the water can be properly disposed of. Steam cleaning will not be performed over bare ground, but will always be conducted so that rinsate can be collected and disposed of properly. Wherever applicable, equipment will be disassembled to permit adequate cleaning of the internal portions.

5.1 Drilling and Large Equipment

The following procedure will be used for decontamination of large pieces of equipment. These include well casings, auger flights, drill pipes and rods, and those portions of the drill rig that may stand directly over a boring or well location, or that may come into contact with casing, auger flights, pipes, or rods.

- Establish a decontamination area large enough to contain the equipment and any decontamination waste
- Place equipment on sawhorse or equivalent, if possible.

- Steam clean the external surfaces and internal surfaces, as applicable, on equipment using high-pressure steam cleaner from an approved water source. If necessary, scrub using brushes and a phosphate-free detergent (e.g., Alconox™), or equivalent laboratory-grade detergent until all visible dirt, grime, grease, oil, loose paint, rust, etc., have been removed.

- Rinse with potable water

- Remove equipment from decontamination pad and allow to air dry

- Record date and time of equipment decontamination

5.2 Soil and Groundwater Sampling Equipment

The following procedure will be used to decontaminate sampling equipment such as split-spoon samplers; brass sleeves; continuous core barrels; scoops; hand augers; non-dedicated bailers; submersible pumps, bladder pumps; and other sampling equipment that may come into contact with samples. To minimize decontamination procedures in the field, dedicated equipment will be used wherever feasible:

- Wash and scrub equipment with phosphate-free, laboratory-grade detergent (e.g., Alconox™ or equivalent) and off-site distilled water

- Triple-rinse with distilled water

- Air dry

- Wrap in aluminum foil, or store in clean plastic bag or designated casing.

- Record date and time of equipment decontamination

Personnel involved in decontamination activities will wear appropriate protective clothing as defined in the project-specific health and safety plan.

5.3 Monitoring Equipment

The following procedure will be used to decontaminate monitoring devices such as slug-test equipment, groundwater elevation and free product thickness measuring devices, and water quality checking instruments. Note that organic solvents can not be used to decontaminate free product measuring devices because they will cause damage to the probes. Spray bottles may be used to store and dispense distilled water.

- Wash equipment with laboratory-grade, phosphate-free detergent (e.g., Alconox™ or equivalent) and distilled water
- Triple-rinse with distilled water
- Store in clean plastic bag or storage case.
- Record date and time of equipment decontamination

6.0 QUALITY ASSURANCE AND QUALITY CONTROL

All equipment decontamination must be documented in the field logbooks and/or field forms, including rationales deviations from this SOP. The Field Team Leader or designated QA reviewer will check and verify that field documentation has been completed per this procedure and other procedures referenced herein.

To assess the adequacy of decontamination procedures, field rinsate blanks may be collected. The specific number of rinsate blanks will be defined in a FSP or work plan or by the Project

Manager. In general, at least one field rinsate blank should collected per sampling event or per day.

Rinsate blanks with elevated or detected contaminates will be evaluated by the Project Manager, who will relay the results to the site workers. Such results may be indicative of inadequate decontamination procedures that require corrective actions (e.g., retaining).

7.0 PROCEDURE FOR WASTE DISPOSAL

All decontamination water that has come into contact with contaminated equipment will be handled, labeled, stored and disposed according to SOP 12. Unless otherwise specified in the FSP, waste generated from other sources and classified as non-hazardous waste (e.g., PPE, pastic sheeting, rope and misc. debris) will be disposed into trash receptacles.

8.0 REFERENCES

Agency for Toxic Substances and Disease Registry. 2006. *Asbestos Exposure and Your Health.*

U.S. Environmental Protection Agency, *RCRA Ground-Water Monitoring: Draft Technical Guidance*, November 1992. Page 7-17.

Libby Superfund Site Operable Unit 3 Standard Operating Procedure

Date: September 26, 2007 OU3 SOP 8 (Rev. 0)

Title: SAMPLE HANDLING AND SHIPPING

APPROVALS:

TEAM MEMBER	SIGNATURE/TITLE	DATE
EPA Remedial Project Manager		9/26/07
SOP Author		9/26/07

Revision Number	Date	Reason for Revision
0	09/26/2007	--

1.0 INTRODUCTION

This standard operating procedure (SOP) is based on MWH SOP-09, Sample Handling and Shipping, Revision 1.0, March 2004, modified for use at the Libby Asbestos Superfund Site OU3. This SOP describes the requirements for sample handling, storage and shipping. The purpose of this SOP is to define sample management activities as performed from the time of sample collection to the time they are received by the laboratory.

2.0 HEALTH AND SAFETY WARNING

All personnel engaged in soil sampling must follow health and safety protocols described in the health and safety plan. Asbestos fibers are thin and long fibers so small that they cannot be seen by the naked eye. Asbestos fibers are easily inhaled when disturbed and when embedded in the lung tissue can cause health problems. Significant exposure to asbestos increases the risk of lung cancer, mesothelioma, asbestosis (non-cancerous lung disease), and other respiratory diseases (ATSDR 2006).

3.0 DEFINITIONS

Chain-of-Custody: An accurate written record of the possession of each sample from the time of collection in the field to the time the sample is received by the designated analytical laboratory.

Sample: Physical evidence collected for environmental measuring and monitoring. For the purposes of this SOP, sample is restricted to solid, aqueous, air, or waste matrices. This SOP does not cover samples collected for lithologic description nor does it include remote sensing imagery or photographs (refer to SOP-9 for field documentation procedures).

Sampler: The individual who collects environmental samples during fieldwork.

4.0 RESPONSIBILITIES

This section presents a brief definition of field roles, and the responsibilities generally associated with them. This list is not intended to be comprehensive and often additional personnel may be involved. Project team member information will be included in project-specific plans (e.g., work plan, field sampling plan (FSP), quality assurance plan, and etc.), and field personnel will always consult the appropriate documents to determine project-specific roles and responsibilities. In addition, one person may serve in more than one role on any given project.

Project Manager: The Project Manager is responsible for ensuring that the requirements for sample management are included in the appropriate project plans. The Project Manager is responsible for coordinating sample management efforts with input from other key project staff and applicable government agencies.

Quality Control Manager: Overall management and responsibility for quality assurance and quality control (QA/QC). Selects QA/QC procedures for the sampling and analytical methods, performs project audits, and ensures that data quality objectives are fulfilled.

Field Team Leader and/or Field Hydrogeologist, Geologist or Engineer: Implements the sampling program, supervises other sampling personnel, and ensures compliance with SOPs and QA/QC requirements. Prepares daily logs of field activities.

Field Technician: Responsible for sample collection, documentation, packaging, and shipping. Assists the FTL and/or geologist, hydrogeologist, or engineer in the implementation of tasks.

5.0 PROCEDURES

5.1 Applicability

The information in this SOP may be used by direct reference or incorporated into project-specific plans. Deviations or modifications to procedures addressed herein must be brought to the attention of, and approved by, applicable government agencies.

5.2 Sample Management

Sample Containers: The sample containers to be used will be dependent on the sample matrix and analyses desired, and are specified in the project FSP. Only certified pre-cleaned sample containers will be used. Sample containers will be filled with adequate headspace (approximately 10 percent) for safe handling upon opening, except containers for volatile organic compound (VOC) analyses, which will be filled completely with no headspace. This no-headspace requirement applies to both soil and groundwater samples.

Once opened, the containers will be used immediately. If the container is used for any reason in the field (e.g., screening) and not sent to the laboratory for analysis, it will be discarded. Prior to discarding the contents of the used container and the container, disposal requirements will be evaluated. When storing before and after sampling, the containers will remain separate from solvents and other volatile organic materials. Sample containers with preservatives added by the laboratory will not be used if held for an extended period on the job site or exposed to extreme heat conditions. Containers will be kept in a cool, dry place. For preserved samples (except VOCs), the pH of the sample will be checked following collection of the sample. If the pH is not at the required level, additional preservative (provided by the laboratory) will be added to the sample container.

Numbering and Labeling: Refer to OU3 SOP-9.

Custody Seals. Custody seals with the date and initials of the sampler will be used on each shipping container to ensure custody. The custody seal will be placed on opposites sides of the cooler across the seam of the lid and the cooler body. Alternatively, if the sample containers are all placed inside a liner bag within the cooler, the custody seal may be placed across the seal of the liner bag inside of the cooler.

Chain-of-Custody: COC procedures require a written record of the possession of individual samples from the time of collection through laboratory analyses. A sample is considered to be in custody if it is:

- In a person's possession
- In view after being in physical possession
- In a secured condition after having been in physical custody
- In a designated secure area, restricted to authorized personnel

The COC record will be used to document the samples taken and the analyses requested. Refer to SOP-9 Attachment 2 for the OU3-specific COC form. Information recorded by field personnel on the COC record will include the following:

- Sample identifier (Index ID)
- Date and time of collection
- Sample matrix
- Preservation
- Type of analyses requested
- Unique COC number
- Lab being shipped to
- Signature of individuals involved in custody transfer (including date and time of transfer)
- Airbill number (if appropriate)
- Any comments regarding individual samples (e.g., organic vapor meter readings, special instructions).

COC records will be placed in a waterproof plastic bag (e.g., Ziploc®), taped to the inside lid of the cooler or placed at the top of the cooler, and transported with the samples. Signed airbills will serve as evidence of custody transfer between the field sampler and courier, as well as between the courier and laboratory. If a carrier service is used to ship the samples (e.g., Federal Express, etc.), custody will remain with the courier until it is relinquished to the laboratory. Upon receiving the sample cooler, a laboratory representative should sign in the receiving box of the COC, thus establishing custody. The sampler will retain copies of the COC record and airbill.

Sample Preservation/Storage: The requirements for sample preservation are dependent on the desired analyses and the sample matrix, and are specified in the FSP.

5.3 Sample Shipping

The methods and procedures described in this SOP were developed from these sources:

- 49 CFR 173. Shippers – Shippers – General Requirements for Shipping. United States Code of Federal Regulations available online at http://www.gpoaccess.gov/cfr/index.html
- 49 CFR 178. Specifications for Packaging. United States Code of Federal Regulations available online at http://www.gpoaccess.gov/cfr/index.html
- ASTM D 4220. Standard Practice for Preserving and Transporting Soil Samples. American Society for Testing and Materials available online at http://www.astm.org/
- ASTM D 4840. Standard Practice for Sampling Chain-of-Custody Procedures. American Society for Testing and Materials available online at http://www.astm.org/

Procedures for packaging and transporting samples to the laboratory are dependent on the chemical, physical, and hazard properties of the material. The procedures may also be based on an estimation of contaminant concentrations/properties in the samples to be shipped. Samples will be identified as environmental samples, excepted quantities samples, limited quantities samples, or standard hazardous materials. Environmental samples are defined as solid or liquid samples collected for chemical or geotechnical analysis. Excepted quantities involve the shipment of a few milliliters of either an acid or base preservative in an otherwise empty sample container. Limited quantities are restricted amounts of hazardous materials that may be shipped in generic, sturdy containers. Standard hazardous material shipments require the use of stamped/certified containers. All samples will be packaged and shipped or hand delivered to the laboratories the same day of sample collection, unless otherwise specified in the project-specific FSPs.

The following paragraphs describe standard shipping procedures for different types of samples. Any exceptions to these procedures will be defined in the FSP. It is the responsibility of the sampler to refer to the U.S. Department of Transportation (DOT) (http://hazmat.dot.gov/regs/rules.htm) regulations when dealing with a substance not addressed in this SOP for requirements and limitations associated with the shipment.

Sample Shipping via Commercial Carrier:

Aqueous or Solid Samples: Samples will be packaged and shipped to the laboratories the same day of sample collection, unless otherwise specified in the FSP and depending on holding time requirements for individual samples. For aqueous or solid samples that are shipped to the laboratory via a commercial carrier the following procedures apply:

- Sample labels will be completed and attached to sample containers.
- The samples will be placed upright in a waterproof metal (or equivalent strength plastic) ice chest or cooler.
- For shipments containing samples for volatile organic analysis, include a trip blank.
- Ice in double Ziploc® bags (to prevent leakage) will be placed around, among, and on top of the sample bottles. Enough ice will be used so that the samples will be chilled and maintained at 4°C ± 2°C during transport to the laboratory. Dry ice or blue ice will not be used.
- To prevent the sample containers from shifting inside the cooler, the remaining space in the cooler will be filled with inert cushioning material, such as shipping peanuts, additional bubble pack, or cardboard dividers, such that the sample containers remain upright and do not break.
- Tape shut the cooler's drain plug
- The original copy of the completed COC form will be placed in a waterproof plastic bag and taped to the inside of the cooler lid or placed at the top of the cooler.
- The lid will be secured by wrapping strapping tape completely around the cooler in two locations.

- Mark the cooler with arrow labels indicating the proper upright position of the cooler.

- Custody seals consisting of security tape with the date and initials of the sampler will be used on each shipping container to ensure custody. Two signed custody seals will be placed on the cooler, one on the front and one on the back.

- A copy of the COC record and the signed air bill will be retained for the project files.

- Affix a label containing the name and address of the shipper to the outside of the cooler

Hand-Delivered Samples: For aqueous or solid samples that will be hand carried to the laboratory, the same procedures apply.

Excepted Quantities: Usually, corrosive preservatives (e.g., hydrochloric acid, sulfuric acid, nitric acid, or sodium hydroxide) are added to otherwise empty sample bottles by the analytical laboratory prior to shipment to field sites. However, if there is an occasion whereby personnel are required to ship bottles with these undiluted acids or bases, the containers will be shipped in the following manner:

1. Each individual sample container will have not more than 30 milliliters of preservative.

2. Collectively, the preservative in these individual containers will not exceed a volume of 500 milliliters in the same outer box or package.
3. Despite the small quantities, only chemically compatible material may be placed in the same outer box, (e.g., sodium hydroxide, a base, must be packaged separately from the acids).

4. Federal Express will transport nitric acid only in concentrations of 40 percent or less.

5. A "Dangerous Goods in Excepted Quantities" label will be affixed to the outside of the outer box or container. Information required on the label includes:

   - Signature of Shipper
   - Title of Shipper
   - Date
   - Name and Address of Shipper
   - Check of Applicable Hazard Class
   - Listing of UN Numbers for Materials in Hazard Classes

Limited Quantities: Occasionally, it may become necessary to ship known hazardous materials, such as pure or floating product. DOT regulations permit the shipment of many hazardous materials in "sturdy" packages, such as an ice chest or cardboard box (not a specially constructed and certified container), provided the following conditions are met:

1. Each sample bottle is placed in a plastic bag, and the bag is sealed. Each VOC vial will be placed in a sealable bag. As much air as possible is squeezed from the bag before sealing. Bags may be sealed with evidence tape for additional security.

2. Or each bottle is placed in a separate paint can, the paint can is filled with vermiculite, and the lid is affixed to the can. The lid must be sealed with metal clips, filament, or evidence tape. If clips are used, the manufacturer typically recommends six clips.

3. The cans are placed upright in a cooler that has had the drain plug taped shut inside and outside, and the cooler is lined with a large plastic bag. Approximately 1 inch of adsorbent material sufficient to retain any liquid that may be spilled, is placed in the bottom of the liner. Only containers having chemically compatible material may be packaged in each cooler or other outer container.

4. The COC record is sealed inside a plastic bag and placed inside the cooler. The sampler retains one copy of the COC record. The laboratory will be notified if the

sample is suspected of containing any substance for which the laboratory personnel should take safety precautions.

5. The cooler is shut and sealed with strapping tape (filament type) around both ends. Two signed custody seals will be placed on the cooler, one on the front and one on the back. Additional seals may be used if the sampler and/or shipper consider more seals to be necessary. Wide, clear tape will be placed over the seals to ensure against accidental breakage.

6. The following markings are placed on the side of the cooler:

   - Proper Shipping Name (Column B, List of Dangerous Goods, Section 4, IATA Dangerous Goods Regulations [DGR])
   - UN Number (Column A, List of Dangerous Goods, Section 4, IATA DGR)
   - Shipper's name and address
   - Consignee's name and address
   - The words "LIMITED QUANTITY"
   - Hazard Labels (Column E, List of Dangerous Goods, Section 4, IATA DGR)
   - Two Orientation (Arrow) labels placed on opposite sides.

7. The Airbill/Declaration of Dangerous Goods form is completed as follows:

   - Shipper's name and address
   - Consignee's name and address
   - Services, Delivery & Special Handling Instructions
   - Cross out "Cargo Aircraft Only" in the Transport Details Box
   - Cross out "Radioactive" under Shipment Type
   - Nature and Quantity of Dangerous Goods

- Proper Shipping Name (Column B, List of Dangerous Goods, Section 4, IATA DGR)
- Class or Division (Column C, List of Dangerous Goods, Section 4, IATA DGR)
- UN Number (Column A, List of Dangerous Goods, Section 4, IATA DGR)
- Packing Group (Column F, List of Dangerous Goods, Section 4, IATA DGR)
- Subsidiary Risk, if any (Column D, List of Dangerous Goods, Section 4, IATA DGR)
- Quantity and type of packing (number and type of containers: for example, "3 plastic boxes", and the quantity per container, "2 L", is noted as "3 Plastic boxes X 2 L" This refers to 3 plastic boxes (coolers are referred to as plastic boxes) with 2 liters in each box.
- Packing Instructions (Column G, List of Dangerous Goods, Section 4, IATA DGR).
- Note: Only those Packing Instructions in Column G that begin with the letter "Y" may be used. These refer specifically to the Limited Quantity provisions.
- Authorization (Write in the words Limited Quantity)
- Emergency Telephone Number (List 800-535-5053. This is the number for INFOTRAC.)
- Printed Name and Title, Place and Date, Signature.

Standard Hazardous Materials: Shipment of standard hazardous materials presents the most difficulty and expense. However, there may be occasion whereby a hazardous material cannot be shipped under the Limited Quantity provisions, (e.g., where there is no Packing Instruction in Column G, List of Dangerous Goods, IATA Dangerous Goods Regulations, that is preceded by the letter "Y").

In such cases, the general instructions noted above but for non-Limited Quantity materials will apply, with one important difference: standard hazardous materials shipment requires the use of certified outer shipping containers. These containers have undergone rigid testing and are, therefore, designated by a "UN" stamp on the outside, usually along the bottom of a container's side. The UN stamp is also accompanied by codes specifying container type, packing group rating, gross mass, density, test pressure, year of manufacturer, state of manufacturer, and manufacturer code name. The transport of lithium batteries in Hermit Data Loggers is an example of a standard hazardous material where only a designated outer shipping container may be used.

5.4 Holding Times

The holding times for samples will depend on the analysis and the sample matrix. Refer to the FSP for holding times requirements.

6.0 QUALITY ASSURANCE AND QUALITY CONTROL

All sample shipments must be documented in the field logbooks and/or field forms, including rationales deviations from this SOP. The Field Team Leader or designated QA reviewer will check and verify that handling and shipment documentation has been completed per this procedure and other procedures referenced herein.

7.0 DECONTAMINATION

All shipment coolers shall be maintained clean of sampled material to avoid exposure during shipment. Any investigation-derived waste generated in the sampling process shall be managed in accordance with the procedures outlined in SOP-12.

8.0 REFERENCES

Agency for Toxic Substances and Disease Registry. 2006. Asbestos Exposure and Your Health.

Enforcement Considerations for Evaluations of Uncontrolled Hazardous Waste Disposal Sites by Contractors, Draft, Appendix D, April 1980.

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Libby Superfund Site Operable Unit 3 Standard Operating Procedure

Date: February 22, 2008 OU3 SOP 9 (Rev. 2)

Title: FIELD DOCUMENTATION

APPROVALS:

TEAM MEMBER	SIGNATURE/TITLE	DATE
EPA Remedial Project Manager	RPM	3/20/08
SOP Author		3/19/08

Revision Number	Date	Reason for Revision
0	09/26/2007	--
1	10/5/2007	• Add section for "Corrections and Modifications" and Field Modification Approval form (Attachment 3) • Update Labeling section and COC (Attachment 2) to reflect non-asbestos analysis and container details • Update FSDS forms (Attachment 1) based on field team input
2	2/22/08	• Incorporate changes to FSDS forms (Attachment 1) based on field input • Remove OU3 phase specificity in SOP text

1.0 INTRODUCTION

This Standard Operating Procedure (SOP) is a general guidance document for the required documentation to be completed by field personnel during field investigations. This SOP is based on MWH SOP-04, Field Documentation, Revision 1.0, March 2006, modified for use at the Libby Mine Site. Documentation in the form of field logbooks, reports, and forms shall be completed for every activity in the field. Records shall be maintained on a daily basis as the work progresses. All field documentation shall be accurate and legible because it is deliverable to the client as potentially a legal document.

2.0 HEALTH AND SAFETY WARNING

All personnel engaged in soil sampling must follow health and safety protocols described in the site health and safety plan. Asbestos fibers are thin and long fibers so small that they cannot be seen by the naked eye. Asbestos fibers are easily inhaled when disturbed and when embedded in the lung tissue can cause health problems. Significant exposure to asbestos increases the risk of lung cancer, mesothelioma, asbestosis (non-cancerous lung disease), and other respiratory diseases (ATSDR 2006). All personnel engaged in soil sampling must follow health and safety protocols described in the health and safety plan.

3.0 RESPONSIBILITIES

This section presents a brief definition of field roles, and the responsibilities generally associated with them. This list is not intended to be comprehensive and often, additional personnel may be involved. Project team member information shall be included in project-specific plans (e.g., work plan, field sampling plan, quality assurance plan, etc.), and field personnel shall always consult the appropriate documents to determine project-specific roles and responsibilities. In addition, one person may serve in more than one role on any given project.

Project Manager: Selects project-specific field documentation with input from other key project staff.

Quality Control Manager: Overall management and responsibility for quality assurance and quality control (QA/QC). Selects QA/QC procedures for the sampling and analytical methods, performs project audits, and ensures that data quality objectives are fulfilled.

Field Team Leader (FTL) and/or Field Geologist, Hydrogeologist, or Engineer: Implements the sampling program, supervises other sampling personnel, and ensures compliance with SOPs and QA/QC requirements. Prepares daily logs of field activities.

Field Technician (or other designated personnel): Assists the FTL and/or field geologist, hydrogeologist, or engineer in the implementation of field tasks and field documentation.

Field Sample/Data Manager: Responsible for proper handling and shipping of all samples collected by the field crew, electronic data entry of field sample data sheet (FSDS) and chain-of-custody (COC) forms, and scanning/posting of field documentation PDFs (FSDS, COC, field logbooks, digital photographs) to a dedicated FTP site.

4.0 FIELD DOCUMENTATION PROCEDURES

Field documentation serves as the primary foundation for all field data collected that will be used to evaluate the project site. There are two main forms of field documentation – field logbooks and FSDS forms. All field documentation shall be accurate, legible and written in indelible black or blue ink. Absolutely no pencils or erasures shall be used. Incorrect entries in the FSDS forms or field logbooks will be corrected by crossing out the incorrect entry with one line, the individual making the correction will initial and date next to the correction.

4.1 Field Logbooks

The field logbook shall be a bound, weatherproof book with numbered pages, and shall serve primarily as a daily log of the activities carried out during the fieldwork. All entries shall be made in indelible black or blue ink. A field logbook shall be completed for each operation undertaken during the field tasks. To further assist in the organization of the field log books, the project name and the date shall be recorded on top of each page along with the significant

activity description (e.g., surface sample or soil boring number). All original field documentation shall be retained in the project files.

Skipped pages or blank sections at the end of a field log book page shall be crossed out with an "X" covering the entire page or blank section; "No Further Entries," initials, and date shall be written by the person crossing out the blank section or page. The responsible field team member shall write his/her signature, date, and time after the day's last entry.

Field activities vary from project to project; however, the concept and general information that shall be recorded are similar. The descriptions of field data documentation given below serve as an outline; individual activities may vary in documentation requirements. A detailed description of two basic example logbooks, suitable for documentation of field activities, is given below. These field logbooks include the FTL logbook and the field geologist/sampling team logbook.

FTL Logbook: The FTL's responsibilities include the general supervision, support, assistance, and coordination of the various field activities. As a result, a large portion of the FTL's day is spent rotating between operations in a supervisory mode. Records of the FTL's activities, as well as a summary of the field team(s) activities, shall be maintained in a logbook. The FTL's logbook shall be used to fill out daily/weekly reports and daily quality control reports (DQCRs), and therefore, shall contain all required information. Entries shall be preceded with time in military units for each observation. Items to be documented include:

- Record of tailgate meetings
- Personnel and subcontractors on job site and time spent on the site
- Field operations and personnel assigned to these activities
- Site visitors
- Log of FTL's activities: time spent supervising each operation and summary of daily operations as provided by field team members
- Problems encountered and related corrective actions
- Deviations from the sampling plan and reasons for the deviations
- Records of communications; discussions of job-related activities with the client, subcontractor, field team members, and project manager

- Information on addresses and contacts
- Record of invoices signed and other billing information
- Field observations

Field Geologist/Sampling Team Logbook: The field geologist or sampling team leader shall be responsible for recording the following information in a logbook:

- Health and Safety Activities
  - Calibration records for health and safety equipment (e.g., type of PID, calibration gas used, associated readings, noise dosimeters, etc.)
  - Personnel contamination prevention and decontamination procedures
  - Record of daily tailgate safety meetings
- Weather
- Calibration of field equipment
- Equipment decontamination procedures
- Personnel and subcontractors on job site and time spent on the site
- Station identifier
- Sampling activities
  - Sample location (sketch)
  - Equipment used
  - Names of samplers
  - Date and time of sample collection
  - Sample interval
  - Number of samples collected
  - Analyses to be performed on collected samples
- Disposal of contaminated wastes (e.g., PPE, paper towels, Visqueen, etc.)
- Field observations
- Problems encountered and corrective action taken
- Deviations from the sampling plan and reason for the deviations
- Site visitors

4.2 Field Sample Documentation

Sample Labels: A unique sample identification label shall be affixed to all sample containers. All samples will be labeled in a clear, precise way for proper identification in the field and for tracking in the laboratory. At the time of collection, each sample will be labeled with a unique 5-digit sequential identification (ID) number, referred to as the Index ID. The Index ID for all samples collected as part of OU3 sampling activities will have a two-character prefix specific to the sampling Phase (e.g., Phase 1 samples will have a "P1" prefix, P1-12345) as specified in the applicable SAP. Index ID labels will be ½ inch x 1 ¾ inch in size and pre-printed for use in the field. For each Index ID, multiple labels will be printed to allow for multiple containers of the same sample (i.e., for different analyses).

Index ID Label Example: **P1-12345**

Each collection container will be labeled with a container label that enables the field team member to record the container specific details, such as the method of sample preparation (e.g., filtered/unfiltered), method of preservation, and the analytical methods that will be requested. Container labels will be 2 inch x 4 inch in size and pre-printed for use in the field. Any container-specific information shall be written in indelible ink.

Container Label Example:

Station ID: _____ Date/Time: _____
Index ID:

Media (circle one): AQ SO AA BK DB TC
For AQ, Filtered? (circle one): Yes No
Container: _____
Preservation: _____
Analyses: _____
_____
Lab QC: _____ ☐ Archive

Media acronyms: AQ – aqueous media, SO – solid media, AA – ambient air, BK – tree bark, DB – organic debris, TC – tree age core

After labels have been affixed to the sample container, the labels will be covered with clear packaging tape to ensure permanence during shipping.

Any unused Index ID labels should be crossed out to avoid the possibility of using unused labels for a different sample.

Field Sample Data Sheet (FSDS) Forms: Data regarding each sample collected as part of the OU3 sampling will be documented using Libby-specific FSDS forms (provided as Attachment 1). These FSDS forms are media-specific and designed to facilitate data entry of station location, sample details, and field measurements needed for the OU3 investigation.

