Sampling and Analysis Plan/Quality Assurance Project Plan: Sediment Porewater Study of Kootenai River Tributaries Libby Asbestos Superfund Site, Operable Unit 4 Libby, Montana

Revision 0 – May 2013

Contract No. EP-W-05-049
Work Assignment No. 329-RICO-08BC

Prepared for:

U.S. ENVIRONMENTAL PROTECTION AGENCY
Region 8

CDM Smith

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Sediment Porewater Study of Kootenai River Tributaries
Libby Asbestos Superfund Site, Operable Unit 4
Libby, Montana

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Approvals:

Date: 5/9/13

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CDM Smith, Project Manager

Date: 5/9/13

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List of Acronyms and Abbreviations

Ago	area of a grid opening
APP	Accident Prevention Plan
ASTM	American Society of Testing and Materials
CARB	California Air Resources Board
CB&I	CB&I Federal Services, LLC
CDM Smith	CDM Federal Programs Corporation
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CHISQ	chi-squared
COC	chain-of-custody
Cw	water concentration
DE Tool	data entry tool
DEQ	Montana Department of Environmental Quality
DQO	data quality objective
EDD	electronic data deliverable
EDS	Energy Dispersive Spectroscopy
EFA	effective filter area
EL	effect level
EPA	U.S. Environmental Protection Agency
ERT	Environmental Response Team
ESAT	Environmental Services Assistance Team
f/L	fibers per liter
FSDS	field sample data sheet
FTL	field team leader
FWP	Fish Wildlife and Parks
GIS	geographic information system
GOx	number of grid openings examined
GPS	global positioning system
H&S	health and safety
HAZWOPER	Hazardous Waste Operations and Emergency Response
HDPE	high-density polyethylene
ID	identification
IDW	investigation-derived waste
KDC	Kootenai Development Corporation
L	liters
LA	Libby amphibole
LADT	Libby Asbestos Data Tool
LC	laboratory coordinator
LRC	lower Rainy Creek
MFL	million fibers per liter
mm^2	square millimeters
N	number of asbestos structures counted

ND	non-detect
NEL	no-effect level
NFG	National Functional Guidelines
NIOSH	National Institute for Occupational Safety and Health
NIST	National Institute of Standards and Technology
NVLAP	National Voluntary Laboratory Accreditation Program
OSHA	Occupational Safety and Health Administration
OU	Operable Unit
PE	performance evaluation
PLM	polarized light microscopy
PLM-VE	polarized light microscopy visual area estimation
PLM-Grav	polarized light microscopy gravimetric
PM	Project Manager
QA	quality assurance
QAM	Quality Assurance Manager
QAPP	quality assurance project plan
QATS	Quality Assurance Technical Support
QC	quality control
RAC	Remedial Action Contract
ROM	record of modification
RPM	Regional Project Manager
SAP	sampling and analysis plan
Site	Libby Asbestos Superfund Site
SPF	Sample Preparation Facility
SOP	standard operating procedure
SRM	standard reference material
TEM	transmission electron microscopy
μm	micrometers
USFWS	United States Fish and Wildlife Service
USGS	U.S. Geological Survey
UV	ultraviolet
V	volume of water applied to the filter
WVB	Whitlock Vibert Box

A Project Management

A3. Distribution List

Copies of this completed/signed sampling and analysis plan/quality assurance project plan (SAP/QAPP) should be distributed to:

U.S. Environmental Protection Agency, Region VIII
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- Rebecca Thomas, Thomas.Rebecca@epa.gov (1 hard copy, 1 electronic copy)
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- Damon Repine, repineDL@cdmsmith.com (electronic copy)

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555 17th Street, Suite 1100
Denver, Colorado 80202
- Nathan Smith, smithNT@cdmsmith.com (electronic copy)

Copies of this SAP/QAPP will be distributed to the individuals above by CDM Federal Programs Corporation (CDM Smith), either in hard copy or in electronic format (as indicated above). The CDM Smith Project Manager (or their designee) will distribute updated copies each time a SAP/QAPP revision occurs.

A4. Project Task Organization

Figure A-1 presents an organizational chart that shows lines of authority and reporting responsibilities for this project. The following sections summarize the entities and individuals that will be responsible for providing project management, technical support, and quality assurance (QA) for this project.

A4.1 Project Management

The U.S. Environmental Protection Agency (EPA) is the lead regulatory agency for Superfund activities within the Libby Asbestos Superfund Site (Site). The EPA Region 8 Libby Asbestos Project Team Leader is Rebecca Thomas. The EPA Regional Project Manager (RPM) for this sampling effort is Elizabeth Fagen. The EPA Region 8 Onsite RPM for this sampling effort is Mike Cirian.

The Montana Department of Environmental Quality (DEQ) is the support regulatory agency for Superfund activities at the Site. The DEQ Project Manager (PM) for this sampling effort is Carolyn Rutland. The EPA will consult with DEQ as provided for by the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), the National Contingency Plan, and applicable guidance in conducting Superfund activities.

A4.2 Technical Support

A4.2.1 SAP/QAPP Development

This SAP/QAPP was developed by CDM Smith at the direction of and with oversight by the EPA. This SAP/QAPP contains all the elements required for both a SAP and a QAPP and has been developed in general accordance with the *EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5* (EPA 2001) and the *Guidance on Systematic Planning Using the Data Quality Objectives Process, EPA QA/G4* (EPA 2006). This SAP/QAPP was prepared and field activities will be performed under the Remedial Action Contract (RAC), Contract No. EP-W-05-049, Work Assignment No. 329-RICO-08BC. As noted above, the CDM Smith Project Manager (or their designee) is responsible for distributing updated copies of the SAP/QAPP if a revision occurs.

A4.2.2 Field Sampling Activities

CDM Smith will also be responsible for conducting all field sampling activities in support of the sampling program described in this SAP/QAPP. Key CDM Smith personnel that will be involved in this sampling program include:

- Nathan Smith, Project Manager
- Dominic Pisciotta, Field Team Leader
- Tracy Dodge, Field Sample Coordinator
- Diane Rode, Field Data Manager
- Terry Crowell, Quality Assurance Coordinator
- Damon Repine, Health and Safety Manager

A4.2.3 Asbestos Analysis

All samples of sediment porewater, surface water, and sediment collected as part of this project will be sent for preparation and analysis for asbestos at laboratories selected and approved by the EPA to support the Site. The EPA Environmental Services Assistance Team (ESAT) is responsible for procuring all analytical and preparation laboratory services and providing direction to the analytical laboratories. Don Goodrich (EPA Region 8) is responsible for managing the ESAT laboratory support contract for asbestos. The ESAT Region 8 Team Manager at TechLaw, Inc. is Mark McDaniel. He is also the designated laboratory coordinator (LC) for the Libby project that is responsible for directing the analytical laboratories, prioritizing analysis needs, and managing laboratory capacity.

A4.2.4 Data Management

All data generated as part of this sampling effort will be managed and maintained in Scribe. The EPA Environmental Response Team (ERT) is responsible for the administration of all Scribe data management aspects of this project. Joseph Schafer is responsible for overseeing the ERT data management support contract. ERT is responsible for the development and management of Scribe and the project-specific data reporting requirements for the Libby project.

The CDM Smith field data manager (Diane Rode) is responsible for uploading sample information to the field Scribe project database. ESAT is responsible for uploading new analytical results to the analytical Scribe project database. The ESAT project data manager for the Libby project is Janelle Lohman (TechLaw, Inc.).

A4.3 Quality Assurance

There is no individual designated as the EPA Quality Assurance Manager for the Libby project. Rather, for SAP/QAPPs prepared under the RAC, the Region 8 QA program has delegated authority to the EPA RPMs for the Site. This means that the EPA RPMs have the ability to review and approve governing investigation documents developed by Site contractors. It is the responsibility of the designated EPA Quality Assurance Reviewer (Dania Zinner), who is independent of the entities planning and obtaining the data, to ensure that this SAP/QAPP has been prepared in accordance with the EPA QA guidelines and requirements. The EPA RPM is responsible for managing and overseeing all aspects of the quality assurance/quality control (QA/QC) program for this sampling effort. In this regard, the RPM is supported by the EPA Quality Assurance Technical Support (QATS) contractor, CB&I Federal Services, LLC (CB&I). The QATS contractor will evaluate and monitor laboratory QA/QC and is responsible for performing annual audits of each preparation and analytical laboratory.

Terry Crowell (CDM Smith) is the field QA Coordinator for this project. Ms. Crowell is responsible for evaluating and monitoring field QA/QC, for providing oversight of field sampling and data collection activities, and for designating a qualified individual to conduct the field audit (see Section C1.1).

A5. Problem Definition/Background

A5.1 Site Background

Libby is a community in northwestern Montana located seven miles southwest of a vermiculite mine that operated from the 1920s until 1990. The mine began limited operations in the 1920s and was operated on a larger scale by the W.R. Grace Company from approximately 1963 to 1990. Studies revealed that the vermiculite from the mine contains amphibole-type asbestos, referred to as Libby amphibole (LA).

Epidemiological studies revealed that workers at the mine had an increased risk of developing asbestos-related lung disease (McDonald *et al.* 1986, 2004; Amandus and Wheeler 1987; Amandus *et al.* 1987; Whitehouse 2004; Sullivan 2007). Additionally, radiographic abnormalities were observed in 17.8 percent (%) of the general population of Libby including former workers, family members of workers, and individuals with no specific pathway of exposure (Peipins *et al.* 2003; Whitehouse *et al.* 2008; Antao *et al.* 2012; Larsen *et al.* 2010, 2012a, 2012b). Although the mine has ceased operations, historic or continuing releases of LA from mine-related materials could be serving as a source of ongoing exposure and risk to current and future residents and workers in the area. The Site was listed on the EPA National Priorities List in October 2002.

Previous investigations conducted at the Site have demonstrated that LA is present in environmental source media (e.g., surface water, sediments) at locations in and around the Site.

High levels of LA have been measured in surface water and sediment in lower Rainy Creek (LRC) (EPA 2009). In addition, a recent sampling effort determined the presence of LA in surface water and/or sediment of the Kootenai River and several of its tributaries (EPA 2013b). The presence of LA in these aquatic media has the potential to adversely impact ecological receptors that inhabit these rivers and creeks.

A5.2 Reasons for this Project

In 2012, contractors for W.R. Grace & Co.-Conn. and the Kootenai Development Corporation (KDC) performed an in-stream caged fish (eyed egg and alevin) study to provide information on potential effects of exposures in LRC to trout (EPA 2012a). Eggs and pre-swim-up alevins reside in the stream gravel, so the exposure medium of chief concern is the gravel porewater. Results from this study demonstrated that measured gravel (sediment) porewater concentrations of LA were much higher than the overlying surface water concentrations (SRC, Inc. 2013). Results from this study also suggested that there might be a decrease in overall survival of trout in LRC, but these results were considered to have low reliability due several study limitations (SRC, Inc. 2013). Due to these limitations, the in-stream eyed egg study will be repeated in 2013, modifying the study design and protocol to address the issues that occurred during the 2012 study (EPA 2013a). It is expected that this study will be used to identify an LA concentration in sediment porewater that is a no-effect level (NEL) and/or an effect level (EL), depending upon the study outcome.

This SAP/QAPP describes the sampling program to collect sediment porewater samples from tributaries to the Kootenai River. These porewater samples will be analyzed for LA and the measured porewater LA concentrations will be compared to the NEL and/or the EL levels (as established by the results of the 2013 in-stream eyed egg study) to determine the potential for LA effects on fish in an ecological risk assessment. The measured porewater LA concentrations will also be used to characterize the nature and extent of LA in porewater at the Site. Detailed information on the study design is presented in Section B1.

A5.3 Applicable Criteria and Action Limits

There are no established criteria to evaluate ecological risk from exposures to LA. A review of available ecological toxicity data for asbestos in the literature reveals that it appears that effects thresholds for freshwater fish range from about 10,000-1,000,000 fibers per liter (f/L) in surface water.

Aquatic invertebrate sediment toxicity tests performed using sediment from Operable Unit 3 (OU3) at levels of up to 5% LA in sediment revealed that no adverse effects on survival, growth, or reproduction occurred in exposed aquatic invertebrates (Parametrix 2009a, 2009b).

Currently, there are no studies that provide information on effects thresholds for LA in sediment porewater. However, it is expected that the 2013 in-stream eyed egg study in LRC will provide an NEL and /or EL that can be used to evaluate potential risks to fish from exposures to LA in sediment porewater.

A6. Project/Task Description

A6.1 Task Summary

The basic tasks that are required to implement this SAP/QAPP are the collection of porewater samples for the same Kootenai River tributary creeks sampled in 2012 as part of the *Nature and Extent Study in Surface Water and Sediment* (EPA 2012b), with the addition of three sampling locations (two in O'Brien Creek and one in Fisher River). To ensure comparability, the porewater samples collected as part of this study will be collected using the same collection techniques that will be used to collect porewater during the 2013 in-stream eyed egg study in LRC (EPA 2013a). At the three new sampling locations in O'Brien Creek and Fisher River, surface water and sediment samples will also be collected. Sediment porewater, surface water, and sediment samples will be analyzed for LA.

A6.2 Work Schedule

As noted above, sediment porewater will be collected using the same sampling procedures as will be used in the 2013 in-stream eyed egg study in LRC (EPA 2013a). In brief, porewater will be collected from inside the chamber of a Whitlock-Vibert box (WVB) that has been buried in the stream channel. WVBs will be placed during the peak or falling limb of the hydrograph, which is expected to occur about mid-May. Actual sampling times may vary depending upon site conditions. Work should not be attempted if the water flow and/or depth are too high and wading into the water for installation of the WVBs cannot be performed safely. If this occurs, work should be performed as soon as conditions are deemed safe.

Sediment porewater samples will be collected from each sampling location about one to two days after WVB placement into the stream. Surface water and sediment samples will be collected concomitant with porewater collection at a subset of sampling locations (new locations that were not sampled previously in 2012).

After collection, porewater and surface water samples will be delivered to the analytical laboratory for treatment and analysis. There are no holding time requirements for the analysis of LA in water. But, it is desirable to perform treatment within 48 hours of sample collection. Sediment samples will be delivered to the Sample Preparation Facility (SPF) in Troy, Montana for preparation and subsequent shipment to the analytical laboratory. Analytical turn-around time will be negotiated between EPA and the analytical laboratory; it is anticipated analytical results will be provided approximately 2-3 weeks after sample collection.

A6.3 Locations to be Evaluated

The locations where sediment porewater, surface water, and sediment samples will be collected are described in Section B1.1.

A6.4 Resources and Time Constraints

There are both resource and time constraints associated with this sampling program. To limit expenditures of field resources, this sampling program will include a single sampling event. Because available sediment porewater data collected from LRC suggest that LA concentrations in porewater tend to be higher during high flow, porewater must be collected during the time period of high flow conditions, which is expected to occur in mid-May. Because some sampling locations are critical habitat for the bull trout, sampling should not occur during times of spawning (September) or egg incubation (October to April). Sampling should only be performed when flow conditions are such that sampling can be performed safely by field personnel. Thus, as appropriate sampling timing may be adjusted for field conditions, provided that sampling does not overlap with bull trout spawning/egg incubation timeframes.

A7. Quality Objectives and Criteria

A7.1 Data Quality Objectives

Data quality objectives (DQOs) are statements that define the type, quality, quantity, purpose, and use of data to be collected. The design of a study is closely tied to the 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 types of analyses to be performed. The EPA has developed a seven-step process for establishing DQOs to help ensure that data collected during a field sampling program will be adequate to support reliable site-specific decision-making (EPA 2001, 2006).

Appendix A provides the detailed implementation of the seven-step DQO process associated with this SAP/QAPP.

A7.2 Performance Criteria

As noted previously, there are currently no asbestos criteria or action limits that apply specifically to sediment porewater. However, it is expected that the 2013 in-stream eyed egg study will provide an NEL and/or EL that can be used to evaluate potential risks to fish from exposures to LA in sediment porewater. For the purposes of this sampling effort, the analytical requirements established in Section B4 are equivalent to the analytical requirements specified in the 2013 in-stream eyed egg study for the analysis of LA in sediment porewater to ensure results from this study will be comparable.

A7.3 Precision

In a transmission electron microscopy (TEM) analysis, the precision of asbestos measurements is determined mainly by the number (N) of asbestos structures counted in each sample. The coefficient of variation resulting from random Poisson counting error is equal to $1/N^{0.5}$. In general, when good precision is needed, it is desirable to count a minimum of 3-10 structures per sample, with counts of 20-25 structures per sample being optimal.

Field duplicates of porewater samples will be collected (see Section B5.1.5). Analysis of these field duplicates will provide a measure of the precision of the sampling and analysis process. Recount and repreparation analyses will be performed as part of the TEM analysis (see Section B5.3.4). These analyses will provide information on analysis reproducibility and precision (both inter- and intra-laboratory).

A7.4 Bias and Representativeness

There is no established set of reference materials or spiked standards that can be used to assess accuracy of TEM analyses of LA in water. Results for field blanks and laboratory blanks will be utilized to ensure that water sample results are not biased as a consequence of cross-contamination due to field sampling procedures or preparation and analysis methods.

Measured data in surface water and sediment porewater in Site streams have shown that concentrations change as a function of flow (higher concentrations are observed under high flow conditions). Thus, sampling of porewater will be performed during the peak surface water flow or just after (falling limb of the hydrograph) in mid-May. Targeting this timeframe for sampling will ensure that results are representative of the high-end of potential exposure conditions.

A7.5 Completeness

Target completeness for this project is 100%. If any samples of porewater, surface water, and sediment are not collected, or if LA analysis is not completed successfully, this could result in that portion of the study providing no useful information. In this event, additional sampling may be needed to support EPA risk management decision-making.