In the field, one field team member will be responsible for recording all sample details onto the appropriate FSDS form. At the time of sample labeling, one Index ID label will be affixed to the FSDS form in the appropriate field. All written entries on the FSDS form shall be accurate, legible and written in indelible black or blue ink.

Once the FSDS form is complete, written entries will be checked by a second field team member. These two field team members will initial the bottom of the FSDS form in the appropriate field to document who performed the written data entry and who performed the QC check of the FSDS form.

On a weekly basis (or more frequently as conditions permit), information from the hard copy FSDS form will be manually entered into a field-specific OU3 database using electronic data entry screens by the Field Sample/Data Manager. Once electronic data entry is complete, QC of all data entry will be completed by the FTL or their designate. The Field Sample/Data Manager and the FTL will initial in the appropriate field on the paper FSDS form to document who performed the data entry into the database and who performed the QC check.

4.3 Photologs

Photologs are often used in the field to document site conditions and sample location characteristics. While photographs may not always be required, they shall be used wherever applicable to show existing site conditions at a particular time and stage of the investigation or related site activity. Photolog information shall include:

- station location identifier
- Index ID (if applicable)
- date and time of photo
- direction/orientation of the photo
- description of what the photo is intended to show

An engineer's scale or tape shall be included in any photographs where scale is necessary. Any wasted frames or images in a roll of film or sequence of digital images shall be so noted in the field logbook.

4.4 Chain-of-Custody Records

Custody Seals: Custody seals with the date and initials of the sampler will be used on each shipping container to ensure custody. The custody seal will be placed on opposites sides of the cooler across the seam of the lid and the cooler body. Alternatively, if the sample containers are all placed inside a liner bag within the cooler, the custody seal may be placed across the seal of the liner bag inside of the cooler.

Chain-of-Custody Forms: COC procedures allow for the tracking of possession and handling of individual samples from the time of field collection through to laboratory analysis. Documentation of custody is accomplished through a COC form that lists each sample and the individuals responsible for sample collection and shipment, sample preparation, and receipt by the analytical laboratory. The COC form also documents the analyses requested for each sample. Whenever a change of custody takes place, both parties will sign and date the COC form, with the relinquishing party retaining a copy of the form. The party that accepts custody will inspect the COC form and all accompanying documentation to ensure that the information is complete and accurate. Any discrepancies will be noted on the COC form. Shipping receipts shall be signed and filed as evidence of custody transfer between field sampler(s), courier, and laboratory.

Attachment 2 provides an example of the COC form that will be used for all samples collected as part of OU3 sampling. This form will be printed as a carbonless triplicate form to facilitate

retention of COC copies by relinquishing parties. As seen, the COC form includes the following information:

- sample identifier (Index ID)
- date and time of collection
- method of sample preparation and preservation
- number of sample containers
- analyses requested
- shipping arrangements and airbill number, as applicable
- recipient laboratories
- signatures of parties relinquishing and receiving the sample

On a daily basis, the Field Sample/Data Manager will package samples for shipping, complete hard copy COC forms, and ship all samples as outlined in SOP No. 8. On a daily basis, information from the hard copy COC form necessary for sample tracking will be manually entered into a field-specific OU3 database using electronic data entry screens by the Field Sample/Data Manager. Once electronic data entry is complete, QC of all data entry will be completed by the FTL or their designate.

5.0 FIELD DATA TRANSMITTAL

Copies of all FSDS forms, COC forms, and field log books will be scanned and posted in portable document format (PDF) to a project-specific file transfer protocol (FTP) site daily. This FTP site will have controlled access (i.e., user name and password are required) to ensure data access is limited to appropriate project-related personnel. File names for scanned FSDS forms, COC forms, and field log books will include the sample date in the format YYYYMMDD to facilitate document organization (e.g., FSDS_20070831.pdf).

Electronic copies of all digital photographs will also be posted weekly (or more frequently as conditions permit) to the project-specific FTP site. File names for digital photographs will

include the station identifier, the sample date, and photograph identifier (e.g., ST-1_20070831_12459.tif).

A copy of the field-specific OU3 database will be posted to the project-specific FTP site on a weekly basis (or more frequently as conditions permit). The field-specific OU3 database posted to the FTP site will include the post date in the file name (e.g., FieldOU3DB_20070831.mdb).

6.0 CORRECTIONS AND MODIFICATIONS

6.1 Field Deviations and Modifications

It is recognized that deviations and modifications from the standard operating procedures may be necessary based on site conditions. Any requested field modifications will be submitted by Robert Marriam (Remedium Group, Inc. - W.R. Grace contractor) to Bonita Lavelle (EPA Region 8 - Remedial Project Manager) for review and approval. All modification requests will be recorded in a Field Modification Approval Form (see Attachment 3).

6.2 Corrections to Hard Copy Forms

If an error is identified on an FSDS or COC form prior to entry into the field-specific OU3 database, the information should be corrected on the hard copy form by crossing out the incorrect entry with one line, the individual making the correction will initial and date next to the correction. Data entry into the field-specific OU3 database and scanning/posting of the hard copy forms should proceed following the data entry procedures described above.

If an error is identified on an FSDS or COC form after entry into the field-specific OU3 database, the information should be corrected on the hard copy form by crossing out the incorrect entry with one line, the individual making the correction will initial and date next to the correction. The corrected form should be scanned and posted to the project-specific FTP site. File names for corrected FSDS forms will include the Index ID of the corrected sample to facilitate document organization (e.g., FSDS_C_P1-12345.pdf). File names for corrected COC forms will include the COC ID of the corrected COC form to facilitate document organization

(e.g., COC_C_OU3-36512.pdf). Necessary data corrections will be made to the master OU3 database by the database manager.

If changes are made to a COC form, the analytical laboratory should be provided with a corrected COC form.

7.0 REFERENCES

Agency for Toxic Substances and Disease Registry. 2006. Asbestos Exposure and Your Health.

RCRA Ground-Water Monitoring: Draft Technical Guidance, November 1992.

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ATTACHMENT 1

OU3 FIELD SAMPLE DATA SHEET (FSDS) FORMS

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Sheet No.: SWS2- _____

LIBBY OU3 PHASE 2 FIELD SAMPLE DATA SHEET (FSDS) rev1
SURFACE WATER AND SEDIMENT

Field Logbook No: _____ Page No: _____

Station ID: _____ Station Type (circle one): Stream Pond Seep Sampling Date: _____

GPS Coordinate System: UTM Zone 11 North, WGS84 datum, meters

X coord: _____m Y coord: _____m

Elevation Coordinate System: WGS84, meters Elevation: _____m

Sampling Team: MWH Sampler Initials: _____

Station Comments: _____

WATER QUALITY PARAMETERS

Time (hh:mm)	Temp. (°C)	pH	Specific Conductance (mS/cm)		Diss. O_2 (mg/L)	ORP (mV)	Turbidity (NTU)	Sampling Depth (ft)	Total Depth (ft)	Discharge (cfs)
			@ Field Temp	@ 25°C						
Water Quality Comments:										

SURFACE WATER AND SEDIMENT SAMPLES

Data Item	Sample 1	Sample 2	Sample 3
Index ID (place pre-printed label in field provided)			
Media (circle one):	Surface Water Sediment	Surface Water Sediment	Surface Water Sediment
Field QC Type (circle one):	FS (field sample) FD (field duplicate) For FD, Parent ID: _____ EB (equip blank) FB (field blank) TB (trip blank) Cooler:_____ PE (perf. eval.) ID:_____	FS (field sample) FD (field duplicate) For FD, Parent ID: _____ EB (equip blank) FB (field blank) TB (trip blank) Cooler:_____ PE (perf. eval.) ID:_____	FS (field sample) FD (field duplicate) For FD, Parent ID: _____ EB (equip blank) FB (field blank) TB (trip blank) Cooler:_____ PE (perf. eval.) ID:_____
Archive Blank (circle)	Yes No	Yes No	Yes No
Sample Time (hh:mm)			
For Sediment:			
Sample Type (circle one):	Grab Composite # of Comp: _____	Grab Composite # of Comp: _____	Grab Composite # of Comp: _____
Sample Depth	Start Depth (in): _____ End Depth (in): _____	Start Depth (in): _____ End Depth (in): _____	Start Depth (in): _____ End Depth (in): _____
Field Comments:			
Cooler:			
Entered by (Provide initials):		**Validated by (Provide initials):**	

For Data Entry Completion (Provide Initials)	Completed by	QC by

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ATTACHMENT 2

OU3 CHAIN OF CUSTODY FORM

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LIBBY OU3 – PHASE II CHAIN-OF-CUSTODY RECORD/REQUEST FOR ANALYSIS

COC No. _____

PAGE: _____ OF: _____

ENTERED BY (Signature): _____ PROJECT MANAGER: _____ DATE: _____

METHOD OF SHIPMENT: _____ CARRIER/WAYBILL NO.: _____ DESTINATION: _____

SAMPLES							ANALYSIS REQUEST																													
							Asbestos			Non-Asbestos (a)																										
Index ID	Date	Time	Media*	Air Volume (L) or Tree Bark Sample Area (cm^2)	Filtered	Archive	TEM-ISO 10312 (b,c)	PLM (d)		TAL Metals+Boron	Mercury	TOC	DOC	Paste pH	Fluoride	Chloride, Sulfate	Total Phosphorus	Cyanide	VPH	EPH	OPP Pesticides	Chlorinated Pesticides	Herbicides	PCBs	SVOCs	VOCs	TDS, TSS, Nitrite, Alkalinity	Ammonia, Nitrate, TKN	Orthophosphate	Radiochemistry	Radium, Uranium	Hardness			Age core (e)	Remarks

TOTAL NUMBER OF CONTAINERS

LABORATORY COMMENTS/CONDITION OF SAMPLES

Cooler Temp:

RELINQUISHED BY:			DATE	TIME	RECEIVED BY:		
SIGNATURE	PRINTED NAME	COMPANY			SIGNATURE	PRINTED NAME	COMPANY

* Media: AQ - Aqueous SO – Solid AA – Ambient Air BK – Tree Bark DB – Organic Debris TC – Tree Age Core

Notes –

(a) Method, container, and preservation details are provided in the attached tables

(b) With Libby-specific modifications. See Phase II OU3 SAP for counting and stopping rules

(c) For tree bark, preparation by TREE-LIBBY-OU3 rev0. For organic debris, preparation by DEBRIS-LIBBY-OU3 rev0

(d) Preparation by ISSI-LIBBY-01 rev8 and analysis by SRC-LIBBY-01 rev2 (PLM-Grav) and SRC-LIBBY-03 rev2 (PLM-VE)

(e) In accordance with procedures in Phipps (1985).

DISTRIBUTION: PINK: Field Copy YELLOW: Laboratory Copy WHITE: Return to Originator

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ATTACHMENT 3

OU3 FIELD MODIFICATION APPROVAL FORM

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FIELD MODIFICATION APPROVAL FORM
LFM-OU3-_____
Libby OU3 Phase II Sampling & Analysis Plan

Requested by:_____ Date:_____

Description of Deviation:

_____
_____
_____
_____
_____
_____
_____
_____
_____
_____
_____

☐ EPA Region 8 has reviewed this field modification approves as proposed.

☐ EPA Region 8 has reviewed this field modification and approves with the following exceptions:

_____
_____
_____
_____
_____
_____
_____

☐ EPA Region 8 has reviewed this field modification and does not agree with the proposed approach for the following reasons:

_____
_____
_____
_____
_____
_____

_____ _____
Bonita Lavelle, EPA RPM Date

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Date: January 31, 2008

OU3 SOP 10 (Rev 1)

Title: FIELD EQUIPMENT CALIBRATION

APPROVALS:

EPA Remedial Project Manager — Date: 3/20/08

SOP Author — Date: 3/20/2008

Revision Number	Date	Reason for Revision
0	9/26/07	
1	1/31/08	Update based on field team input

LIBBY ASBESTOS SUPERFUND SITE OU3

STANDARD OPERATING PROCEDURE No.10

FIELD EQUIPMENT CALIBRATION

1.0 INTRODUCTION

This standard operating procedure (SOP) provides general guidelines on calibration and operating procedures for typical field equipment. This SOP is based on two documents: (1) MWH FMC SOP-01, Field Equipment Calibration, Revision 1.0, March 2004, modified for use at the Libby Asbestos Superfund Site OU3 and (2) USGS Survey Techniques of Water-Resources Investigations, Book 9. Field monitoring instruments are used to measure chemical parameters in situ and when data quality objectives specify screening-level analytical support. Screening-level data are collected for on-site, real-time measurements; evaluation of existing conditions; refinement of sampling locations; and health and safety evaluations. Field measurements are generally used to refine sampling programs and to estimate the extent of contamination at the site. This type of support also provides real-time data for health and safety purposes.

The purpose of this SOP is to define the calibration and operating procedures for equipment used for field monitoring.

2.0 DEFINITIONS

Conductivity: Is a measure of the quantity of electricity transferred across a unit area, per unit potential gradient, per unit time. Conductivity is measured by dipping a probe directly into the water source or into a separate sample aliquot.

Dissolved Oxygen (DO): Is a measure of the quantity of oxygen dissolved in water. DO data is collected in the field using direct measurement probes.

pH: Is measured directly using a probe and is the acidity or alkalinity of a solution; numerically equal to 7 for neutral solutions, increasing with increasing alkalinity and decreasing with increasing acidity.

Temperature: An indicator of the thermal energy contained in a solid or fluid. Units are degrees Centigrade (°C) or degrees Fahrenheit (°F). Temperature measurements are made with a mercury-filled thermometer, bimetallic-element thermometer, or electrical thermistor.

Turbidity: a measure of cloudiness in water due to suspended and colloidal organic and inorganic material. Turbidity is measured by using a field portable nephlometer capable of reading down to 0.1 Nephelometric Turbidity Units (NTU).

3.0 RESPONSIBILITIES

This section presents a brief definition of field roles, and the responsibilities generally associated with them. This list is not intended to be comprehensive and often, additional personnel may be involved. Project team member information will be included in project-specific plans (e.g., work plan, field sampling plan, quality assurance plan, etc.), and field personnel will always consult the appropriate documents to determine project-specific roles and responsibilities. In addition, one person may serve in more than one role on any given project.

Project Manager: Responsible for identifying the appropriate equipment necessary for adequate site characterization and the requirements for the project-specific tasks.

Quality Control Manager: Performs field program audits and ensures project data quality objectives are fulfilled.

Field Team Leader (FTL) and/or Field Geologist, Hydrogeologist, or Engineer: Implements the field program, and supervises other field staff to ensure proper calibration and use of field equipment through the duration of the project.

Field Technician (or other designated personnel): Responsible for providing requested instrumentation and basic instructions on its calibration and use. Assists the FTL with the implementation of tasks and is responsible for regular equipment maintenance and calibration.

4.0 GUIDELINES

This SOP provides a summary of the calibration and operating procedures in accordance with the various manufacturers' instruction manuals, which accompany each piece of equipment. This SOP will be reviewed and used in conjunction with the manufacturer's instruction manual by field team members when using field equipment.

4.1 APPLICABILITY

Field equipment must be kept in designated cases, packaged properly, and secured during transport to prevent equipment damage, which may result in inaccurate readings. Decontaminate and calibrate all equipment prior to use. As part of the calibration process, standard laboratory procedures of decontamination shall be followed; prior to calibration and between calibration buffers, solution vessels and probes shall be rinsed a minimum of three times with distilled/deionized (DI) water and a minimum of one time with the calibration buffer solution or sample solution.

Always calibrate meters according to the manufacturer's instructions before the start of each workday and whenever equipment drift is suspected. Consult the specific instruments' instruction manual for further calibration details.

4.2 pH METERS

Determining pH is critical for predicting and interpreting the reactions and migration of dissolved chemical constituents in groundwater or surface water. Whenever groundwater or surface water samples are collected, pH may be measured using a flow-

through type meter or another type of pH probe. Meters used will have temperature and slope adjustments and a repeatability of ± 0.01 standard pH units.

4.2.1 Calibration: Calibration standard buffers shall not be used beyond the date specified by the manufacturer, and calibration standard buffers shall be stored in a manner that protects the integrity and precision of the solution. Proper decontamination of equipment shall be performed following standard lab practices (refer to section 4.1) prior to calibration.

Two pH standard buffers will be used for calibration of the electrode and are to bracket the anticipated pH of the water samples. For example, if the anticipated pH of sample water is 6, calibration will be conducted with pH 4 and pH 7 buffer solutions; for an anticipated pH of 8, calibrate with pH 7 and pH 10 buffers. Three buffer solutions can be used to calibrate over a larger pH range. Because pH is temperature dependent, buffers and samples should be kept at similar temperature. The temperature of buffer solutions must be known, and temperature-correction factors must be applied before calibration adjustments are made. Theoretically, buffer solutions are stable indefinitely; however, they are susceptible to contamination. Therefore, old, partially full bottles will be replaced and solutions will not be used past the manufacturer's recommended expiration date. The instrument calibration will be checked periodically against a standard solution.

Meters with microprocessors have reliable autocalibration functions and will automatically compensate for buffer temperatures and indicate Nernst slope. For such meters, follow the manufacturer's calibration instructions precisely and completely.

- Check the records of electrode performance before each calibration and sampling event. Electrode response is optimum between approximately 98 percent and 99.5 percent. A slope of 94 percent indicates possible electrode deterioration. Do not use the electrode is the response slope is below 90 percent.
- Calibrate or check the temperature sensor calibration at least annually, and tag the sensor with the date of in-house certification. Do not use the automatic

temperature compensating function of a pH meter if it has not been certified within the past 12 months.

- Record calibration in the instrument log book and on field forms at the time of instrument calibration.

Procedure: Calibration and operating procedures differ with instrument systems—always check the manufacturer's instructions.

1. Equilibrate Equipment to Temperature (this is recommended, even if using an automatic compensating meter).

a. Bring the pH buffers, thermometer (if necessary), container, and electrode to the temperature of the sample.

- To equilibrate to stream temperature, place the buffer bottles in a minnow bucket or mesh bag and suspend them in the stream.

b. Allow 15 to 30 minutes for the buffers to adjust to the sample temperature.

- When making temperature corrections, use the correction factors provided by the buffer manufacturer (temperature coefficients can vary with buffer manufacturer).

2. Inspect the pH Electrode.

a. Check for damage to the electrode bulb, body, or cables.

b. Rinse any precipitate off of the electrode with DI water (the measurement can be affected if precipitate falls into the buffer or sample).

c. Slide the protective sleeve up or down to uncover the filling hole.

d. Gently shake or tap the electrode to dislodge and remove air bubbles trapped in the sensing tip of the electrode and to remove excess deionized water. Do not wipe the electrode.

3. Calibration Rinse.

a. Rinse the electrode, thermometer or automatic temperature compensating (ATC) sensor, and a container large enough to hold the sensors and buffer with pH buffer 7. Discard the used pH buffer into a waste container.

b. Pour fresh pH 7 buffer into the buffer-rinsed container that holds the electrode and thermometer. Allow the instruments to equilibrate for 1 minute (if necessary), then discard the buffer into a waste container.

4. Calibration.

a. Pour fresh pH 7 buffer into the container that holds the electrode and thermometer or ATC sensor.

- The bulb of the pH electrode must not touch the bottom or side of the container.
- Add enough pH buffer to cover the reference junction.

b. Swirl the sample gently or stir carefully with the electrode. If using a magnetic stirrer, stir slowly enough so that a vortex is not created. Place a thin piece of insulating material (styrofoam or cardboard) between the magnetic stirrer and beaker to prevent transfer of heat to the buffer solution.

c. Measure the temperature of the buffer solution; remove the thermometer (it is not necessary to remove the ATC sensor).

d. Determine the theoretical pH of the buffer from the temperature-correction tables.

e. Note and record the pH temperature readings. Adjust the meter reading to the pH value using the "standardize" function on the meter (usually a knob or pressure pad). Record the adjusted pH value for the 7.0 buffer and associated millivolt reading.

f. Remove the electrode and ATC sensor (some instruments require that the meter be switched to the standby or off position before removing the electrode from the solution).

- Repeat the calibration steps using fresh portions of reference buffer solution until two successive readings are obtained at the adjusted pH value for pH 7 buffer without further adjustment to the system.

- Discard the used pH 7 buffer into a waste container.

5. Slope Adjustment Rinse.

a. Rinse the electrode and thermometer or ATC sensor thoroughly with DI water.
b. Rinse a clean container, electrode, and thermometer with a second buffer (usually pH 4 or 10) that brackets the expected pH value of the sample; discard the used buffer into a waste container.
c. Pour the second buffer into a container holding the electrode and thermometer or ATC sensor. Allow the temperature to equilibrate for 1 minute, then discard the used buffer into a waste container.

6. Slope Adjustment. ***Ncte: For most modern meters, this step is automated—if so, skip to step seven.

a. Pour a fresh portion of the second pH buffer into a container holding the electrode and thermometer or ATC sensor.
b. Stir slowly (no vortex) or swirl manually. Follow the directions in 4b, above.

c. Measure the temperature and pH of the buffer solution and check the pH value of the buffer on temperature coefficient tables. Record the pH and temperature readings.
d. Adjust the slope to the value of the second pH buffer at known temperature. (Some meters have separate slope-adjustment knobs, pressure pads, or other devices, whereas others have to be adjusted by use of a temperature knob.) Record the adjusted pH value and associated millivolt reading.
e. Discard the used buffer into a waste container.
f. Repeat steps 6(a) through 6(e) using successive portions of the buffer solution until two successive readings are obtained without further adjustment.

7. Rinse the electrode and thermometer or ATC sensor thoroughly with DI water.

8. If using a non-compensating or non-automated meter, repeat the calibration rinse (step 3) and calibration procedures [steps 4(a) through 4(d)] to ensure that the slope adjustments did not affect the calibration adjustment.

- This step is a check only; no adjustment should be needed, but the result should be recorded. If adjustment is needed, repeat the entire calibration procedure.
- If adjustment is still needed, a systematic problem is likely (see 6.4.4). Inspect the instrument system, clean the electrode or add filling solution, or use a spare electrode or meter.

9. Calibration Check Rinse.

a. Rinse the electrode and thermometer or ATC sensor with DI water.

b. Rinse another clean container, electrode, and thermometer with a third buffer (pH 4 or 10) and discard the used buffer into a waste container.

c. Pour the third buffer into a container holding the electrode and thermometer or ATC sensor. Allow the temperature to equilibrate for 1 minute, then discard the used buffer into a waste container.

10. Calibration Range Check.

a. Pour a fresh portion of third pH buffer into a container holding the electrode and thermometer or ATC sensor.

b. Stir without forming a vortex or swirl slowly (see step 4b).

c. Measure the temperature of the buffer solution (remove the liquid-filled thermometer and check the temperature-adjusted pH value), if necessary for the meter being used.

d. The pH instrument system should read the value of the third buffer at a known temperature within ±0.1 pH units.

- Meters reading to three or more places to the right of the decimal may not provide better accuracy than ±0.05 units, and their accuracy must be verified.

- If it checks, the instrument system is calibrated over a range of pH 4 to 10 and is ready for ANC or alkalinity titrations as well as pH measurement.
- If the instrument system does not check over the entire range, recalibrate before measuring the sample pH. Recalibrate before an alkalinity/ANC titration if the sample has a pH greater than 7.0.

e. Discard the used buffer into a waste container.

f. Rinse the electrode and thermometer (or ATC sensor) with DI water

4.2.2 Measurement

It is generally preferable to measure pH in situ rather than on a sample taken from a splitter or compositing device. If stream conditions are such that water would pass the in situ pH sensor at a very high rate of flow, however, streaming-potential effects could affect the accuracy of the measurement. For such conditions, it is preferable to withdraw a discrete sample directly from the stream or compositing device and use the sub-sample measurement procedures described below. The pH instrument system should be set up on board the boat or on-shore so that pH is measured at the time of sample collection.

The pH of a water sample can change significantly within hours or even minutes after sample collection as a result of degassing (such as loss of carbon dioxide, hydrogen sulfide, and ammonia); mineral precipitation (such as formation of calcium carbonate); temperature change; and other chemical, physical, and biological reactions. The electrometric method of pH measurement described below applies to filtered or unfiltered surface water and ground water, from fresh to saline.

Field conditions, including rain, wind, cold, dust, and direct sunlight can cause measurement problems. To the extent possible, shield the instrument and measurement process from the effects of harsh weather.

Record the pH variation from a cross-sectional profile, if possible, to determine if pH is uniform at any given discharge, and select the sampling method appropriate for study objectives.

- To sample a shallow flowing stream, wade to the location where pH is to be measured.
- To sample a stream or river too deep to wade, lower a weighted pH sensor with a calibrated temperature sensor (if needed) from a bridge, cableway, or boat. Do not attach the weight to a sensor or sensor cables.
- To sample under still water conditions, measure pH at multiple depths at several points in the cross-section.

Procedure:

1. Ensure that the instrument is calibrated.
2. Immerse the pH probe in the water to the correct depth and hold it there for at least 60 seconds to allow for temperature equilibration.
3. Record the pH and temperature values without removing the sensor from the water.
   - Values generally stabilize quickly within ±0.05 to 0.1 standard pH unit, depending on the instrument system.
   - Record the median of the observed values.
   - If readings do not stabilize after extending the measurement period, note this on the field forms along with the pH readings, and record the median value of the last five or more readings.
   - After measurements are completed, rinse the pH probe with deionized water and continue with further sampling.
4. For EWI (Equal Width Increment) or EDI (Equal Depth Increment) measurements—Proceed to the next station in the cross section. Repeat steps 3 through 5. After all stations in the cross section have been measured, rinse the sensors with deionized water and store them.
5. Record the mean or median stream pH on the field forms
   - **In still water**—median of three or more sequential values.

- **EDI**—mean value of all subsections measured (use the median if measuring one vertical at the centroid of flow).
- **EWI**—mean or median of all subsections measured.

4.3 SPECIFIC CONDUCTANCE METERS

Specific conductance is used as an indicator of water quality. It is a simple indicator of the change within a system and provides useful information for site characterization. Any meter used to collect field specific conductance measurements will be equipped with a temperature compensator, and read directly in micromhos per centimeter (µmhos/cm) corrected to 25°C. The meter will be calibrated to record values over the anticipated range of conductivity values during measurement.

4.3.1 Calibration

Reagent-grade potassium chloride (KCl) will be used for the calibration of specific conductance equipment. Calibration standards will not be used beyond the date specified by the manufacturer. Consult the manufacturer's instruction manual for further details. Specific conductance readings will be reported on the field logs in micromhos/centimeter (µm/cm) or millimhos/cm. The instrument calibration will be checked before every water-quality field trip and periodically throughout the sampling event against a standard solution of KCl.

Procedure: Calibration and operating procedures differ with instrument systems—always check the manufacturer's instructions.

1. Inspect the instrument and the conductivity sensor for damage. Check the battery voltage. Make sure that all cables are clean and connected properly.
2. Turn the instrument on and allow sufficient time for electronic stabilization.
3. Select the correct instrument calibration scale for expected conductivity.

4. Select the sensor type and the cell constant that will most accurately measure expected conductivity.

5. Select two conductivity standards that will bracket the expected sample conductivity. Verify that the date on the standards has not expired.

6. Equilibrate the standards and the conductivity sensor to the temperature of the sample.

   a. Put bottles of standards in a minnow bucket, cooler, or large water bath that is being filled with ambient water.

   b. Allow 15 to 30 minutes for thermal equilibration. Do not allow water to dilute the standard.

7. Rinse the conductivity sensor, the thermometer (liquid-in-glass or thermistor), and a container large enough to hold the dip-type sensor and the thermometer.

   a. First, rinse the sensor, the thermometer, and the container three times with deionized water.

   b. Next, rinse the sensor, the thermometer, and the container three times with the standard to be used.

8. Put the sensor and the thermometer into the rinsed container and pour in fresh calibration standard.

9. Measure water temperature. Accurate conductivity measurements depend on accurate temperature measurements or accurate temperature compensation.

   a. If the sensor contains a calibrated thermistor, use this thermistor to measure water temperature.

   b. If using a manual instrument without a temperature display or temperature compensation, adjust the instrument to the temperature of the standard using a calibrated liquid-in-glass or a thermistor thermometer.

10. Agitate a submersible-type conductivity sensor up and down under the solution surface to expel air trapped in the sensor. Read the instrument display. Agitate the sensor up and down under the solution surface again, and read the display. Repeat the procedure until consecutive readings are the same.

11. Record the instrument reading and adjust the instrument to the known standard value.

    a. For non temperature-compensating conductivity instruments, apply a temperature-correction factor to convert the instrument reading to conductivity at 25°C.

    b. The correction factor depends to some degree on the specific instrument used—use the temperature-correction factor recommended by the manufacturer.

    c. If an instrument cannot be adjusted to a known calibration standard value, develop a calibration curve. After temperature compensation, if the percentage difference from the standard exceeds 5 percent, refer to the instrument troubleshooting guide.

12. Record in the instrument log book and on field forms:

    a. The temperature of the standard solution.

    b. The known and the measured conductivity of the standard solution (including ± variation).

    c. The temperature-correction factor (if necessary).

13. Discard the used standard into a waste container. Thoroughly rinse the sensor, thermometer, and container with deionized water.

14. Repeat steps 7 through 13 with the second conductivity standard.

    a. The purpose for measuring a second standard is to check instrument calibration over the range of the two standards.

    b. The difference from the standard value should not exceed 5 percent.

    c. If the difference is greater than 5 percent, repeat the entire calibration procedure. If the second reading still does not come within 5 percent of standard value, refer to a troubleshooting guide or calibrate a backup instrument.

    d. Switching instrument calibration scales could require re-calibration.

15. Record in the instrument log book and on field forms the calibration data for the second standard.

4.3.2 Measurement

In situ measurement is preferred for determining conductivity of surface water. Conductivity measurements should be performed immediately upon sample collection at the field site.

Field conditions (rain, wind, cold, dust, direct sunlight) can cause measurement problems—Shield the instrument to the extent possible and perform measurements in a collection chamber in an enclosed vehicle or an on-site laboratory.

For waters susceptible to significant gain and loss of dissolved gases, make the measurement within a gas-impermeable container (Berzelius flask) fitted with a stopper—Place the sensor through the stopper and work quickly to maintain the sample at ambient surface-water or ground-water temperature.

Avoid contamination from the pH electrode filling solution—Measure conductivity on a separate discrete sample from the one used for measuring pH; in a flowthrough chamber, position the conductivity sensor upstream of the pH electrode.

The conductivity measurement reported must account for sample temperature. If using an instrument that does not automatically temperature compensate to 25°C, record the uncompensated measurement in your field notes, along with the corrected conductivity value. Use correction factors supplied by the instrument manufacturer.

Conductivity measurements in flowing surface water should represent the cross-sectional mean or median conductivity at the time of observation. Any deviation from this convention must be documented in the data base and with the published data.

Before beginning, take a cross-sectional conductivity profile to determine the degree of system variability if feasible. A submersible sensor works best for this purpose.

Procedure:

1. Calibrate the conductivity instrument system at the field site after equilibrating the buffers with stream temperature.

2. Record the conductivity variation from a cross-sectional profile on a field form and select the sampling method.

- Flowing, shallow stream—wade to the location(s) where conductivity is to be measured.
- Stream too deep or swift to wade—lower a weighted conductivity sensor from a bridge, cableway, or boat. Do not attach weight to the sensor or the sensor cable.
- Still-water conditions—measure conductivity at multiple depths at several points in the cross section.

3. Immerse the conductivity and temperature sensors in the water to the correct depth and hold there (no less than 60 seconds) until the sensors equilibrate to water conditions.

4. Record the conductivity and corresponding temperature readings without removing the sensors from the water.

- Values should stabilize quickly to within 5 percent at conductivity ≤100 µS/cm and within 3 percent at conductivity >100 µS/cm.
- Record the median of the stabilized values on field forms.
- If the readings do not meet the stability criterion after extending the measurement period, record this difficulty in the field notes along with the fluctuation range and the median value of the last five or more readings.

5. For EWI or EDI measurements, proceed to the next station in the cross section and repeat steps 3 and 4. Record on field forms the mean (or median, if appropriate) value for each subsection measured.

6. When the measurement is complete, remove the sensor from the water, rinse it with deionized water, and store it.

7. Record the stream conductivity on the field forms:

- **In still water**—median of three or more sequential values.

- **EDI**—mean value of all subsections measured (use the median if measuring one vertical at the centroid of flow).
- **EWI**—mean or median of all subsections measured (see NFM 6.0).

Sub-sample measurement

Representative samples are to be collected and split or composited according to approved USGS methods. Measure the conductivity of samples as soon as possible after collection. If the sample cannot be analyzed immediately, fill a bottle to the top, close it tightly, and maintain the sample at stream temperature until measurement.

Reported conductivity values normally are determined on an unfiltered sample. Large concentrations of suspended sediment can be a source of measurement error—record such conditions in the field notes.

- If sediment concentrations are heavy, measure conductivity on both unfiltered and filtered sub-samples and record both values on the field form.
- If the conductivity value differs significantly between the filtered and unfiltered samples, report the filtered value as sample conductivity and identify it as a "filtered sample."

1. Calibrate the conductivity instrument system at the field site.

2. Select the sampling method (see NFM 6.0) and collect a representative sample.

3. Withdraw a homogenized sub-sample from a sample splitter or compositing device. Rinse the sample bottles three times with the sample—rinse them with sample filtrate, for filtered samples.

4. Rinse the conductivity sensor, the thermometer (liquid-in-glass or thermistor), and a container large enough to hold the dip-type sensor and the thermometer.

   a. First, rinse the sensor, the thermometer, and the container three times with deionized water.

b. Next, rinse the sensor, the thermometer, and the container using sample water.

5. Allow the sensors to equilibrate to sample temperature, then discard the used sample water. Pour fresh sample water into a container holding the sensor and the thermometer. When using a dip-type sensor, do not let the sensor touch the bottom or sides of the measuring container.

6. Measure water temperature.

- If the conductivity sensor contains a calibrated thermistor, use this thermistor to measure water temperature.
- If the instrument is not temperature compensating, use a calibrated thermistor or a liquid-in-glass thermometer.
- Adjust the instrument to the sample temperature (if necessary) and remove the thermometer.

7. Measure conductivity.

a. Remove any air trapped in the sensor by agitating the sensor up and down under the water surface.

b. Read the instrument display.

c. Agitate the sensor up and down under the water surface, and read the display again.

d. Repeat the procedure until consecutive readings are the same.

8. Record the conductivity and the sample temperature on field forms.

- If the instrument is not temperature compensating, record the raw data and convert the values to conductivity at 25°C using temperature-correction factors provided by the manufacturer.
- Report the median of the readings to three significant figures on the field forms.
- Discard the sample into a waste container and dispose according to regulations.

9. Quality control-

- Repeat steps 3 through 8 with at least two fresh subsamples, rinsing the instruments once only with sample water.
- Subsample values should be within ±5 percent for conductivity ≤100 µS/cm, or ±3 percent for conductivity >100 µS/cm.
- If criteria cannot be met: filter the samples, report the median of 3 or more samples, and record this difficulty in field notes.

10. Rinse the sensor, the thermometer, and the container with deionized water. If another measurement is to be made within the next day or two, store the sensor in deionized water. Otherwise, store the sensor dry.

4.4 TURBIDITY METERS

Turbidity meters measure the amount of light scattered at right angles from a beam of light passing through the test sample. Turbidity readings are the measure of the interaction of light with suspended solid particles in the sample. Test results are read directly in Nephelometric Turbidity Units (NTUs) on an LCD digital readout.

4.4.1 Calibration

The turbidity meter is pre-calibrated in the factory, and a simple standardization is the only step required prior to testing.

4.4.2 Measurement

Turbidity measurements should be repeated three to five times to ensure accuracy and replication within the precision of the instrument.

Benchtop determination of turbidity is especially susceptible to negative bias from particle settling. Visually check for the presence of coarse material (sand or coarse silt) in the sample. Gently agitate the sample, then set it down. If particles rapidly settle to the

bottom (within 3-5 seconds), then coarse materials are present and the sample cannot be measured accurately using the static method. Static measurements made on such samples therefore must be coded to indicate that accuracy is qualified when being entered into a database.

Procedure:

Preliminary steps for benchtop turbidity determination:

1. Warm up the turbidimeter according to the manufacturer's instructions. Put on powderless laboratory gloves.

2. Rinse a clean, dry, scratch-free, index marked cell with a turbidity calibrant within the range of interest.

3. Gently agitate the calibrant, pour the calibrant into the sample cell to the fill mark, and dry the cell exterior with a lint-free cloth. When using a meter recently calibrated with an acceptable calibrant turbidity solution (formazin or styrene-divinylbenzene polymer), a verification calibrant may be used for this check measurement.

4. Follow the manufacturer's instructions for readout of turbidity value and record the turbidity of the calibrant used and the turbidity value measured in the calibration logbook. If readings are not within specifications for the indicated range, recalibrate the instrument for the turbidimeter using accepted calibration turbidity solutions.

For samples with turbidity less than 40 turbidity units:

1. Measure sample turbidity immediately or as soon as possible upon sample withdrawal.

    a. If discrete sub-samples are to be taken from a churn splitter or other sample-compositing device, remove samples for turbidity measurement along with other whole water samples. Avoid pouring the sample into a cuvette from a bottle, if possible. If not possible, then invert the bottle 25 times using a 1-second inversion cycle and pour off the sample immediately to capture suspended particles.

b. For drinking water, use an instrument that complies with EPA Method 180.1 or GLI Method 2. Measurements are reported in NTU or NTRU for EPA 180.1, or in FNMU for GLI Method 2.

2. Rinse a freshly cleaned cell with the sample to be tested.

3. For a discrete (static) sample, complete the following sequence of steps (through step 4a) without hesitation (skip to step 4 for flowthrough cell measurement).

a. Gently invert—do not shake—the sample 25 times to completely disperse the solids, taking care not to entrain air bubbles. Allow air bubbles to disappear before filling the sample cell.

b. Rapidly pour the sample into a sample cell to the line marked (to the neck if there is no line). Do not touch cell walls with fingers.

c. Remove condensation from the cell with a clean, soft, lint-free cloth or tissue. If condensation continues, apply a thin coating of silicon oil to the outside of the cell about every third time the cell is wiped dry of moisture. Allow samples to equilibrate to ambient temperature, if necessary, before sub-sampling to help minimize condensation problems. Note: warming the sample may change particle associations in the water matrix.

d. Before inserting the sample cell into the meter, ensure that no air bubbles are present in the cell. If necessary, degas the sample according to the manufacturer's instructions. Air bubbles can cause significant positive bias in turbidity measurements.

e. Orient the calibration cell in the cell holder according to the index marks—the calibration cell and sample cell must have identical orientation when in the instrument measurement chamber.

4. Determine the measured turbidity value of the sample directly from the instrument scale or by using the instrument value and calibration curve, as is appropriate for the

instrument being used. For samples with less than 1 turbidity unit, see the Technical Note under step 4d.

a. Record the very first readings after placement of the sample cell in the measurement chamber. If readings are unstable, then particle settling may be occurring. If so, gently re-invert the cell 25 times and record at least three readings over a short, defined time interval (for example, 30 seconds to 1 minute).

b. Repeat at least twice with fresh sample, until three or more sample values fall within ±10 percent.

c. Samples that contain significant color should be diluted if using EPA Method 180.1 (for samples with turbidity greater than 40 units see below "For samples, including drinking water, with turbidity greater than 40 turbidity units," step 3). Results of diluted samples must be qualified with a "d" in the "Value Qualifier Code" field for data entered into the USGS NWIS database.

d. Report the median of the three or more sequential readings that fall within ±10 percent.

Technical Note: When using low-level reporting scales, you may need to subtract a correction factor from the reading to correct for stray light. For example, the Hach Company reports the correction for the 0.2-NTU scale to be on the order of 0.04 NTU for the Hach 2100P. The stray-light correction is determined by reading turbidity from an empty instrument (without cuvette).

5. Record the data. If particle settling or instability in initial readings was a problem, add documentation to field notes in the log book.

For samples, including drinking water, with turbidity greater than 40 turbidity units:

1. Select an appropriate instrument.

- For drinking water, use EPA Method 180.1, a compliant instrument, and NTU or NTRU reporting units; alternatively, select the GLI Method 2, a compliant instrument, and FNMU reporting units. Reporting units for these methods must be remarked with an "E" code in NWIS for turbidities greater than 40.
- For study objectives other than drinking water, choose instruments according to study objectives.

2. Obtain a discrete sample.

- For drinking-water samples, proceed to step 3.
- For non-drinking-water samples, skip to step 4.

3. For drinking-water samples, dilution is required to comply with USEPA regulations.

a. Dilute the sample with one or more equal volumes of turbidity-free water until turbidity is less than 40 turbidity units after mixing and degassing.

b. Record the volume of turbidity-free water used for dilution. Follow steps 1-5 from the previous section for samples with turbidity less than 40 turbidity units.

c. Skip to step 5, below

4. For non-drinking-water samples (where USEPA compliance is not required), with 100 and 1,000 turbidity-unit ranges only — place a cell riser (if available) into the cell holder before inserting the sample cell. This decreases the length of the light path in order to improve the linearity of measurements. Do not use the cell riser for the lower turbidity ranges.

a. For turbidimeters with adjustable ranges and signal-processing capabilities (for instance, ratio mode to compensate for high particle densities), select the desired configuration and operate according to manufacturer's recommendations. Some instruments will automatically switch to different modes (for example, ratio mode) or to a different light source. Record instrument mode on field sheets.

b. Select the desired range on the turbidimeter.

5. Fill the cell with sample water:

a. Hold the cell by the rim (top lip), not beneath the lip.

b. Gently agitate the sample 25 times. Without hesitation, carefully but rapidly pour sample water into the cell to the fill mark.

c. Wipe the exterior of the cell using a soft, lint-free cloth or tissue to remove moisture (condensation) from cell walls.

d. If necessary, apply a thin layer of silicon oil onto the exterior of the cell to reduce condensation on the cell and mask slight scratches and nicks.

e. If rapid particle settling is occurring, steadily invert the cell 25 times, taking care not to shake too vigorously, which could entrain gases in the sample.

6. Record the sample turbidity.

Most modern turbidimeters will adjust initial sample readings directly into a final reading based on the previous calibration. If the meter does not have this capability, you will need to read values from a calibration curve constructed previously.

a. Record the very first readings after placement of the sample cell in the measurement chamber. If readings are unstable, particle settling may be occurring: gently re-invert the cell 25 times and record at least three readings over a defined time interval (for example, 30 seconds to 1 minute).

b. Repeat at least twice with fresh sample until three or more sample values fall within ±10 percent.

c. Samples that contain significant color should be diluted if using EPA Method 180.1. Results of diluted samples must be qualified with a "d" in the "Value Qualifier Code" field for data entered into the USGS NWIS database.

d. Report the median of the three or more sequential readings that fall within ±10 percent.

- For diluted water samples, the measured turbidity must be converted based on the amount of dilution, according to the following equation:

$$T_s = T_d \times (V_o+V_s)/V_s$$

where *T*s= turbidity of the environmental sample, *T*d=turbidity of the diluted sample, *V*o = volume of turbidity-free water in the diluted mixture, and *V*s= volume of the environmental sample in the diluted mixture.

EXAMPLE: If five volumes of turbidity-free water were added to one volume of sample, and the diluted sample showed a turbidity of 30 units, then the turbidity of the original sample is computed as 180 units.

e. Report turbidity as follows, using method codes as described in http://water.usgs.gov/owq/turbidity_codes.xls:

- For EPA Method 180.1, use NTU or NTRU.
- For GLI Method 2, use FNMU.
- For non-diluted, non-USEPA-compliant measurements, use the appropriate reporting units.

4.5 DISSOLVED OXYGEN METERS

Dissolved oxygen (DO) meters measure the quantity of oxygen dissolved in water. In a typical DO meter, the tip of the probe consists of a cell enclosed by a selective membrane in a protective holder containing the electrolyte and electrodes.

4.5.1 Calibration

Always calibrate the instrument according to the manufacturer's specifications. For an accurate calibration, the probe may require immersion in water in an airtight container. If an open container is used for calibration, the margin of error is approximately 0.1 ppm. If the calibration is performed above sea level, a correction will be made for the difference in altitude. Certain table listings are available for oxygen solubility as a function of temperature and salinity. Refer to the manufacturer's specifications regarding slope calibration.

Instrument systems for the amperometric or the luminescent-sensor methods must be properly calibrated and tested before each field trip and cleaned in the field after each use.

- *Amperometric instruments*

  Different manufacturers recommend different calibration frequencies for membrane-electrode DO meters; however, virtually all state that optimum instrument performance and data quality will be obtained by frequent calibration. Calibration and operation procedures for the amperometric method differ among instrument types and makes—refer to the manufacturer's instructions.

- *Luminescent-sensor instruments*

  Luminescent-based sensors are pre-calibrated by the manufacturer and most manufacturers' literature suggests that no further calibration is warranted. The accuracy of factory calibrations, however, may not satisfy the data-quality objectives of a specific program. Frequency of calibration can have a significant effect on the overall accuracy and precision of DO measurements; therefore, users of these meters are advised to make frequent calibration checks and to recalibrate as frequently as required to meet specific data-quality objectives.

- *One-point and two-point calibrations*

  Calibration for most amperometric DO instruments and some luminescent-sensor instruments can only be checked with a 1-point calibration at 100-percent saturation. For these instruments, a zero DO check should be performed routinely as an evaluation of sensor performance. Because the sensors on DO instruments may be slow to respond after the zero check, the sensor should be thoroughly rinsed with deionized water before use.

Some instruments allow for 2-point calibrations at 0-percent and 100-percent saturation. Follow the manufacturer's instructions for those instruments with 2-point calibration functionality. Verifying instrument performance at zero DO and using a 2-point calibration can be particularly important for data accuracy when the instrument will be used to measure low DO concentrations (less than 5 mg/L).

- *Correction for atmospheric pressure*

Atmospheric pressure, the temperature of the water or water vapor, and the conductivity (or salinity) of the water must be known to determine the theoretical amount of oxygen that can be dissolved in water. Record all calibration information in instrument log books and copy calibration data onto field forms at the time of calibration.

Ambient atmospheric pressure is true atmospheric pressure at the measurement site, not that which has been adjusted to sea level. Atmospheric pressure reported by the National Weather Service generally is not the true (ambient) value. Weather Service atmospheric readings usually are adjusted to sea level and must be adjusted back to the elevation of the weather station. Upon request, a weather station may provide ambient atmospheric pressure.

- Use a calibration-checked pocket altimeter-barometer to determine ambient atmospheric pressure to the nearest 1 millimeter (mm) of mercury.
- Check the accuracy of all field barometers before each field trip, and record readings and adjustments in the log book. If possible, check barometer accuracy with information from an official weather station.
- Use Table 1 and Figure 1 if the value used for atmospheric pressure has been adjusted to sea level.
- To correct weather station readings adjusted to sea level to ambient atmospheric pressure: subtract appropriate values shown (Table 1, Figure 1) from atmospheric readings adjusted to sea level (shown in millimeters of mercury).

Although atmospheric pressure does not decrease linearly with increases in elevation, linear interpolation is acceptable within the elevation ranges given in Table 1. Alternatively, plot the values from Table 1 and extrapolate subtraction factors directly from the graph (Figure 1). Many instruments have the pressure-temperature algorithm stored in internal memory. Interactive tables also are available for user-specified temperature, pressure, and salinity at http://water.usgs.gov/software/dotables.html.

Table 1. Factors used to correct atmospheric pressures adjusted at sea level NGVD, National Geodetic Vertical Datum of 1929

Elevation of weather station (in feet, NGVD)	Value to subtract (mm of Hg)
0	0
1000	27
2000	53
3000	79
4000	104
5000	128
6000	151

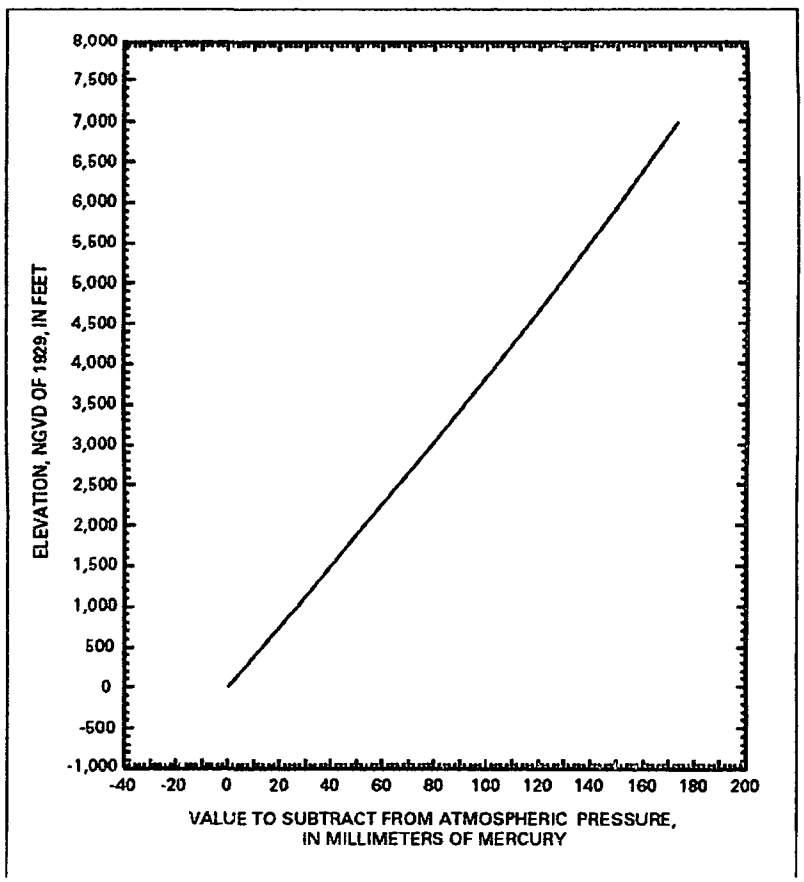

Figure 1. Factors used to correct atmospheric pressures adjusted to sea level.

Procedure 1—Air-calibration chamber in air

This calibration method is most commonly recommended by manufacturers of amperometric instruments. Calibration chambers are either built into the instrument case or are provided as separate components by the manufacturer. Use the calibration chamber provided or recommended by the manufacturer.

1. Wet the inside of the calibration chamber with water. Then pour out the water (but leave a few drops). Remove any water droplets on the sensor membrane and insert the sensor into the chamber (this ensures 100-percent humidity).

2. If using an amperometric instrument, allow 10 to 15 minutes for the DO sensor and the air inside the calibration chamber to equilibrate.

3. Using your calibration pocket altimeter-barometer, read the ambient atmospheric pressure checked to the nearest 1 mm of mercury.

4. Measure the temperature in the calibration chamber and observe the readings until the instrument stabilizes. Read the temperature to the nearest 0.1°C. The temperature inside the chamber should approximate the water temperature, measured with a calibrated thermometer.

> **Technical Note for Amperometric Instruments:** Most instrument manufacturers recommend calibrating at temperatures that are at least within 10°C of the ambient water temperature. The most accurate calibration will be achieved if the temperature difference between the environmental water and the calibration chamber is minimized as much as possible.

5. Use Table1 to determine the DO saturation at the measured temperature and atmospheric pressure.

6. Following the manufacturer's instructions, adjust the calibration control until the instrument reads the DO saturation value determined from Table 1. Verify that the instrument reading is within ±0.2 mg/L of the computed saturation value, or use more stringent accuracy criteria that reflect the data-quality requirements of the study. The luminescent-sensor instrument is now calibrated and ready for use.

7. When working with an amperometric instrument, remove the sensor from the calibration chamber and check to see if any water droplets are on the membrane. Water droplets on the membrane cause improper calibration. If water droplets are present, recalibrate the instrument; otherwise the instrument is now calibrated and ready for use.

Procedure 2—Calibration with air-saturated water

In this procedure, the DO sensor or instrument system is calibrated against water that is saturated with oxygen at a known temperature and ambient atmospheric pressure.

1. The temperature of water used for calibration should be about the same as the temperature and conductivity of the water to be measured.

- **If working at the field site**—obtain about 1 liter (L) of water from the water body to be measured.
- **If working in the laboratory**—obtain about 1 L of deionized water or tap water.

2. Place the DO sensor and calibration water in a large beaker or open-mouth container. (Some manufacturers supply an air-saturated water-calibration vessel.)

- Allow the sensor to come to thermal equilibrium with the water temperature.
- Shield the beaker or container from direct sunlight and wind to minimize temperature variations.

3. Aerate the water for 5 to 10 minutes. Using a battery-operated aquarium pump or minnow-bucket aerator and a short piece of tubing, attach a gas diffusion stone to the end of the tubing and place it at the bottom of the beaker of calibration water. Avoid placing the instrument in the stream of air bubbles.

4. Determine if the water is 100-percent saturated with oxygen.

- Observe the instrument reading while aerating the calibration water.
- When no change in the DO reading is observed on the instrument for 4 to 5 minutes, assume that the water is saturated.

5. Using your pocket altimeter-barometer, read the ambient atmospheric pressure to the nearest 1 mm of mercury.

6. Read the temperature of the calibration water to the nearest 0.1°C.

7. Using oxygen solubility Table 1, determine the DO saturation value at the measured temperature and atmospheric pressure of the calibration water.

*****Skip to Step 9 if using an amperometric instrument*****

8. **For luminescent-sensor instruments:** Following the manufacturer's instrument calibration instructions, verify that the instrument reading is within ±0.2 mg/L of the computed saturation value. (Alternatively, apply a more stringent accuracy criterion that reflects study data-quality requirements.) **The luminescent-sensor instrument is now calibrated and ready for use.**

9. **For amperometric instruments:** Adequate flow of water across the surface of the membrane is required for accurate measurements. Recommendations for flow velocity vary by manufacturer, with most recommending about 1 foot per second (ft/s).

- Provide suitable turbulence in the air-saturated water by physical or mechanical means to maintain the required flow rate past the membrane, avoiding the creation of air bubbles at the water-sensor interface.
- Maintain this flow rate when making measurements and adjusting instrument calibration.

10. **For amperometric instruments**: Turn off the aerator and take care to prevent any air bubbles from adhering to the membrane. Following the manufacturer's instructions, set or adjust the calibration control until the instrument reads a saturation value of DO as determined above. Verify that the instrument reading is within ±0.2 mg/L of the computed saturation value, or use more stringent accuracy criteria that reflect the data-quality objectives of the study.

Procedure 3—Air-calibration chamber in water

This calibration method is applicable only to amperometric instruments. An air-calibration chamber permits calibration of the DO sensor at the temperature of the water in which the DO concentration is to be measured. This calibration procedure minimizes errors caused by temperature differences. Air-calibration chambers for in-water

calibrations currently are not available on the open market and one of the most common, the YSI 5075A calibration chamber, is no longer manufactured. For most multi-parameter water-quality instruments, the manufacturer-provided ground-water flow cell may be modified and used as an air-calibration chamber in water. The modification requires the cell to be mounted on the sonde with one port of the cell plugged and the other port vented to the atmosphere with tubing.