A7.6 Comparability

The data generated during this study will be obtained using standard sample collection protocols and analytical methods for LA that have been established for use at the Site, and will yield data that are comparable to existing and future analyses of LA in sediment porewater, surface water, and sediment. In particular, sediment porewater will be collected using techniques that are identical to those that will be used in the 2013 in-stream eyed egg study to ensure the resulting data are directly comparable.

A7.7 Method Sensitivity

The method sensitivity (analytical sensitivity) needed for LA in sediment porewater, surface water, and sediment is discussed in Section B4.

A8. Special Training/Certifications

A8.1 Field

Asbestos is a hazardous substance that can increase the risk of cancer and serious non-cancer effects in people who are exposed by inhalation. Therefore, all individuals involved in the collection, packaging, and shipment of samples must have appropriate training. Prior to starting any field work, any new field team member must complete the following, at a minimum:

Training Requirement	Location of Documentation Specifying Training Requirement Completion
Read and understand the governing Accident Prevention Plan (APP) and specific activity hazard analysis form for this event	APP signature sheet
Attend an orientation session with the field health and safety (H&S) manager (Libby site primer/asbestos awareness)	Orientation session attendance sheet
Sample collection techniques	Orientation session attendance sheet

All training documentation will be stored in the CDM Smith field office. It is the responsibility of the field H&S manager to ensure that all training documentation is up-to-date and on-file for each field team member.

Prior to beginning field sampling activities, a field planning meeting will be conducted to discuss and clarify the following:

- Objectives and scope of the fieldwork
- Equipment and training needs
- Field operating procedures, schedules of events, and individual assignments
- Required QC measures
- Health and safety requirements

It is the responsibility of each field team member to review and understand all applicable governing documents associated with this sampling program, including this SAP/QAPP, all associated standard operating procedures (SOPs) (see **Appendix B**), and the applicable APP.

A8.2 Laboratory

A8.2.1 Certifications

All analytical laboratories participating in the analysis of samples for the Libby project are subject to national, local, and project-specific certifications and requirements. Each laboratory is accredited by the National Institute of Standards and Technology (NIST)/National Voluntary Laboratory Accreditation Program (NVLAP) for the analysis of airborne asbestos by TEM and/or analysis of bulk asbestos by polarized light microscopy (PLM). This includes the analysis of NIST/NVLAP standard reference materials (SRMs), or other verified quantitative standards, and successful participation in two proficiency rounds per year each of bulk asbestos by PLM and airborne asbestos by TEM supplied by NIST/NVLAP.

Copies of recent proficiency examinations from NVLAP or an equivalent program are maintained by each participating analytical laboratory. Many of the laboratories also maintain certifications from other state and local agencies. Copies of all proficiency examinations and certifications are also maintained by the LC.

Each laboratory working on the Libby project is also required to pass an on-site EPA laboratory audit. The details of this EPA audit are discussed in Section B5.3.3. The LC also reserves the right to conduct any additional investigations deemed necessary to determine the ability of each laboratory to perform the work. Each laboratory also maintains appropriate certifications from the state and possibly other certifying bodies for methods and parameters that may also be of interest to the Libby project. These certifications require that each laboratory has all applicable state licenses and employs only qualified personnel. Laboratory personnel working on the Libby project are reviewed for requisite experience and technical competence to perform asbestos analyses. Copies of personnel resumes are maintained for each participating laboratory by the LC in the Libby project file.

A8.2.2 Laboratory Team Training/Mentoring Program

<u>Initial Mentoring</u>

The orientation program to help new laboratories gain the skills needed to perform reliable analyses at the Site involves successful completion of a training/mentoring program that was developed for new laboratories prior to their analysis of Libby field samples. All new laboratories are required to participate in this program. The training program includes a rigorous 2-3 day period of on-site training provided by senior personnel from those laboratories already under contract on the Libby project, with oversight by the QATS contractor. The tutorial process includes a review of morphological, optical, chemical, and electron diffraction characteristics of LA, as well as training on project-specific analytical methodology, documentation, and administrative procedures used on the Libby site. The mentor will also review the analysis of at least one sample by each type of analytical method with the trainee laboratory.

Site-Specific Reference Materials

TEM

Because LA is not a common form of asbestos, U.S. Geological Survey (USGS) prepared Site-specific reference materials using LA collected at the Libby mine site (EPA 2008a). Upon entry into the Libby program, each laboratory is provided samples of these LA reference materials. Each laboratory is required to analyze multiple LA structures present in these samples by TEM in order to become familiar with the physical and chemical appearance of LA and to establish a reference library of LA energy dispersive spectroscopy (EDS) spectra. These laboratory-specific and instrument-specific LA reference spectra (EPA 2008b) serve to guide the classification of asbestos structures observed in Libby field samples during TEM analysis.

PLM

USGS has also prepared Site-specific reference materials of LA in soil for use during PLM using the Libby-specific visual area estimation method (PLM-VE) (EPA 2008a). These reference materials were prepared by adding aliquots of LA spiking material to uncontaminated Libby soils to obtain nominal LA concentrations of 0.2% and 1.0% (by weight). Each laboratory was provided with samples of these reference materials for use in training PLM-VE analysts in the visual area estimation of LA levels in soil. In addition, aliquots of these reference materials (as well as other spiked soils) are also utilized as performance evaluation (PE) standards to evaluate PLM-VE laboratory accuracy.

Regular Technical Discussions

On-going training and communication is an essential component of laboratory QA for the Libby project. To ensure that all laboratories are aware of any technical or procedural issues that may arise, a regular teleconference is held between the EPA, their contractors, and each of the participating laboratories. Other experts (e.g., USGS) are invited to participate when needed. These calls cover all aspects of the analytical process, including sample capacity, information processing, technical issues, analytical method procedures and development, documentation issues, project-specific laboratory modifications, and pertinent asbestos publications.

Professional/Technical Meetings

Another important aspect of laboratory team training has been the participation in technical conferences. The first of these technical conferences was hosted by USGS in Denver, Colorado, in February 2001, and was followed by another held in December 2002. The Libby laboratory team has also convened on multiple occasions at the American Society of Testing and Materials

(ASTM) Johnston Conference in Burlington, Vermont, including in July 2002, July 2005, July 2008, and July 2011, and at the ASTM Michael E. Beard Asbestos Conference in January 2010 and January 2013. In addition, members of the Libby laboratory team attended an EPA workshop to develop a method to determine whether LA is present in a sample of vermiculite attic insulation held in February 2004 in Alexandria, Virginia. These conferences enable the Libby laboratories and technical team members to have an on-going exchange of information regarding all analytical and technical aspects of the project, including the benefits of learning about developments by others.

A8.2.3 Analyst Training

TEM

All TEM analysts for the Libby project undergo extensive training to understand TEM theory and the application of standard laboratory procedures and methodologies. The training is typically performed by a combination of personnel, including the laboratory manager, the laboratory quality assurance manager (QAM), and senior TEM analysts.

In addition to the standard TEM training requirements, trainees involved with the Libby project must familiarize themselves with Site-specific method deviations, project-specific documents, and visual references. Standard samples that are often used during TEM training include known pure (traceable) samples of chrysotile, amosite, crocidolite, tremolite, actinolite and anthophyllite, as well as fibrous non-asbestos minerals such as vermiculite, gypsum, antigorite, kaolinite, and sepiolite. New TEM analysts on the Libby project are also required to perform an *EDS Spectra Characterization Study* (EPA 2008b) on the LA-specific reference materials provided during the initial training program to aide in LA mineralogy recognition and definition. Satisfactory completion of each of these tasks must be approved by a senior TEM analyst.

All TEM analysts are also trained in the Site-specific laboratory QA/QC program requirements for TEM (see Section B5.3.4). The entire program is discussed to ensure understanding of requirements and responsibilities. In addition, analysts are trained in the project-specific reporting requirements and data reporting tools utilized in transmitting results. Upon completion of training, the TEM analyst is enrolled as an active participant in the Libby laboratory program.

A training checklist or logbook is used to assure that the analyst has satisfactorily completed each specific training requirement. It is the responsibility of the laboratory QAM to ensure that all TEM analysts have completed the required training requirements.

PLM

All PLM analysts for the Libby project are expected to be familiar with routine chemical laboratory procedures, principles of optical mineralogy, and proficient in EPA Method 600/R-93/116, National Institute for Occupational Safety and Health (NIOSH) Method 9002, California Air Resources Board (CARB) Method 435, and Site-specific SOPs SRC-LIBBY-01 and SRC-LIBBY-03. Analysts with less than one year of experience specific to the Libby project are required to participate in the laboratory mentoring program to obtain additional guidance and instruction. This training is provided by the laboratory managers and/or senior PLM analysts that are familiar with the types of asbestos and analytical challenges encountered at the Site. Before performing any Site analyses, the analyst must demonstrate the ability to generate acceptable accuracy and precision for the LA-specific reference materials.

Satisfactory completion of each of these training tasks must be approved by a senior PLM analyst. A training checklist or logbook is used to ensure that the analyst has satisfactorily completed each specific training requirement. It is the responsibility of the laboratory QAM to ensure that all analysts have completed the required training requirements.

A9. Documentation and Records

Field teams will record sample information on the most current version of the Site-specific field sample data sheets (FSDSs) developed for water and sediment[1]. Section B3.1.2 provides detailed information on the documentation requirements for FSDS forms. In brief, the FSDS forms document the unique sample identifier assigned to all pore water, surface water, and sediment samples collected as part of this program. In addition, the FSDSs provide information on whether the sample is representative of a field sample or a field-based QC sample (e.g., field blank, field duplicate).

All analytical data for asbestos generated in the analytical laboratory will be documented on Site-specific laboratory bench sheets. Section B4.4 provides detailed information on the requirements for laboratory documentation and records. In brief, the data recorded on the bench sheets are entered into a Site-specific electronic data deliverable (EDD) template spreadsheets developed for recording TEM results for water and PLM for sediment[2].

It is the also responsibility of the field team, preparation laboratory, and analytical laboratory staff to maintain logbooks and other internal records throughout the sample lifespan as a record of sample handling procedures. Significant deviations (i.e., those that impact or have the potential to impact investigation objectives) from this SAP/QAPP, or any procedures referenced herein governing sample handling, will be discussed with the EPA Project Manager

[1] The most recent version of the FSDS forms are provided in the Libby Field eRoom.
[2] The most recent version of the TEM EDD for water and PLM-VE EDD are provided in the Libby Lab eRoom.

(or their designee) and the CDM Smith Project Manager prior to implementation. Such deviations will be recorded on a Record of Modification (ROM) form. Sections B5.1.2, B5.2.2, and B5.3.2 provide detailed information on the procedures for preparing and submitting ROMs by field, preparation laboratory, and analytical laboratory personnel, respectively.

B Data Generation and Acquisition

B1. Study Design

B1.1 Sampling Locations

Sampling locations for sediment porewater collection will be the same tributary locations as sampled during the 2012 *Nature and Extent Study in Surface Water and Sediment* (EPA 2012b; 2013b), with the addition of three locations (two in O'Brien Creek and one in the Fisher River). Sampling locations were added in O'Brien Creek because this tributary is critical habitat for the bull trout. Although Fisher River was sampled in the 2012 Nature and Extent Study, an additional sampling location has been added further upstream for this study because the headwaters were thought to be more representative of potential trout spawning habitat. Because these three new sampling locations have not been sampled for surface water and sediment, these media will be collected as part of this study to provide additional data to support the characterization of the extent of LA in the sediments and surface water.

The EPA consulted with Montana Fish, Wildlife, and Parks (FWP) and the U.S. Fish and Wildlife Service (USFWS) concerning the location of the sampling locations for this study relative to critical habitat and followed recommendations concerning timing and placement of the WVBs. **Table B-1** provides a description of each sampling location. **Figures B-1** and **Figure B-2** provide a map that shows the location of each sampling station near Libby and Troy, respectively. For tributaries where removal actions have been conducted, this figure also shows the approximate location of where past removal actions occurred.

At each sampling location, the area selected for WVB deployment (see Section B2.1.1) and collection of porewater samples should (ideally) be or approximate a natural redd (gravel/cobble area that fish are using or could use for spawning). If no redds are found then the bottom of the area excavated for deployment of the WVB will be layered with natural (or commercial) spawning gravel on which the deployed cages will rest. Sampling locations will be prepared over a 1-2 day period prior to porewater sample collection.

If necessary, any changes in sampling locations should be documented in the field logbook and new global positioning system (GPS) location coordinates will be recorded on the FSDS form. If any sampling locations become inaccessible, this information should be documented in the field logbook.

B1.2 Sampling Schedule

Porewater samples will be collected from the installed WVBs approximately 1 to 2 days after installation (to allow conditions inside the WVBs to equilibrate). One porewater sample will be collected from each sampling location (see **Figure B-1** and **Figure B-2**). For the new sampling locations (in O'Brien Creek and the Fisher River), one surface water sample and one sediment sample will be collected concomitant with the porewater collection event. Sample collection methods are discussed in Section B.2.1.

B1.3 Study Variables

As demonstrated in **Figure B-3**, asbestos concentrations in porewater are influenced by flow variations. Measured LA data in surface water and sediment pore water in LRC have shown that concentrations change as a function of flow (higher concentrations are observed under high flow conditions). Based on flow monitoring conducted in creeks within OU3, flow rates begin to increase in late April, peak in mid-May, and decrease in late May (see **Figure B-4**). It is assumed that most of the Kootenai tributaries will follow a similar time-trend. Thus, sampling will be conducted in mid- to late-May to capture higher flow conditions and anticipated corresponding higher LA concentrations. However, sampling may be postponed based on field conditions (water flow and/or depth) to ensure the safety of field personnel. Based on consultation with Montana FWP and the USFWS, sampling should not occur during the time period of bull trout spawning (September) or egg incubation (October to April).

B1.4 Critical Measurements

Critical measurements associated with this project include the measurement of LA in sediment porewater. The collection of surface water and sediment at the three new sampling locations in O'Brien Creek and Fisher River, while desirable, are not critical for the purposes of supporting the ecological risk assessment.

The analysis of asbestos in water (surface water and porewater) may be achieved using several different types of microscope, but the EPA generally recommends using TEM because this technique has the ability to clearly distinguish asbestos from non-asbestos structures and to classify different types of asbestos (i.e., LA, chrysotile). Prior to analysis, water samples will be treated with ozone/ultraviolet (UV) to remove any organic materials that would cause LA to clump and bind to the container walls and ensure that concentrations are not biased low (see Section B4).

B1.5 Data Reduction and Interpretation

B1.5.1 Sediment Porewater and Surface Water

Following ozone/UV treatment, water samples collected in the field will be filtered by the analytical laboratory and the resulting filter will be used to prepare grids for TEM examination (see Section B4). From this examination, the total number of asbestos structures for each type of asbestos is determined and the water concentration is calculated as follows:

$$C_w = (N \cdot EFA) / (GOx \cdot Ago \cdot V \cdot 1E{+}06)$$

where:

C_w	= Water concentration, in million fibers per liter (MFL)
N	= Number of asbestos structures observed (fibers)
EFA	= Effective filter area, in square millimeters (mm^2)
GOx	= Number of grid openings examined
Ago	= Area of a grid opening (mm^2)
V	= Volume of water applied to the filter (L)
1E+06	= Conversion factor (fibers per liter [f/L] --> MFL)

Data on LA concentrations in porewater collected as part of this SAP/QAPP will be used to compare to the NEL and/or the EL levels (as identified from the results of the 2013 in-stream eyed egg study in LRC) to determine the potential for LA effects on fish in an ecological risk assessment. The measured porewater and surface water LA concentrations will also be used to characterize the nature and extent of LA in Kootenai tributaries.

B1.5.2 Sediment

Sediment samples will be analyzed for LA using the Libby-specific analytical method for PLM-VE. The results of the PLM-VE analysis will provide a semi-quantitative estimate of the amount of LA in the sediment on a weight percent basis.

Data on LA concentrations in sediment samples collected as part of this study will be used to compare to levels measured in sediment to the NEL established by the Site-specific aquatic invertebrate sediment toxicity tests performed in OU3 (Parametrix 2009a, 2009b). The measured sediment LA concentrations will also be used to characterize the nature and extent of LA in sediment.

B2. Sampling Methods

B2.1 Sample Collection

B2.1.1 Sediment Porewater

All sediment porewater samples will be collected using the procedures described in SOP EPA-LIBBY-2013-01, *Sediment Porewater Sample Collection Using a Modified Whitlock-Vibert Box* (see **Appendix B**), with the following investigation-specific modification:

- The excavated WVB placement area will be allowed to sit undisturbed for 15 minutes to flush away fine sediments prior to actual installation of the WVBs.

In brief, at each sampling location (see **Figure B-5**), a modified WVB will be placed into a metal cage that has been filled with cobble and buried into the sediment in an area to approximate a natural redd (i.e., a gravel/cobble area that fish are using to spawn or could use to spawn). The WVB will be modified such that a porewater sampling port extends from the inside of the WVB chamber to above the water surface. A metal bailer will be used to sample water from within the WVB chamber. Collected water will placed into a 500-mL capacity high-density polyethylene (HDPE) wide-mouth bottle, or equivalent container. Headspace should be left in the container to ensure there is ample room at the top to accommodate ozone/UV treatment (see Section B4.1).

B2.1.2 Surface Water

All surface water samples will be collected using the procedures described in SOP EPA-LIBBY-2012-08, *Surface Water Sampling* (see **Appendix B**). In brief, water will be collected using direct sampling methods from the river or creek bank and placed into a 500-mL capacity HDPE wide-mouth bottle, or equivalent container, as detailed in Section 5.2.1 of the SOP. Headspace should be left in the container to ensure there is ample room at the top to accommodate ozone/UV treatment (see Section B4.1). To minimize affects of field collection activities to subsequent locations downstream, water samples will be collected from downstream to upstream. In addition, water samples will be collected prior to the collection of other media (i.e., sediment, porewater).