1. Insert the sensor probe into the rings of the DO wand and dip this calibration chamber into the surface or ground water to be measured, allowing the temperature readings to stabilize. Remove the wand and pour out the excess water, leaving a few drops.

- Check for and remove any water droplets on the sensor membrane.
- Insert the DO sensor into the wet chamber (this ensures 100 percent humidity).
- If a YSI model 5739 sensor is used, the pressure-compensating diaphragm on the side of the sensor must be enclosed within the calibration chamber during calibration.
- Check that no water can leak into the calibration chamber and that the membrane does not have droplets of water adhering to it. The water droplets reduce the rate of oxygen diffusion through a membrane, producing erroneous results.

2. Immerse the calibration chamber into the water to be measured. Allow 10 to 15 minutes for the air temperature inside the chamber to equilibrate with the water (see the **Technical Note** in Procedure 1).

- For streams, choose an area of the stream that closely approximates mean stream temperature. In shallow streams, try to place the chamber in an area that represents the stream but that is shaded from direct sunlight.
- For ground water, use temperature-stabilized purge water or other clean water having a temperature that closely approximates that of the ground water.

3. Using a calibration-checked pocket altimeter-barometer, determine the ambient atmospheric pressure to the nearest 1 mm of mercury.

4. Read the temperature within the chamber to the nearest 0.1°C, using a calibrated thermometer.

- The temperature inside the chamber should approximate the water temperature.
- If the two temperatures do not match, allow additional time for equilibration of the chamber with the water temperature.
- If the temperature of the chamber still does not approximate the water temperature, the thermistor in the DO sensor might be malfunctioning. Compare water temperature measured by the DO meter and a calibrated field thermometer. If the two measurements vary by more than ±0.2°C, the calibration should be discontinued and the DO meter thermistor should be repaired following the manufacturer's recommendations.

  Technical Note: Most instrument manufacturers recommend calibrating at temperatures that are at least within 10°C of the ambient water temperature. The most accurate calibration will be achieved if the temperature difference between the environmental water and the calibration chamber is minimized as much as possible.

5. Use Table 1 to determine the DO saturation value at the measured water temperature and atmospheric pressure.

6. Following the manufacturer's instructions, set or adjust the calibration control until the instrument reads a DO saturation value determined from oxygen solubility (Table 1). Verify that the instrument reading is within 0.2 mg/L of the computed saturation value, or use more stringent accuracy criteria per the data-quality objectives of the study. The instrument is now calibrated and ready for use. Remove the sensor from the calibration chamber.

4.5.2 Measurement

The solubility of oxygen in water depends on the partial pressure of oxygen in air, the temperature of the water, and the dissolved-solids content of the water.

Standard determinations of dissolved oxygen in surface water represent the cross-sectional median or mean concentration of dissolved oxygen at the time of observation.

Measuring the DO concentration at one distinct spot in a cross section is valid only for flowing water with a cross-sectional DO variation of less than 0.5 mg/L. Discerning such variation requires a cursory cross-section measurement. The effort involved in collecting this cross-section information is only slightly less than making an equal-width-increment (EWI), equal-discharge-increment (EDI), or multiple-vertical cross-sectional measurement. Measurements made at multiple locations in the cross section are recommended when possible.

- Determining DO for a single vertical at the centroid of flow at the midpoint of the vertical only represents the cross section under ideal mixing conditions.
- Do not measure DO in or directly below sections with turbulent flow, in still water, or from the bank, unless these conditions represent most of the reach or are required by the study objectives.
- Apply a salinity correction to the saturation values after the DO measurement, if needed

Dissolved oxygen must be measured in situ. Never measure DO from a sample splitter.

Procedure:

1. Calibrate the DO instrument at the field site and check that the temperature thermistor has been certified by the USGS Water Science Center within the past 4 months
2. Record the DO variation from the cross-sectional profile and select the sampling method
   - **Flowing, shallow stream**—Wade to the location(s) where DO is to be measured.
   - **Stream too deep or swift to wade**—Lower a weighted DO sensor with a calibrated temperature sensor from a bridge, cableway, or boat. (Do not attach the weight directly to the sensors or sensor cables, because this could damage the sensors or sensor cables.)
   - **Still-water conditions**—Measure DO at multiple depths at several points in the cross section.
3. Immerse the DO and temperature sensors directly into the water body and allow the sensors to equilibrate to the water temperature (no less than 60 seconds).

Notes for amperometric instruments only:

If the water velocity at the point of measurement is less than about 1 ft/s, use a stirring device or stir by hand to increase the velocity. (To hand stir, raise and lower the sensor at a rate of about 1 ft/s, but do not break the surface of the water.) The stir-by-hand method may not be appropriate in lakes, reservoirs, or slow-moving waters (for example, bayous) as these water bodies may be stratified at the point of measurement, making accurate DO measurements impossible. This could be especially problematic in areas where DO

concentrations change substantially over short distances, such as near the thermocline or bottom sediments.

- High stream velocity can cause erroneous DO measurements.

4. Record the temperature without removing the sensor from the water.

5. After the instrument reading has stabilized, record the median DO concentration

The reading should stabilize to within ±0.2 mg/L.

6. For EWI, EDI, or multiple-vertical measurements, proceed to the next station in the cross section and repeat steps 3 through 5. When measurements for the stream have been completed, remove the sensor from the water, rinse it with deionized water, and store it according to the manufacturer's instructions.

7. Record DO concentrations on the field forms:

- **In still water**—median of three or more sequential values.
- **EDI**—mean value of all subsections measured (use the median if measuring one vertical at the centroid of flow).
- **EWI—mean (or median)** of all subsections measured.

5.0 QUALITY ASSURANCE AND QUALITY CONTROL

All equipment calibration data must be documented in the field logbooks and/or field forms, including rationales deviations from this SOP or manufacturer's recommendations. The Field Team Leader or designated QA reviewer will check and verify that field documentation has been completed per this procedure and other procedures referenced herein. All equipment must be operated according to the manufacturer's specifications, including calibration and maintenance.

6.0 DECONTAMINATION

All equipment used in the sampling process shall be decontaminated prior to field use and between sample locations. Decontamination procedures are presented in SOP-7. Personnel shall don appropriate personal protective equipment as specified in the health and safety plan. Any investigation-derived waste generated in the calibration process shall be managed in accordance with the procedures outlined in SOP-12.

7.0 REFERENCES

Beta Technology Incorporated. Hydac Instruction Manual for the Hydac Conductivity Temperature pH Tester, Beta Technology Incorporated, 545 Moose Lodge Road, Cambridge, MD 21613-0265.

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Date: January 31, 2008

OU3 SOP 11 (Rev 1)

Title: GPS DATA COLLECTION

APPROVALS:

EPA Remedial Project Manager — Date 3/20/08

SOP Author — Date 3/20/2008

Revision Number	Date	Reason for Revision
0	9/26/07	
1	1/31/08	Update based on field team input

LIBBY ASBESTOS SUPERFUND SITE OU3

STANDARD OPERATING PROCEDURE No. 11

GPS DATA COLLECTION

1.0 INTRODUCTION

This Standard Operating Procedure (SOP) is a general guidance document for the collection of coordinates of point locations using a Global Positioning System (GPS) unit. The GPS is a worldwide, satellite-based system with location positioning capabilities. The system is administered and managed by the Department of Defense. It is comprised of:

- a space segment of approximately 24 operational satellites in complimentary orbit,
- a ground control segment made up of a network of control stations around the globe, and
- a user segment, which includes anyone who uses GPS to collect locational information.

The system utilizes precise time and radio signals to determine distances from satellites to user GPS receivers. Distances are most commonly calculated by using the time it takes for a radio signal code to be transmitted from the satellite and received by the GPS unit. Precise time is critical to the successful operation of the system. The control stations ensure that the satellites employ synchronized, atomic clock-derived universal time coordinates (UTC), commonly known as Greenwich Mean Time (GMT). Receiver units collecting four satellite signals can determine the geodetic (x, y, z) location through a process of mathematical triangulation. The satellite signals contain precise time and satellite position information.

GPS technology is used as a method of accurately determining the coordinates of point locations. The three-dimensional position, or the x, y, and z geodetic coordinates, are determined for the point locations; however, only the x and y values are primarily used. This is due to the processes involved in the system; the vertical GPS coordinates are approximately

half as accurate as the horizontal GPS coordinates. The position reported by the GPS unit is based on the geodetic model selected. The vertical, or z coordinate, value is not as accurate as the reported position due to the geometry of the satellite constellation relative to the receiver's position on the earth.

GPS is one of the arrays of tools for accurately determining location in the field. The collection of x, y, and z coordinates (for gross data collection) for locations in the field using GPS is useful for a variety of purposes, including accurate sample locations, locational correlation of remotely sensed data with ground truth locations, and efficiently collecting better spatial data.

3.0 RESPONSIBILITIES

This section presents a brief definition of field roles, and the responsibilities generally associated with them. This list is not intended to be comprehensive and often, additional personnel may be involved. Project team member information will be included in project-specific plans (e.g., work plan, field sampling plan, quality assurance plan, etc.), and field personnel will always consult the appropriate documents to determine project-specific roles and responsibilities. In addition, one person may serve in more than one role on any given project.

Project Manager: Selects project-specific field documentation with input from other key project staff, and appropriate oversite agencies.

Quality Control Manager: Overall management and responsibility for quality assurance and quality control (QA/QC). Selects QA/QC procedures for the sampling and analytical methods, performs project audits, and ensures that data quality objectives are fulfilled.

Field Team Leader (FTL) and/or Field Geologist, Hydrogeologist, or Engineer: Implements the sampling program, supervises other sampling personnel, and ensures compliance with SOPs and QA/QC requirements. Prepares daily logs of field activities.

Field Technician (or other designated personnel): Assists the FTL and/or field geologist, hydrogeologist, or engineer in the implementation of field tasks and field documentation.

4.0 PROCEDURES

There are three major types of GPS units available: survey-grade units, mapping-grade units, and recreational-grade units. For the collection of environmental/natural resource data, mapping-grade and recreational-grade units are usually sufficient. The type of GPS unit employed should meet the data collection needs as outlined in the project work plan.

Recreational-grade GPS units can be used to acquire location information (generally points) when spatial accuracy is not paramount to the project. Recreational GPS units do not have data dictionaries for storing attribute information with the point location. The procedures described here are geared toward hand-held recreational GPS units. Consult the specific instrument's instruction manual for details on operation.

There has been, and will continue to be, a considerable and rapid evolution in GPS techniques and technologies. Adjustments to the following operational procedures may be necessary to reflect these rapid changes in technology.

4.1 Method

Planning

If a recreational-grade GPS meets the criteria of the project, the unit chosen must have the capability of downloading collected data to a personal computer. This is usually accomplished with a parallel or USB cable connection.

Much of the data collected by GPS will eventually reside in a relational database. Each GPS feature collected should contain a unique identifier that relates the feature to an associated record in a database. Since recreational GPS units have only one text field for input, careful consideration should be given to the use of this field and the design of unique identifiers.

Data Collection

Locational data are captured by recreational-grade GPS units as *waypoints*. When taking a waypoint, enter the Location ID in the text field provided. It is also recommended that reference points be collected occasionally. These reference point positions should be taken at known

locations (e.g., site headquarters office, stream confluences) which can later be used in GIS to QC the accuracy of waypoint data.

If navigation to preset waypoints is applicable to a project, they must be loaded onto the GPS unit before departure to the field. It is also recommended to have printed topographic maps of the waypoint locations in order to maximize field time and efficiently navigate between waypoints.

Data Processing

GPS units should be downloaded once a day or after each field session. Data should be downloaded both as a text file and a shapefile. Points should be checked for reasonable spatial accuracy and errors. Subsequent downloads should be error-checked in the same manner. When data collection is finished, all files should be compiled into one spatial file.

Additional Documentation

Regardless of the type of GPS unit used to collect locational data, all resulting GIS datasets need to have information documenting how the GPS data were collected. Documentation can be recorded at the time of data point collection and/or can be stored along side the electronic data set with a simple readme text file.

The following details are suggested as items to include in data collection:

- Name of project
- Name(s) of data collectors
- Coordinate system (projection, datum & zone)
- Type (or types) of GPS units used
- The range of field collection dates

Below is an example of typical GPS data collection using a hand-held recreational-use GPS device. It serves only as an example of data collection and is not intended to provide detailed step-by-step instructions operation. Always refer to the owner's manual for specific instructions on device operation and data collection.

- Turn GPS unit on by pressing and releasing the power key.
- Following the Welcome Page the Satellite Page will be next.
- After sufficient satellites have been acquired, change to Position Page.
- When locating a sample location, use the sample identification as described in the field sampling plan.
- To record a location press the Mark key; the longitude, latitude, time, and date will be saved. Record the information into a field log book, and save the information in the GPS unit with a unique identification name and/or number to be downloaded later.
- Then enter OK to return to the Position Page.
- To turn off the GPS unit press and hold down the power key.

5.0 QUALITY ASSURANCE AND QUALITY CONTROL

Only data with high geometrical strength or low percent dilution of position (PDOP) will be used to ensure high accuracy. The Field Team Leader or designated QA reviewer will check and verify that the GPS coordinates are collected using the appropriate Datum, are entered into a field logbook or electronic database on a daily basis and that coordinates entered into project records match those recorded in the GPS-unit memory. If any corrections are necessary, the field team lead or other field personnel will make those corrections before coordinates are transmitted to data users.

All GPS equipment must be operated according to the manufacturer's specifications, including calibration and maintenance.

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Garmin GPSmap 60CSx Operators Manual

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Libby Superfund Site Operable Unit 3 Standard Operating Procedure

Date: September 26, 2007 OU3 SOP 12 (Rev. 0)

Title: INVESTIGATION DERIVED WASTE (IDW) MANAGEMENT

APPROVALS:

TEAM MEMBER	SIGNATURE/TITLE	DATE
EPA Remedial Project Manager		9/26/07
SOP Author		9/26/07

Revision Number	Date	Reason for Revision
0	09/26/2007	–

1.0 INTRODUCTION

This SOP is based on MWH SOP-07, Investigation-Derived Waste (IDW) Management, Revision 1.0, April 2007, modified for use at the Libby Asbestos Superfund Site OU3. IDW may be generated during field investigations at the Libby Asbestos Superfund Site OU3. The National Contingency Plan (NCP), codified in 40 Code of Federal Regulations (CFR) 300, requires that IDW be handled to attain all the applicable or relevant and appropriate requirements (ARARs) to the extent practicable, considering the urgency of the situation. The purpose of this SOP is to present procedures to be followed in the management of IDW generated during the field investigations.

Typical IDW generated during field activities are solid wastes and may include (but are not limited to) the following media and waste types:

Fluids	Solids
Purge water and groundwater	Soils and soil cuttings
Drilling mud	Plastic tarps or sheeting
Grout	Drill pipe and well casing/screen
Decontamination fluids and wastewater	Decontamination solids
	Disposable equipment (i.e., rope, bailers, sampling equipment, & other consumables)
	Spent personal protective equipment (PPE)
	Used containers, sample bottles
	Packaging materials

The above wastes may or may not be encountered, generated or managed while performing field investigations. However, all solid waste streams will be characterized to determine if they are hazardous wastes per 40 CFR § 262.11 for the purposes of handling and disposal. Guidance from this document shall be used as part of project planning to estimate total volumes of IDW likely to be generated as well as how the IDW will be managed and disposed.

2.0 HEALTH AND SAFETY WARNING

All personnel engaged in IDW handling must follow health and safety protocols described in the health and safety plan. Asbestos fibers are thin and long fibers so small that they cannot be seen by the naked eye. Asbestos fibers are easily inhaled when disturbed and when embedded in the lung tissue can cause health problems. Significant exposure to asbestos increases the risk of lung cancer, mesothelioma, asbestosis (non-cancerous lung disease), and other respiratory diseases (ATSDR 2006).

3.0 DEFINITIONS

Area of Contamination (AOC) unit: The AOC unit concept is critical to the IDW management at a CERCLA investigation site. Although EPA has not promulgated a definition of an AOC unit, an AOC unit is generally an area within a CERCLA investigation site with similar characteristics with respect to contamination and the associated risks to human health and the environment. A CERCLA investigation site may contain one or more AOC units.

Decontamination fluids: Any fluids, including aqueous wash water, solvents, and contaminants that are used or generated during decontamination procedures.

Decontamination solids: Any solids, including soils and soil cuttings, fill materials, and contaminants that are generated during decontamination procedures.

Grout: A fluid mixture of cement and water (neat cement) of a consistency that can be forced through a pipe and placed as required.

Hazardous waste: A solid waste that meets the definition of a hazardous waste under RCRA as defined in 40 CFR § 261.3.

Investigation-derived waste (IDW): Solid wastes, as defined in 40 CFR § 261.2, directly generated as result of performing the field activities.

Nonhazardous waste: A solid waste that does not meet the definition of a hazardous waste as defined in 40 CFR § 261.3 or is excluded from hazardous waste regulation per 40 CFR § 261.4(b).

Soils and soil cuttings: Solid material generated from excavation or drilling processes. Soils may include native soils, fill materials, and/or other historical plant waste streams used as fill materials on the site.

Solid waste: Any waste stream (solid, liquid or containerized gas) that meets the definition of solid waste under RCRA as defined in 40 CFR § 261.2.

4.0 RESPONSIBILITIES

This section presents a brief definition of the field team roles and responsibilities for management of IDW generated while conducting field investigations. This list is not intended to be a comprehensive list as additional personnel may be involved. Project team member information shall be included in project-specific plans (e.g., work plan, field sampling plan (FSP), quality assurance plan, etc.), and field personnel shall always consult the appropriate documents to determine project-specific roles and responsibilities. In addition, one person may serve in more than one role on any given project.

Project Manager: Responsible to ensure that all field team members are properly trained per their responsibilities associated with IDW and that appropriate equipment and facilities are available for appropriate IDW management.

Field Team Leader (FTL): Implements the field program and supervises all field team members in the appropriate management of IDW. Ensures that only properly trained personnel are managing IDW on the site.

Environmental, Health and Safety (EHS) Officer: Assists the Team Leader in the supervision of all IDW management on site. The EHS officer shall be responsible for all IDW identification

and characterization, on site disposal, off site shipment and disposal, waste accumulation, emergency response and contingency planning, IDW training, and IDW reporting and recordkeeping.

Project Team Members: Ensure that they are properly trained prior to any IDW management as well as follow the appropriate IDW procedures and training.

5.0 REGULATORY BASIS AND GUIDANCE

IDW encountered, generated, or managed during the field investigations may contain hazardous substances as defined by CERCLA. Some IDW may be hazardous wastes under RCRA while others may be regulated under other federal laws such as TSCA. These regulatory requirements may be applicable or relevant and appropriate requirements (ARARs) which impact how the IDW is managed.

5.1 EPA Guidance on IDW Management

The management of IDW shall be in accordance with EPA Guidance "Management of Investigation-Derived Wastes During Site Inspections", May 1991 (EPA, 1991). The specific elements of EPA's guidance for IDW management are as follows:

- Characterizing IDW through the use of existing information (manifests, MSDSs, previous test results, knowledge of the waste generation process, and other relevant records) and best professional judgement.

- Delineating an AOC unit for leaving RCRA hazardous soil cuttings within the unit.

- Containerizing and disposing of RCRA hazardous groundwater, decontamination fluids, PPE, and disposable equipment at RCRA Subtitle C facilities.

- Leaving on-site RCRA nonhazardous soil cuttings, groundwater, and decontamination fluids preferably without containerization and testing.

In general, EPA does not recommend removal of wastes from sites, in particular, from those sites where IDW do not pose any immediate threat to human health or the environment. Actions taken during field investigations with respect to IDW that leave conditions essentially unchanged should not require a detailed analysis of ARARs or assurance that conditions at the site after field investigations will comply with the ARARs. At the same time, field personnel ensure that their handling of IDW does not create additional hazards at the site.

In brief, compliance with the NCP can generally be assured by:

1) Identifying contaminants, if any, present in the IDW based upon existing information and best professional judgement; testing is not required in most circumstances.

2) Determining ARARs and the extent to which it is practicable to comply with them.

3) Delineating an AOC unit based upon existing information and visual observation if soil cuttings are RCRA hazardous.

4) Burying RCRA hazardous soil cuttings within the AOC unit, so long as no increased hazard to human health and the environment will be created. Containerization and testing are not required.

5) Containerizing RCRA hazardous groundwater and other RCRA hazardous IDW such as PPE, disposable sampling equipment, and decontamination fluids for off-site disposal.

5.2 Hazardous Waste Regulation

The RCRA hazardous waste regulations are clearly ARARs for hazardous IDW generated and managed during field investigations. However, with the application of EPA IDW guidance, RCRA requirements apply to management of IDW in the following manner:

- If RCRA hazardous IDW is stored or disposed off-site, then comply with all RCRA (and other ARAR) requirements.

- If RCRA hazardous IDW is stored on-site, then comply with RCRA (and other ARAR) requirements to the extent practicable.

For these field investigations, the following general guidance is expected to be practicable and therefore followed, recognizing that each situation will be evaluated against EPA IDW guidance (EPA, 1991) as well as RCRA hazardous waste requirements and other ARARs:

- IDW may be assumed not to be a "listed" hazardous waste under RCRA 40 CFR 261 Subpart D, unless available information about the site suggests otherwise.

- IDW characterization to determine if the IDW exhibits RCRA hazardous waste characteristics do not typically require testing if the characterization can be made by "applying knowledge of the hazardous characteristics in light of the materials or processes used" or by historical testing consistent with 40 CFR § 262.11(c).

- Compliance with the RCRA hazardous waste generator requirements of 40 CFR Part 262 for all RCRA hazardous IDW generated and/or managed (with exception of soil cuttings managed in accordance with the EPA IDW guidance). It is presumed that the RCRA hazardous IDW generated will fall within the large quantity generator (LQG) requirements.

- Land disposal does <u>not</u> occur (and thus the Land Disposal Restrictions [LDR] of 40 CFR Part 268 are <u>not</u> applicable) when IDW soil cutting wastes are:

- Moved, stored or left in place within a single AOC unit;
- Capped in place;
- Treated in situ (without moving the IDW to another AOC unit for treatment); or
- Processed within the AOC unit to improve structural stability (without placing the IDW into another AOC unit for processing).

- Conversely, land disposal does occur (and the LDR of 40 CFR Part 268 are applicable) when IDW soil cutting wastes are:

  - Moved from one AOC unit to another AOC unit for disposal;
  - Moved outside an AOC unit for treatment or storage and returned to the same AOC unit for disposal;
  - Excavated from an AOC unit and placed in a container, tank, surface impoundment, etc. and then re-deposited back into the same AOC.

5.3 TSCA PCB Regulation

IDW containing PCBs at detectable levels may be generated, although the concentration of PCBs in any IDW generated is expected to be far below 50 ppm. However, IDW generated will be evaluated for PCBs and managed according to the following per the requirements of 40 CFR Part 761 Subpart D:

- Liquid IDW at concentrations greater than or equal to 50 ppm PCBs will be incinerated off-site at a TSCA-approved incinerator site.

- Nonliquid IDW at concentration greater than or equal to 50 ppm PCBs may be incinerated, treated by an equivalent TSCA-approved method, or disposed in a TSCA chemical landfill off-site.

- IDW at concentrations less than 50 ppm are generally not regulated under TSCA, and may be disposed in an acceptable Subtitle D facility.

6.0 DESCRIPTION OF ANTICIPATED IDW MANAGEMENT

The following subsections provide a description of the anticipated IDW to be encountered, generated, and/or managed at the Libby Asbestos Superfund Site OU3 during field activities and the anticipated management of each. It should be noted that this information is provided for planning purposes, and will be evaluated and may need to be revised based upon actual experience while on site.

6.1 Soil and Soil Cuttings

During field investigations, surface soil samples, samples of mine waste rock, and samples of fine tailings will be collected. Only a small portion of material will be collected for analysis. While the soil and soil cuttings IDW generated will be evaluated on a case-by-case basis, the general approach will follow the EPA guidance for IDW (EPA, 1991) which includes:

- Characterizing the IDW through the use of existing information (previous test results, previous waste characterization, knowledge of the waste generation process, and other relevant records) and best professional judgement.

- Soil and soil cuttings which are not used directly for sample makeup will not be taken outside of the AOC unit in which they were generated.

- Soil and soil cuttings within the AOC where they are generated will be placed back into the same investigation pit, trench, or bore hole and in the same order from which the material was removed, to the extent practicable and unless noted otherwise in the FSP.

- Soil cuttings potentially requiring RCRA disposal will be handled per the procedures presented in Section 7.0 below and disposed in an off-site RCRA facility.

6.2 Spent Sampling-Related Equipment

During field investigations, spent sampling-related equipment will be generated. This may include (but not limited to) plastic sheeting/tarps, rope, bailers, sampling equipment, spent PPE, sample bottles, used containers, packaging materials, and other consumables. Although the vast majority of the spent sampling-related equipment is expected to be nonhazardous, these IDW may contain a listed hazardous waste (e.g., spent solvents) or may exhibit a hazardous waste characteristic (e.g., toxicity from metals).

While the spent sampling-related equipment will be evaluated on a case-by-case basis, the general approach to be followed for spent sampling-related equipment IDW will follow the EPA guidance for IDW (EPA, 1991) which includes:

- Containerizing the spent sampling-related equipment, typically in a satellite accumulation station.

- Characterizing the spent sampling-related equipment IDW through the use of existing information (previous test results, previous waste characterization, knowledge of the contaminants present, and other relevant records) and best professional judgement. This characterization will be documented and maintained as part of the solid/hazardous waste determination records.

- Those spent sampling-related equipment IDW that are determined to be nonhazardous will be disposed of onsite or as municipal waste.

- Those spent sampling-related equipment IDW that are determined to be hazardous will be managed per the procedures presented in Section 7.0 below and disposed in an off-site RCRA facility.

6.3 Decontamination Fluids and Solids

During field investigations, decontamination fluids and solids will be generated. Typically, these will be generated at a common decon area, although there may be more than one decon

area. Typically, the decontamination IDW will include (but not limited to) washwater from vehicles/equipment, and cleaning agents. Although the vast majority of decontamination IDW is expected to be nonhazardous, this IDW may contain a listed hazardous waste (e.g., spent solvents) or may exhibit a hazardous waste characteristic (e.g., toxicity from metals).

While the decontamination IDW will be evaluated on a case-by-case basis, the general approach to be followed for decontamination IDW will follow the EPA guidance for IDW (EPA, 1991) which includes:

- Containment of decontamination fluids (typically washwater) as generated. The washwater will be segregated from solids to the extent practicable (i.e., solids will be allowed to settle out of the washwater on the decontamination containment pad). Washwater will then be containerized to await waste determination. Solids will also be containerized in a separate container to await waste determination.

- Other decontamination solids such as cleaning utensils and PPE will also be containerized to await waste determination.

- Characterizing the decontamination IDW through the use of existing information (previous test results, previous waste characterization, knowledge of the contaminants present, and other relevant records) and best professional judgement. This characterization will be documented and maintained as part of the solid/hazardous waste determination records.

- The decontamination solids IDW that are determined to be nonhazardous will be disposed of onsite.

- The decontamination liquids IDW that are determined to be nonhazardous will be disposed as a nonhazardous solid waste, preferably on-site.

- The decontamination IDW (either liquid or solid) that are determined to be hazardous will be managed per the procedures presented in Section 7.0 below and disposed in an off-site RCRA facility.

6.4 Drilling, Well Purging, and Development Water

Generally, water at the Site that is extracted from boreholes, wells or piezometers for the purpose of drilling, development, sampling, or hydraulic testing is considered non-hazardous and will be discharged to designated shallow sumps away from the boreholes or wells at the site. If the water generated is determined to be hazardous will be managed per the procedures presented in Section 7.0 below and disposed in an off-site RCRA facility.

7.0 PROCEDURES FOR HAZARDOUS IDW MANAGEMENT

The following procedures apply to all IDW that have been determined to be hazardous except for soil cuttings IDW that remain with the AOC unit.

7.1 Introduction

Once an IDW has been determined to be hazardous, the federal RCRA Subtitle C waste management requirements apply to that waste. The scope of this procedure covers the requirements for large quantity generators (LQG) of hazardous IDW which manage the hazardous IDW on site such that RCRA permitting is not required.

7.2 Determine Land Disposal Restrictions

The 1984 amendments to the RCRA law included a prohibition of land disposal of certain hazardous wastes without first meeting some treatment standards. For the most part, all listed and characteristic hazardous wastes must be treated according to the treatment levels and technologies outlined in 40 CFR Part 268 to reduce the toxicity and/or mobility of hazardous constituents prior to being disposed of on the land, i.e., landfilled. Therefore, a generator must

determine if the waste is a "restricted waste" under the land ban rules, and if so, off site treatment and disposal is limited. Note that these rules apply only to wastes destined for land disposal which is defined as: placement in or on the land including a landfill, surface impoundment, waste pile, injection well, land treatment facility, salt dome formation, salt bed formation, underground mine or cave, or concrete vault or bunker. Wastes which are shipped off site for disposal other than land disposal are not regulated under the land disposal restriction regulations of 40 CFR Part 268.

Generators of hazardous wastes must determine if the waste is restricted from land disposal under 40 CFR Part 268. The following reporting and recordkeeping requirements apply.

- If a generator determines that he is managing a restricted waste and the waste does not meet the applicable treatment standards, with each shipment of waste, the generator must notify the treatment or storage facility in writing of the appropriate treatment standards;
- If the generator determines that he is managing a restricted waste and the waste can be disposed without further treatment, with each shipment of waste, the generator must submit to the treatment, storage or disposal facility a notice and certification stating that the waste meets the applicable treatment standards;
- If the generator determines that he is managing a waste subject to an exemption from a prohibition on the type of land disposal method utilized for the waste, with each shipment of waste, the generator must submit to the receiving facility a notice stating that the waste is not prohibited from land disposal;
- If the generator is managing prohibited waste in tanks, containers, or containment buildings regulated under 40 CFR 262.34, and is treating such waste in such tanks, containers, or containment buildings to meet applicable treatment standards, the generator must develop a waste analysis plan which describes the procedures the generator will carry out to comply with the treatment standards; and

- If the generator determines whether the waste is restricted based solely on his knowledge of the waste, all supporting data used to make this determination must be retained on-site in the generator's files.

The generator must retain on-site a copy of all notices, certifications, demonstrations, waste analysis data, and other documentation produced pursuant to these requirements for at least three years from the date the waste was last shipped from the site. It should also be noted that it is prohibited to dilute a hazardous waste in order to circumvent the land disposal prohibitions (40 CFR 268.3). Once a waste is determined to be a "restricted waste", an appropriate Treatment, Storage, and Disposal Facility (TSDF) can be selected to properly treat and dispose of the waste.

7.3 On-Site Accumulation

As discussed in Section 5.0 above for each IDW generated, a large quantity generator (LQG) must make the appropriate hazardous waste determination per 40 CFR Part 262.11. If the IDW is determined to be hazardous, then the IDW will typically be stored on-site prior to shipment off-site for disposal. The following requirements apply to all hazardous IDW being stored on-site prior to shipment.

7.3.1 EPA Identification Number (40 CFR Part 262.12)

Any facility which is a LQG of hazardous wastes must not treat, store, dispose, transport or offer for transportation any hazardous waste without first obtaining a EPA identification number from EPA (or the authorized state). Hazardous wastes cannot be offered to transporters or to treatment, storage or disposal facilities that have not received a EPA identification number.

7.3.2 On-Site Hazardous Waste Accumulation (Storage) (40 CFR 262.34(d))

Two types of accumulation areas for hazardous waste are permissible for a LQG without RCRA interim status or a Part B permit. These are the "90-day storage area" and the "satellite accumulation station" (SAS). The SAS requirements are discussed below. With regards to a

"90-day storage area", a LQG may store hazardous wastes on-site for up to 90 days or less in a storage area, provided that the following conditions are met:

- If the waste is placed in containers, the requirements of 40 CFR Part 265 Subpart I (container requirements) are met. See below for container requirements;
- If the waste is placed in tanks, the requirements of 40 CFR 265 Subpart J (tank requirements) are met. See below for the tank requirements.
- At closure, the generator closes the storage area per the requirements of 40 CFR 265.111 and 40 CFR 265.114;
- The date which the hazardous waste is placed in the storage area is clearly marked on the container, and the container is clearly marked as "Hazardous Waste";
- The facility complies with 40 CFR Part 265 Subpart C, Preparedness and Prevention (See Section 6.3.3 below);
- The facility complies with 40 CFR Part 265 Subpart D, Contingency Plan and Emergency Procedures (See Section 6.3.4);
- The facility complies with 40 CFR Part 265.16 training requirements (See Section 6.6 below);
- Any hazardous wastes which are stored longer than 90 days must first be granted an extension by EPA (or authorized state).

90-Day Storage Area Container Requirements (40 CFR Part 265 Subpart I)

Hazardous waste stored in containers must meet the following requirements:

- Containers must be in good condition, free of leaks;
- Hazardous wastes must be compatible with container (or liner) material;
- Containers must always be kept closed except to add or remove wastes;

- Containers must be handled in a manner to avoid ruptures;
- The storage area must be inspected at least weekly to check for container deterioration; and
- Incompatible wastes must be stored separately with separate secondary containment.

Incompatible wastes are wastes that are unsuitable for co-mingling because the co-mingling could result in any of the following:

- Extreme heat or pressure generation;
- Fire;
- Explosion or violent reaction;
- Formation of substances that have the potential to react violently;
- Formation of toxic dusts, mists, fumes, gases, or other chemicals; and/or
- Volatization of ignitable or toxic chemicals due to heat generation.

90-Day Storage Area Tank Requirements (40 CFR Subpart J)

LQGs that accumulate or store hazardous wastes in tanks or tank systems must meet the following requirements:

- For tanks existing prior to July 14, 1986, an assessment of tank must be performed and certified by an independent, qualified, licensed engineer. The written certification must be kept on file at the facility (40 CFR 265.191);
- New tank systems (those built after July 14, 1986) must meet tank technical standards and have been certified by an independent, qualified, licensed engineer. The written certification must be kept on file at the facility (40 CFR 265.192);

- New tank systems must have adequate secondary containment and leak detection systems. Existing tanks must be upgraded to meet these standards by the time the tank is 15 years of age (40 CFR 265.193);
- Tanks must be operated to prevent system failure, overflow and spills. Tanks must be operated with sufficient freeboard to prevent overtopping (40 CFR 265.194);
- Inspect the tanks at least once each operating day for the following:
  - Discharge control equipment;
  - Monitoring equipment and controls;
  - Tank level; and
  - Evidence of leaks or spills. (40 CFR 265.195)
- Inspect the tanks at least weekly for corrosion, erosion or leaks;
  - The tank must meet the closure and post-closure care provisions of 40 CFR 265.197; and
  - Store incompatible wastes separately (40 CFR 265.199).

Satellite Accumulation Station (SAS) Requirements (40 CFR 262.34(c))

A SAS is a container placed at or near the point of waste generation for the purpose of collecting the waste as it is being generated. For example, a container may be placed in the quality control laboratory for collection of hazardous wastes generated in the laboratory. This SAS may collect up to 55 gallons of hazardous waste or 1 quart of acute hazardous waste. The SAS does not need to meet the requirements of a storage area, provided the following conditions are met:

- The amount of hazardous waste accumulated at the SAS does not exceed 55 gallons (or 1 quart of acute hazardous waste);

- The SAS is located at or near the point of generation where the waste is initially accumulated and is under the control of the operator of the process generating the waste;
- The container used is in good condition, is compatible with the wastes being accumulated, and is kept closed except to add or remove wastes;
- The container is marked with the words "Hazardous Waste" or other words to identify the contents; and
- Once the 55-gallon limit is reached, the date is marked on the container and the container is moved from the SAS within three days to a proper location. For example, the wastes must either be moved to the storage area or be picked up by a waste transporter and moved off-site.

7.3.3 Preparedness and Prevention (40 CFR Part 265 Subpart C)

The following preparedness and prevention steps must be taken concerning the hazardous waste storage area:

- The storage area must be operated and maintained to minimize the possibility of fire, explosions or releases of hazardous waste;
- The facility must have appropriate communication systems, fire-fighting equipment, spill control equipment and decontamination equipment;
- All emergency response systems and equipment must be tested monthly with documentation and maintained to assure proper operation;
- Persons handling hazardous wastes must have immediate access to alarms and/or communication systems;
- The storage area shall have adequate aisle space for emergency response activities; and

- The facility must attempt to make arrangements with the local police, fire departments, emergency response teams, and local hospitals to assure readiness for potential emergencies associated with the storage area.

7.3.4 Contingency Plan and Emergency Procedures (40 CFR Subpart D)

A LQG that accumulates or stores hazardous waste on site in a 90-day storage area must develop and keep current a contingency plan for the facility. The purpose of the contingency plan is to provide an organized plan of action and delegation of responsibilities and authority to specific facility personnel to respond to emergency situations that may require both the facility and/or outside resources. The contingency plan is designed to minimize hazards to humans or the environment from fires, explosion or any unplanned sudden or non-sudden release of hazardous waste/hazardous waste constituent to air, soil or surface water in compliance with the requirements of 40 CFR 265 Subpart D. A Contingency Plan will be maintained on the site if hazardous IDW are accumulated on-site.

The key components of the contingency plan include the following (40 CFR 265.52):

- A description of the emergency response organization, including designation of the Emergency Coordinator and alternates;
- Response procedures;
- Emergency notification;
- Arrangements with local authorities;
- List of names, addresses and phone numbers of designated emergency personnel and alternates;
- List of emergency response communication equipment and locations;
- Evacuation procedures, routes and alternates; and
- Procedures for amending the plan.

Copies of the plan must be sent to (40 CFR 265.53):

- The Project Manager;
- Lincoln County Sheriff's department;
- Libby fire department; and
- Other agencies as deemed appropriate.

The emergency coordinator (EC) is the key person facilitating emergency preparedness and response. The EC or designated alternate shall be on-site or on-call at all times. The EC and alternates must be trained and thoroughly familiar with the contingency plan, emergency response activities and operation of the facility. The EC must know the locations and characteristics of all waste generated, location of all records within the facility and the facility layout. The EC must have the authority to commit the resources needed to carry out the spill response plan. Any person or department who first discovers any spill of a hazardous waste/material is responsible for notifying the spill response/emergency response coordinator. The EC for the Libby Mine Site field investigations will be the EHS Officer with the Field Team Leader and the Project Manager as alternates.

The contingency plan should be reviewed and immediately amended when:

- Changes in applicable regulations occur;
- The plan fails in an emergency;
- Changes are made to emergency procedures;
- Changes occur in emergency personnel list; or
- Changes occur in emergency equipment list.

7.4 Pre-Transportation Requirements

Prior to transporting hazardous wastes or offering hazardous wastes for transportation off-site, the generator must comply with the following:

- Package the hazardous wastes in DOT-approved containers per 49 CFR Parts 173, 178 and 179. DOT-approved containers (such as drums) are usually marked as being DOT-approved);
- Label the hazardous wastes according to DOT labeling requirements per 49 CFR Part 172;
- Mark each container (of 110 gallons or less) used in transportation with the following:

  HAZARDOUS WASTE - Federal Law Prohibits Improper Disposal. If found, contact the nearest police or public safety authority or the EPA.

  - Generator's Name and Address
  - Manifest Document Number
- Ensure that the initial transporter placards the transport vehicle with the appropriate placard in accordance with 49 CFR Part 172 Subpart F.

7.5 Manifesting Off-Site Shipments of Hazardous IDW

Any generator which transports or offers for transportation hazardous waste for off-site treatment, storage or disposal must prepare a manifest according to manifest instructions for each shipment of similar hazardous wastes. The manifest must be carefully filled out with each shipment. Take care to follow the instructions and use the terms as listed in the instructions. A generator must designate on the manifest one facility (designated facility) which is permitted to handle the waste described on the manifest (40 CFR 262.20).

The generator must determine if the state to which the wastes are destined (consignment state) requires use of its own manifest. If so, then the consignment state's manifest must be used. If the consignment state does not require use of its manifest, and the state in which the waste

shipment originates (generator state) does, then the manifest from the generator state must be used. If both states have manifests, use the consignment state manifest, making sure that there are sufficient copies to meet the generator state distribution requirements. If neither state requires use of its manifest, then any uniform hazardous waste manifest may be used (40 CFR 262.21).

The manifest must contain at least enough copies such that the generator gets two copies, the transporter gets one copy and the designated facility gets one copy. Some states require additional copies to be sent to the state. At the time of shipment, the generator must keep one copy (the generator copy) of the completed, signed manifest and give the remaining copies to the transporter. Each copy must have the signature of the generator and the transporter at the time of shipment. The original manifest shall be returned to the generator once the shipment reaches the designated facility and the manifest is signed by the designated facility (40 CFR 262.21).

If the original, signed manifest is not received by the generator within a certain number of days, action by the generator is required. These requirements are discussed in the following sections:

- If, after 35 days from the date of shipment, the original manifest copy is not yet received by the LQG, the LQG must contact the transporter and/or the designated disposal facility to determine the status of the hazardous waste (40 CFR 262.42(a)(1)).
- If after 45 days from the date of shipment, the original manifest copy is not yet received by the LQG, the LQG must submit an exception report to the U.S. EPA (or authorized state). The exception report must include a copy of the manifest along with an explanation of efforts to locate the hazardous wastes and the result of these efforts (40 CFR 262.42(a)(2)).

7.6 Personnel Training

Any person, and their immediate supervisor(s), involved in waste management at a LQG facility which stores hazardous waste in a 90-day storage area must undergo initial and annual training

for hazardous waste management (40 CFR 262.34(a)(4) and 40 CFR 265.16). Facility personnel are required to successfully complete a program of classroom instruction or on-the-job training that teaches them to perform hazardous waste management duties relevant to their jobs. The program must be directed by a person trained in hazardous waste management procedures.

The training must be designed to enable personnel to effectively respond to emergencies by becoming familiar with emergency procedures, emergency equipment and emergency systems, including the following;

- Procedures for using, inspecting, repairing and replacing facility emergency and monitoring equipment;
- Communications or alarm systems;
- Response to fires or explosions; and
- Off-site communication.

Employee training is to be held at regular intervals. Emergency planning information, e.g., the Contingency Plan, also should be provided to state and local emergency response agencies at regular intervals (40 CFR 265.37 and 265.53). Employees required to receive the training cannot work unsupervised until they have completed the training requirements (either classroom or on-the-job training). In addition, facility personnel must take part in an annual review of the initial training.

The following records must be maintained at the facility for employees affected by this training:

- Job title for each position and name of employee filling each job;
- Job descriptions for each position related to hazardous waste management;
- Written description of type and amount of initial and continuing training that will be given to each person filling the various job positions; and

- Documentation that necessary training has been given and completed by each affected personnel.

Training records are required to be kept on current personnel until closure of the facility. For former employees, training records must be kept for at least three years from the date the employee last worked at the facility and may be transferred if the employee stays within the same company (40 CFR 265.16(e).

7.7 Reporting and Recordkeeping

The following reports are required of a LQG:

- Manifest exception reports as discussed in Section 6.5 above.
- A LQG must submit a Biennial Report to the EPA (or authorized state) every even numbered year by March 1, e.g., March 1, 2008 for the 2007 reporting year. The Biennial Report is to be submitted on EPA form 8700-13A.

The following records are required to be kept for a minimum of three years by the LQG:

- The signed original manifests;
- Biennial reports;
- Exception reports;
- All records pertaining to hazardous waste determinations; and
- Land disposal determination records, notification and certification records.

8.0 QUALITY ASSURANCE AND QUALITY CONTROL

All IDW data must be documented in the field logbooks, field forms, manifests, including rationales deviations from this SOP. The Field Team Leader or designated QA reviewer will

check and verify that IDW documentation has been completed per this procedure and other procedures referenced herein.

9.0 REFERENCES

Agency for Toxic Substances and Disease Registry. 2006. Asbestos Exposure and Your Health.

EPA, 1991. Management of Investigation-Derived Wastes During Site Inspections, EPA May 1991, EPA/540/G-91/009

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Libby Site OU3 SOP No. 14
Rev. No. 0

Automated Water Sampling

SOP will be prepared by MWH.

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Date: January 31, 2008 OU3 SOP 15 (Rev 0)

Title: INSTALLATION, OPERATION, AND MAINTENANCE OF PERMANENT FLUMES

APPROVALS:

EPA Remedial Project Manager — Date: 3/20/08

SOP Author — Date: 3/20/2008

Revision Number	Date	Reason for Revision
0	1/31/08	

LIBBY ASBESTOS SUPERFUND SITE OU3

STANDARD OPERATING PROCEDURE No. 15

INSTALLATION, OPERATION, AND MAINTENANCE OF PERMANENT FLUMES

1.0 INTRODUCTION

This Standard Operating Procedure (SOP) describes the protocols to be followed when installing, operating, and/or maintaining a permanent flume used for flow measurements within a stream.

This document focuses on methods and equipment that are readily available and typically applied for the installation, operation, and maintenance of permanent flumes. It is not intended to provide an all-inclusive discussion regarding permanent flow measurement structures. Specific installations and operational problems may require the adaptation of existing equipment or design of new equipment. Such innovations shall be clearly described in the project-specific sampling plan and approved by the Project Manager and the Quality Manager.

2.0 HEALTH AND SAFETY WARNING

All personnel engaged in installation, operations, and/or maintenance of a permanent flume must adhere to health and safety protocols described in the health and safety plan. Asbestos fibers are thin and long fibers so small that they cannot be seen by the naked eye. Asbestos fibers are easily inhaled when disturbed and when embedded in the lung tissue can cause health problems. Significant exposure to asbestos increases the risk of lung cancer, mesothelioma, asbestosis (non-cancerous lung disease), and other respiratory diseases (ATSDR 2006).

3.0 DEFINITIONS

Parshall Flume: A permanent flow measurement device that resides in the streambed where the entire stream flow is directed through a narrow opening (the throat). The water height at the entrance can be measured and correlated—using predefined calibration curves—to the flow rate through the device.

Pressure Transducer: An instrument component which detects a fluid pressure and produces an electrical, mechanical, or pneumatic signal related to the pressure detected.

Data Logger: A stand alone electronic device that records data over time either with a built in instrument/sensor or via external instruments/sensors. It is programmable to accommodate various sample rates and the data stored can be retrieved for analysis.

4.0 RESPONSIBILITIES

This section presents a brief definition of field roles, and the responsibilities generally associated with them. This list is not intended to be comprehensive and often additional personnel may be involved. Project team member information shall be included in project-specific plans (e.g., work plan, field sampling plan (FSP), quality assurance plan, etc.), and field personnel shall always consult the appropriate documents to determine project-specific roles and responsibilities. In addition, one person may serve in more than one role on any given project.

Project Manager: Selects the location for the flume, coordinates the purchase and delivery of the flume and equipment, and obtains the proper permits for construction of the flume in the stream bed with input from other key project staff.

Quality Control Manager: Overall management and responsibility for quality assurance and quality control (QA/QC). Selects QA/QC procedures for the installation of the flume, performs project audits, and ensures that data quality objectives are fulfilled.

Field Team Leader (FTL) and/or Field Geologist, Hydrogeologist, or Engineer: Provides oversight for the flume installation, supervises other personnel, and ensures compliance with SOPs and QA/QC requirements. Prepares daily logs of field activities.

Sampling Technician (or other designated personnel): Assists the FTL, geologist, hydrogeologist, or engineer in the implementation of tasks. Performs the actual flume installation, calibration, and documentation.

5.0 FLUME INSTALLATION, OPERATION, AND MAINTENANCE PROCEDURES

This section describes typical permanent flume installation, operation, and maintenance procedures.

5.1 Installation

Installation of a permanent flume includes the following four steps: (1) identify an appropriate location for the flume, (2) obtain the required federal and state permits to work within a stream and place a permanent structure in a waterway, (3) construct the flume base, diversion structure, and place the flume, and (4) check the calibration of the flume and calibrate the pressure transducers. These steps are described within this Section.

5.1.1 Identify a Location

An ideal location for a permanent flume will consist of a narrow segment of the creek with a shallow upstream slope (<2%) and a steeper downstream slope. It will have minimal alluvial material beneath where the flume will be placed and be accessible to construction equipment. The upstream conditions should promote laminar flow at the inlet of the flume while the downstream conditions should not permit submerged flow (i.e., backwater conditions). The upstream conditions should also be free of waves,

curves, and projections. An approach velocity of at least 1 foot per second will minimize the emergence of aquatic pests, insects, and sediment deposition.

The actual location will be identified by the Project Manager and other key personnel who know the objectives of the flow measurements recorded by the flume. The actual location may not be the ideal location due to the objectives. If it is necessary to raise the flume off the streambed to ensure no submerged flow, a 1:4 sloping ramp should be used upstream. If submerged flow conditions cannot be avoided, they will need to be corrected mathematically by using the results from the secondary stilling well/pressure transducer. Alternatively, a different type of flume (e.g., long-throated flume) may be substituted for the Parshall flume if submergence cannot be avoided.

5.1.2 Obtain Permits

State and federal permits may be required before any construction activities can commence. These permits may include (MT DNRC 2001):

- Montana Natural Streambed and Land Preservation Act (310)
- Montana Stream Protection Act (SPA 124 Permit)
- Montana Floodplain and Floodway Management Act (Floodplain Development Permit)
- Federal Clean Water Act (404 Permit)
- Federal Rivers and Harbors Act (Section 10 Permit)
- Short-Term Water Quality Standard for Turbidity (318 Authorization)
- Montana Land-Use License or Easement on Navigable Waters
- Montana Water Use Act (Water Right Permit and Change Authorization
- Montana Water Use Act (Water Reservations)
- Stormwater Discharge General Permits
- Streamside Management Zone Law

5.1.3 Construction

The manufacturer's instructions should be followed during installation. This section contains an overview of a typical installation.

There are four major rules that must be followed during flume construction:

1. The flume must be installed at the location and elevation specified by the Project Manager.
2. The flume must be installed with the crest level in both directions.
3. The flume must not float out of position due to grout pressure.
4. The flume's internal dimensions must not be distorted due to grout pressure.

The first construction step is pouring a concrete vault into the streambed to anchor the flume and reduce the potential for underflow. The concrete vault incorporates the approach structure (e.g., wingwalls, approach bottom slope, etc), a vault around the flume (allow roughly 18-inches of clearance between the vault wall and both sides of the flume), and an outlet structure (e.g., expansion to reduce the potential for submergence and erosion). All-thread used to secure the flume could be imbedded at this stage or drilled and epoxied into place after the concrete has set.

The second construction step is placing the flume, leveling the flume, and grouting the flume in place. The all-thread is used to ensure the crest is level but blocks (or poured piers) may be necessary to ensure that the crest is level both laterally and longitudinally since weight will be necessary to counteract the buoyancy effects of the grout (which without blocks would cause the middle of the crest to sag). Grouting must be completed in stages. The first pour should just barely cover the bottom. Chaining or vibrator sticks must be used to ensure there are no air pockets underneath the flume crest that could cause settlement in the future. The number of remaining stages depends on the flume height but in general, each pour should not be greater that 2 feet and the interior walls of the flume must be braced to reduce the chance of wall distortion.

The third construction step is backfilling around the vault and armoring the outlet structure. Backfilling around the vault should be completed with the consideration that these areas may likely be submerged during low-probability storm events (e.g., 100yr 24hr storms) and will be subjected to erosion and scouring during those events. It is also important that these backfill areas have low permeability to reduce the potential for water to seep around the flume. The outlet structure and discharge area must be armored to reduce scouring and erosion due to the increased velocity of the water exiting the flume that may cause undermining of the flume's concrete vault.

5.1.4 Calibration

Calibration of the flume includes verifying that the crest is level and that the pressure transducers are reading the same depth as the manual crest gauge.

On small flumes, a standard construction level can be used to check the crest but on large flumes, a laser level or survey equipment is necessary. If the crest is not level, the slope and angle of the crest must be recorded and these measurements must be given to the project engineer. It is possible in some cases to mathematically correct for an out-of-level crest when calculating the flow rate.

The pressure transducers are best calibrated via the manufacturer's instructions but a brief description is provided in this section. The pressure transducer must first be initialized by connecting it with the data logger and verifying communication. It is then placed within the crest stilling well and anchored to keep it from shifting within the well. Once the pressure has stabilized, the manual crest head measurement is taken and this head measurement is used to calibrate the pressure transducer. Once the calibration is stored in the data logger, the system is ready for operation.

5.2 Operation

Operating the flume consists mainly of either manually recording the head gauge within the crest or extracting the records from the data logger attached to the pressure

transducer(s). Before readings are taken, general maintenance should be completed to ensure accurate flow measurements.

Manual recording of the head gauge within the converging section (Ha) requires simply reading and recording the water level on the gauge. If excessive wave action is present on the gauge, record the average reading over a period of time—however, the presence of wave action is an indication that the approach channel needs maintenance or reconstruction. The head gauge within the diverging section (Hb) must also be recorded. Then the submergence ratio can be calculated (Hb/Ha) and compared to the manufacturer's submergence ratio criteria to determine which calculation will be most accurate for calculating flow. The head measurements are then used along with flume-specific equations or tables to calculate flow. Date, time, head measurements, and calculated flow must be recorded in the field logbook or field sheets as per SOP 9 – Field Documentation.

Automated recording of the head gauges can be used to collect data from the flume over extended periods of time. Automated recording requires two pressure transducers that are within stilling wells that have communication with the flume at the corresponding points where manual measurements are made (i.e., Ha and Hb) and a data logger. The data logger must be set up as per the manufacturer's recommendations to record pressure measurements at an interval that will achieve the objectives defined in the Sampling and Analysis Plan. The information recorded on the data logger can be downloaded and evaluated via a spreadsheet or database using the same technique that is described above for manual readings. The maximum retrieval interval (download frequency) will be based on the data logger capacity and the recording interval.

5.3 Maintenance

Maintenance of flumes includes:

- Checking the approach zone to ensure that there is no excessive sedimentation, debris, aquatic pests, or undermining. This should be completed during each visit or semi-annually, which ever is shortest. If there is any of the above identified,

the problem should be resolved by cleaning or armoring the approach zone. After rehabilitation of the zone, the flow velocity should be checked (>1 ft/sec if possible) and it should not change the flow dynamics (i.e., it should remain laminar) within the approach zone.

- Checking the flume to ensure that there is no sedimentation, debris, or aquatic pests lining the bottom and/or sides of the flume as well as the ports connecting the stilling wells to the flume. This should be completed during each visit or quarterly, which ever is shortest. Clean the flume if any of the above is present.

- Checking the flume crest for level. This should be completed annually. This can be accomplished by measuring the water depth at various locations within the crest. Any discrepancies from level must be relayed to the project engineer for correction calculations to be used in the future when calculating flow.

- Checking the discharge zone for undermining and erosion. This should be completed during each visit or semi-annually, whichever is shorter. If any problems are noted, the project engineer should be contacted and additional armoring should be added as per the engineer's recommendations.