B2.1.3 Sediment

All sediment samples will be collected using the procedures described in SOP EPA-LIBBY-2012-09, *Sediment Sampling* (see **Appendix B**), with the following investigation-specific modifications:

- Sediment samples will be collected after surface water sampling has been completed at each location in order to minimize effects of field collection activities.

- A single surficial sediment sample will be collected from each tributary location with each sample consisting of a homogeneous mixture, or composite, of five grab samples 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). The five grab samples will be collected over a reach that is within 100 feet upstream or 100 feet downstream of the specified station.

- The mass of sediment collected may be estimated by visual assessment of sediment volume. If the mass of sediment from the inundated areas is not sufficient for the analyses that are required, sediment will be collected from within the active high-flow channel, but no sediments will be collected from over-bank areas. After homogenization, the composite sample may be used to fill the appropriate container.

B2.2 Global Positioning System Coordinate Collection

GPS location coordinates will be recorded for each sampling location in basic accordance with Site-specific SOP CDM-LIBBY-09, *GPS Coordinate Collection and Handling* (see **Appendix B**). For the purposes of this study, GPS coordinates for porewater locations should be collected from the actual WVB deployment location (within the stream channel), not from the stream bank. Field-collected GPS data are converted to a usable geographic information system (GIS) format using the general processes described in SOP CDM-LIBBY-09. After the conversion from GPS points to GIS files, 100% of the data is checked visually to identify any potential data entry errors.

B2.3 Equipment Decontamination

Equipment used to collect, handle, or measure environmental samples will be decontaminated in basic accordance with SOP EPA-LIBBY-2012-04, *Field Equipment Decontamination at Nonradioactive Sites* (see **Appendix B**). Materials used in the decontamination process will be disposed of as investigation-derived waste (IDW) as described below. This SOP specifies the minimum procedural requirements for equipment decontamination. Additional equipment decontamination procedures are also specified in the media collection SOPs (see **Appendix B**).

B2.4 Handling Investigation-derived Waste

Any disposable equipment or other IDW will be handled in basic accordance with the procedures specified in SOP EPA-LIBBY-2012-05, *Guide to Handling of Investigation-Derived Waste* (see **Appendix B**). In brief, IDW will be double-bagged, with the outer bag being a clear heavy-weight trash bag that has been pre-printed with 'IDW' on the outside. If pre-printed IDW bags are not available, the outer bag needs to be clearly labeled (once) using an indelible marker or a taped label. All IDW generated during this investigation will enter the waste stream at the local class IV asbestos landfill.

B3. Sample Handling and Custody

B3.1 Sample Identification and Documentation

B3.1.1 Sample Labels

Samples will be labeled with sample identification (ID) numbers supplied by field administrative staff and will be signed out by the sampling teams. Labels for porewater, surface water, and sediment samples from the tributaries will be affixed to the outside of the sample container and covered with a piece of clear packaging tape.

Sample ID numbers will identify the samples collected during this sampling effort using the following format:

PT-#####

where:

PT = Prefix that designates samples collected under this SAP/QAPP
= A sequential five-digit number

B3.1.2 Field Sample Data Sheets

As noted previously in Section A9, field teams will record sample information on the most current version of the Site-specific FSDS for each environmental medium. Use of standardized forms ensures consistent documentation across samplers. Hard copy FSDSs are location-specific and allow for the entry of up to three individual samples from the same location on the same FSDS form. If columns are left incomplete due to fewer than three samples being recorded on a sheet, the blank columns will be crossed out, dated, and signed by the field team member completing the FSDS. Erroneous information recorded on a hard copy FSDS will be corrected with a single line strikeout, initial, and date. The correct information will be entered in close proximity to the erroneous entry.

FSDS information will be completed in the field before field personnel leave the sampling location. To ensure that all applicable data is accurately entered and all fields are complete, a different field team member will check each FSDS. The team member completing the hard copy form and the team member checking the form will initial the FSDS in the proper fields. In addition, the field team leader (FTL) will also complete periodic checks of FSDSs prior to relinquishment of the samples to the field sample coordinator. Once FSDSs and samples are relinquished to the field sample coordination staff, the FSDSs are again checked for accuracy and completeness when data are input into the local Scribe field database.

If a revision is required to the hard copy FSDS during any of these checks, it will be returned to the field team member initially responsible for its completion. The error will be explained to the team member and the FSDS corrected. If the team member is no longer on site, revisions will be made by sample coordination staff or the FTL. It is the responsibility of the field data manager to make the appropriate change in the local Scribe field database.

Each hard copy FSDS is assigned a unique sequential number. This number will be referenced in the field logbook entries related to samples recorded on individual sheets. Field administrative staff will manage the hard copy FSDSs in their respective field office. Original FSDSs will be filed by medium and FSDS number. Hard copies of all FSDS forms will also be sent to the CDM Smith office in Denver, Colorado for archive.

B3.1.3 Field Logbooks

The field logbook is an accounting of activities at the Site and will duly note problems or deviations from the governing documents. Field logbooks will be maintained in general conformance with SOP EPA-LIBBY-2012-01, *Field Logbook Content and Control* (see **Appendix B**). Separate field logbooks will be kept for each investigation and the cover of each field logbook will clearly indicate the name of the investigation and its sequence number. Field logbooks will be completed for each investigation activity prior to leaving a sampling location. Field logbooks will be checked for completeness and adherence to SOP requirements on a daily basis by the FTL or their designee for the first week of each investigation. When incorrect field logbook completion procedures are discovered during these checks, the errors will be discussed with the author of the entry and corrected. Erroneous information recorded in a field logbook will be corrected with a single line strikeout, initial, and date. The correct information will be entered in close proximity to the erroneous entry.

The field administrative staff will manage the field logbooks by assigning unique identification numbers to each field logbook, tracking to whom and the date each field logbook was assigned, the general investigation activities recorded in each field logbook (e.g., ambient air monitoring), and the date when the field logbook was returned. As field logbooks are completed, originals will be catalogued and maintained by the field administrative staff in their respective field office. Scanned copies of field logbooks will be maintained on the local servers for the CDM Smith offices in Libby and Denver.

B3.2 Field Sample Custody

All teams will ensure that samples, while in their possession, are maintained in a secure manner to prevent tampering, damage, or loss. All samples and FSDSs will be relinquished to the sample coordinator or designated secure sample storage area. The field team will be responsible for documenting this transfer of sample custody in the field logbook.

B3.3 Chain-of-Custody Requirements

The chain-of-custody (COC) is used as physical evidence of sample custody and control. This record system provides the means to identify, track, and monitor each individual sample from the point of collection through final data reporting. A complete COC record is required to accompany each shipment of samples. COC procedures will follow the requirements as stated in SOP EPA-LIBBY-2012-06, *Sample Custody* (see **Appendix B**).

At the end of each day, all samples will be relinquished to the field sample coordinator or a designated secure storage location by the sampling team following COC procedures, and an entry will be made into the field logbook indicating the time samples were relinquished and the sample coordinator who received the samples. The field sample coordinator will follow COC procedures to ensure proper sample custody between acceptance of the sample from the field teams to delivery or shipment to the laboratory.

A member of the sample coordination staff will manually enter sample information from the hard copy FSDS into the local Scribe field project database using a series of standardized data entry forms developed in Microsoft Access® by ESAT, referred to as the sample Data Entry Tool, or the "DE Tool". The DE Tool has a variety of built-in QC functions that improve accuracy of data entry and help maintain data integrity. After the data entry is checked against the hard copy FSDSs (by a different sample coordination staff member than completed the original data entry), the DE Tool is used to prepare an electronic COC. A three-page carbon copy COC will be generated from the electronic COC. The field sample coordinator will retain one hard copy of the COC for the project file; the other two hard copies of the COC will accompany the sample shipment. A copy of the investigation-specific Analytical Requirements Summary Sheet (see **Appendix C**) will also accompany each COC.

If any errors are found on a COC after shipment, the hard copy of the COC retained by the field sample coordinator will be corrected with a single strikeout, initial, and date. A copy of the corrected COC will be provided to the LC for distribution to the appropriate laboratory. It is the responsibility of the field data manager to make any corrections to the local Scribe field project database. Sample and COC information will be published to Scribe.NET regularly from the local Scribe field project database by the field data manager (see Section B10.1 for additional details).

B3.4 Sample Packaging and Shipping

Samples will be packaged and shipped in general accordance with SOP EPA-LIBBY-2012-07, *Packaging and Shipping of Environmental Samples* (see **Appendix B**). In brief, a custody seal will be placed over at least two sides of the shipping cooler and then secured by tape. Prior to sealing the shipping container, the sample coordinator will perform a final check of the contents of the shipment with the COC, sign and date the designated spaces at the bottom of the COC. The field sample coordinator will then place the custody seals on the shipping container.

The field sample coordinator will be responsible for sending samples to the appropriate location, as specified by the LC. Porewater and surface water samples will be hand-delivered to the EMSL Mobile Laboratory in Libby, Montana. Sediment samples will be sent to the Troy SPF for preparation and subsequent shipment to the appropriate analytical laboratory, or archive.

Samples will be hand-delivered, picked up by a courier service, or shipped by a delivery service to the designated location, as applicable. For hand-deliveries and courier pickups, samples will be packaged for transit such that they are contained and secure (i.e., will not be excessively jostled). Clean plastic totes with the lids secured or sample coolers may be used for this purpose. For samples requiring shipment, an established overnight delivery service provider (e.g., Federal Express) will be used.

B3.5 Holding Times

In general, there are no holding time requirements for asbestos. Because sample preparation will include techniques to address any issues related to holding time (see Section B4.1), there are no holding time requirements for water and sediment samples collected as part of this sampling program. However, to limit organic growth, it is desirable to perform ozone/UV treatment of water samples within 48 hours of sample collection.

B3.6 Archival and Final Disposition

All samples will be maintained in storage at the Troy SPF or analytical laboratory unless otherwise directed by the EPA. All filters will be maintained in storage at the analytical laboratory for a period of six months. After this time, filters will be sent to the SPF in Troy, Montana for final archival. All prepared grids will be maintained in storage at the analytical laboratory until authorized by EPA. When authorized by the EPA, the laboratory will be responsible for proper disposal of any remaining grids, 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.

B4. Analytical Methods

An analytical requirements summary sheet (**PWTBOU4-0513**), which details the specific analytical requirements for asbestos analyses associated with this sampling investigation, is provided in **Appendix C**. A copy of this summary sheet will be submitted with each COC.

B4.1 Analysis of LA in Water

B4.1.1 Sample Preparation

All water samples should be prepared for asbestos analysis in basic accordance with the techniques in EPA Method 100.2, as modified by Libby Laboratory Modification[3] LB-000020A. In brief, all water samples will be prepared using an ozone/UV treatment that oxidizes organic matter that is present in the water or on the walls of the bottle, destroying the material that causes clumping and binding of asbestos structures. Following treatment, an aliquot of water (generally about 50 milliliters) will be filtered through a 25-millimeter diameter polycarbonate filter with a pore size of 0.1 micrometers (µm) with a mixed cellulose ester filter (0.45 µm pore size) used as a support filter.

[3] Copies of all Libby Laboratory Modifications are available in the Libby Lab eRoom.

B4.1.2 Analysis Method

Approximately one quarter of the filter will be used to prepare a minimum of three grids using the grid preparation techniques described in Section 9.3 of International Organization for Standardization (ISO) Method 10312:1995(E). Grids will be examined by TEM in basic accordance with the recording procedures described in ISO 10312:1995(E), as modified by the most recent versions of Libby Laboratory Modifications LB-000016, LB-000029, LB-000066, LB-000067, and LB-000085.

B4.1.3 Counting Rules

All structures with fibrous morphology, an selected area electron diffraction pattern consistent with amphibole asbestos, an energy dispersive spectrum consistent with LA or other amphibole asbestos types, length greater than or equal to 0.5 µm, and an aspect ratio (length:width) greater than or equal to 3:1 will be counted and recorded. These counting rules will enable the calculation of water concentrations based on both total LA and LA structures longer than 10 µm. If observed, chrysotile structures will be recorded, but chrysotile structure counting may stop after 25 structures have been recorded.

B4.1.4 TEM Stopping Rules

Appendix A provides detailed information on the derivation of the stopping rules for water samples analyzed by TEM. The TEM stopping rules for all water samples from this investigation are as follows:

1. Examine a minimum of two grid openings from each of two grids.
2. Continue examining grid openings until one of the following is achieved:
    a. The target analytical sensitivity of 50,000 L^{-1} has been achieved.
    b. 25 LA structures have been observed.
    c. A total filter area of 1.0 mm^2 has been examined (this is approximately 100 grid openings).

When one of these criteria has been satisfied, complete the examination of the final grid opening and stop.

B4.2 Analysis of LA in Sediment

B4.2.1 Sample Preparation

All sediment samples collected for asbestos analysis will be submitted to the Troy SPF for preparation. Samples will be prepared in accordance with Libby-specific SOP ISSI-LIBBY-01. 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 µm or less and this fine-ground portion is split into 4 aliquots.

B4.2.2 Analysis Method

Each sediment sample will be analyzed for LA in accordance with Libby Site-specific SOPs for PLM. The coarse fraction (if any) will be examined using stereomicroscopy, and any particles of LA will be removed and weighed in accordance with SOP SRC-LIBBY-01, referred to as "PLM-Grav". One of the fine ground fraction aliquots will be analyzed by PLM using the visual area estimation method in accordance with SOP SRC-LIBBY-03, referred to as "PLM-VE". PLM-VE is a semi-quantitative method that utilizes Libby-specific LA reference materials to allow assignment of fine ground samples into one of four semi-quantitative "bins", as follows:

- Bin A (ND): non-detect
- Bin B1 (Trace): LA detected at levels lower than the 0.2% LA reference material
- Bin B2 (< 1%): LA detected at levels lower than the 1% LA reference material but greater than or equal the 0.2% LA reference material
- Bin C (≥ 1%): LA detected at levels greater than or equal to the 1% LA reference material, a quantitative estimate of the concentration is reported

B4.3 Analytical Data Reports

An analytical data report will be prepared by the laboratory and submitted to the appropriate LC after the completion of all required analyses within a specific laboratory job (or sample delivery group). This analytical data report may vary by laboratory and analytical method but generally includes 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 copies of the signed COC forms, analytical data summaries, a QC package, and raw data. Raw data is to consist of instrument preparation logs, instrument printouts, and QC sample results including, instrument maintenance records, COC check in and tracking, raw data instrument print outs of sample results, analysis run logs, and sample preparation logs. The laboratory will provide an electronic scanned copy of the analytical data report to the LC and others, as directed by the LC.

B4.4 Laboratory Data Reporting Tools

Standardized data reporting tools (i.e., EDDs) have been developed specifically for the Libby project to ensure consistency between different laboratories in the presentation and submittal of analytical data. In general, unique Libby-specific EDDs have been developed in Microsoft Excel® for each analytical method and medium. Since the beginning of the Libby project, each EDD spreadsheet has undergone continued development and refinement to better accommodate current and anticipated future data needs and requirements. EDD refinement continues based on laboratory and data user input. Electronic copies of all current EDD templates are provided in the Libby Laboratory eRoom.

For TEM analyses, detailed raw structure data will be recorded and results will be transmitted using the standard Libby project EDD for reporting TEM results for water samples. For PLM analyses, weight percent estimates of LA and optical property details will be recorded on the standard project EDDs for reporting PLM-VE and PLM-Grav results. Standard project data reporting requirements will be met for TEM and PLM analyses. EDDs will be transmitted electronically (*via* email) to the following:

- Doug Kent, Kent.Doug@epa.gov
- Holly Sprunger, Sprunger.Holly@epa.gov
- Janelle Lohman, Lohman.Janelle@epa.gov
- Tracy Dodge, DodgeTA@cdmsmith.com
- Phyllis Haugen, HaugenPJ@cdmsmith.com
- Libby project email address for CDM Smith, libby@cdmsmith.com

ESAT has developed a Site-specific analytical results reporting tool, referred to as the Libby Asbestos Data Tool (LADT). This tool is a relational Microsoft Access® database with a series of standard data entry forms specific to each analytical method. The LADT creates a Microsoft Excel® export file that can be directly uploaded into an analytical Scribe project database (see Section B10.4). Laboratories have the option of using LADT as a replacement for the Libby-specific EDDs.

B4.5 Analytical Turn-around Time

Analytical turn-around time will be negotiated between the EPA LC and the laboratory based on data needs and laboratory capacity. It is anticipated that a turn-around times of 2-3 weeks are acceptable for these surface water and sediment samples. This may be revised as determined necessary by the EPA.

B4.6 Custody Procedures

Specific laboratory custody procedures are provided in each laboratory's *QA Management Plan*, which have been independently reviewed at the time of laboratory procurement. While specific laboratory sample custody procedures may differ between laboratories, the basic laboratory sample custody process is described briefly below.

Upon receipt at the facility, each sample shipment will be inspected to assess the condition of the shipment and the individual samples. This inspection will include verifying sample integrity. The accompanying COC record will be cross-referenced with all of the samples in the shipment. The laboratory sample coordinator will sign the COC record and maintain a copy for their project files.

Depending upon the laboratory-specific tracking procedures, the laboratory sample coordinator may assign a unique laboratory identification number to each sample on the COC. This number, if assigned, will identify the sample through all further handling at the laboratory. It is the responsibility of the laboratory manager to ensure that internal logbooks and records are maintained throughout sample preparation, analysis, and data reporting.

B5. Quality Assurance/Quality Control

B5.1 Field

Field QA/QC activities include all processes and procedures that have been designed to ensure that field samples are collected and documented properly, and that any issues/deficiencies associated with field data collection or sample processing are quickly identified and rectified. The following sections describe each of the components of the field QA/QC program implemented at the Site.