- Checking the pressure transducers for normal operation and calibration. This should be completed during each visit. If the calibration is off, they must be re-calibrated. The battery condition must be checked and it must be verified that the remaining battery capacity is at least sufficient to last until the next scheduled visit. They should be cleaned following the manufacturer's cleaning instructions. If they are not functioning properly, they should be troubleshot using the manufacturer's guidelines. If they cannot be fixed, they must be replaced.

All maintenance activities must be recorded in the field logbook or on field forms following SOP 9—Field Documentation.

6.0 QUALITY ASSURANCE AND QUALITY CONTROL

Parshall flumes have a typical measurement error of 3-5% when free-flow conditions exist but this error increases to 20% when submerged conditions exist. Debris, aquatic pests, sedimentation within the flume, changes in the approach section, and settlement causing the flume to not be level can all be responsible for flumes to lose their accuracy.

Maintenance activities must be evaluated to determine if the problems were severe enough to warrant qualifying the records obtained. This is more important when evaluating the data quality from automated systems than from manual readings because maintenance will have taken place prior to recording a manual reading. Submerged conditions should also be noted and should be used to qualify the readings as estimates.

7.0 DECONTAMINATION

All equipment used in the construction or sampling process shall be decontaminated prior to field use and after field use. Decontamination procedures are presented in SOP-7. Personnel shall don appropriate personal protective equipment as specified in the health and safety plan. Any investigation-derived waste generated shall be managed in accordance with the procedures outlined in SOP-12.

8.0 REFERENCES

Agency for Toxic Substances and Disease Registry (ATSDR). 2006. Asbestos Exposure and Your Health.

Montana Department of Natural Resources and Conservation (MT DNRC). June 2001. Montana Stream Permitting: A Guide for Conservation District Supervisors and Others.

U.S. Environmental Protection Agency (US EPA). 2007. Guidance for Preparing Standard Operating Procedures.

U.S. Department of the Interior. Bureau of Reclamation. 2001. Water Measurement Manual: A Water Resources Technical Publication. 3rd Edition.

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Date: January 31, 2008 OU3 SOP 16 (Rev 0)

Title: SURFACE WATER SAMPLING USING DEPTH-INTEGRATED SAMPLERS

APPROVALS:

EPA Remedial Project Manager — Date: 3/20/08

SOP Author — Date: 3/20/2008

Revision Number	Date	Reason for Revision
0	1/31/08	

LIBBY ASBESTOS SUPERFUND SITE OU3

STANDARD OPERATING PROCEDURE No. 16

Surface Water Sampling Using Depth-Integrated Samplers

1.0 INTRODUCTION

This Standard Operating Procedure (SOP) describes the protocols to be followed when sampling surface water using depth-integrated samplers.

This document focuses on methods and equipment that are readily available and typically applied for sampling surface water using depth-integrated samplers. It is not intended to provide an all-inclusive discussion regarding this type of sampling. Specific sampling situations may require the adaptation of existing equipment or design of new equipment. Such innovations shall be clearly described in the project-specific sampling plan and approved by the Project Manager and the Quality Manager.

2.0 HEALTH AND SAFETY WARNING

All personnel engaged in surface water discharge measurements must follow health and safety protocols described in the health and safety plan. Asbestos fibers are thin and long fibers so small that they cannot be seen by the naked eye. Asbestos fibers are easily inhaled when disturbed and when embedded in the lung tissue can cause health problems. Significant exposure to asbestos increases the risk of lung cancer, mesothelioma, asbestosis (non-cancerous lung disease), and other respiratory diseases (ATSDR 2006).

3.0 DEFINITIONS

Centroid: The point in the increment at which discharge is equal on both sides of the point.

Isokinetic: Constant velocity.

4.0 RESPONSIBILITIES

This section presents a brief definition of field roles, and the responsibilities generally associated with them. This list is not intended to be comprehensive and often additional personnel may be involved. Project team member information shall be included in project-specific plans (e.g., work plan, field sampling plan, quality assurance plan, etc.), and field personnel shall always consult the appropriate documents to determine project-specific roles and responsibilities. In addition, one person may serve in more than one role on any given project.

Project Manager: Selects site-specific sampling methods, sample locations, and constituents to be analyzed with input from other key project staff.

Quality Control Manager: Overall management and responsibility for quality assurance and quality control (QA/QC). Selects QA/QC procedures for the sampling and analytical methods, performs project audits, and ensures that data quality objectives are fulfilled.

Field Team Leader (FTL) and/or Field Geologist, Hydrogeologist, or Engineer: Implements the sampling program, supervises other sampling personnel, and ensures compliance with SOPs and QA/QC requirements. Prepares daily logs of field activities.

Sampling Technician (or other designated personnel): Assists the FTL, geologist, hydrogeologist, or engineer in the implementation of tasks. Performs the actual sample

collection, packaging, and documentation (e.g., sample label and log sheet, chain-of-custody record, etc).

5.0 DEPTH-INTEGRATED SAMPLING PROCEDURES

5.1 BACKGROUND

At flowing-water sites, collection of an isokinetic, depth-integrated, discharge weighted sample is standard procedure; however, site characteristics, sampling-equipment limitations, or study objectives constrain how a sample is to be collected and could necessitate use of other methods. If the QC plan calls for collection of concurrent samples, then the relevant procedures and equipment needs must be reviewed before field work begins.

Isokinetic, depth-integrated methods are designed to produce a discharge-weighted (velocity-weighted) sample; that is, each unit of stream discharge is equally represented in the sample, either by dividing the stream cross section into intervals of equal width (EWI) or equal discharge (EDI). The analyte concentrations determined in a discharge-weighted sample are multiplied by the stream discharge to obtain the analyte loading rate. If used correctly and the sample is collected within the limitations of the sampling device being used, the EWI and EDI methods result in samples that have identical constituent concentrations.

5.2 DEPTH-INTEGRATED SAMPLE COLLECTION

The Equal Discharge Interval (EDI) Method of depth-integrated sampling will be employed at the Libby Asbestos Site. The objective of the EDI method is to collect a discharge-weighted sample that represents the entire flow passing through the cross section by obtaining a series of samples, each representing equal volumes of stream discharge. The EDI method requires that flow in the cross section be divided into increments of equal discharge. Equal-volume, depth-integrated samples are collected at the centroid of each of the equal-discharge increments along the cross section.

5.2.1 EQUIPMENT

A depth-integrated surface water sample will be collected according to one of the following, or similar, techniques.

1. Direct Method -- Sample bottle is uncapped and inverted, submerged to the specified depth, turned upright pointing upstream, removed from the water, and then capped. Add preservative, if any, after sample collection.

2. Bailer Method -- A appropriate sampling bailer with a ball check valve is submerged to the desired sample depth, either directly or by suspending the bailer on a rope from a pole.

3. Peristaltic Pump Method -- The sample is collected through a section of new, clean, flexible Tygon (polyvinylchloride) tubing. The tubing intake will be secured manually or by attaching weights. This procedure may be modified to collect the sample through a Teflon tube into a sample flask by running the pump on a vacuum.

4. Kemmerer Bottle – Use a properly decontaminated Kemmerer bottle. Set the sampling device so that the upper and lower stoppers are pulled away from the body, allowing water to enter the tube. Lower the pre-set sampling device to the predetermined depth. Avoid disturbing the bottom. Once at the required depth, send the weighted messenger down the suspension line, closing the device. Retrieve the sampler and discharge the first 10-20 mL from the drain to clear water that may not be representative of the sample. Repeat as needed to fill collect the needed volume.

5. Van Dorn Sampler – Set the device so that the end stoppers are pulled away from the body allowing surface water to enter the tube. Lower the sampler to the predetermined depth. Once at the required depth send the weighted messenger down the suspension line, closing the sampling device. Retrieve the sampler and decant the first 10-20 mL from the drain to clear water that may not be representative of the sample from the valve. Repeat as needed until the required volume to fill sample bottles is collected.

6. Bacon Bomb Sampler -- Lower the bacon bomb sampler carefully to the desired depth, allowing for the trigger to remain slack at all times. When the desired depth is reached, pull the trigger line until taught. This will allow the sampler to fill. Release the trigger line and retrieve the sampler. Decant the first 10-20 mL from the drain to clear water that may not be representative of the sample from the valve. Repeat as needed until the required volume to fill sample bottles is collected.

The selection of sampling equipment listed above depends on the site conditions and sample type required. In addition, the following equipment is needed to collect surface water samples:

- Field notebook, indelible marker
- Global Positioning System (GPS) unit
- Marking stakes
- Digital Camera
- Compass
- 100 m measuring tapes
- Detergent solution (0.1-0.3 % Alconox)
- Distilled water
- Latex gloves
- Ziploc bags
- Paper Towel
- Chain of custody and sample labels
- Coolers
- Sample bottles
- Plastic sheeting

5.2.2 SAMPLING PROCEDURE

There are four steps to collecting a depth-integrated sample: (1) prepare for sampling, (2) select the number and location of equal-discharge increments, (3) select the transit rate, and (4) collect sample. These steps are discussed in more detail below.

1. Prepare for Sampling
    a. Upon arrival at the field site, set out safety equipment such as traffic cones and signs. Park vehicle (if using) in a location and direction so as to prevent sample contamination from vehicle emissions.
    b. Assemble equipment needed and set up a clean work space.

- **Organic compounds**. Select equipment with fluorocarbon polymer, glass, or metal components if components will directly contact samples to be analyzed for organic compounds. **Do not use plastics other than fluorocarbon polymers.**
- **Inorganic constituents**. Select equipment with components made of fluorocarbon polymer or other relatively inert and uncolored plastics or glass if components will directly contact samples to be analyzed for inorganic constituents. **Do not use metal or rubber components for trace-element sampling.**

2. Select the number and location of equal-discharge increments.

The number of increments to sample should be stated in the Sampling and Analysis Plan.

3. Select the transit rate.
   a. Determine the sampling depth (e.g., water surface to sediment) and the mean stream velocity at the centroid of each equal-discharge increment.
   b. Determine the transit rate of the sampler for each centroid that will yield sub-samples with approximately the same volume (within 10 percent) using sampling depth, mean stream velocity, and sampler intake flow rates (available from the manufacturer). When compositing sub-samples, ensure that the same volume is composited from each sub-sample.
4. Collect sample water.

The procedures are the same whether you are wading or using a reel-and-cable suspension method.

- Collect sub-samples at EDI centroids as many times as necessary to ensure collection of sufficient sample volume for analysis. If the sample is to be composited, care must be taken to obtain approximately the same total volume (± 10 percent) from each EDI centroid so that the composited cross-sectional sample will be proportional to flow at the time of sampling.

- Stay within the isokinetic transit-rate range of the sampler at each centroid. If flow velocity is less than the isokinetic transit-rate range of the sampler, a discharge-

weighted sample still can be obtained by collecting equal volumes at each centroid; however, this sample will not be isokinetic.

a. Move sampling and support equipment to the centroid of the first increment to be sampled. Properly decontaminate the sampling equipment following SOP-7 (Equipment Decontamination).
b. Read and record the starting gage height. Record sampling start time.
c. Lower the sampler at the predetermined transit rate until slight contact is made with the streambed.
    - Do not pause upon contacting the streambed. Raise the sampler immediately at a constant transit rate to complete the vertical traverse.
    - Take care not to disturb the streambed with the sampler. Disturbing the streambed could cause bed material to enter the sampler, resulting in erroneous data.
    - Ensure that the sampler container has not overfilled. If the sampler container is overfilled, then repeat the sampling process using a quicker transit time or a reduce number of passes.

d. Inspect each sub-sample, looking for overfilling and (or) the presence of anomalously large amounts of particulates that might have been captured because of excessive streambed disturbance during sample collection. If you note either or both of these conditions, discard the sample, making sure there are no residual particulates left in the container, and resample.
e. Ensure that the sampler container is not underfilled (that the minimum volume has been collected). Underfilling will result in a sub-sample that is not isokinetically collected—usually because the maximum transit rate has been exceeded.
f. Depending on study objectives, either process and (or) analyze the sub-sample collected at the initial centroid as a separate sample, composite this sub-sample with other sub-samples collected along the cross section, or split the sub-sample for further processing.

- If the total volume of the sub-samples that will be collected will exceed the operational capacity of the churn or cone splitter, decrease the number of increments or use a smaller nozzle.
- Ensure that all particulates in the sampler bottle or bag are transferred with the sample by swirling the sample gently to keep particulates suspended, and quickly pouring the sample into the churn or cone splitter.

g. Move equipment to the next vertical.
   - Determine the transit rate for this vertical. If the sub-samples are composited, the total volume collected at each centroid must be equal.
   - Repeat procedures, steps 4 c-f.
   - Repeat this process at the remaining verticals along the cross section.

h. Record the following information after all samples have been collected:
   - Sampling end time.
   - Ending gage height.
   - All field observations and any deviations from standard sampling procedures.

i. Process Samples
   - Process the samples according to the SAP and SOP-8 (Sample Handling).

j. Clean Equipment
   - Clean and decontaminate all equipment following SOP-7 (Equipment Decontamination).

6.0 QUALITY ASSURANCE AND QUALITY CONTROL

Field splits, equipment rinsates, and matrix spike samples shall be collected at the frequencies documented in the field sampling plan. Calibration checks shall be performed at least once prior to and at least once following each day of instrument use in the field and the results documented on the Sampling Record for each sampling station in accordance with SOP 10 (Field Equipment Calibration). All sampling data

must be documented in the field logbooks and/or field forms, including rationale deviations from this SOP. The Field Team Leader, or designated QA reviewer, shall check and verify that field documentation has been completed per this procedure and other procedures referenced herein. All equipment must be operated according to the manufacturer's specifications, including calibration and maintenance.

7.0 DECONTAMINATION

All equipment used in the sampling process shall be decontaminated prior to field use and after field use. Decontamination procedures are presented in SOP-7 (Equipment Decontamination). Personnel shall don appropriate personal protective equipment as specified in the health and safety plan. Any investigation-derived waste generated shall be managed in accordance with the procedures outlined in SOP-12 (Investigation Derived Waste Management).

8.0 REFERENCES

Agency for Toxic Substances and Disease Registry. 2006. Asbestos Exposure and Your Health.

US Geological Survey (USGS). 2006. National Field Manual for the Collection of Water-Quality Data. U.S. Geological Survey Techniques of Water-Resource Investigations, Book 9, Chapter A4. Collection of Water Samples. Available online at http://water.usgs.gov/owq/FieldManual/chapter4/pdf/Chap4_v2.pdf

Libby Site OU3 SOP No. 17
Rev. No. 0

Precipitation Monitoring

SOP will be prepared by MWH.

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NOTE: This SOP has been prepared for use at the Libby Asbestos Superfund Site. The applicability of this SOP at other sites should be evaluated by the site team with regard to site-specific goals and objectives.

Date: December 6, 2007 SOP No. ISSI-LIBBY-01 (Rev. 10)

Title: SOIL SAMPLE PREPARATION

SYNOPSIS: A standardized method for preparation of soil samples for asbestos analysis at the Libby Asbestos Superfund Site is described.

Original Author: William Brattin Syracuse Research Corporation[1]

Received by QA Unit:

APPROVALS:

TEAM MEMBER	SIGNATURE/TITLE	DATE
EPA Region 8:	[illegible]	12/11/07
Syracuse Research Corp.	W S Brattin	12/10/07

[1] This SOP was originally prepared by ISSI Consulting Group. ISSI is no longer in existence, and finalization of the SOP was performed by Syracuse Research Corporation (SRC).

REVISION LOG

Revision Number	Revision Date	Reason for Revision
1	1/7/00	Incorporation of sieving to the sample preparation.
2	7/12/00	Revision in sieve size, other minor edits.
3	5/7/02	Incorporate minor edits
4	8/1/02	Modify sieving procedure, add grinding step
5	3/6/03	Incorporate modifications to the procedure and documentation requirements
6	3/24/03	Incorporate modifications to the log-sheets to conform with electronic data storage requirements and add grinder blank requirements.
7	8/5/03	Incorporate modifications to drying and sample storage procedures
8	5/4/04	Incorporate modifications to drying batch size and recording of preparation information
9	5/14/07	Incorporate modifications so as to expand use to other Operable Units (removed references to OU4 / CSF, changed Index ID to Sample ID). Repair formatting. Remove reference to missing Figure 1. Add optional use of electronic logs. Oven temperature set to 90±10 degrees C. Lowered inventory batch size from ~120 to ~50 samples so that one inventory batch can fit in one tub. Designate drying batch as one batch per oven (~20 samples). Allow for optional use of disposable drying pans. Remove direction to NOT move grinding plates during decontamination (new BICO design allows plates to be separated for decontamination without adjusting gap). Ovens will be calibrated daily. *[Note: Revision 9 was an unsigned version that reflects changes made at the Troy Preparation Laboratory. Some of the changes in Revision 9 are retained in Revision 10, below].*
10	12/06/07	Incorporate modifications so as to expand use to other Operable Units. Designate drying batch as ~20 samples. Allow for optional use of disposable drying pans. Allow alternative methods for decontamination of plate grinder. Clarify and modify QC requirements. General editing for clarity.

1.0 PURPOSE

This Standard Operating Procedure (SOP) has been prepared by the United States Environmental Protection Agency (USEPA) Region 8 to standardize the methods used to prepare soil samples from the Libby Asbestos Superfund Site for the analysis of asbestos content. This procedure is intended for use by employees of USEPA Region 8 and by contractors and subcontractors supporting USEPA Region 8 projects and tasks for the Remedial Investigation work performed at the Libby site. Deviations from the procedures outlined in this document must be reviewed and approved by the USEPA Region 8 Remedial Project Manager or Regional Chemist.

2.0 RESPONSIBILITIES

Each laboratory that performs soil preparation activities under this SOP must have a designated Preparation Laboratory Project Leader (PL^2). The PL^2 may be an USEPA employee or contractor. The PL^2 is responsible for ensuring that all personnel in the laboratory who perform work under this SOP are familiar with the SOP, and for ensuring that all work performed satisfies the requirements of this SOP and any other relevant laboratory-specific operating procedures. It is also the responsibility of the PL^2 to communicate and document the need for any deviations from the SOP with the appropriate USEPA Region 8 Remedial Project Manager or Regional Chemist.

All laboratory personnel preparing Libby soil samples are responsible for reading and understanding the requirement of this SOP, and for performing all applicable tasks in accordance with this SOP. Any laboratory worker who identifies any issues or encounters any difficulties in implementation of this SOP is responsible for promptly communicating the issue or difficulty to the PL^2. In addition, all laboratory personnel are responsible for reading and understanding the Health and Safety Plan (HASP) applicable to the soil preparation activities in that laboratory, and performing all tasks in accord with the requirements of that HASP.

3.0 EQUIPMENT

- General purpose laboratory oven - capable of maintaining a constant temperature of approximately 90°C.

- Analytical balance - capable of measuring in a range of 0.1 g to at least 2000 g, calibrated and accurate to the tolerance limits indicated in Attachment 2.

- Riffle splitter - with 3/4 inch chutes to split samples.

- Plate grinder - capable of accepting soil particles of approximately 1/4 inch diameter and grinding to produce particles of approximately 250 µm.

- HEPA Vacuum - A portable vacuum unit equipped with a high efficiency particulate air (HEPA) filter to remove any asbestos fibers and other soil particles from the exhaust air. Used to decontaminate equipment and maintain general laboratory cleanliness.

- Metal scoop or spoon - for transferring samples. Plastic scoops or spoons are not acceptable.

- 1/4 inch metal sieve and catch pan - for coarse sieving samples. Plastic sieves and pans are not acceptable.

- 60 mesh (250 µm) and 200 mesh (74 µm) metal sieves - for verification of the plate grinder settings. Plastic sieves are not acceptable.

- Clean quartz sand - required for preparation of grinding and drying blank samples and for decontamination of grinder.

- Clean soil - required for calibration of grinder.

- Drying pans with lids - used during the sample drying process, lids used to cover samples during transfer

- Sample containers - plastic ziplock bags (pint and gallon size).

- Gloves - for personal protection and to prevent cross-contamination of samples. May be plastic or latex. Disposable, powderless.

- Personal Protective Equipment - as specified in the applicable Health and Safety Plan for the soil preparation laboratory.

- Laboratory notebook and pen - used to record progress, any problems or observations and deviations. All information in the laboratory notebook must be recorded in pen (not pencil).

- Sample Drying Log Sheets - (Attachment 1). Used to record all sample drying information.

- Sample Preparation Log Sheets - (Attachment 1). Used to record all sample preparation information (splitting, sieving and grinding).

- Equipment Calibration and Maintenance Logs for:

  - Analytical Balance (Attachment 2)
  - Plate Grinder (Attachment 3)
  - Ventilation Hood (Attachment 4)
  - HEPA Vacuum (Attachment 5)
  - Drying Oven (Attachment 6)

  These logs are used to record all maintenance and calibration records for the listed equipment. If hard copy, all entries must be recorded in pen, and the logs must be organized and maintained in a laboratory notebook.

- Sample Labels – Self-adhesive labels for attachment to sample bags.

- Trash Bags - used to dispose of gloves, wipes and other investigation derived waste.

- Indelible Marking Pen - used to record sample information onto plastic ziplock bags and to record logbook information.

4.0 METHOD SUMMARY

Figure 1 provides an overview of the steps in the soil preparation process. Soil samples received from the field are first dried in a laboratory oven and are then split into a preparation sample and an archive sample. The preparation sample is sieved to separate coarse material (> 1/4 inch) from fine material (< 1/4 inch). The fine material is ground to a particle size of less than 250 µm, and this fine ground material is split into several aliquots. This grinding step is needed to achieve a reasonable degree of homogeneity in the sample, and to allow for preparation of slides for microscopic analysis. The coarse fraction (if any) and one aliquot of the fine ground material are then sent to an analytical laboratory for asbestos analysis by methods specified in the project-specific Sampling and Analysis Plan. At present, the fine-ground sample is generally analyzed by Phase Contrast Microscopy (Visual Area Estimation) (PLM-VE) in accord with the most recent version of SOP SRC-LIBBY-03, and the coarse material is examined by stereomicroscopy and any observable particles of asbestos are removed and weighted in accord with the most recent version of SOP SRC-LIBBY-01.

It should be noted that this preparation method, coupled with these analytical techniques, is intended to estimate the total mass fraction of asbestos that is present in a sample, without regard

to the current size distribution of the asbestos particles. That is, no distinction is drawn between asbestos that is presently in a large "lump" that is non-respirable and free asbestos fibers that are readily released to air and inhaled. Because of this, concentration values based on this approach may tend to overestimate the amount of currently releasable fibers, but do provide an estimate of the total amount of fibers that may be releasable in the future.

5.0 SOIL STORAGE

Upon receipt at the soil preparation facility, samples will be grouped into an inventory batch of 50-120 samples. Samples will be archived according to the inventory batch they are assigned to and filed by the Inventory Batch ID (box number) noted in the Sample Drying Log and Sample Preparation Log (Attachment 1).

6.0 BULK SOIL DRYING

6.1 Equipment Calibration

Samples will be weighed prior to and following drying activities. The analytical balance used for drying activities will be calibrated on days when samples are loaded into, or unloaded from, the oven. Before weighing samples, calibrate the balance using S-1 class weights and record all measurements, any required maintenance, and the balance number in the Analytical Balance Calibration and Maintenance Log (Attachment 2).

All drying activities will be performed under a negative pressure HEPA filtered hood or similar containment box. Prior to loading the oven, the ventilation hood will be calibrated to ensure that the ventilation system is operating properly. Ventilation hood calibration and any required maintenance will be documented in the Ventilation Hood Calibration and Maintenance Log (Attachment 4).

A HEPA vacuum will be used to decontaminate the oven following the removal of dried samples. Vacuum calibration will be performed daily, prior to drying activities. All system checks, required maintenance and the vacuum number will be recorded in the Vacuum Maintenance Log (Attachment 5).

Oven temperature calibration will be performed on a daily basis (during periods of operation). Oven temperature calibration and any required maintenance will be documented in the Oven Temperature Calibration and Maintenance Log (Attachment 6).

6.2 Drying Procedure

- Prior to unsealing and drying each sample, record on the Sample Drying Log the starting sample mass to the nearest 0.1 g. Include the technicians initials and the date.

- Group samples into drying batches of approximately 20 samples per batch. Assign each batch a drying batch number, and record this number on the Sample Drying Log, along with the SOP and Revision Number and the oven number used to dry the samples.

- Include one preparation blank in each drying batch. See Section 12.1 for more details regarding preparation blanks.

- Set the oven temperature to approximately 90±1°C. For every drying batch, check the oven temperature to verify that proper temperature[2] has been reached and document the start date/time and temperature in the Sample Drying Log.

- Transfer each sample to be dried from its ziplock storage bag into a clean drying pan. Each sample should be transferred to its respective drying pan under the negative pressure HEPA filtered hood. Label each drying pan with the Index ID[3] of the sample. Place each sample in the oven.

- Leave the samples in the oven for approximately 24-48 hours or until completely dry. Verify that each sample is dry by squeezing a portion of the soil with a freshly gloved thumb and forefinger to test the cohesiveness. Once it is confirmed that samples are dry, record the technician's initials, and the date and time of completion, in the Sample Drying Log.

- Turn off the oven and allow the samples to cool in the oven. Once the samples are cooled, unload each sample and transfer each sample volume to a clean ziplock bag, re-bag the sample with another clean ziplock bag and identify the dried sample with the Index ID. All samples should be transferred to ziplock bags under the negative pressure HEPA filtered hood to prevent potential exposure to fibers that might be released from the sample.

- Record the sample mass of each dried and bagged sample to the nearest 0.1 g along with the technician's initials and the date in the Sample Drying Log.

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[2] Drying temperatures in the range of 80-100°C will not compromise sample integrity, but monitoring of oven temperature to ± 1°C is needed to allow early detection of any problems with the oven temperature control.

[3] Unique sample identifiers at the Libby site are referred to as "Index ID" numbers rather than "Sample ID" numbers. However, the meaning is the same.

6.3 Decontamination

Decontaminate the inside of the hood and the inside of the drying oven by HEPA vacuuming and wet wiping all surfaces before loading a new batch for drying.

If drying pans are to be re-used, decontaminate all sample drying pans under the ventilation hood using compressed air and a HEPA vacuum to remove any residual organic material left on the pans. Wet wipe or brush off any visible material that is not removed using the vacuum.

7.0 DIVISION OF ARCHIVE AND PREPARATION SAMPLES

All dried samples are mixed and split into two portions: one portion is held in archive, and the second portion is prepared for asbestos analysis. The sections below describe the sample splitting procedure.

7.1 Equipment Calibration

Prior to any splitting, sieving, or grinding activities, calibrate the ventilation hood to ensure that the ventilation system is operating properly. Document ventilation hood calibration and any required maintenance in the Ventilation Hood Calibration and Maintenance Log.

7.2 Procedure for Sample Splitting

Splitting must be performed in the hood to prevent potential exposure to fibers that might be released from the sample. Samples will be divided using the following steps:

- Place the cooled, re-bagged samples in the hood, and knead the contents of the bag to break up any soil clumps.

- Place one collection pan on each side of the riffle splitter. Pour the sample from its plastic bag through the splitter in order to divide the sample into two equal sub-parts.

- After splitting, set aside one portion for sample preparation, as described below. If the mass of the portion for preparation is larger than about 200 grams, split the preparation sample again so that 3/4 of the original sample will be archived and 1/4 will be set aside for processing.

- Place the remaining portion(s) into a clean, ziplock bag, re-bag the sample in another clean ziplock bag, and store as an archive sample in the event additional analyses are required in the future. Identify the archive sample with the Index ID and the suffix "A" (for archive fraction). Record the technician's initials and date in the Sample Preparation

Log. Store the archive portion in the numbered inventory box noted in the Sample Preparation Log.

7.3 Preparation Duplicate Samples

One preparation duplicate sample will be prepared for every 20 field samples processed. A preparation duplicate is generated by using the riffle splitter to divide the preparation fraction into two equivalent portions ("parent" and "duplicate"). The duplicate portion is assigned an independent Index ID and both the parent sample and the duplicate sample are then processed in an identical fashion and are each submitted to the laboratory blind. For further information on preparation and processing of preparation duplicates, refer to Section 12.4.

7.4 Performance Evaluation Samples

Performance Evaluation (PE) samples are used to assess the accuracy of the analytical laboratory and to check for any potential contamination or loss of asbestos during processing. For further information on preparation and processing of PE samples, refer to Section 12.3.

7.5 Decontamination

The splitter need not be decontaminated following this step if the next use of the splitter will be the division of the fine ground fraction of the same samples into four fractions (see Section 10, below). If for any reason the next use of the splitter is division of material from a different sample, the riffle splitter must be decontaminated as follows.

- Use a HEPA vacuum and compressed air to decontaminate the splitter and brush or wipe off any visible material that is not removed by the air blast. The splitter is now ready to process the next sample.

8.0 SIEVING THE PREPARATION SAMPLE

All preparation samples are sieved prior to grinding to separate out the coarse and fine fractions. The sample sieving procedure is described in the sections below.

8.1 Equipment Calibration

All sieving activities will take place in the hood. Refer to Section 6.1 for details regarding the frequency of ventilation hood calibration.

Samples are weighed during sieving activities. The analytical balance will be calibrated daily with S-1 class weights before processing begins. All measurements, any required maintenance,

and the analytical balance number will be recorded in the Analytical Balance Calibration and Maintenance Log.

8.2 Sample Sieving Procedure

Samples will be sieved using the procedure outlined below.

- Pour the sample onto a clean 1/4 inch stainless-steel sieve with a clean pre-weighed catch pan. Shake the screen until all particles <1/4 inch in size have passed through the screen into the pan. When needed, a pestle may be used to gently break up any remaining soil clumps to ensure all particles <1/4 in size pass through the screen.

- Pour all material which does not pass through the screen (>1/4 inch) into a new, tared, sample bag. This is the Coarse Fraction.

- Weigh and record the mass of the coarse fraction to the nearest 0.1 g in the Sample Preparation Log and record the technician's initials and the date. If all of the material passes through the screen, such that there is no coarse fraction, record a mass of zero for the coarse fraction in the Sample Preparation Log.

- Double-bag the coarse sample portion and identify the sample with the Index ID and "C" suffix on the sample bag. Coarse fraction samples are now ready to be packaged for shipment to the analytical laboratory or archived as directed.

- All material that passes through the 1/4 inch screen is the Fine Fraction. Weigh and record the mass of the fine fraction to the nearest 0.1 g in the Sample Preparation Log.

Whenever possible, immediately process the fine fraction material in accord with the approach described in Section 9.3 (below). If processing cannot occur immediately, pour the fine fraction material into a new ziplock bag and identify the fine sample material with the Index ID and the suffix "F" (for "fine fraction"). Double-bag the sample and identify the sample with the Index ID and suffix on the outside of the bag.

8.3 Decontamination

All non-disposable pans and sieves will be decontaminated between samples. Decontaminate sieves and pans (and the pestle, if used) under the ventilation hood using compressed air. Wipe or brush off any visible material that is not removed from the air blast. A HEPA vacuum may also be used to remove any residual material.

9.0 GRINDING THE FINE FRACTION

The fine fraction of each preparation sample will be ground to produce a material of about 250 µm[4]. The procedure for grinding the fine fraction is outlined below.

9.1 Equipment Calibration

All grinding activities will take place in the hood. Refer to Section 7.1 for details regarding the frequency of ventilation hood calibration.

A HEPA vacuum will be used to decontaminate the hood and processing equipment, following the preparation of each sample. Vacuum calibration will be performed daily, prior to grinding activities. All system checks, required maintenance and the vacuum number will be recorded in the Vacuum Maintenance.

A plate grinder will be used to process samples. The grinder will be calibrated daily or after any adjustments are made to the plates. To verify proper particle size (approximately 250 µm), and demonstrate that samples will not be over-processed, grind a sample of clean soil (rather than quartz sand) and sieve using stacked sieves. Clean soil will be provided by the United States Geological Survey (USGS). Unlike the coarseness of quartz sand, clean soil will more accurately approximate the typical grain size and texture of the Libby samples being processed and will reduce the chance of over-processing.

The grinder is adjusted acceptably if, after grinding of the clean soil sample, all material passes through a 60-mesh (250 µm) screen and is substantially retained by a 200-mesh (74 µm) sieve. If a significant amount of the ground clean soil sample is retained on the 60-mesh screen, or if a substantial fraction of the material passes through the 200-mesh screen, adjust the plates of the grinder until these targets are achieved. If the required particle size cannot be achieved even after plate adjustment, other grinder maintenance such as plate replacement may be required. Regardless, grinding of field samples cannot resume until the desired particle size is achieved. Document the grinder number, verification of acceptable adjustment and any observations in the Grinder Calibration and Maintenance Log.

Samples will be weighed following grinding activities. The analytical balance will be calibrated daily with S-1 class weights before processing begins. All measurements, any required maintenance, and the analytical balance number will be recorded in the Analytical Balance Calibration and Maintenance Log.

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[4] Note that the particle size is cited as "approximately 250 µm". This is due to the nature of grinding asbestos material. Some material that is longer than 250 µm may pass through the grinder if its longest side is parallel with the vertical grinder plates. The material that comes in contact more nearly perpendicular to the vertical grinder plates will be ground to <250 µm

9.2 Grinding Blanks

One grinding blank per grinder will be prepared daily, and will be associated with all samples prepared by that grinder on that day. For further information on grinding blanks refer to Section 12.2.

9.3 Grinding of Fine Field Samples

The sample portion that was sieved to < 1/4 inch will be ground to a particle size of approximately 250 µm. Set up a catch pan under the grinder to collect all the ground material. Take the fine sample set aside in Section 8.2, load the grinder hopper, and allow the fine sample to pass through the plate grinder into the catch pan. Note the technician's initials, date of grinding, and grinder number in the Sample Preparation Log.

The net recovery of fine ground material must not be less than 90% of the mass of fine material placed into the grinder. If recovery is less than 90%, soil grinding must be stopped and the grinder re-adjusted until the mass recovery of test sand and/or soil samples exceeds 90%.

9.4 Decontamination

Plate Grinder

The details of decontamination of the plate grinder and it associated containers and equipment may vary depending on the model of grinder that is being used.

If the plate grinder can be readily disassembled for cleaning without altering its grinding properties, disassemble the grinder and clean the chutes and plates with the HEPA vacuum and compressed air. Then, if needed, use wet wipes to ensure decontamination. If wet wipes are used, the plates and chutes must be thoroughly dried before reassembly. If the grinder is not easily disassembled, clean the grinder with the HEPA vacuum and several blasts of compressed air, paying special attention to areas where dust from the grinding process is known to accumulate (e.g., between the plates and areas adjacent to the catch pan clamps). Then, pass an aliquot of approximately 20 g of quartz sand through the grinder to clean out any residual soil. Discard the quartz sand and re-clean the grinder with the vacuum and another round of high pressure air blasts. After this decontamination procedure, the grinder is ready to process the next sample.

In general, all soil containers, hoppers and catch pans associated with use of the grinder should be decontaminated by using a HEPA vacuum and/or wet wipes, followed by a blast of high pressure air.

Calibration Sieves

The stacked sieves used to calibrate the plate grinder will be decontaminated using a HEPA vacuum and compressed air between calibration uses.

10.0 SPLITTING OF THE FINE GROUND SAMPLE

The fine ground soil sample should be distributed into four approximately equal subsamples using a splitter. All splitting activities will be performed in the hood. Refer to Section 7.1 for details regarding the frequency of ventilation hood calibration.

10.1 Splitting Procedure for Fine Ground Sample

The following method for splitting a soil sample was adapted from EPA 540-R-97-028 (USEPA, 1997):

- Set up one receiving pan on each side of the splitter. Load the soil from the grinder catch pan (Section 9.3) into the splitter, collecting the sample in two receiving pans.

- Tap the catch pan vigorously several times to free any remaining material. Tap the splitter to facilitate the flow of all material through the chutes into the receiving pans.

- Empty one receiving pan into the grinder catch pan and the other receiving pan into the sieve catch pan. Set the sieve catch pan aside; this portion of fine ground sample will be split again later.

- Replace the receiving pans under the splitter. Take the grinder catch pan, containing half of the fine ground sample, and re-load the contents into the splitter as detailed above. Repeat the process of dispersing the sample material by shaking the catch pan and tapping the splitter to uniformly distribute the sample. The resulting splits are the "FG1" and "FG2" portions in the Sample Preparation Log.

- Take these two portions and carefully transfer each into a clean, tared, ziplock sample bag. Re-bag one sample portion in another clean ziplock sample bag and identify this fine ground sample with the Index ID, the suffix "FG" (for "fine fraction, ground") and the fraction number 1, (ex. CS-12345-FG1 for fine ground fraction #1). Identify the bagged second portion with the Index ID, the suffix "FG" and the fraction number 2 and set aside to be re-bagged with the following fine ground portions:

- Place the two empty receiving pans from the "FG1" and "FG2" portion next to the splitter. Repeat the splitting procedure using the other fine ground portion set aside in the sieve pan and split the remaining sample material to create the "FG3" and "FG4" portions.

- Take the remaining "FG3" and "FG4" portions and carefully transfer each into a clean, tared, ziplock sample bag, identify each remaining fine ground sample with the Index ID as noted above.

- Weigh each sample portion (FG1 through FG4), and record each mass along with the technician's initials and date in the Sample Preparation Log.

Combine all of the bagged coarse and fine portions of the sample into one large clean, ziplock sample bag.

Coarse and fine ground samples are now ready to be packaged for shipment to the analytical laboratory or archived as directed. When samples are requested for shipment, the "FG1" fraction will be sent first. If further analyses are required for the fine ground portion, the subsequent fractions will be double bagged and sent (i.e., FG-2 then FG-3, etc.). All archived fine ground portions will be filed in the appropriate inventory archive box noted in the Sample Preparation Log.

10.2 Decontamination

The splitter must be decontaminated between each sample. Use the vacuum and/or wet wipes followed by a blast of compressed air to decontaminate the splitter and brush or wipe off any visible material that is not removed by the vacuum or air blast. The splitter is now ready to process the next sample.

11.0 DOCUMENTATION

Index ID numbers are recorded in the Sample Drying Log, Sample Preparation Log and on all sample containers. Sample Drying Logs and Sample Preparation Logs will be filed or archived according to their associated dry batch and preparation batch number. If revisions to the Sample Drying Log and/or Sample Preparation Log are necessary, the appropriate parties will be notified of the changes, however, these changes will not necessitate revision to the current standard operating procedure, a modification form will be filled out to document the revisions.

As mentioned above, the following equipment calibration and maintenance logs will also be maintained:

- Daily analytical balance calibration using S-1 class weights (Attachment 2)
- Daily grinder setting verification for calibration check and/or post-adjustment verification, grinder maintenance as necessary (Attachment 3)
- Daily ventilation hood operating condition verification (i.e., inline filter checks, changes) (Attachment 4)
- HEPA vacuum maintenance and bag changes (Attachment 5)
- Weekly oven temperature calibration, oven maintenance as necessary (Attachment 6)

In addition, a laboratory notebook will be maintained by each individual or team that is preparing samples. For each day that samples are processed, the following information should be collected:

- Date
- Time
- Personnel
- Personal protective equipment (PPE)
- SOP (including revision number) and any other laboratory-specific governing plan being followed
- Descriptions of any deviations to the SOP, the reason for the deviation and/or any modification forms being followed
- Summary of laboratory activities (including number of samples prepared, and equipment calibrated and used)

12.0 QUALITY CONTROL

Quality control (QC) samples are inserted into the sample train to monitor for potential contamination introduced during the preparation process or to assess accuracy of analysis that may be affected due to preparation procedures. If samples results indicate the occurrence of contamination or inconsistent results, the PL^2 will be notified. The PL^2 will then notify the EPA Regional Project Manager and the Regional Chemist in order to review laboratory procedures and identify any changes in preparation laboratory methods and procedures that may be necessary. Any such reviews and resultant changes will be documented accordingly by the PL^2.

12.1 Preparation Blanks

A preparation blank is a sample of 200-400 grams of clean quartz sand that is treated identically to a field soil sample. That is, the preparation sample is dried in the oven along with the field soil samples, split into archive and preparation fractions using a riffle splitter, screened through a ¼ inch screen (even though there are no particles larger than ¼ inch), and ground by passing through the plate grinder. This type of sample is intended to detect contamination that may occur at any stage of the soil preparation procedure.

At least one preparation blank will be processed with each drying batch of approximately 20 field samples. Preparation blanks will be assigned a random and unique Index ID and will be submitted to the laboratory blind. The Index ID assigned to each preparation blank must be in accord with the numbering system specified in the program-specific project plan.

Detection of asbestos fibers (any type) in any preparation blanks at a level greater than Non-detect (Bin A) by PLM-VE should be taken as a sign of potential cross-contamination, and all field samples associated with the preparation batch for the preparation blank having detectable asbestos (> Bin A) will be reviewed and qualified appropriately if detectable levels of asbestos are also found in any of the corresponding field samples. If the overall fraction of preparation blanks that contains detectable asbestos (> Bin A) exceeds 1%, a review of laboratory procedures should be undertaken to identify and address the source of the contamination.

12.2 Grinding Blanks

A grinding blank consists of 100-200 grams of clean quartz sand that is passed through the plate grinder. The purpose of this type of sample is to evaluate the effectiveness of decontamination procedures for the plate grinder.

One grinding blank per grinder will be prepared for each day that field samples are being ground. Each grinder used in the laboratory will be assigned a number and all samples processed will be associated with the grinder used for preparation. The grinder number used for each sample will be noted in the Sample Preparation Log. Grinding blanks will not be dried, split for archive, or sieved. Rather, a grinding blank will only be ground and split into four fine ground samples. The grinding blank is assigned a random and unique Index ID and is submitted to the laboratory blind. The Index ID assigned to each grinding blank must be in accord with the numbering system specified in the program-specific project plan.

Detection of asbestos fibers (any type) in any grinding blank at a level greater than Non-detect (Bin A) should be taken as a sign of potential cross-contamination, and all field samples associated with the grinding blank that reports detectable asbestos (> Bin A) will be reviewed and qualified appropriately if detectable levels of asbestos are also found in any pf the corresponding field samples. If the overall fraction of grinding blanks that contains detectable asbestos (> Bin A) in a soil preparation facility exceeds 1%, steps should be taken to develop an improved method for grinder decontamination.

12.3 Performance Evaluation Samples

Performance Evaluation (PE) samples are samples of Libby soil that have been spiked with a known amount of Libby Amphibole (LA) asbestos. These samples were prepared by the USGS

for use at the Libby site by spiking uncontaminated soil from Libby with a known mass of LA fibers collected at the mine site, and then grinding the sample to a particle size of ≤ 250 um as described above. Several different concentration values of PE samples were prepared, ranging from < 0.1% to 2%. Each bottle contains about 100 grams of the PE material.

PE samples will be utilized in two ways.

First, the soil preparation facility will insert untreated PE samples into the analytical sample train sent to the laboratory for PLM-VE analysis. This type of PE sample is intended to evaluate the performance of the analytical laboratory (rather than the preparation facility).

Second, the soil preparation laboratory will process PE samples in the same way that field soil samples are processed, as detailed below. This type of PE sample is intended to determine if there is any loss of asbestos during sample processing. In addition, considered in conjunction with a grinding blank that is passed through the decontaminated grinder immediately following the PE sample, the PE sample will also be used to facilitate assessment of grinder decontamination procedures.

The frequency of each type of PE sample (unprocessed and processed) should be one per month for each month in which soil processing is occurring. These should be distributed approximately evenly between the different concentration values that are available for PE samples.

Each month that soil processing is occurring, the procedure to be followed for generation and submittal of PE samples is as follows:

1. Select a PE bottle for inclusion.
2. Thorough mix the contents of the PE bottle by inversion (a minimum of 10 times) and/or rolling (a minimum of 10 minutes).
3. Remove an aliquot of about 20 grams and package this for submission to the analytical laboratory without any processing. If more than one laboratory is analyzing samples, rotate the submittal of unprocessed samples so that all laboratories receive approximately equal total number of unprocessed PE samples.
4. Take the remainder of the PE bottle (about 80 grams) and carry this material through the full sequence of steps applied to each field sample, starting with oven drying. After splitting the dried sample with the riffle splitter, recombine the samples so that the full 80 grams is screened through the ¼ inch sieve and passed through the plate grinder. Thus, there is no archive split for PE samples. After grinding and splitting, this should result in four sub-samples of processed PE sample. Prepare three of these for submittal to the analytical laboratories, and hold one sample in archive.

Results of PE samples processed by the soil preparation laboratory are evaluated by comparing the reported results for LA to the nominal results. Deviations from nominal may be the result of variations either in soil processing procedures and/or in the analytical procedure. If the frequency of strongly discordant results (i.e., the results of the PE sample differ by more than one bin from the nominal result) exceeds 10%, then the source of the inconstancy should be investigated and remedied.

12.4 Preparation Duplicates

A preparation duplicate is prepared by using a riffle splitter to divide a field soil sample into two approximately equal portions, creating a parent and duplicate sample. Both samples are then processed in the same fashion. The preparation duplicate is assigned a unique Index ID, and is submitted to the laboratory blind. The Index ID assigned to each preparation duplicate must be in accord with the numbering system specified in the program-specific project plan.

One preparation duplicate sample will be processed for every 20 field samples prepared (5%). Results from duplicate samples serve to evaluate the precision of the combined sample preparation process and the laboratory analysis. Inconsistent results between parent and duplicate may be due either to variability in sample preparation, sample analysis, and/or to small scale variability in the sample that is not fully controlled by mixing and splitting. If the overall frequency of strongly discordant results (i.e., the results for the parent sample and duplicate are different by more than one bin) is greater than 10%, steps should be taken to identify and address the source of the variability in the sample preparation procedure.

13.0 DECONTAMINATION

All non-disposable equipment used during soil sample preparation must be decontaminated prior to use. Scoops, spoons, splitters, sieves and drying pans that are re-used must be decontaminated with a HEPA vacuum, compressed air, wet-wiping and/or by brushing off any residual material. If soil particles are visible on any of the equipment, repeat the decontamination procedure until the equipment is clean. To reduce the potential for human exposure in the laboratory, COMPRESSED AIR SHOULD BE USED CAREFULLY AND ONLY UNDER VENTED HOODS.

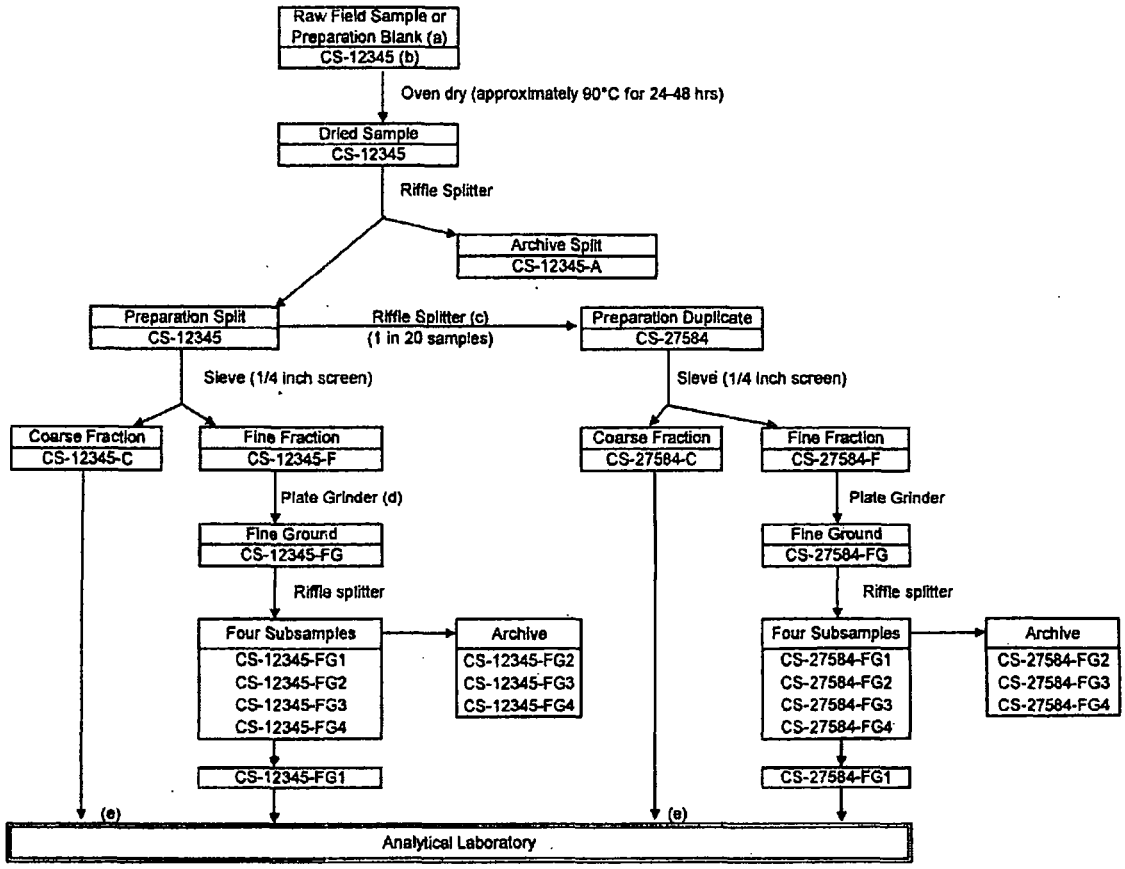
Detailed decontamination procedures for specific equipment are noted in Sections 6.3, 7.5, 8.3, 9.4, and 10.2.

14.0 REFERENCES

American Society for Testing and Materials. 1998. Standard Practice for Reducing Samples of Aggregate to Testing Size, ASTM Designation: C 702 - 98, 4 p.

USEPA. 1997. Superfund Method for the Determination of Releasable Asbestos in Soils and Bulk Materials. EPA 540-R-97-028.

FIGURE 1 SOIL PREPARATION FLOW DIAGRAM



NOTES:

(a) A preparation blank (200-400 grams of clean silica sand) is prepared in the same way as field samples at a rate of 5%

(b) Example Index ID (sample number) shown to illustrate naming conventions

(c) A preparation duplicate is prepared at a rate of 5%

(d) A grinding blank (100-200 grams of clean sand) is passed through the plate grinder and split into 4 sub-samples at a rate of 5%

(e) Coarse sample will be returned to EPA for archive after laboratory analysis

ATTACHMENT 1

SAMPLE DRYING AND SAMPLE PREPARATION LOG SHEETS

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Sample Drying Log Sheet

Laboratory Name: _____ Sheet No.: _____

Drying Begun: date _____ time _____

Drying Complete: date _____ time _____

Oven number: _____

Oven temp: _____°C

	Index ID	Inventory ID No.	SOP and Rev No.	Sample mass (g)			Original Sample ID and Notes (indicate if preparation blank)	QC
				Before drying	After Drying	Initials and date		Initials and Date
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								
11								
12								
13								
14								
15								
16								
17								
18								
19								
20								
21								
22								
23								
24								
25								

Sample Preparation Log Sheet

Laboratory Name: _____ Sheet No.: _____

Preparation Batch: _____

	Index ID	SOP and Rev No.	Inventory ID	Drying Batch ID	Archive Sample Splitting	Duplicate Sample Splitting	Sieving			Sample Grinding		Sample Splitting					Original Sample Identification and Notes (indicate if grind blank, prep blank, or duplicate pair. For duplicate pair enter the parent ID)	QC
							Sample Mass (g)					Sample Mass (g)						
					Initials and date	Initials and date	Coarse Fraction > 1/4"	Fine Fraction < 1/4"	Initials and Date	Initials and Date	Grinder #	FG1	FG2	FG3	FG4	Initials and Date		Initials and Date
1																		
2																		
3																		
4																		
5																		
6																		
7																		
8																		
9																		
10																		
11																		
12																		
13																		
14																		
15																		
16																		
17																		
18																		
19																		
20																		

The following preparation steps require Technician Initials and Date to document activity: Sample Drying, Archive Sample Splitting, Preparation Duplicate Splitting, Sieving, Homogenization, Sample Splitting

ATTACHMENT 2

ANALYTICAL BALANCE CALIBRATION AND MAINTAINANCE LOG SHEET

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Preparation Laboratory = _____

Balance # = _____

Measurement Number		S - 1 Class Weight Measurements				Measurement within range? Yes or No	If "No" Recalibrate	Technician Initials	QC check Initials
	Calibration Weights	0.1 g	1 g	10 g	100 g				
	Tolerance Limit Range	0.05 - 0.15 g	0.90 - 1.10 g	9.75 - 10.25 g	99.00 - 101.00 g				
	Date								
1									
2									
3									
4									
5									
6									
7									
8									
9									
10									
11									
12									
13									
14									
15									
16									
17									
18									
19									
20									
21									
22									
23									
24									
25									

The analytical balance calibration will be verified daily.
All tolerance limits are standard tolerance limits for Class S-1 weights.
After 20 measurements, the tolerance range will be evaluated for reasonableness.
Weights falling outside the range require that the balance be recalibrated using all S-class weights

Sheet No.: Balance - _____

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ATTACHMENT 3

GRINDER CALIBRATION AND MAINTAINANCE LOG SHEET

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Preparation Laboratory = _____

Grinder # = _____

Date	Activity Type (D, A, M)	Sieve Test 60 mesh acceptable Y or N	Sieve Test 200 Mesh acceptable Y or N	Notes (Include description of action/maintenance)	Technician Initials	QC Check Initials

D = daily calibration
A = Adjustment Calibration Verification
M = Maintenance Only
For the 60 mesh sieve test to be acceptable all of the clean soil must pass through the sieve.
For the 200 mesh sieve test to be acceptable a substantial portion of the ground soil must be retained on the sieve.
Failure of either sieve test requires adjustment of the plates followed by adjustment verification prior to grinding samples.

Sheet No.: Grinder - _____

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ATTACHMENT 4

VENTILATION HOOD CALIBRATION AND MAINTAINANCE LOG SHEET

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Preparation Laboratory = _____

Ventilation Hood # = _____

Date	In-line HEPA indicators acceptable?	Notes (Include description of action/maintenance)	Technician Initials	QC Check Initials

Ventilation system operating conditions should be verified daily
In-line system should indicate the velocity/negative flow is 100 ft/min. If not, the problems with the system must be corrected prior to processing samples. Note actions or maintenance taken.

Sheet No.: Hood - _____

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ATTACHMENT 5

HEPA VACUUM CALIBRATION AND MAINTAINANCE LOG SHEET

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Preparation Laboratory = ______________________________

Vacuum # = _________________

Date	System Check Acceptable? Yes or No	HEPA filter clean? Yes or No	Bag fill level acceptable? Yes or No	Notes (Include description of action/maintenance performed)	Technician Initials	QC Check Initials

Physically check the vacuum suction and note audible change in motor daily. If significant reduction in the vacuum suction or motor "strain" is audibly noted then the system check is unacceptable. If unacceptable, perform and document the HEPA filter and bag maintenance checks above and perform maintenance.