B5.1.1 Training

Before performing field work in Libby, field personnel are required to read all governing field guidance documents relevant to the work being performed and attend a field planning meeting specific to the this sampling effort. Additional information on field training requirements is provided in Section A8.1.

B5.1.2 Modification Documentation

All field deviations from and modifications to this SAP/QAPP will be recorded on the Libby field ROM Form[4]. The field ROM forms will be used to document all permanent and temporary changes to procedures contained in guidance documents governing investigation work that have the potential to impact data quality or usability. Any minor deviations (i.e., those that will not impact data quality or usability) will be documented in the field logbooks. ROMs are completed by the FTL overseeing the investigation/activity, or by assigned field or technical staff. As modifications to governing documents are implemented, the FTL will communicate the changes to the field teams conducting activities associated with the modification.

Each completed field ROM is assigned a unique sequential number (e.g., LFO-000026) by the CDM Smith field QA Coordinator. A ROM tracking log for all field modifications is maintained by the field QA Coordinator. This tracking log briefly describes the ROM being documented, as well as ROM author, the reviewers, and date of approval. Once a form is prepared, it is

[4] The most recent version of the field ROM form is provided in the Libby Field eRoom.

submitted to the appropriate EPA RPM for review and approval. Copies of approved ROMs are maintained on the CDM Smith server in Libby.

B5.1.3 Field Surveillances

Field surveillances consist of periodic observations made to evaluate continued adherence to investigation-specific governing documents. A field audit is planned for this investigation and therefore, it is not anticipated that a field surveillance will be performed. Field surveillances may be conducted if field processes are revised or other QA/QC procedures indicate potential deficiencies.

B5.1.4 Field Audits

Field audits are broader in scope than field surveillances. Audits are evaluations conducted by qualified technical or QA staff that are independent of the activities audited. Field audits can be conducted by field contractors, internal EPA staff, or EPA contracted auditors. It is the responsibility of the EPA RPM to ensure that field auditing requirements are met for each investigation. It is anticipated that a field audit will be performed for this investigation.

B5.1.5 Field QC Samples

Field-based QC samples are collected in the field to provide information on data quality and submitted to the laboratory in a blind fashion. That is, the laboratory is not aware the sample is a QC sample, and will treat the sample as a field sample.

Sediment Porewater and Surface Water

Two types of field QC samples will be collected for porewater and surface water as part of this sampling investigation – field blanks and field duplicates.

<u>Field Blank</u>

A field blank is a sample of the same medium as field samples, but which does not contain any contaminant. A field blank for water shall be prepared by placing 400 mL of clean water (e.g., store bought drinking water) into the same type of sample collection container as the field samples. Field blanks will be collected at a frequency of one per field team per day. It is the responsibility of the FTL to ensure that the appropriate number of field blanks is collected. Field blanks will be given a unique sample number and will be specified as a field blank on the FSDS. One field blank per sampling event, chosen at random by the sample coordinator, is analyzed for each investigation. The field blanks will be analyzed for asbestos fibers by the same method as will be used for field sample analysis.

If asbestos is observed on the analyzed field blank, all other field blanks collected by that team will be submitted for analysis to determine the potential impact on the related sample results. If any asbestos structures are observed on a field blank, the FTL and/or laboratory manager will be notified and will take appropriate measures to ensure staff are employing proper sample handling techniques. In addition, a qualifier of "FB" will be added to the related field sample results in the project database to denote that the associated field blank had asbestos structures detected.

Field Duplicate

Field duplicates for water are a second 400-mL water sample collected sequentially from the same station as the parent sample. The field duplicate is collected using the same collection technique as the parent sample. Water field duplicate samples will be collected at a rate of 1 field duplicate per 5 field samples (20%). It is the responsibility of the FTL to ensure that the appropriate number of field duplicates is collected. Each field duplicate is given unique sample number, and field personnel record the sample number of the associated co-located sample in the parent sample number field of the FSDS. The same station location is assigned to the field duplicate sample as the parent field sample. Field duplicates will be sent for analysis by the same method as the field samples.

Field duplicate results will be compared to the original parent field sample using the Poisson ratio test using a 90% confidence interval (Nelson 1982). Because field duplicate samples are expected to have inherent variability that is random and may be either small or large, typically, there is no quantitative requirement for the agreement of field duplicates. Rather, results are used to determine the magnitude of this variability to evaluate data usability.

Sediment

Field duplicate samples will be collected as part of the sediment sampling for this study. Field duplicates for sediment are collected from the same area as the parent sample but from different individual sampling points. These samples are collected independent of the original field sample with separate sampling equipment and submitted for analysis along with the collected field samples. The field duplicate contains the same number of subsamples as the parent sample (i.e., if the parent sample is a 30-point composite, the field duplicate sample is also a 30-point composite).

Sediment field duplicate samples will be collected at a rate of 1 field duplicate per 10 field samples (10%). It is the responsibility of the FTL to ensure that the appropriate number of field duplicates is collected. Each field duplicate is given a unique sample number, and field personnel record the sample number of the associated co located sample in the parent sample number field of the FSDS. The same station location is assigned to the field duplicate sample as the parent field sample. Field duplicates will be sent for analysis by the same method as the field samples.

Field duplicate results analyzed by PLM-VE will be considered concordant if the reported semi-quantitative bin result for the field duplicate is within one bin of the original parent field sample. The variability between the field duplicate and the associated parent field sample reflects the combined variation in sample heterogeneity and the variation due to measurement error. Because field duplicate samples are expected to have inherent variability that is random and may be either small or large, typically, there is no quantitative requirement for the agreement of field duplicates. Rather, results are used to determine the magnitude of this variability to evaluate data usability.

B5.2 Preparation Laboratory

As noted above, prior to analysis by PLM, sediment samples will be dried, sieved, and ground at the Troy SPF. The sections below provide detailed information on QA/QC procedures for the Troy SPF, which is maintained by adherence to standard preparation procedures, submission of preparation QC samples, facilities monitoring, and audits.

B5.2.1 Training/Certifications

Personnel performing sample preparation activities must have read and understood the *Soil Sample Preparation Work Plan*, the *SPF Health and Safety Plan*, and all associated SOPs and governing documents for soil preparation (e.g., SOP ISSI-LIBBY-01). In addition, all personnel must have completed 40-hour Occupational Safety and Health Administration (OSHA) Hazardous Waste Operations and Emergency Response (HAZWOPER) training, annual updates, annual respirator fit tests, and annual or semi-annual physicals, as required.

Prior to performing activities at the Troy SPF, new personnel will be instructed by an experienced member of the SPF staff and training sessions will be documented in the SPF project files. It is the responsibility of the SPF QAM to ensure that all personnel have completed the required training requirements.

B5.2.2 Modification Documentation

When changes or revisions are needed to improve or document specifics about sample preparation procedures used by the Troy SPF, these changes are documented using a laboratory ROM form[5]. The ROM form provides a standardized format for tracking procedural changes in sample preparation and allows project managers to assess potential impacts on the quality of the data being collected. SPF ROMs will be completed by the appropriate SPF or technical staff. Once a form is prepared, it is submitted to the ESAT QAM (or their designee) for review. Final review and approval is provided by the appropriate EPA RPM. Copies of approved SPF ROMs are available in the Libby Lab eRoom.

[5] The most recent version of the laboratory ROM form is provided in the Libby Lab eRoom.

B5.2.3 Soil Preparation Facility Audits

Internal audits of the SPF are conducted by the SPF QAM periodically to evaluate personnel in their day-to-day activities and to ensure that all processes and procedures are performed in accordance with governing documents and SOPs. All aspects of sample preparation, as well as sample handling, custody, and shipping are evaluated. If any issues are identified, SPF personnel are notified and retrained as appropriate. Audit reports will be completed following each laboratory audit. A copy of the internal audit report, as well as any corrective action reports, will be provided to the LC and the QATS contractor.

Internal audits will be conducted following any significant procedural changes to the soil preparation processes or other SPF governing documents, to ensure the new methods are implemented and followed appropriately.

The Troy SPF is also required to participate in an annual on-site laboratory audit carried out by the EPA through the QATS contract. Audits consist of an evaluation of facility practices and procedures associated with the preparation of soil samples. A checklist of requirements, as derived from the applicable governing documents and SOPs, is prepared by the auditor prior to the audit, and used during the on-site evaluation. Evaluation of the facility is made by reviewing SPF documentation, observing sample processing, and interviewing personnel.

It is the responsibility of the QATS contractor to prepare an On-site Audit Report following the SPF audit. The On-site Audit Report includes both a summary of the audit results and completed checklist(s), as well as recommendations for corrective actions, as appropriate. Responses from each SPF to any deficiencies noted in the On-site Audit Report are also maintained with the respective reports.

B5.2.4 Preparation QC Samples

Four types of preparation QC samples are collected during the soil/sediment preparation process: sand blanks, drying blanks, grinding blanks, and preparation duplicates. Each type of preparation QC sample is described in more detail below.

<u>Sand Blank</u>

A sand blank is a sample of store-bought quartz sand that is analyzed to ensure that the quartz sand matrix used for drying and grinding blanks is asbestos-free. Detailed procedures for this certification process are provided in ESAT SOP PLM-02.00, *Blank Sand Certification by Polarized Light Microscopy*. In brief, about 800 grams of sand are split into 40 sand blank aliquots of roughly equal size. Each sand blank is evaluated using stereomicroscopic examination and analyzed by PLM-VE. If a sand blank has detected asbestos, it is re-analyzed by a second PLM analyst to verify the presence of asbestos. The sand is certified as asbestos-free if all 40 sand blanks are non-detect for asbestos. The sand is rejected for use if any asbestos is detected in the sand blanks. Only sand that is certified as asbestos-free will be utilized in the SPF.

Drying Blank

A drying blank consists of approximately 100 to 200 grams of asbestos-free quartz sand that is processed with each batch of field samples that are dried together (usually this is approximately 125 samples per batch). The drying blank is then processed identically to field samples. Drying blanks determine if cross-contamination between samples is occurring during sample drying. One drying blank will be processed with each drying batch per oven. It is the responsibility of the SPF QAM to ensure that the appropriate number of drying blanks is collected. Each drying blank is given unique sample number that is investigation-specific, as provided by the field sample coordinator (i.e., a subset of sample numbers for each investigation will be provided for use by the SPF). SPF personnel will record the sample number of the drying blank on the sample drying log sheet.

It is the responsibility of the QATS contractor to review the drying blank results and notify the SPF QAM immediately if drying blank results do not meet acceptance criteria and if corrective actions are necessary. If asbestos is detected by PLM-VE in the drying blank (i.e., result is not Bin A), a qualifier of "DB" will be added to the related field sample results in the project database that were dried at the same time as the detected drying blank to denote that the associated drying blank had detected asbestos. In addition, the drying oven will be thoroughly cleaned. If asbestos continues to be detected in drying blanks after cleaning occurs, sample processing must stop and the drying method and decontamination procedures will be evaluated to rectify any cross-contamination issues.

Grinding Blank

A grinding blank consists of asbestos-free quartz sand and is processed along with the field samples on days that field samples are ground. Grinding blanks determine if decontamination procedures of laboratory soil processing equipment used for sample grinding and splitting are adequate to prevent cross-contamination. Grinding blanks are prepared at a frequency of one per grinding batch per grinder per day. It is the responsibility of the SPF QAM to ensure that the appropriate number of grinding blanks is collected. Each grinding blank is given unique sample number that is investigation-specific, as provided by the field sample coordinator. SPF personnel will record the sample number of the grinding blank on the sample preparation log sheet.

It is the responsibility of the QATS contractor to review the grinding blank results and notify the SPF QAM immediately if drying blank results do not meet acceptance criteria and if corrective actions are necessary. If any asbestos is detected by PLM-VE in the grinding blank (i.e., result is not Bin A), a qualifier of "GB" will be added to the related field sample results in the project database that were ground at the same time as the detected grinding blank to denote that the associated grinding blank had detected asbestos. In addition, the grinder will be thoroughly cleaned. If asbestos continues to be detected in grinding blanks after cleaning occurs, sample processing must stop and the grinding method and decontamination procedures will be evaluated to rectify any cross-contamination issues.

Preparation Duplicate

Preparation duplicates are splits of field samples submitted for sample preparation. The preparation duplicates are used to evaluate the variability that arises during the soil preparation and analysis steps. After drying, but prior to sieving, a preparation duplicate is prepared by using a riffle splitter to divide the field sample (after an archive split has been created) into two approximately equal portions, creating a parent and duplicate sample.

Preparation duplicate samples are prepared at a rate of 1 per 20 samples (5%) of samples prepared. It is the responsibility of the SPF QAM to ensure that the appropriate number of preparation duplicates is prepared. Each preparation duplicate is given unique sample number that is investigation-specific, as provided by the field sample coordinator. SPF personnel will record the sample number of the preparation duplicate and its associated parent field sample on the sample preparation log sheet. Preparation duplicates are submitted blind to the laboratory for analysis by the same analytical method as the parent sample.

Preparation duplicate results will be considered concordant if the reported PLM-VE bin for the preparation duplicate is within one bin of the original parent field sample. The variability between the preparation duplicate and the associated field sample reflects the combined variation due to sample preparation and due to measurement error. Results for preparation duplicate samples are evaluated by the QATS contractor or their designee. If the concordance rate for preparation duplicate samples is less than 10%, the QATS contractor will notify the SPF QAM to determine if corrective action is needed.

B5.2.5 Performance Evaluation Standards

The USGS has prepared several Site-specific reference materials of LA in soil that are utilized as PE standards to evaluate PLM-VE laboratory accuracy. These PE standards are kept in storage at the Troy SPF and are inserted into the sample train during soil sample processing. In accordance with SOP ISSI-LIBBY-01, PE standards are inserted both pre- and post-processing. PE standards of varying nominal levels will be inserted on a quarterly basis at a rate of at least one PE standard per PLM-VE laboratory.

It is the responsibility of the SPF QAM to ensure that the appropriate number of PE standards is inserted. Each PE standard is given unique sample number that is investigation-specific, as provided by the field sample coordinator. SPF personnel will record the sample number of the PE standard, the nominal level of the PE standard, and whether it was inserted pre- or post-processing on the sample preparation log sheet. PE standards are submitted blind to the laboratory for analysis by the same analytical method as the field samples.

Results for PE standards will be evaluated by the QATS contractor (or their designee). PE standard results are ranked as acceptable if the correct semi-quantitative bin is reported, as determined by the nominal concentration of the PE standard. The LC should be notified if PE standard results do not meet acceptance criteria. Corrective action will be taken if the PE standards demonstrate issues with accuracy and/or bias in PLM-VE results reporting. Examples of corrective actions that may be taken include reanalysis and/or repreparation, collaboration between and among laboratories to address potential differences in analysis methods, and analyst re-training.

B5.3 Analytical Laboratory

Laboratory QA/QC activities include all processes and procedures that have been designed to ensure that data generated by an analytical laboratory are of high quality and that any problems in sample preparation or analysis that may occur are quickly identified and rectified. The following sections describe each of the components of the analytical laboratory QA/QC program implemented at the Site.

B5.3.1 Training/Certifications

All analytical laboratories participating in the analysis of samples for the Libby project are subject to national, local, and project-specific certifications and requirements. Additional information on laboratory training and certification requirements is provided in Section A8.2.

Laboratories handling samples collected as part of this sampling program will be provided a copy of and will adhere to the requirements of this SAP/QAPP. Samples collected under this SAP/QAPP will be analyzed in accordance with standard EPA and/or nationally-recognized analytical procedures (i.e., Good Laboratory Practices) in order to provide analytical data of known quality and consistency.

B5.3.2 Modification Documentation

All deviations from project-specific and method guidance documents will be recorded on the Libby Laboratory ROM Form[6]. The ROM will be used to document all permanent and temporary changes to analytical procedures. ROMs will be completed by the appropriate laboratory or technical staff. As ROMs are completed, it is the responsibility of the LC to communicate any changes to the project laboratories. When the project management team determines the need, this SAP/QAPP will be revised to incorporate necessary modifications. Copies of approved ROMs for this SAP/QAPP will be made available in the Libby Lab eRoom.

[6] The most recent version of the laboratory ROM form is provided in the Libby Lab eRoom.

B5.3.3 Laboratory Audits

Each laboratory working on the Libby project is required to participate in an annual on-site laboratory audit carried out by the EPA through the QATS contract. These audits are performed by EPA personnel (and their contractors), that are external to and independent of, the Libby laboratory team members. These audits ensure that each analytical laboratory meets the basic capability and quality standards associated with analytical methods for asbestos used at the Libby site. They also provide information on the availability of sufficient laboratory capacity to meet potential testing needs associated with the Site.

<u>External Audits</u>

Audits consist of several days of technical and evidentiary review of each laboratory. The technical portion of the audit involves an evaluation of laboratory practices and procedures associated with the preparation and analysis of samples for the identification of asbestos. The evidentiary portion of the audit involves an evaluation of data packages, record keeping, SOPs, and the laboratory *QA Management Plan*. A checklist of method-specific requirements for the commonly used methods for asbestos analysis is prepared by the auditor prior to the audit, and used during the on-site laboratory evaluation.

Evaluation of the capability for a laboratory to analyze a sample by a specific method is made by observing analysts performing actual sample analyses and interviewing each analyst responsible for the analyses. Observations and responses to questions concerning items on each method-specific checklist are noted. The determination as to whether the laboratory has the capability to analyze a sample by a specific method depends on how well the analysts follow the protocols detailed in the formal method, how well the analysts follow the laboratory-specific method SOPs, and how the analysts respond to method-specific questions.

Evaluation of the laboratory to be sufficient in the evidentiary aspect of the audit is made by reviewing laboratory documentation and interviewing laboratory personnel responsible for maintaining laboratory documentation. This includes personnel responsible for sample check-in, data review, QA procedures, document control, and record archiving. Certain analysts responsible for method quality control, instrument calibration, and document control are also interviewed in this aspect of the audit. Determination as to the capability to be sufficient in this aspect is made based on staff responses to questions and a review of archived data packages and QC documents.

It is the responsibility of the QATS contractor to prepare an On-site Audit Report for each analytical laboratory participating in the Libby program. These reports are handled as business confidential items. The On-site Audit Report includes both a summary of the audit results and completed checklist(s), as well as recommendations for corrective actions, as appropriate. Responses from each laboratory to any deficiencies noted in the On-site Audit Report are also maintained with the respective reports.

It is the responsibility of the QATS contractor to prepare an On-Site Audit Trend Analysis Report on an annual basis. This report shall include a compilation and trend analysis of the on-site audit findings and recommendations. The purpose of this reported is to identify common asbestos laboratory performance problems and isolate the potential causes.

Internal Audits

Each laboratory will also conduct periodic internal audits of their specific operations. Details on these internal audits are provided in the laboratory *QA Management Plan*. The laboratory QAM should immediately contact the LC and the QATS contractor if any issues are identified during internal audits that may impact data quality.

B5.3.4 Laboratory QC Analyses

TEM

The Libby-specific QC requirements for TEM analyses of asbestos are patterned after the requirements set forth by NVLAP. In brief, there are three types of laboratory-based QC analyses for TEM – laboratory blanks, recounts, and repreparations. Detailed information on the Libby-specific requirements for each type of TEM QC analysis, including the minimum frequency rates, selection procedures, acceptance criteria, and corrective actions are provided in the most recent version of Libby Laboratory Modification LB-000029.

With the exception of inter-laboratory analyses, it is the responsibility of the laboratory manager to ensure that the proper number of TEM QC analyses are completed. Inter-laboratory analyses for TEM will be selected *post hoc* by the QATS contractor or their designee in accordance with the selection procedures presented in LB-000029. The LC will provide the list of selected inter-laboratory analyses to the laboratory manager and will facilitate the exchange of samples between the analytical laboratories.

PLM

Laboratory QC for PLM-Grav is ensured through compliance with laboratory-based QC requirements for the NIOSH Method 9002, as specified by NVLAP. No additional project-specific QC requirements have been established for PLM-Grav.

Laboratory-based QC requirements for PLM-VE are specified in SOP SRC-LIBBY-03 and Libby Laboratory Modification LB-000073. Three types of laboratory-based QC analyses will be performed for PLM-VE, including laboratory duplicates, inter-laboratory analyses, and PE standards. Detailed information on the Libby-specific requirements for each type of PLM-VE QC analysis, including the minimum frequency rates, selection procedures, acceptance criteria, and corrective actions are provided in SOP SRC-LIBBY-03 and LB-000073.

It is the responsibility of the laboratory manager to ensure that the proper number of PLM-VE laboratory duplicate analyses is completed. Inter-laboratory analyses for PLM-VE will selected *post hoc* by the QATS contractor (or their designee) in accordance with the selection procedures presented in LB-000073. The LC will provide the list of selected inter-laboratory analyses to the laboratory manager and will facilitate the exchange of samples between the analytical laboratories. It is the responsibility of the SPF QAM to ensure that the appropriate number of PE standards is inserted.

B6/B7. Instrument Maintenance and Calibration

B6/B7.1 Field Equipment

All field equipment (e.g., GPS) should be maintained and calibrated in basic accordance with manufacturer specifications. When a piece of equipment is found to be operating incorrectly, the piece of equipment will be labeled "out of order" and placed in a separate area from the rest of the sampling equipment. The person who identified the equipment as "out of order" will notify the FTL overseeing the investigation activities. It is the responsibility of the FLT to facilitate repair of the out-of-order equipment. This may include having appropriately trained field team members complete the repair or shipping the malfunctioning equipment to the manufacturer. Field team members will have access to basic tools required to make field acceptable repairs. This will ensure timely repair of any "out of order" equipment.

B6/B7.2 Laboratory Instruments

All laboratory instruments used for this project will be maintained and calibrated in accordance with the manufacturer's instructions. If any deficiencies in instrument function are identified, all analyses shall be halted until the deficiency is corrected. The laboratory shall maintain a log that documents all routine maintenance and calibration activities, as well as any significant repair events, including documentation that the deficiency has been corrected.

B8. Inspection/Acceptance of Supplies and Consumables

B8.1 Field

In advance of field activities, the FTL will check the field equipment/supply inventory and procure any additional equipment and supplies that are needed. The FTL will also ensure any in-house measurement and test equipment used to collect data/samples as part of this SAP/QAPP is in good, working order, and any procured equipment is acceptance tested prior to use. Any items that the FTL determines unacceptable will be removed from inventory and repaired or replaced as necessary.

The specialized equipment necessary for this sampling program includes the modified WVBs and sampling port bailers, as well as an adequate supply of 500-mL capacity HDPE sampling containers.

B8.2 Laboratory

The laboratory manager is responsible for ensuring that all reagents and disposable equipment used in this project is free of asbestos contamination. This is demonstrated by the collection of laboratory blank samples, as described in Section B5.

B9. Non-Direct Measurements

There are no non-direct measurements that are anticipated for use in this project.

B10. Data Management

The following subsections describe the field, Troy SPF, and analytical laboratory data management procedures and requirements for this investigation. These subsections also describe the project databases utilized to manage and report data from this investigation. Detailed information regarding data management procedures and requirements can be found in the *EPA Data Management Plan* for the Libby Asbestos Superfund Site (EPA 2012).

B10.1 Field Data Management

Scribe is a software tool developed by ERT to assist in the process of managing environmental data. A Scribe project is a Microsoft Access database. Data for the Site are captured in various Scribe projects. Additional information regarding Scribe and the Libby Scribe project databases is discussed in Section B10.3.

The field data manager utilizes a "local" field Scribe project database (i.e., LibbyCDM_Field.mdb) to maintain field sample information. The term "local" denotes that the database resides on the server or personal computer of the entity that is responsible for the creating/managing the database. It is the responsibility of the field data manager to ensure that all local field Scribe project databases are backed-up nightly to a local server.

Field sample information from the FSDS is manually entered by a member of the field sample coordination staff using a series of standardized data entry forms (i.e., DE Tool). This tool is a Microsoft Access® database that was originally developed by ESAT. The DE Tool is currently maintained by CDM Smith and resides on the local server in the Libby field office. This tool is used to prepare an electronic COC. Data in the DE Tool are imported into the local field Scribe project database by the field data manager.

It is the responsibility of the field data manager to "publish" sample and COC information from the local field Scribe database to Scribe.NET on a daily basis. It is not until a database has been published via Scribe.NET that it becomes available to external users.

B10.2 Troy SPF Data Management

The Troy SPF utilizes a local SPF Scribe project database to maintain soil/sediment sample preparation information. Preparation information from the preparation log sheets is entered into the local SPF Scribe project database by SPF personnel. After the data entry is checked against the original forms, it is the responsibility of the SPF manager (or their designee) to publish sample preparation information from the local SPF Scribe database to Scribe.NET.

B10.3 Analytical Laboratory Data Management

The analytical laboratories utilize several standardized data reporting tools developed specifically for the Libby project to ensure consistency between laboratories in the presentation and submittal of analytical data. In general, a unique Libby-specific EDD has been developed for each analytical method and each medium. Electronic copies of all current EDD templates are provided in the Libby Lab eRoom.

Once the analytical laboratory has populated the EDD with results, the spreadsheet(s) are transmitted *via* email to the ESAT TEM Laboratory Manager, the ESAT project data manager, and the FTL (or their designee). (Other email recipients may also be specified by the LC).

The ESAT project data manager utilizes a local analytical Scribe project database (i.e., LibbyLab2013.mdb) to maintain analytical results information. The EDDs are uploaded directly into the analytical Scribe project database(s). It is the responsibility of the ESAT project data manager to publish analytical results information from the local analytical Scribe database to Scribe.NET.

B10.4 Libby Project Database

As noted above, Scribe is a software tool developed by ERT to assist in the process of managing environmental data. A Scribe project is a Microsoft Access database. Multiple Scribe projects can be stored and shared through Scribe.NET, which is a web-based portal that allows multiple data users controlled access to Scribe projects. Local Scribe projects are "published" to Scribe.NET by the entity responsible for managing the local Scribe project. External data users may "subscribe" to the published Scribe projects via Scribe.NET to access data. Subscription requests are managed by ERT.

All data collected for this investigation will be maintained in Scribe. As discussed above, data will be captured in various Scribe project databases, including a field Scribe project (i.e., LibbyCDM_Field.mdb), Troy SPF preparation Scribe project (i.e., PrepLabLibby.mdb), and an analytical results Scribe project (i.e., LibbyLab2013.mdb).

B10.5 Data Reporting

Data users can access data for the Libby project through Scribe.NET. To access data, a data user must first download the Scribe application from the EPA ERT website[7]. The data user must then subscribe to each of the published Scribe projects for the Site using login and password information that are specific to each individual Scribe project. Scribe subscriptions for the Libby project are managed by ERT. Using the Scribe application, a data user may download a copy of any published Scribe project database to their local hard drive. It is the responsibility of the data user to regularly update their local copies of the Libby Scribe projects *via* Scribe.NET.

The Scribe application provides several standard queries that can be used to summarize and view results within an individual Scribe project. However, these standard Scribe queries cannot be used to summarize results across multiple Scribe projects (e.g., it is not possible to query both the "LibbyCDM_Field" project and the "LibbyLab2013" project using these standard Scribe queries).

If data users wish to summarize results across multiple published Scribe projects, there are two potential options. Data users may request the development of a "combined" Scribe project from ERT. This combined project compiles tables from multiple published Scribe projects into a single Scribe project. This allows data users to utilize the standard Scribe queries to summarize and view results.

Alternatively, data users may download copies of multiple published Scribe project databases for the Site and utilize Microsoft Access® to create user-defined queries to extract the desired data across Scribe projects. This requires that the data user is proficient in Microsoft Access® and has an intimate knowledge of proper querying methods for asbestos data for the Site.

It is the responsibility of the data users to perform a review of results generated by any data queries and standard reports to ensure that they are accurate, complete, and representative. If issues are identified by the data user, they should be reported to the EPA Region 8 data manager for resolution *via* email (Mosal.Jeffrey@epa.gov). It is the responsibility of the EPA Region 8 data manager to notify the appropriate entity (e.g., field, Troy SPF, analytical laboratory) in order to rectify the issue. A follow-up email will be sent to the party reporting the issue to serve as confirmation that a resolution has been reached and any necessary changes have been made.

[7] http://www.ertsupport.org/scribe_home.htm

C Assessment and Oversight

C1. Assessment and Response Actions

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.

C1.1 Assessments

System assessments are qualitative reviews of different aspects of project work to check the use of appropriate QC measures and the general function of the QA system. Field and office system assessments will be performed under the direction of the CDM Smith QAM, with support from the CDM Smith QA Coordinator. As noted previously, it is anticipated that a field audit will be conducted for this investigation. Field surveillances may be conducted if field processes are revised or other QA/QC procedures indicate potential deficiencies. Findings of the field audit will be shared with the EPA RPM and the QATS contractor.

System assessments/audits of the analytical laboratories will be conducted by the QATS contractor, as coordinated by the EPA.

C1.2 Response Actions

Corrective response actions will be implemented on a case-by-case basis to address quality problems. Minor actions taken to immediately correct a quality problem will be documented in the applicable field or laboratory logbooks and a verbal report will be provided to the appropriate manager (e.g., the FTL or LC). Major corrective actions will be approved by the EPA RPM and the appropriate manager prior to implementation of the change. Major response actions are those that may affect the quality or objective of the investigation. EPA project management will be notified when quality problems arise that cannot be corrected quickly through routine procedures.

In addition, when modifications to this SAP/QAPP are required, either for field or laboratory activities, a ROM must be completed by field staff and approved by the EPA prior to implementation.

C2. Reports to Management

No regularly-scheduled written reports to management are planned as part of this project. However, QA reports will be provided to management for routine audits and whenever quality problems are encountered. Field staff will note any quality problems on FSDSs or in field logbooks. Further, the field and laboratory managers will inform the EPA RPM upon encountering quality issues that cannot be immediately corrected.

D Data Validation and Usability

D1/D2. Data Review, Verification and Validation

D1/D2.1 Data Review

Data review of Scribe project data typically occurs at the time of data reporting by the data users and includes cross-checking that sample IDs and sample dates have been reported correctly and that calculated analytical sensitivities or reported values are as expected. If discrepancies are found, the data user will contact the EPA data manager, who will then notify the appropriate entity (field, Troy SPF, or laboratory) in order to correct the issue.

D1/D2.2 Criteria for LA Measurement Acceptability

Several factors are considered in determining the acceptability of LA measurements in water samples analyzed by TEM. This includes the following:

- *Evenness of filter loading.* This is evaluated using a Chi-square (CHISQ) test, as described in ISO 10312:1995(E) Annex F2. If a filter fails the CHISQ test for evenness, the result may not be representative of the true concentration in the sample, and the results should be given low confidence.

- *Results of QC samples.* This includes both field and laboratory QC samples, such as field and laboratory blank samples, as well as various types of recount and repreparation analyses. If significant LA contamination is detected in field or laboratory blanks, all samples prepared on that day should be considered to be potentially biased high. If agreement between original analyses and field or laboratory duplicates (i.e., repreparation or recount analyses) is poor, results for those samples should be given low confidence.

For PLM analyses, the following factors will be considered in determining the acceptability of LA measurements sediment samples:

- *Results of performance evaluation (PE) standard analyses.* PLM-VE accuracy is evaluated using LA-specific PE standards. If the results for these PE standards are not within the project-specific acceptance criteria, results should be given low confidence.

- *Results of QC samples.* This includes field, preparation, and laboratory QC samples. If LA contamination is detected in any blanks, associated samples should be considered to be potentially biased high. If agreement between original and repeat analyses (i.e., duplicate analyses, inter-laboratory analyses) is strongly discordant, results for those samples should be given low confidence.

D1/D2.3 Data Verification Method

Data verification includes checking that results have been transferred correctly from the original hand-written, hard copy field and analytical laboratory documentation to the project databases. The goal of data verification is to identify and correct data reporting errors.

For analytical laboratories that utilize the Libby-specific EDD spreadsheets, data checking of reported analytical results begins with automatic QC checks that have been built into the spreadsheets. In addition to these automated checks, a detailed manual data verification effort will be performed for 10% of all sample information and analytical results from this study. This data verification process utilizes Site-specific SOPs developed to ensure TEM results, PLM results, and field sample information in the project databases are accurate and reliable (see **Appendix B**):

- EPA-LIBBY-09 – *SOP for TEM Data Review and Data Entry Verification* – This Site-specific SOP describes the steps for the verification of TEM analyses, based on a review of the laboratory benchsheets, and verification of the transfer of results from the benchsheets into the project database.

- EPA-LIBBY-10 – *SOP for PLM Data Review and Data Entry Verification* – This Site-specific SOP describes the steps for the verification of PLM analyses, based on a review of the laboratory benchsheets, and verification of the transfer of results from the benchsheets into the project database.

- EPA-LIBBY-11 - *SOP for FSDS Data Review and Data Entry Verification* – This Site-specific SOP describes the steps for the verification of field sample information, based on a review of the FSDS form, and verification of the transfer of results from the FSDS forms into the project database. An FSDS review is performed on all samples selected for TEM or PLM data verification.

The data verification review ensures that any data reporting issues are identified and rectified to limit any impact on overall data quality. If issues are identified during the data verification, the frequency of these checks may be increased as appropriate.

Data verification will be performed by appropriate technical staff that are familiar with project-specific data reporting, analytical methods, and investigation requirements. The data verifier will prepare a data verification report (template reports are included in the SOPs) to summarize any issues identified and necessary corrections. A copy of this report will be provided to the appropriate project data manager, LC, and the EPA RPM. The data verifier will also transmit the results of the data verification, including any electronic files summarizing identified discrepancies, *via* email to the EPA Region 8 data manager (Mosal.Jeffry@epa.gov) for resolution. A follow-up email will be sent to the data verifier to serve as confirmation that a resolution has been reached on any issues identified.

It is the responsibility of the EPA Region 8 data manager to coordinate with the FTL and/or LC to resolve any project database corrections and address any recommended field or laboratory procedural changes from the data verifier. The EPA Region 8 data manager is also responsible for electronically tracking in the project database which data have been verified, who performed the verification, and when.

D1/D2.4 Data Validation Method

Unlike data verification, where the goal is to identify and correct data reporting errors, the goal of data validation is to evaluate overall data quality and to assign data qualifiers, as appropriate, to alert data users to any potential data quality issues. Data validation will be performed by the QATS contractor (or their designee), with support from technical support staff that are familiar with project-specific data reporting, analytical methods, and investigation requirements.

Data validation for asbestos will be performed in basic accordance with the draft *National Functional Guidelines (NFG) for Asbestos Data Review* (EPA 2011), and should include an assessment of the following:

- Internal and external field audit/surveillance reports
- Field ROMs
- Field QC sample results
- Internal and external laboratory audit reports
- Laboratory contamination monitoring results
- Laboratory ROMs
- Internal laboratory QC analysis results
- Inter-laboratory analysis results
- Performance evaluation results
- Instrument checks and calibration results
- Data verification results (i.e., in the event that the verification effort identifies a larger data quality issue)

A comprehensive data validation effort should be completed quarterly and results should be reported as a technical memorandum. This technical memorandum shall detail the validation procedures performed and provide a narrative on the quality assessment for each type of asbestos analysis, including the data qualifiers assigned, and the reason(s) for these qualifiers. The technical memorandum shall detail any deficiencies and required corrective actions.

The QATS contractor will also prepare an annual addendum to the *Quality Assurance and Quality Control Summary Report for the Libby Asbestos Superfund Site* (CDM Smith 2011) to summarize results of the quarterly data validation efforts. This addendum should include a summary of any data qualifiers that are to be added to the project database to denote when results do not meet NFG guidelines and/or project-specific acceptance criteria. This addendum should also include recommendations for Site QA/QC program changes to address any data quality issues.