Sheet No.: Vacuum - ______________

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ATTACHMENT 6

OVEN CALIBRATION AND MAINTAINANCE LOG SHEET

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Preparation Laboratory = _____

Oven # = _____

Date	Certified Temperature Reading (°C)	Thermometer ID	System Check Acceptable? (90+/-1 °C) Yes or No	Notes (Include description of action/maintenance performed)	Technician Initials	QC Check Initials

Sheet No.:Oven - _____

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LIBBY SUPERFUND SITE STANDARD OPERATING PROCEDURE

APPROVED FOR USE IN LIBBY SUPERFUND SITE ONLY

Date: April 21, 2004 SOP No. SRC-LIBBY-01 (Rev. 2)

Title: QUALITATIVE ESTIMATION OF ASBESTOS IN COARSE SOIL BY VISUAL EXAMINATION USING STEREOMICROSCOPY AND POLARIZED LIGHT MICROSCOPY

Author Sally M. L. Gibson Syracuse Research Corporation

SYNOPSIS: A standardized method is described for the examination of the coarse fraction (>1/4") of soil samples using stereomicroscopy and polarized light microscopy (PLM) to identify, segregate, and estimate the mass percent of asbestos in the sample matrix.

Received by QA Unit:

APPROVALS:

TEAM MEMBER	SIGNATURE/TITLE	DATE
EPA Region 8	Valerie Waldrade	4/26/04
Syracuse Research Corp.	W S Brattin	4/26/04

Revision	Date	Reason for Revision
0	11/12/02	--
1	5/20/03	Provided clarification on dealing with very small particles.
2	4/21/04	Included statements on limitations of intended use

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide a standardized screening method for the visual examination of the coarse fraction of previously sieved soil samples for evidence of asbestos mineral content using stereomicroscopy with confirmation of asbestos content by polarized light microscopy (PLM). This SOP incorporates salient components of EPA Test Method 600/R-93/116 *Method for Determination of Asbestos in Bulk Building Materials* and National Institute of Occupational Safety and Health (NIOSH) Method 9002 *Asbestos (bulk) by PLM*, Issue 2.

This procedure will be used by employees of contractors/subcontractors supporting USEPA Region 8 projects and tasks for the Libby, Montana, site. Deviations from the procedure outlined in this document must be approved by the USEPA Region 8 Remedial Project Manager or Regional Chemist prior to initiation of sample analysis.

2.0 PREREQUISITE TRAINING

Visual examination will be performed according to this SOP by a laboratory accredited by the National Voluntary Laboratory Accreditation Program (NVLAP) and by analysts proficient either by education or experience in asbestos mineral identification by stereomicroscopy and PLM. Analyst familiarity with the procedural applications prescribed in EPA Test Method 600/R-93/116 and NIOSH Method 9002 is required.

Training as described in the Sampling and Analysis Plan, Remedial Investigation, Contaminant Screening Study, Libby Asbestos Site, Operable Unit 4, (CSS SQAPP [CDM 2002]) will be provided to laboratory personnel or laboratories with less than one year of project-specific experience by "mentors" from either Reservoir Environmental Services, Inc. or EMSL.

3.0 RESPONSIBILITIES

The CDM Laboratory Coordinator (LC) is responsible for overseeing the activities of the CDM Soil Preparation Laboratory and subcontracted laboratories performing sample analysis for the Libby, Montana, project. The LC is also responsible for checking all work performed and verifying that the work satisfies the specific tasks outlined by this SOP and the CSS SQAPP. It

is the responsibility of the LC to communicate with the project personnel and subcontracted laboratory regarding specific analysis objectives and anticipated situations that require any deviation from the CSS SQAPP SOPs. In addition, it is the responsibility of the LC to communicate the need for any deviations from this SOP with the CDM Project Manager, USEPA Region 8 personnel (Remedial Project Manager or Regional Chemist.)

Subcontracted laboratory analysts performing the visual examination are responsible for adhering to the applicable tasks outlined in this SOP and substantiating components of the reference procedures (EPA 1993; NIOSH 1994) with the modifications contained herein.

4.0 EQUIPMENT

- Analytical balance - accurate to 0.01 g, range of 0.01 g to 1000 g (for weighing total sample)
- Analytical balance - accurate to 1 mg (for weighing asbestos)
- Traceable standards - major asbestos types
- Microscope - binocular stereomicroscope, 5-60X approximate magnification
- Microscope - polarized light, binocular or monocular with a cross hair reticle (or functional equivalent) and magnification of at least 8X
  - 10X, 20X, and 40X objectives
  - 360 degree rotatable stage
  - substage condenser with iris diaphragm
  - polarizer and analyzer which can be placed at 90 degrees to one another and calibrated relative to the cross-line reticle in the ocular
  - port for wave plates and compensators
  - wave retardation plate (Red I Compensator) with ~550 nanometer retardation and known slow and fast vibration directions
- Light Sources - incandescent or fluorescent

- Tweezers, dissecting needles, scalpels, probes, razor knives, etc. - standard sample manipulation instruments/tools

- Microscope slides and cover slips

- Refractive index liquids

- Pre-tared glassine paper, glass plates, weigh boats, petri dishes, watchglasses, etc. - laboratory sample containers

- HEPA-filtered or Class 1 biohazard hood negative pressure

- Three-ring binder book- binders will contain Microscopic Examination Logbook Sheets (Attachment 1)

5.0 METHOD

Soils from the Libby, Montana site will be dried, sieved, and prepared according to the most recent revision of SOP ISSI-LIBBY-01, Soil Sample Preparation. The coarse fraction of the soil sample is defined as that portion of the sample which does not pass through a 1/4" sieve. The coarse fraction will be weighed, placed in a zip-top plastic bag, and labeled as described in Camp, Dresser, and McKee (CDM) SOP 1-3 (with project-specific modifications). The samples will be packaged and shipped by the soil preparation laboratory as described in CDM SOP 2-1 (with project-specific modifications) and transferred to the laboratory via chain-of-custody procedures described in CDM SOP 1-2 (with project-specific modifications).

The following sections describe the stereomicroscopic and PLM examination. Materials tentatively characterized as asbestos by stereomicroscopy will be isolated and subjected to confirmation by PLM. The mass % of Libby amphibole asbestos, other amphibole asbestos, and chrysotile asbestos in the coarse soil fraction will be calculated from the mass of each asbestos type positively identified by PLM and the original sample weight. Figure 1 provides an overview of the process.

5.1 Stereomicroscopic Examination

The laboratory will receive the coarse fraction soil samples from the CDM Soil Preparation Laboratory. The entire sample will be weighed and placed in an appropriate container. The weight of each coarse sample will be recorded, along with the sample identification, on the Microscope Examination Logbook Sheet. The sample will be subject to stereomicroscopic examination and particle segregation as depicted Figure 1. The stereomicroscopic examination to identify and segregate asbestos includes:

- using multiple fields of view over the entire sample
- probing the sample by turning pieces over and breaking clumps where possible
- manipulating the sample using appropriate instruments/tools
- observing homogeneity, texture, friability, color and extent of any observed asbestos in the sample(s)

NOTE: Although the coarse fraction is prepared by sieving with a 1/4" screen, particles smaller than 1/4" may be present in the fraction due to adherence between coarse and fine particles. This may even include some very fine asbestos fibers. Because of the technical difficulty, the analyst should not attempt to physically segregate and weigh particles smaller than about 2-3 mm (1/10 inch). A particle this size is expected to have a mass of about 10-20 mg, which is less than 0.1% of a sample whose total mass is 25 grams. If no particles larger than 2-3 mm are present, this should be noted in the data sheet for each category of asbestos using the following code system:

- ND = No asbestos observed
- Tr = Trace levels of asbestos observed but not quantified

The weight fraction for any asbestos type marked "ND" or "Tr" in a given sample is not calculated and is left blank.

As the sample is examined, the analyst will continue segregation of the sample until the entire coarse soil fraction has been characterized as either "non-asbestos" or "tentatively identified asbestos." The tentatively identified asbestos particles will be examined by PLM, as described below. The stereomicroscopist will initial and date the Microscopy Examination Logbook Sheet.

5.2 PLM

The coarse material tentatively identified as asbestos by stereomicroscopic examination will be subject to confirmation using PLM, as described in SOP SRC-LIBBY-03 (Revision 0) ("Analysis of Asbestos Fibers in Soil by Polarized Light Microscopy"). The PLM examination will be used to confirm that the particles tentatively classified as asbestos are actually asbestos, and will be assign each particles to one of three categories:

LA = Libby amphibole
OA = Other amphibole
C = Chrysotile

If OA is observed, the type of OA observed should be noted in the data sheet using the following code system:

- AMOS = Amosite
- ANTH = Anthophyllite
- CROC = Crocidolite
- UNK = Unknown

The total weight of each type of positively identified asbestos (LA, OA, C) will be determined and recorded on the Microscopic Examination Logbook Sheet, along with the analyst's initials and the date of the examination.

6.0 QUALITY ASSURANCE

Laboratories performing the examination must be accredited by NVLAP. "Calibration" should be verifiable for each microscopist in terms of project-specific training and the successful analysis of materials of known asbestos content (NVLAP test samples, in-house standards) similar to those anticipated to be observed in Libby, Montana soils. Additionally, references such as photographs of the asbestos minerals illustrating distinguishing properties should be available benchside during characterization.

Quality control samples as described in ISSI-LIBBY-01 (i.e., preparation duplicates) will not submitted for the coarse materials samples. The entire coarse fraction will be subject to examination.

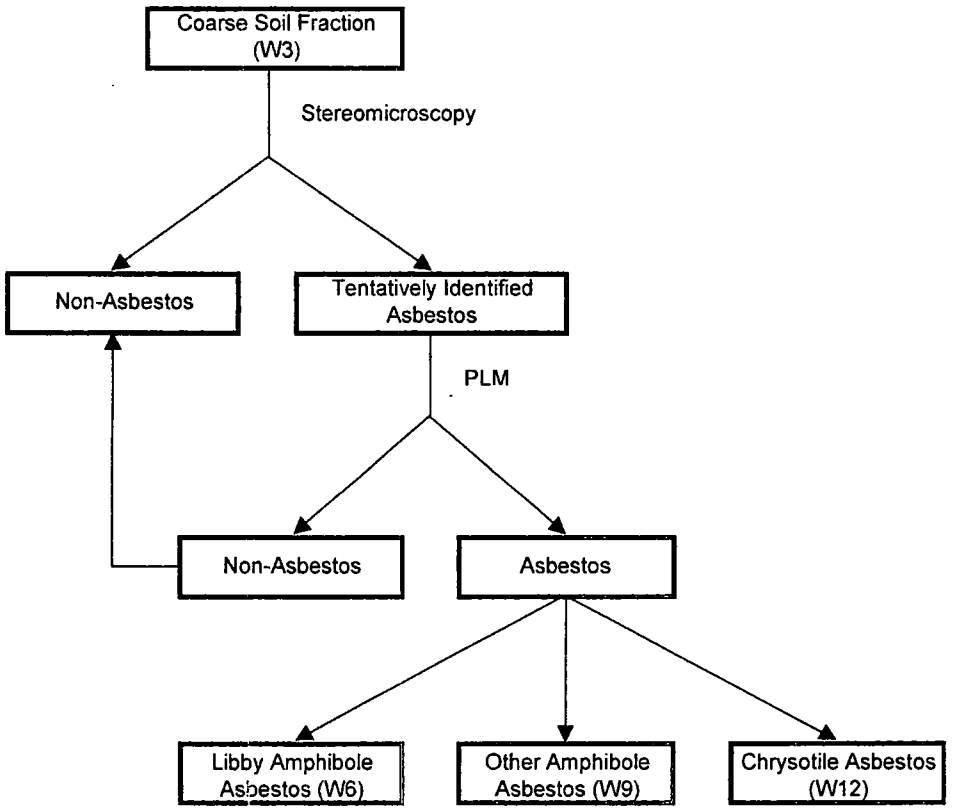
7.0 REFERENCES

CDM 2002. *Sampling and Analysis Plan, Remedial Investigation, Contaminant Screening Study, Libby Asbestos Site, Operable Unit 4.* 3282-116-PP-SAMP-14187. Camp, Dresser and McKee Denver, Colorado. April.

NIOSH 1994. National Institute of Occupational Safety and Health (NIOSH) Method 9002 *Asbestos (bulk) by PLM*, Issue 2.

USEPA 1993. *Method for Determination of Asbestos in Bulk Building Materials.* 600/R-93/116.

Figure 1. Overview of Sample Examination Process



W3 = Original coarse soil fraction mass (g)
W6 = If present in measurable quantities, mass (mg) of Libby amphibole
W9 = If present in measurable quantities, mass (mg) of other amphibole
W12 = If present in measurable quantities, mass (mg) of chrysotile

Codes used in the illustration (e.g., W3) correspond to Data Log Sheet

ATTACHMENT 1

MICROSCOPIC EXAMINATION LOGBOOK SHEET

SRC-LIBBY-01 Data sheet and EDD.xls
(Check with Volpe or SRC to determine the latest version number)

Example hard copy of data entry sheet shown on next page (for illustration purposes only).

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Data Log Sheet v6 for SOP SRC-LIBBY-01

Stereomicroscopic and Gravimetric Analysis of Coarse Soil

Lab Name:

SOP Version:

Lab Job No.

Page _____ of _____

Calculated automatically in the "Electronic Data Entry" form. Do not enter data here.

EPA Index ID	Index Suffix	Lab Job-Sample No	Status (1 = Analyzed, 2 = Missing, 3 = Contam, 4 = Cancelled)	QA Type (Not QA)	Total Sample Weight (g): Tare Weight (g) Empty Container	Total Sample Weight (g): Mass of Sample + Container (g)	Total Sample Weight (g): Mass of Sample (g)	Analysis Details: Analyst Initials	Analysis Date	Mass of Asbestos Fractions (mg): Libby Amphibole (LA) Asbestos: LA Qual* (ND, Tr)	Libby Amphibole (LA) Asbestos: Tare Weight - Container (mg)	Libby Amphibole (LA) Asbestos: Mass of LA + Container (mg)	Libby Amphibole (LA) Asbestos: Mass (mg) LA	Other Amphibole (OA) Asbestos: OA Qual* (ND, Tr)	Other Amphibole (OA) Asbestos: OA Type** (AMOS, ANTH, CROC, UNK)	Other Amphibole (OA) Asbestos: Tare Weight - Container (mg)	Other Amphibole (OA) Asbestos: Mass of OA + Container (mg)	Other Amphibole (OA) Asbestos: Mass (mg) OA	Chrysotile (C) Asbestos: C Qual* (ND, Tr)	Chrysotile (C) Asbestos: Tare Weight - Container (mg)	Chrysotile (C) Asbestos: Mass of C + Container (mg)	Chrysotile (C) Asbestos: Mass (mg) C	% LA	% OA	% C	Comments (see Notes below)

Notes:

*Qualifier codes: ND = No asbestos observed.
Tr = Trace levels observed but not quantified.

**OA Type codes: AMOS = Amosite
ANTH = Anthophyllite
CROC = Crocidolite
UNK = Unknown

Comment Codes (user-defined):

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LIBBY SUPERFUND SITE STANDARD OPERATING PROCEDURE
APPROVED FOR USE IN LIBBY PE STUDY ONLY

ANALYSIS OF SOIL-LIKE MEDIA FOR ASBESTOS BY POLARIZED LIGHT MICROSCOPY

Date: July 21, 2005 SOP No. SRC-LIBBY-03 (Revision 2)

Title: **ANALYSIS OF ASBESTOS FIBERS IN SOIL BY POLARIZED LIGHT MICROSCOPY**

Author: William Brattin

SYNOPSIS: A semi-quantitative method for identifying and quantifying asbestos fibers in soil using polarized light microscopy (PLM) is provided. This method is based on NIOSH Method 9002, EPA Method 600/R-93/116, and CARB Method 435, with project-specific modifications intended specifically for use at the Libby Superfund Site.

APPROVALS:

TEAM MEMBER	SIGNATURE/TITLE	DATE
USEPA Region 8		
Syracuse Research Corp.		

Revision	Date	Principal Changes
0	03/03/03	--
1	12/11/03	Clarify binning assignment of samples at 0.2%
2	07/21/05	Provided more detail on refractive index for oils

1.0 PURPOSE

The purpose of this standard operating procedure (SOP) is to provide a standard approach for semi-quantitative analysis of asbestos in samples of soil or other soil-like materials using polarized light microscopy (PLM). This SOP is specifically intended for application at the Libby Superfund site.

2.0 SCOPE AND APPLICATION

This method is intended mainly for analysis of asbestos in soil or other similar soil-like media. This method is appropriate for the analysis of all types of asbestos fibers, including both chrysotile and amphiboles, including those that are characteristic of the Libby site.

3.0 RESPONSIBILITIES

It is the responsibility of the laboratory supervisor to ensure that all analyses and quality assurance procedures are performed in accord with this SOP, and to identify and take appropriate corrective action to address any deviations that may occur during sample preparation or analysis. The laboratory supervisor should also communicate with project managers at EPA or their oversight contractors any situations where a change from the SOP may be useful, and must receive approval from EPA for any deviation or modification from the SOP before proceeding with sample preparation and analysis.

4.0 METHOD DESCRIPTION

The soil sample to be evaluated for asbestos content by PLM is examined under stereomicroscopy and under PLM (3-5 slides per sample) to estimate the amount of asbestos present. Quantification of the amount of asbestos present may be done either using a visual estimation approach or by a point counting approach, as specified in the Chain of Custody request. In either case, the concentration of Libby amphibole asbestos in the sample is estimated in terms of mass fraction (i.e., percent asbestos by weight) based on the use of project-specific reference materials (calibration standards).

5.0 DETAILED METHOD

5.1 Basic Methods

All qualitative and quantitative analyses are to be performed in general accordance with the methods and techniques specified in NIOSH 9002, EPA 600/R-93/116, and CARB Method 435. Project-specific modification, clarifications, and requirements are provided below.

5.2 Visual Estimation Approach

5.2.1 Classification of Asbestos Mineral Type

Based on fiber attributes (morphology, refractive index, color, birefringence, etc.), asbestos in the sample is classified into one of three categories:

Code	Description	Notes
LA	Libby Amphibole	Refractive index values for LA span the standard values for tremolite/actinolite (EPA 1993), but may include values for other similar amphiboles (e.g., winchite, richterite) characteristic of the mine at Libby. Based on analysis of 4 different samples from the mine (Wylie and Verkouteren 2000; USGS, unpublished data; Verkouteren, personal communication), **observed refractive indices of Libby amphiboles** range from about 1.629-1.640γ and 1.614-1.623α, with a birefringence of about 0.017. The full range of refractive indices of samples from the mine may be somewhat greater. **Based on these data, when confirming the identity of LA, the target refractive index (RI) for oil is about 1.620-1.630. See Attachment 2 for details on preparing oils of a specified RI.**
OA	Other amphibole	Includes amphibole forms (e.g., amosite, crocidolite, anthophyllite) that are not thought to occur in significant amount at the mine in Libby
C	Chrysotile	

5.2.2 Estimation of LA Mass Percent

The visual area estimation is a semi-quantitative approach that requires the microscopist to estimate the area fraction of the total material present in a field of view that consists of asbestos material. Because this estimation may be difficult, especially at low concentration values, and because the desired output is an estimate of mass fraction (rather than area fraction), all visual estimates of Libby amphibole content will be performed using a set of site-specific reference materials (calibration standards) as a frame of reference. These reference material will contain either 0.2 % or 1.0% Libby amphibole by weight[1], and have been prepared for analysis using the same approach as for field samples. Using the two reference concentrations (0.2% and 1.0%) as a visual guide, the microscopist will evaluate the field sample and report the results as follows:

PLM Laboratory Report			Description
Qual	Conc (wt.%)	Bin	
ND		A	Asbestos was not observed in the field sample
Tr		B1	Asbestos was observed in the field sample at a level that appeared to be lower than the 0.2% reference material
<	1	B2	Asbestos was observed in the field sample at a level that appeared to be approximately equal to or greater than the 0.2% reference material but was less than the 1% reference material.
	1, 2, 3, etc	C	Asbestos was observed in the field sample at a level that appeared to equal or exceed the 1% standard. In this case, the mass percent is estimated quantitatively.

[1] The nominal mass fraction of the reference materials (calibration standards) is based on the gravimetric fraction of the material that is soil and the amount that is spiking material, adjusted for the fraction of the spiking material that is LA. For example, if the spiking material were estimated to contain 85% LA by mass, then the 1.0% calibration standard would contain 1.18 grams of spiking material (1.00 grams of LA) per 100 grams of calibration standard. Because the estimate of LA content of the spiking material is approximate, the true concentration of a calibration material may not be precisely equal to the nominal value.

"ND" (not detected) in the Qualifier column is used for all samples in which asbestos is not observed under stereomicroscopy and is also not detected in five (5) different PLM slides prepared using representative sub-samples of the test material. These samples are assigned to **Bin A**.

"Tr" (trace) in the Qualifier column is used for all samples in which asbestos is observed either under stereomicroscopy or in at least one out of 3-5 PLM slides prepared from representative sub-samples of the test material, and in which the amount of asbestos present appears to be less than the 0.2 % reference material. These samples are assigned to **Bin B1**.

"<" (less than) in the Qualifier column and 1 in the Concentration column is used for all samples in which asbestos is observed either under stereomicroscopy or in PLM slides prepared from representative sub-samples of the test material, and in which the amount of asbestos present appears to be equal to or greater than the 0.2 % reference material but less than the 1% reference material. These samples are assigned to **Bin B2**.

A numeric value (1, 2, 3, etc) in the Concentration column without an entry in the Qualifier column is used for all samples in which asbestos is observed either under stereomicroscopy or in PLM slides prepared from representative sub-samples of the test material, and in which the amount of asbestos present appears to be similar to or greater than the 1 % reference material. These samples are assigned to **Bin C**.

Note that because these reference materials are based on Libby amphibole, they are not appropriate for estimating the mass percent of other types of asbestos (chrysotile, other types of asbestos). Therefore, if any asbestos types besides Libby amphibole are observed, the reported values for those samples should be in units of area percent.

5.3 Point Counting Approach

5.3.1 Counting Procedure

Any analysis in which evaluation by point counting is requested will be performed in general accordance with the descriptions provided in EPA/600/R-93/116 and CARB Method 435. The

total number of particles to be counted (generally 400 or 1000) will be specified in the Chain of Custody request.

Take eight sub-samples of the soil sample and mount each separately with the appropriate refractive index liquid. The preparations should not be heavily loaded. Each sample should be uniformly dispersed to avoid overlapping particles and allow 25-50% empty area within the fields of view.

An ocular reticule (point array) or cross-hair is used to visually superimpose points on the microscope field of view. Count 1/8 of the total points required on each of the 8 slides (e.g., 50 non-empty points per slide for a 400 point count and 125 non-empty points per slide for a 1000 point count). For each non-empty point counted, assign the particle that is present at the point into one of four bins:

- Not asbestos
- Libby asbestos (LA)
- Other asbestos (OA)
- Chrysotile asbestos (C)

In order for a particle to be counted as asbestos, the aspect ratio must be ≥ 3:1.

After the required total number of non-empty points have been counted, record the total number of points in the LA, OA and C bins on the point counting data sheet.

5.3.2 Estimation of Mass Percent

Like visual estimation, the output of the point counting approach is an estimate of area fraction, not mass fraction. For this site, point-count estimates of area fraction for Libby amphibole particles will be converted into estimates of mass fraction using a standard curve approach.

The standard curve will be prepared using a series of site-specific reference materials (calibration standards) containing 0%, 0.2%, 0.5%, 1%, or 2% Libby amphibole. The area fraction of each reference material will be estimated by the point counting approach in quadruplicate. The standard curve will be prepared by plotting the mean area fraction

determined by point counting versus the mass percent in the reference material. The mass fraction of a field sample will be determined by measuring the area fraction of the field sample and locating the mass fraction that corresponds to that area fraction on the standard curve.

Because the standard curve is based on Libby amphibole, it is not appropriate to utilize this standard curve for other types of asbestos. Therefore, if any asbestos types besides Libby amphibole are observed, the reported values for those samples should be in units of area percent.

6.0 APPARATUS AND MATERIALS

Polarized light microscope, with lens and filters
Stereomicroscope (approximately 10-45x)
Petri dish for stereomicroscopic sample examination
Spatula and forceps
Glass slides and cover slips
Refractive Index (RI) oils
Reference Materials (Calibration Standards)
- Soil containing 0.2% LA by mass
- Soil containing 0.5% LA by mass
- Soil containing 1.0% LA by mass
- Soil containing 2.0% LA by mass

Laboratory log book
Data recording sheet (Attachment 1)
Liquid calibration conversion table (Attachment 2)

7.0 QUALITY ASSURANCE/QUALITY CONTROL

7.1 Precision and Accuracy

PLM by visual estimation and point counting are both semi-quantitative methods. For the purposes of this project, the accuracy and precision of the method are evaluated by measuring the frequency with which samples are assigned to the correct "bins". Data on precision and accuracy of bin assignment will be collected in the future and used to establish performance criteria for this project.

7.2 Method Proficiency

At present, sufficient data are not available to establish a quantitative procedure for method proficiency demonstration. As results become available, a procedure will be established and applied, based on the analysis of a set of blind Performance Evaluation materials and assessing the frequency of correct bin assignments. If the assignments reported by a laboratory are within acceptance criteria bounds (see Section 7.1), then that laboratory will be deemed proficient. If not, remedial actions must be taken to address the errors before work may begin by that laboratory.

8.0 RECORDS

8.1 PLM Data Forms

Analysts will record analytical results using the electronic data sheets developed for the Libby project, as presented in Attachment 1. Note that there are two different electronic forms; one is for use in visual area estimation, and the other is for use in point counting. Once completed and checked, these spreadsheets are submitted to EPA for upload into the database. The laboratory should retain all original records for use in resolving any questions until otherwise instructed by EPA.

8.2 Instrument Maintenance Logbook

An individual instrument maintenance logbook should be kept for each piece of equipment in use at the laboratory. All maintenance activities must be recorded in the appropriate logbook.

8.3 Data Storage and Archival

Electronic Data. Each day of data acquisition, all electronic files will be saved onto two separate media. For example, the data may be saved onto a computer hard drive, but must also be backed up onto a type of portable media such as CD-ROM, floppy disc, or tape. Portable media will be maintained in a single location with limited access.

Hardcopy Data. All data sheets and micrographs must be stored in a secured location with limited access (e.g., locking file cabinet) when not in use.

Copies (hardcopy and electronic) of the raw analytical data will be submitted to USEPA for archival.

9.0 REFERENCES

CARB 435. California Environmental Protection Agency Air Resources Board, Method 435, Determination of Asbestos Content in Serpentine Aggregate. June 6, 1991.

EPA. 1993. Method for the Determination of Asbestos in Bulk Building Materials. United States Environmental Protection Agency, Office of Research and Development. EPA/600/R-93/116. July 1993.

EPA. 2003. Technical Memo 8. Procedure for Combining Mass Fraction Estimates for Coarse and Fine Fractions of Soil. Prepared by US EPA Region 8 with technical assistance from Syracuse Research Corporation.

NIOSH. 1994. Asbestos (Bulk) by PLM. NIOSH Manual of Analytical Methods, Fourth Edition. National Institute of Occupational Safety and Health. August 15, 1994.

Wylie AG and Verkouteren JR. 2000. Amphibole Asbestos from Libby, Montana: Aspects of Nomenclature. American Mineralogist 85:1540-1542.

ATTACHMENT 1

PLM DATA RECORDING SHEETS

PLM (VE and PC) Data Sheet and ED.xls
(Check with Volpe or SRC to determine the latest version number)

ATTACHMENT 2

RI Liquid Calibration Conversion Tables
Prepared by Dr. Shu-Chun Su, Hercules, Inc.

See attached Excel spreadsheet entitled
"Create_RI_Liquid_Calibration_Conversion_Tables.xls"