The data validator will transmit the results for each data validation effort *via* email to the EPA Region 8 data manager (Mosal.Jeffrey@epa.gov). This email should include an electronic summary of the records that have been validated, the date they were validated, any recommended data qualifiers, and their associated reason codes. It is the responsibility of the EPA Region 8 data manager to ensure that the appropriate data qualifiers and reason codes recommended by the data validator are added to the project database, and to electronically track in the project database which data have been validated, who performed the validation, and when.

In addition to performing quarterly data validation efforts, it is the responsibility of the QATS contractor (or their designee) to perform regular evaluations of all field and laboratory blanks, to ensure that any potential contamination issues are quickly identified and resolved. If any blank contamination is noted, the QATS contractor should immediately contact the appropriate QA Coordinator or QAM to ensure that corrective actions are made.

D3. Reconciliation with User Requirements

It is the responsibility of data users to perform a data usability assessment to ensure that investigation-specific DQOs have been met, and reported investigation results are adequate and appropriate for their intended use. This data usability assessment should utilize results of the data verification and data validation efforts to provide information on overall data quality specific to each investigation.

The data usability assessment should evaluate results with regard to several data usability indicators. **Table D-1** summarizes several indicators of data usability and presents general evaluation methods for each indicator. Depending upon the nature of the investigation, other evaluation methods may also be appropriate. The data usability assessment results and conclusions should be included in any investigation-specific data summary reports.

Non-attainment of project requirements may result in additional sample collection or field observations in order to achieve project needs.

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Sampling and Analysis Plan/Quality Assurance Project Plan: Sediment Porewater Study of Kootenai River Tributaries Libby Asbestos Superfund Site

Revision 0 – May 2013

FIGURES

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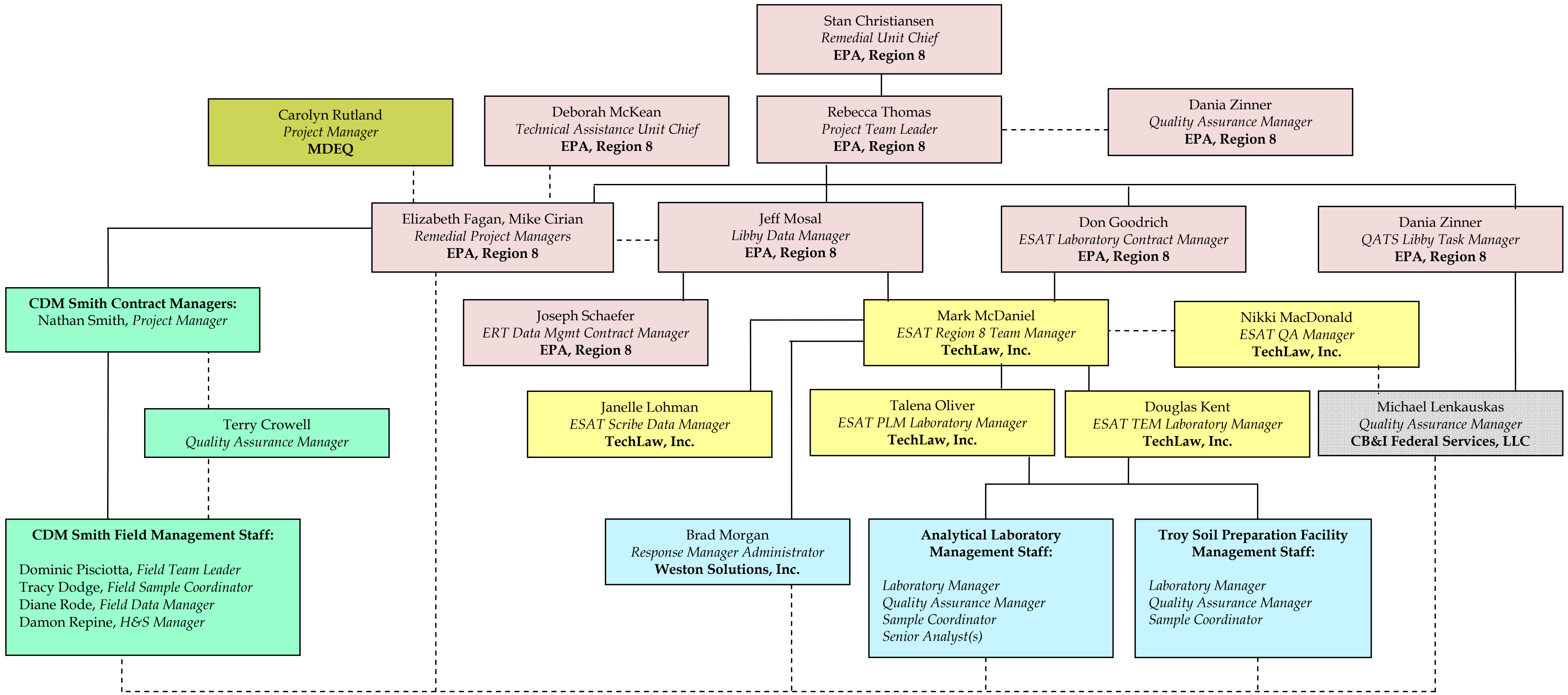
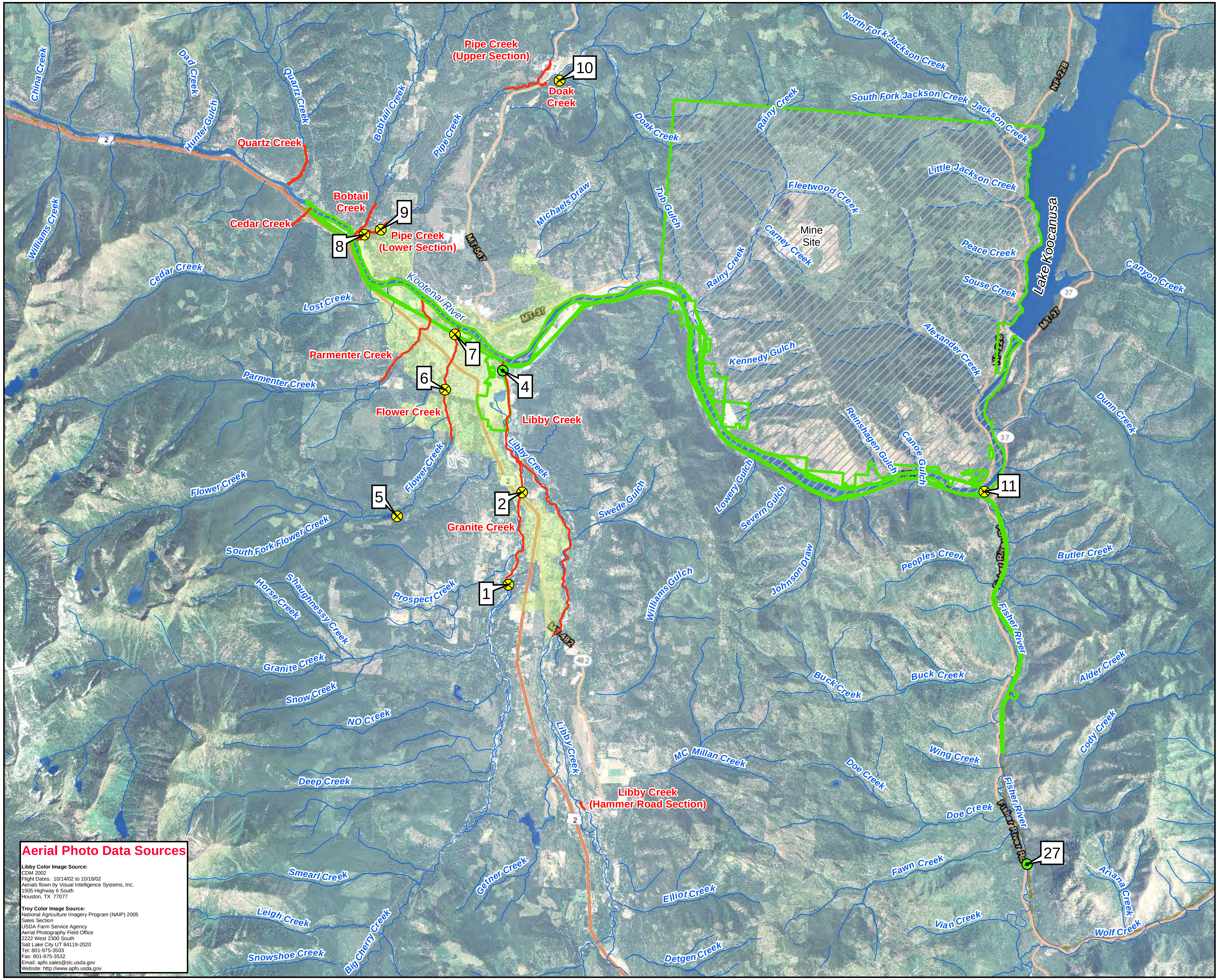
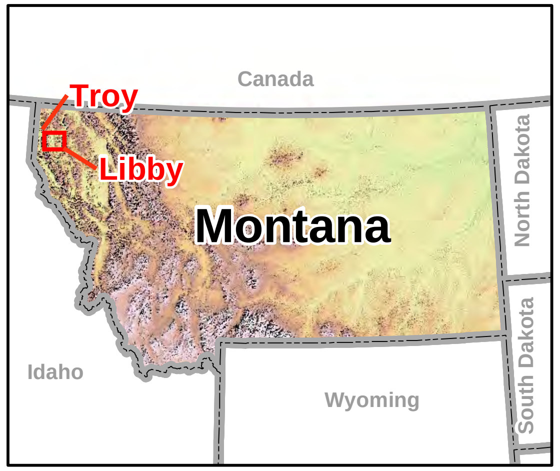


Figure A-1. Organizational Chart for the Tributary Sediment Porewater Study

EPA Region 8 Staff	CB&I Staff
MDEQ Staff	TechLaw Staff
CDM Smith Staff	TechLaw Subcontractors

Lines of authority (solid line)
Lines of communication (dashed line)





Legend

- Porewater Sampling Locations
- Sediment and Porewater Sampling Locations
- Portion of Creek Investigated
- Creeks / Rivers
- OU4 Boundary
- OU3 Boundary
- Populated Areas
- Lake

N
W E
S

Miles
0 1 2 4
1 inch = 1.90196 miles

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Figure B - 1

Libby Sediment Porewater
Sampling Locations
Libby Asbestos Superfund Site, Operable Unit 4
Libby, Montana

CDM Smith

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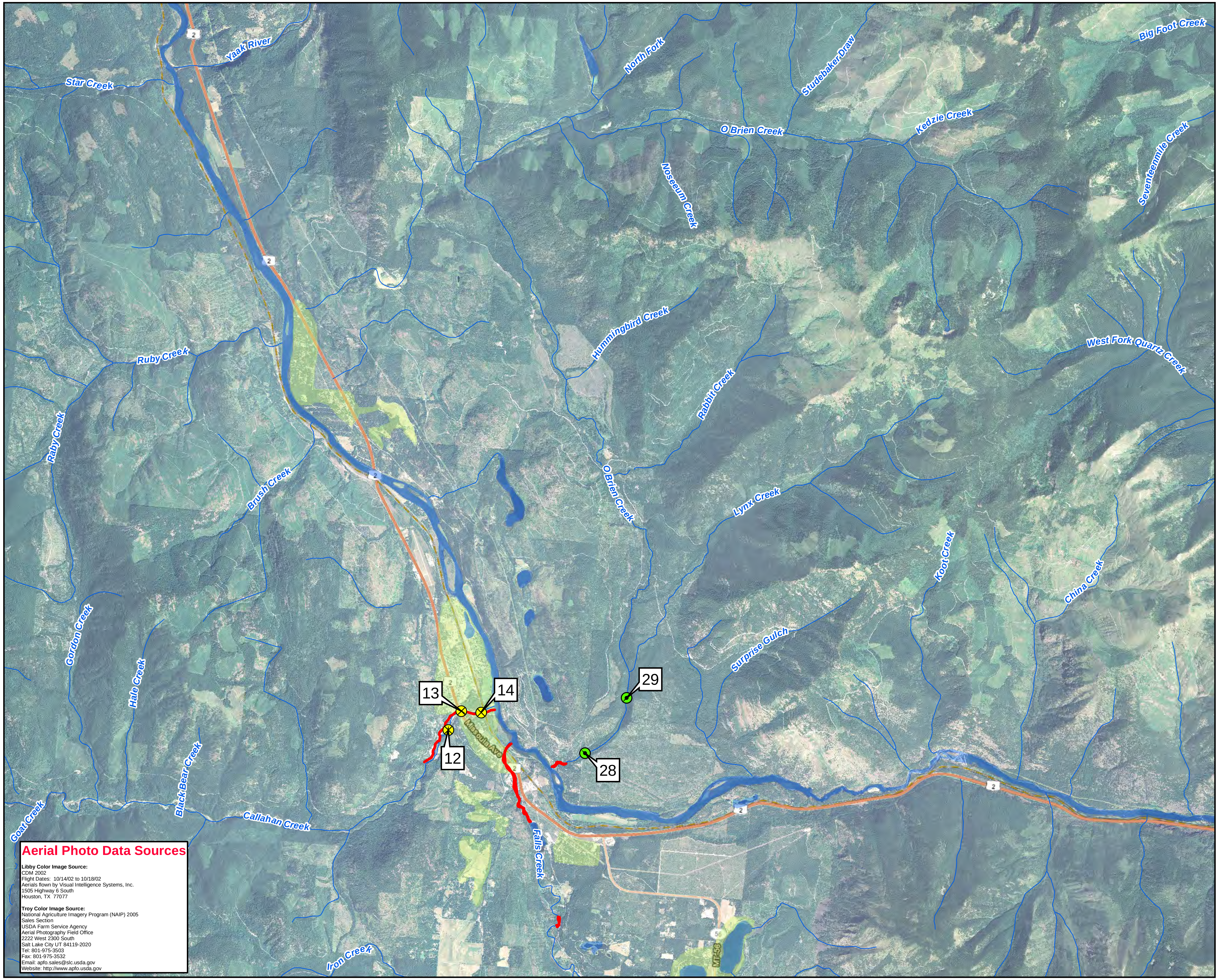

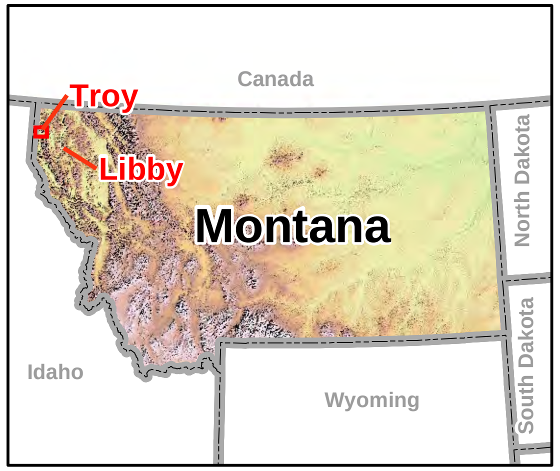

Legend

- Porewater Sampling Locations
- Sediment and Porewater Sampling Locations
- Portion of Creek Investigated
- Creeks / Rivers
- OU4 Boundary
- OU3 Boundary
- Populated Areas
- Lake

N S W E

Miles

0 0.5 1 2

1 inch = 1 miles

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

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Figure B - 2

Troy Sediment Porewater
Sampling Locations
Libby Asbestos Superfund Site, Operable Unit 4
Troy, Montana

CDM Smith

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FIGURE B-3. LA CONCENTRATIONS IN POREWATER DURING THE 2012 EYED EGG STUDY

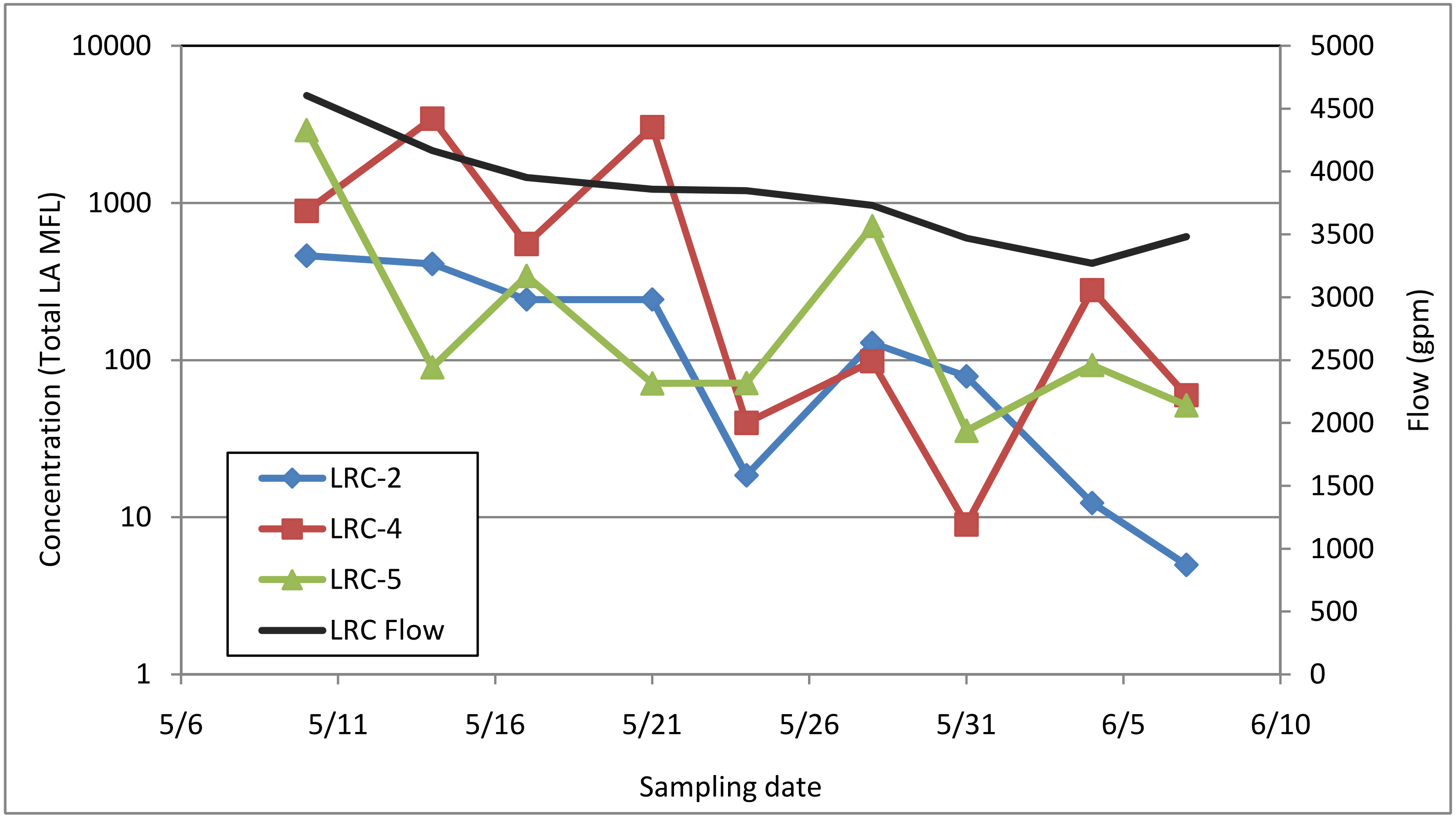
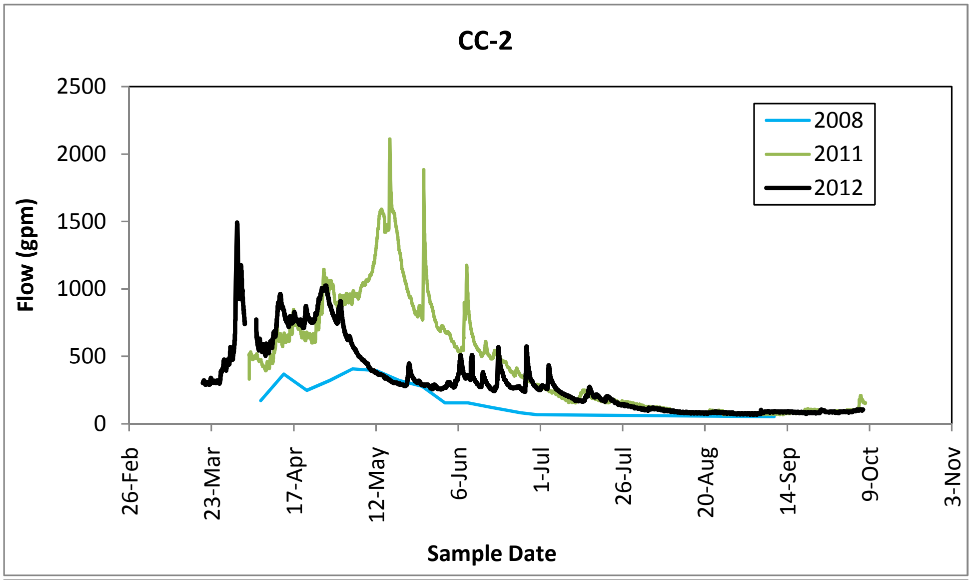
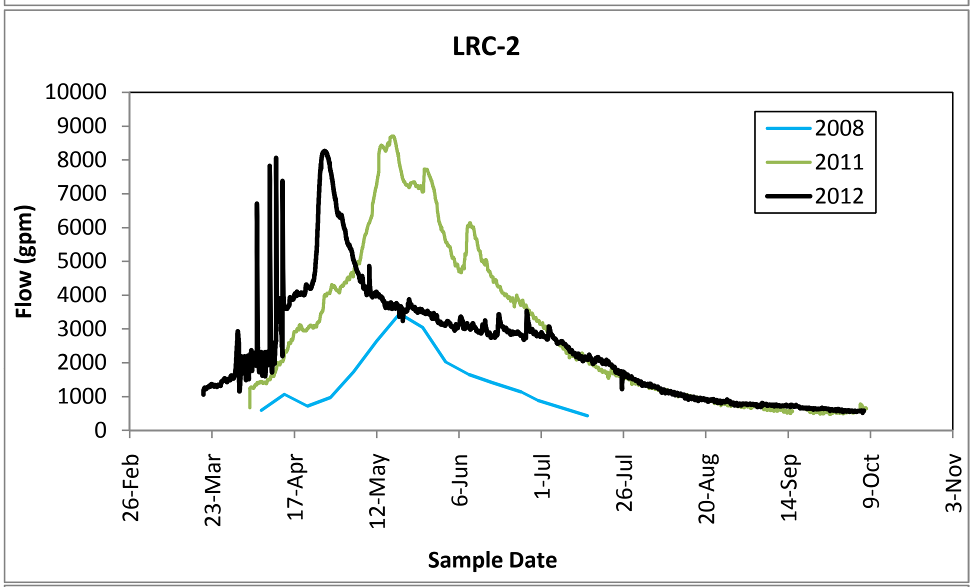
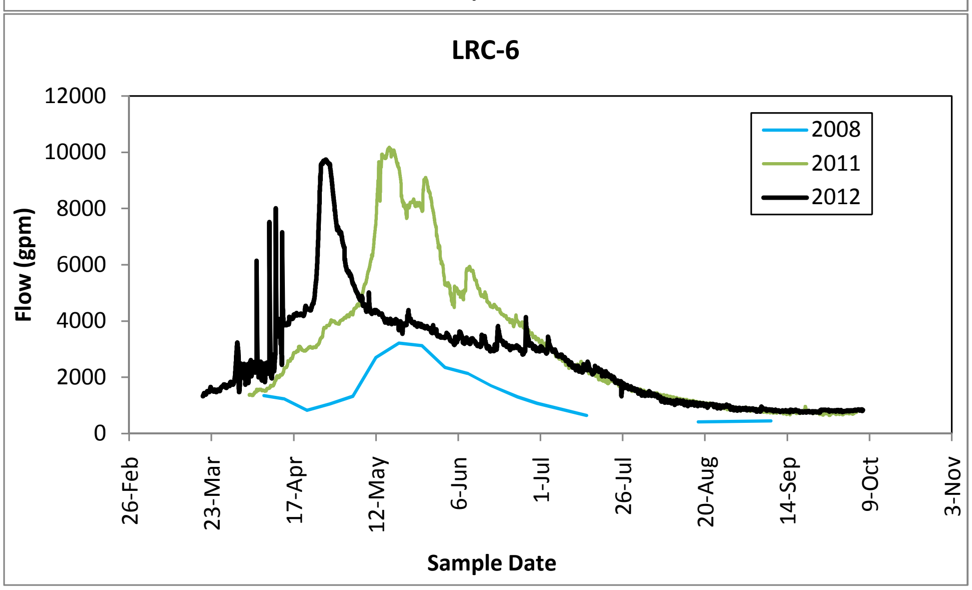


FIGURE B-4. MEASURED SURFACE WATER FLOWS IN OU3 CREEKS







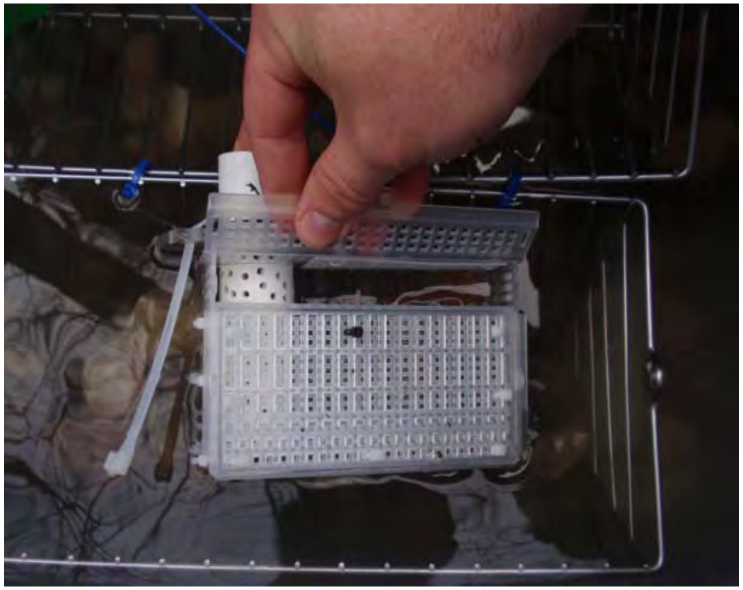


Figure B-5. Example Photos of Porewater Sampling using Modified Whitlock Vibert Boxes

Sampling and Analysis Plan/Quality Assurance Project Plan:
Sediment Porewater Study of Kootenai River Tributaries
Libby Asbestos Superfund Site
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TABLES

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TABLE B-1. SAMPLE LOCATION DESCRIPTIONS

Water Body	Location	Description	Sediment Pore Water	Surface Water	Sediment
Granite Creek	1	Near Granite Creek/Cherry Creek junction	X		
	2	Prior to Libby Creek confluence	X		
Libby Creek	3	Approximately 12 miles south of Libby, US Highway 2 bridge over Libby Creek	X		
	4	Immediately prior to Kootenai River confluence	X		
Flower Creek	5	Outlet of Flower Creek Reservoir (Libby water source)	X		
	6	Near bridge on W. Balsam St.	X		
	7	Immediately prior to Kootenai River confluence	X		
Pipe Creek	8	Immediately prior to Kootenai River confluence	X		
	9	Upstream of Kootenai River Rd.	X		
	10	Approximately 6 miles north on Pipe Creek Rd. North of Red Dog Saloon	X		
Fisher River	11	Immediately prior to Kootenai River confluence	X		
	27	Upstream of Location 11	X	X	X[a]
Callahan Creek	12	Upstream of last residential/commercial property	X		
	13	Northeast of US Highway 2 bridge over creek	X		
	14	Immediately prior to Kootenai River confluence	X		
O'Brien Creek	28	Immediately prior to Kootenai River confluence	X	X	X[a]
	29	Upstream of Location 28	X	X	X[a]

[a] = 5 point composite sample

New location (shaded)

TABLE D-1. GENERAL EVALUATION METHODS FOR ASSESSING ASBESTOS DATA USABILITY

Data Usability Indicator	General Evaluation Method
Precision	Sampling – Review results for co-located samples and field duplicates to provide information on variability arising from medium spatial heterogeneity and sampling and analysis methods. Soil Preparation – Review results for preparation duplicates to provide information on variability arising from sample preparation and analysis methods. Analysis – Review results for PLM laboratory duplicates, TEM recounts, and TEM repreparations to provide information on variability arising from analysis methods. Review results for inter-laboratory analyses to provide information on variability and potential bias between laboratories.
Accuracy/Bias	TEM – Calculate the background filter loading rate and use results to assign detect/non-detect in basic accordance with ASTM 6620-00. For air samples, determine the frequency of indirect preparation. PLM – Review results for LA-specific performance evaluation standards to provide information on direction/magnitude of potential bias. Review results for blanks to provide information on potential contamination.
Representativeness	Review relevant field audit report findings and any field/laboratory ROMs for potential data quality issues.
Comparability	Compare the sample collection SOPs, preparation techniques, and analysis methods to previous investigations.
Completeness	Determine the percent of samples that were able to be successfully collected and analyzed in accordance with the investigation-specific SAP requirements (e.g., 99 of 100 samples, 99%).
Sensitivity	TEM – Determine the fraction of all analyses that stopped based on the area examined stopping rule (i.e., did not achieve the target sensitivity).

ASTM = American Society of Testing and Materials
LA = Libby amphibole
PLM = polarized light microscopy
QATS = Quality Assurance Technical Support
ROM = record of modification
SAP = sampling and analysis plan
SOP = standard operating procedure
TEM = transmission electron microscopy

Sampling and Analysis Plan/Quality Assurance Project Plan:
Sediment Porewater Study of Kootenai River Tributaries
Libby Asbestos Superfund Site
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Appendix A
Data Quality Objectives (DQOs)

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APPENDIX A

DATA QUALITY OBJECTIVES FOR THE SEDIMENT POREWATER STUDY OF KOOTENAI RIVER TRIBUTARIES

Data quality objectives (DQOs) are statements that define the type, quality, quantity, purpose, and use of data to be collected. The design of a study is closely tied to the 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 types of analyses to be performed. EPA has developed a seven-step process for establishing DQOs to help ensure that data collected during a field sampling program will be adequate to support reliable site-specific risk management decision-making (EPA 2001, 2006).

The following sections implement the seven-step DQO process associated with this study.

Step 1: State the Problem

Libby is a community in northwestern Montana located 7 miles southwest of a vermiculite mine that operated from the 1920s until 1990. The mine began limited operations in the 1920s and was operated on a larger scale by the W.R. Grace Company from approximately 1963 to 1990. Studies revealed that the vermiculite from the mine contains amphibole-type asbestos, referred to as Libby amphibole (LA). Previous investigations conducted at the Site have demonstrated that LA is present in environmental source media (e.g., surface water, sediments) at locations in and around the Site. In particular, a recent sampling effort determined the presence of LA in surface water and/or sediment of the Kootenai River and several of its tributaries (EPA 2013a).

In 2012, contractors for W.R. Grace & Co.-Conn. and the Kootenai Development Corporation (KDC) performed an in-stream caged fish (eyed egg and alevin) study to provide information on potential effects of exposures in lower Rainy Creek (LRC) to trout (EPA 2012a). Results from this study suggested that there might be a decrease in overall survival of trout in LRC, but these results were considered to have low reliability due to the number of organisms that went missing during the study and the high mortality in the study controls (SRC, Inc. 2013). Due to these limitations, the in-stream study will be repeated in 2013, modifying the study design and protocol to address the issues that occurred during the 2012 study (EPA 2013b). The results of this study will be used to identify an LA concentration in sediment porewater that is a no-effect level (NEL) and/or an effect level (EL), depending upon the study outcome.

In order to evaluate potential exposures to fish (eyed eggs/alevins) in the Kootenai River tributaries, measured porewater data are needed for comparison to the NEL and/or EL (to be identified from the results of the 2013 in-stream eyed-egg study in LRC).

Step 2: Identify the Goal of the Study

The goal of this study is to collect sediment porewater samples from locations in tributaries to the Kootenai River and analyze these samples for LA. The measured porewater LA concentrations will be compared to the NEL and/or the EL levels to determine the potential for LA effects on fish in an ecological risk assessment. The measured porewater LA concentrations will also be used to characterize the nature and extent of LA in porewater.

Step 3: Identify Information Inputs

The information needed to meet the study goals identified above consists of reliable and representative measurements of LA in sediment porewater from Kootenai River tributaries. The following sections discuss the type of samples that should be collected and the analytical methods that should be used to analyze these samples.

Type of Samples

In order to ensure comparability of the porewater results with the NEL and/or EL from the in-stream eyed-egg test, the porewater samples will be collected using the same collection procedures used in the 2013 in-stream eyed egg study in LRC (EPA 2013b). In brief, sediment porewater samples should be collected using Whitlock-Vibert boxes (WVBs) that have been modified to accommodate a sampling port to allow for the collection of water from within the WVB chamber.

Analysis Method

All collected porewater samples should be analyzed for asbestos using transmission electron microscopy (TEM). EPA generally recommends using TEM because this technique has the ability to clearly distinguish asbestos from non-asbestos structures and to classify different types of asbestos (i.e., LA, chrysotile). Also, the use of TEM will allow comparability of results with those from prior water sampling efforts, as well as the 2013 in-stream eyed egg study. Prior to analysis, water samples should be treated with ozone/ultraviolet (UV) to remove any organic materials that would cause LA to clump and bind to the container walls and ensure that concentrations are not biased low. In addition, because it is possible that there could be various sources of LA present, information on the sodium and potassium content of each LA structure observed, as determined by energy dispersive spectroscopy (EDS), should also be recorded. This requirement is based on the observation of Meeker *et al.* (2003) that most particles from the Libby ore body contain detectable levels of both sodium and potassium, whereas other potential sources of LA may not.

Step 4: Define the Bounds of the Study

Spatial Bounds

Porewater samples should be collected from the same Kootenai River tributary creeks sampled as in 2012 as part of the *Nature and Extent Study in Surface Water and Sediment* (EPA 2012b; 2013a), with the addition of three sampling locations (two in O'Brien Creek and one in Fisher Creek) (see **Figures B-1 and B-2** in the main text). Sampling locations were added based on consultation with Montana Fish, Wildlife, and Parks (FWP) and the U.S. Fish and Wildlife Service (USFWS). Locations were added in O'Brien Creek because this tributary is critical habitat for the bull trout. Although Fisher River was sampled in 2012, an additional sampling location was added further upstream for this study because the headwaters were thought to be more representative of potential trout spawning habitat. Montana FWP and USFWS provided consultation concerning the location of the sampling locations for this study relative to critical habitat and provided recommendations concerning timing and placement of the WVBs to minimize impacts to bull trout.

At each sampling location, the area selected for collection of porewater samples should (ideally) be or approximate a natural redd (gravel/cobble area that fish are using or could use for spawning). If no redds are found, then a synthetic red should be constructed with natural (or commercial) spawning gravel.

Temporal Bounds

Sediment porewater data collected from LRC suggest that LA concentrations tend to be higher during high flow conditions (see **Figure B-3** in the main text). Sediment porewater samples will be collected during the peak or falling limb of the hydrograph, which is expected to occur about mid-May (see **Figure B-4** in the main text). If field conditions (stream water flow/depth) are not safe for sampling then samples should be collected as soon as they are deemed safe. Because some sampling locations are critical habitat for the bull trout, sampling should not occur during times of spawning (September) or egg incubation (October to April).

Step 5: Define the Analytical Approach

The porewater results from this study will be used to evaluate LA exposures and risks for freshwater fish. The measured LA concentrations in porewater will be compared to the NEL and/or the EL levels (to be identified from the results of the 2013 in-stream eyed egg study) to determine the potential for LA effects on fish in an ecological risk assessment. The data interpretation will depend upon the outcome of the 2013 in-stream eyed egg study.

- If the eyed egg study yields an NEL, and if sediment porewater concentrations of LA in tributaries are equal to or lower than the NEL, this would support the conclusion that no adverse effects are expected for fish in these tributaries.
- If the eyed egg study yields an NEL, and if sediment porewater concentrations of LA in tributaries are higher than the NEL, additional information would be needed on the potential EL.
- If the eyed egg study yields an EL, and if sediment porewater concentrations of LA in tributaries are equal to or higher than the EL, this would support the conclusion that there is the potential for adverse effects in fish in these tributaries.
- If the eyed egg study yields an EL, and if sediment porewater concentrations of LA in tributaries are below the EL but above the NEL, potential for adverse effects in fish in these tributaries would depend upon the proximity of the measured porewater concentrations to the NEL.

These comparisons would be used as part of the weight of evidence for evaluating potential risks to fish in the ecological risk assessment. In addition, the measured porewater LA concentrations will be used to characterize the nature and extent of LA in porewater at the Site.

Step 6: Specify Performance Criteria

In making decisions about the risks to ecological receptors from LA in sediment porewater, two types of decision errors are possible:

- A *false negative decision error* would occur if a risk manager decides that exposure to LA is not of concern, when in fact it is of concern.
- A *false positive decision error* would occur if a risk manager decides that exposure to LA is above a level of concern, when in fact it is not.

The EPA is most concerned about guarding against the occurrence of false negative decision errors, since an error of this type may leave receptors exposed to unacceptable levels of LA. To minimize chances of underestimating the true amount of exposure and risk, the EPA generally recommends that risk calculations be based on the 95 percent (%) upper confidence limit (95UCL) of the sample mean (EPA 1992). Use of the 95UCL in risk calculations limits the probability of a false negative decision error to no more than 5%. To support this approach, the EPA has developed a software application (ProUCL) to assist with the calculation of 95UCL values (EPA 2010). However, equations and functions in ProUCL are not designed for asbestos datasets and application of ProUCL to asbestos datasets is not recommended (EPA 2008). The EPA is presently working to develop a new software application that will be appropriate for use with asbestos datasets, but the application is not yet available for use. Because the 95UCL cannot presently be calculated with confidence, risk calculations will be based on the sample mean only, as recommended by EPA (2008). This means that risk estimates may be either higher or lower than true values, and this will be identified as a source of uncertainty in the risk assessment.

The EPA is also concerned with the probability of making false positive decision errors. Although this type of decision error does not result in unacceptable exposures, it may result in unnecessary expenditure of resources. The risk of false positive decision errors can be minimized by increasing the number of samples. The number of samples needed depends on the magnitude of between-sample variability and the proximity of exposure concentrations to the decision threshold (NEL and/or EL). If between-sample variability is low, or if the exposure concentration is not near a decision threshold, then the number of samples needed is relatively low. However, if between-sample variability is high and the exposure concentration is relatively near a decision threshold, then the number of samples needed is usually higher. Because it is not possible at present to quantify the uncertainty in the mean of an asbestos dataset as a function of the number of samples, it is not possible to calculate a minimum number of samples required to minimize the risk of false positive decision errors.

Step 7: Develop the Plan for Obtaining Data

A detailed study design for the collection of porewater in Kootenai River tributaries is provided in Section B1 of this SAP/QAPP. Key features of this study design are discussed below.

Sample Locations

Sampling locations for sediment porewater collection will be the same tributaries as those sampled for the *Nature and Extent Study in Surface Water and Sediment* (EPA 2012b; 2013a), with the addition of three locations (two in O'Brien Creek and one in the Fisher River) (see **Figures B-1 and B-2** in the main text).

Sampling Method

To ensure comparability with porewater measurements that will be made during the 2013 in-stream eyed egg study, all porewater samples will be collected using a modified WVB. In brief, at each sampling location, a WVB will be placed into a metal cage that has been filled with cobble and buried into the sediment in an area of a natural or synthetic redd. The WVB will be modified such that a porewater sampling port extends from the inside of the WVB chamber to above the water surface. A metal bailer will be used to sample water from within the WVB chamber. This is the same method that will be used to sample porewater during the 2013 in-stream eyed egg study in LRC (EPA 2013b).

WVB Placement

At each sampling location, the area selected for WVB deployment and collection of porewater samples should (ideally) be or approximate a natural redd (gravel/cobble area that fish are using or could use for spawning). If no redds are found then the bottom of the area excavated for deployment of the WVB will be layered with natural (or commercial) spawning gravel on which the deployed cages will rest.

Sampling Schedule

Porewater sampling will be conducted in mid-May to capture high flow conditions and anticipated corresponding higher LA concentrations. If conditions are not safe for WVB installation, then sampling will be conducted as soon as they are deemed safe. Sampling will not occur during bull trout spawning (September) or egg incubation (October to April). Porewater samples will be collected from the installed WVBs approximately 1 to 2 days after installation (to allow conditions inside the WVBs to equilibrate). One porewater sample will be collected from each sampling location.

Analytical Requirements for Porewater Samples

In general, three alternative stopping rules are specified for TEM analyses to ensure resulting data are adequate:

1. The target analytical sensitivity (TAS) to be achieved
2. A maximum number of structures to be counted
3. A maximum area of filter to be examined

The basis for each of these values for this study is presented below.

Target Analytical Sensitivity

For the purposes of planning this sampling effort, the analytical requirements for LA measurements were derived such that concentrations of LA in porewater will be reliably detected and quantified if present at levels of 0.15 MFL (150,000 f/L). This target sensitivity is consistent with past surface water sampling events (EPA 2012a, 2012b), and is equivalent to the analytical requirements for the 2013 in-stream eyed egg study (EPA 2013b). The TAS was determined by dividing the target concentration by the target number of structures to be observed during the analysis of a sample with a true concentration equal to the target concentration:

TAS = Target Conc / Target Count

The target count is determined by specifying a minimum detection frequency required during the analysis of samples at the target concentration. This probability of detection is given by:

Probability of detection = 1 – Poisson (0,Target Count)

Assuming a minimum detection frequency of 95 percent, the target count is 3 fibers. Based on this, the TAS is:

$$TAS = (150{,}000\ f/L)\ /\ (3\ fibers) = 50{,}000\ L^{-1}$$

In the event that a lower TAS is needed, additional grid openings may be analyzed in the future.

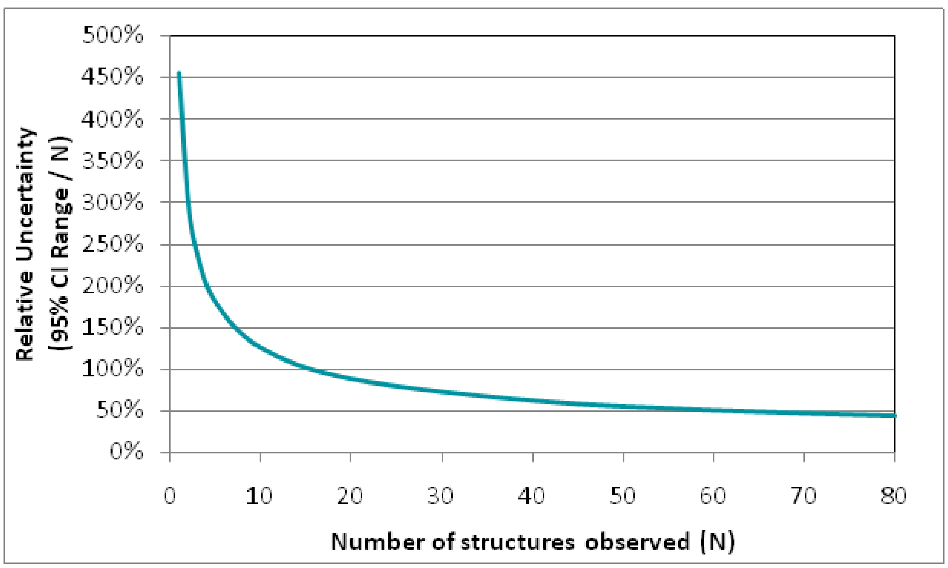
Maximum Number of LA Structures

Ideally, all samples would be examined by TEM until the TAS is achieved. However, for filters that have high asbestos loading, reliable estimates of concentration may be achieved before achieving the TAS. This is because the uncertainty around a TEM estimate of asbestos concentration in a sample is a function of the number of structures observed during the analysis. The 95% confidence interval (CI) around a count of N structures is computed as follows:

$$\text{Lower bound (2.5\%)} = \tfrac{1}{2} \cdot \text{CHIINV}(0.975, 2 \cdot N_{observed} + 1)$$
$$\text{Upper bound (97.5\%)} = \tfrac{1}{2} \cdot \text{CHIINV}(0.025, 2 \cdot N_{observed} + 1)$$

As N_{obs} increases, the absolute width of the CI range increases, but the relative uncertainty (expressed as the CI range divided by N_{obs}) decreases. This concept is illustrated in the figure below.

RELATIONSHIP BETWEEN THE NUMBER OF STRUCTURES OBSERVED AND RELATIVE UNCERTAINTY



CI = confidence interval

The goal is to specify a target N such that the resulting Poisson variability is not a substantial factor in the evaluation of method precision. As shown in the figure, above about 25 structures, there is little change in the relative uncertainty. Therefore, the count-based stopping rule for TEM should utilize a maximum structure count of 25 structures.

Maximum Area to be Examined

The number of grid openings (GOx) that must be examined by TEM to achieve the TAS is calculated as:

$$GOx = EFA / (TAS \cdot Ago \cdot V)$$

where:

GOx = Number of grid openings
EFA = Effective filter area (assumed to be 1295 mm^2)
TAS = Target analytical sensitivity $(L)^{-1}$
Ago = Grid opening area (assumed to be 0.01 mm^2)
V = Water volume applied to the filter (L)

Assuming that 0.1 L of water is able to be applied to the filter, a total of 26 grid openings would need to be examined to achieve the TAS. In the event that less water is able to be applied to the filter (due to water turbidity), the number of grid openings that would need to be examined would increase. In order to limit the level of effort (and cost) for any one analysis, the maximum number of grid openings to be examined for this project is 100 grid openings. Assuming that each grid opening has an area of about 0.01 mm^2, this would correspond to a maximum area examined of about 1.0 mm^2.

Summary of TEM Stopping Rules for Porewater Samples

The TEM stopping rules for all porewater samples from this investigation should be as follows:

1. Examine a minimum of two grid openings from each of two grids.
2. Continue examining grid openings until one of the following is achieved:
    a. The target analytical sensitivity of 50,000 L^{-1} has been achieved.
    b. 25 LA structures have been observed.
    c. A total filter area of 1.0 mm^2 has been examined (this is approximately 100 grid openings).

When one of these criteria has been satisfied, complete the examination of the final grid opening and stop.

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Sampling and Analysis Plan/Quality Assurance Project Plan:
Sediment Porewater Study of Kootenai River Tributaries
Libby Asbestos Superfund Site
Revision 0 – May 2013

Appendix B
Libby-Specific Standard Operating Procedures (SOPs)

SOP ID	SOP Description
Field Procedures	
CDM-LIBBY-09	GPS Coordinate Collection and Handling
EPA-LIBBY-2012-01	Field Logbook Content and Control
EPA-LIBBY-2012-04	Field Equipment Decontamination
EPA-LIBBY-2012-05	Handling Investigation-Derived Waste
EPA-LIBBY-2012-06	Sample Custody
EPA-LIBBY-2012-07	Packaging and Shipping of Environmental Samples
EPA-LIBBY-2012-08	Surface Water Sampling
EPA-LIBBY-2012-09	Sediment Sampling
EPA-LIBBY-2013-01	Sediment Porewater Sampling Using Modified WVBs
Laboratory Procedures	
ISSI-LIBBY-01	Soil Sample Preparation
SRC-LIBBY-01	Qualitative Estimation of Asbestos in Coarse Soil by Visual Examination Using Stereomicroscopy and PLM
SRC-LIBBY-03	Analysis of Asbestos Fibers in Fine Soil by PLM
Data Verification Procedures	
EPA-LIBBY-09	SOP for TEM Data Review and Data Entry Verification
EPA-LIBBY-10	SOP for PLM Data Review and Data Entry Verification
EPA-LIBBY-11	SOP for FSDS Data Review and Data Entry Verification

The most recent versions of all field SOPs are provided electronically in the Libby Field eRoom (https://team.cdm.com/eRoom/R8-RAC/Libby).
The most recent version of all laboratory and data verification SOPs are provided electronically in the Libby Lab eRoom (https://team.cdm.com/eRoom/mt/LibbyLab).

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Sampling and Analysis Plan/Quality Assurance Project Plan: Sediment Porewater Study of Kootenai River Tributaries Libby Asbestos Superfund Site

Revision 0 – May 2013

Appendix C
Analytical Requirements Summary Sheet
[PWTBOU4-0513]

The most recent version of the Analytical Requirements Summary Sheet is provided electronically in the Libby Lab eRoom (https://team.cdm.com/eRoom/mt/LibbyLab).

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SAP ANALYTICAL SUMMARY # PWTBOU4-0513
SUMMARY OF PREPARATION AND ANALYTICAL REQUIREMENTS

SAP Title: Sampling and Analysis Plan/Quality Assurance Project Plan: Sediment Porewater Study of Kootenai River Tributaries, Libby Asbestos Superfund Site, Operable Unit 4, Libby, Montana

SAP Date/Revision: May 2013 (Rev. 0)

EPA Technical Advisor: Elizabeth Fagen (303-312-6095, fagen.elizabeth@epa.gov); Dan Wall (303-312-6560, wall.dan@epa.gov)
(contact to advise on DQOs of SAP related to preparation/analytical requirements)

Sampling Program Overview: The purpose of this SAP/QAPP is to collect sediment porewater from tributaries to the Kootenai River. A small number of surface water and sediment samples will also be collected from new stations not previously evaluated as part of the 2012 Nature & Extent study.

Estimated number and timing of field samples:

>> All samples will be collected in late-May to early June (actual collection dates will depend upon site conditions)
>> Sediment porewater = 17 samples + field QC samples
>> Surface water = 3 samples + field QC samples
>> Sediment = 3 samples + field QC and preparation QC samples

Index ID Prefix: PT-xxxxx

TEM Preparation and Analytical Requirements for Water Samples:

Medium Code	Medium	Preparation Details [a]				Analysis Details			Applicable Laboratory Modifications (current version of)
		Investi-gative?	Indirect Prep? With Ashing	Indirect Prep? Without Ashing	Filter Archive?	Method	Recording Rules	Analytical Sensitivity/ Stopping Rules	
A	Water	Yes	No	No	Yes	Standard TEM; ISO 10312	All asbestos [b]; L: ≥ 0.5 µm AR: ≥ 3:1	Count a minimum of 2 grid openings in 2 grids, then continue counting until one is achieved: i) sensitivity of 50,000 L^{-1} is achieved ii) 25 structures are recorded iii) A total filter area of 1.0 mm^2 has been examined (approx. 100 grid openings)	LB-000016, LB-000029, LB-000066, LB-000067, LB-000085

[a] Sample and filter preparation should be performed in basic accordance with EPA Method 100.2 (as modified by LB-000020B). Grid preparation should be performed in basic accordance with Section 9.3 of ISO 10312:1995(E).
[b] If observed, chrysotile structures should be recorded, but chrysotile structure counting may stop after 25 structures have been recorded.

PLM Preparation and Analytical Requirements for Sediment Samples:

Medium Code	Medium	Preparation Method[c]	Analysis Method	Applicable Laboratory Modifications (current version of)
B	Sediment	ISSI-LIBBY-01 Rev. 11	PLM-Grav: SRC-LIBBY-01 Rev. 3 PLM-VE: SRC-LIBBY-03 Rev. 3	LB-000073

[c] Sample preparation to be performed at the Troy sample preparation facility and shipped to the PLM analytical laboratory.

Laboratory Quality Control Sample Frequencies:

TEM [d]: Lab Blank – 4%
Recount Same – 1%
Verified Analysis – 1%
Repreparation – 1%
Recount Different – 2.5%
Inter-laboratory – 0.5% [f]

[d] See LB-000029 for selection procedure and QC acceptance criteria.
[e] *Post hoc* selection to be performed by the QATS contractor.

PLM [f]: Lab Duplicates – 10% (cross-check 8%; self-check 2%)
Inter-laboratory – 1% [g]

[f] See LB-000073 for selection procedure and QC acceptance criteria.
[g] *Post hoc* selection to be performed by the QATS contractor.

Asbestos Analytical Laboratory Review Sign-off:

- ☐ EMSL – Libby [sign & date:____________________]
- ☐ EMSL – Cinnaminson [sign & date:____________________]
- ☐ EMSL – Beltsville [sign & date:____________________]
- ☐ EMSL – Denver [sign & date:____________________]
- ☐ ESAT [sign & date: ____________________]
- ☐ Hygeia [sign & date:____________________]
- ☐ RESI [sign & date:____________________]

[Checking the box and signing (electronically) above indicates that the laboratory has reviewed and acknowledged the preparation and analytical requirements associated with the specified SAP.]

Requirements Revision:

Revision #:	Effective Date:	Revision Description
0	5/8/13	--