

**General Property Investigation
Quality Assurance Project Plan
Libby Asbestos Site, Operable Units 4 and 7
Libby, Montana**

Revision 4

04/10/2014

Project Period 03/30/2014 to 03/29/2015

Contract No. W9128F-11-D-0023

Task Order No. 0007

Prepared for:



**ENVIRONMENTAL PROTECTION AGENCY
Region VIII**

Prepared under Libby Asbestos Interagency Agreement, Libby, MT (DW96954027) by:



U.S. Army Corps of Engineers
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With technical assistance from:

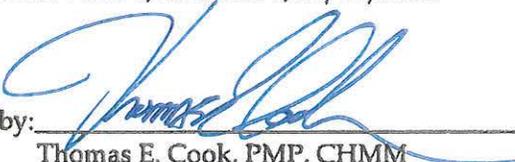


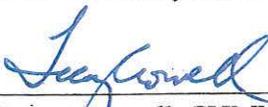
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A. Project Management

A1. Title and Approval Sheet

Title: General Property Investigation Quality Assurance Project Plan, Libby Asbestos Site, Operable Units 4 and 7, Revision 4, 04/10/2014

Reviewed by:  Date: 4/11/14
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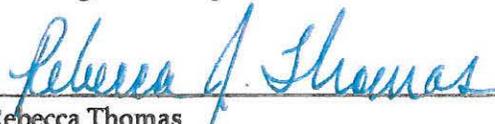
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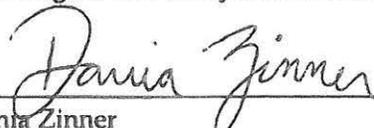
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Document Revision Log

Revision No.	Date	Description
0	04/23/2010	---
1	04/16/2012	<ul style="list-style-type: none"> ▪ Made editorial changes, corrected typographical errors ▪ Sample ID prefix change from 2S- and 2D- to 3G- ▪ Bulk samples will no longer be collected or analyzed ▪ Screening Investigation no longer requires a land survey
2	04/15/2013	<ul style="list-style-type: none"> ▪ Made editorial changes, corrected typographical errors ▪ Changed document format and included EPA Region 8 Quality Assurance Document Review Crosswalk ▪ Updated distribution list ▪ Added section for project/task organization ▪ Added project organizational chart ▪ Added Troy sample preparation facility procedures ▪ Updated data validation and usability section ▪ Updated references ▪ Eliminated Exterior Property Information Form ▪ Sample ID prefix change from 3G- to 4G- ▪ Areas with vermiculite may be sampled at DI at the direction of USACE or the EPA ▪ Incorporated technical details/processes from the 3/8/13 Flowerpot Action Level Memorandum
3	08/22/2013	<ul style="list-style-type: none"> ▪ Addressed the EPA QA Reviewer comments (refer to GPI QAPP Rev3 document crosswalk table) ▪ Document is addressed as a QAPP and no longer referred to as a SAP/QAPP ▪ Updated references ▪ Added Libby project process flow chart
4	04/07/2014	<ul style="list-style-type: none"> ▪ Soil samples will be collected from areas containing vermiculite ▪ Visual inspection will no longer be conducted at a greater frequency than sample aliquot collection for SUAs at screening investigations ▪ Maximum sample areas were established for SUAs at screening investigations ▪ Added bulk material sampling ▪ Clarified laboratory QC analysis requirements

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Acronyms and Abbreviations

%	percent
A&E	architect and engineering contractor
ACM	Asbestos containing material
AHERA	Asbestos Hazard Emergency Response Act
APP	Accident Prevention Plan
AS	analytical sensitivity
ASTM	American Society for Testing and Materials
CAR	corrective action request
CB&I	CB&I Federal Services
CDM Smith	CDM Federal Programs Corporation
COC	chain-of-custody
CFR	Code of Federal Regulations
CSS	Contaminant Screening Study
CUA	common-use area
DEQ	Montana Department of Environmental Quality
DI	detailed investigation
DQOs	data quality objectives
EDD	electronic data deliverable
EDS	energy dispersive spectroscopy
EPA	U.S. Environmental Protection Agency
ERT	EPA Environmental Response Team
ESAT	EPA Environmental Services Assistance Team
f/cc	fibers per cubic centimeter
FPM	field planning meeting
FSDS	field sample data sheet
FTL	Field Team Leader
GIS	geographic information system
GPI	general property investigation
GPS	global positioning system
H&S	health and safety
HAZWOPER	Hazardous Waste Operations and Emergency Response
HVAC	heating, ventilation, and air conditioning
ID	identifier
IDW	investigation-derived waste
IFF	Information Field Form
IPIF	Interior Property Inspection Form
LA	Libby Amphibole asbestos
LADT	Libby Asbestos Data Tool
LC	Laboratory Coordinator
LUA	limited-use area
N	number
NA	not applicable
ND	none detected
NFG	National Functional Guidelines
NIST	National Institute of Standards and Technology

NUA	non-use area
NVLAP	National Voluntary Laboratory Accreditation Program
OIF	Occupant Information Form
OSHA	Occupational Safety and Health Administration
OU	operable unit
PE	performance evaluation
PLM	polarized light microscopy
PLM-Grav	polarized light microscopy - gravimetric
PLM-VE	polarized light microscopy - visual area estimation
POC	Property Operations Coordinator
PPE	personal protective equipment
PSEF	Property Status Evaluation Form
QA	quality assurance
QAM	Quality Assurance Manager
QAPP	quality assurance project plan
QATS	Quality Assurance Technical Support
QC	quality control
RAFU	reasonably anticipated future use
ROM	Record of Modification
RPM	Remedial Project Manager
s/cm ²	structures per square centimeter
SI	screening investigation
Site	Libby Asbestos Superfund Site
SOP	standard operating procedure
SPF	sample preparation facility
SUA	specific-use area
TEM	transmission electron microscopy
TL	Task Leader
TR	Trace (<0.2% by PLM-VE)
USACE	U.S. Army Corps of Engineers
USGS	U.S. Geological Survey
VRP	Voluntary Recruitment Program
Weston	Weston Solutions, Inc.

A3. Distribution List

Copies of this completed and signed quality assurance project plan (QAPP) will be distributed to:

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Libby, Montana 59923

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- Kara McKenzie, mckenzieKE@cdmsmith.com (15 hard copies, electronic copy)
- Terry Crowell, crowellTL@cdmsmith.com (1 hard copy, electronic copy)

Copies of the QAPP will be distributed to the individuals above by the architect and engineering contractor (A&E) (CDM Federal Programs Corporation [CDM Smith]), either in hard copy or in electronic format (as indicated above). The A&E's Project Manager (or their designee) will distribute updated copies each time a QAPP revision occurs. An electronic copy of the final, signed QAPP (and subsequent revisions) will also be posted to the Libby Field eRoom.

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 Remedial Project Manager (RPM) for general property investigations (GPIs) is Elizabeth Fagen. The EPA Region 8 Onsite RPM and Field Team Leader (FTL) for GPIs is Mike Cirian.

The U.S. Army Corps of Engineers (USACE), Omaha District, is the contracting agency for GPI activities at the Site, on behalf of the EPA. The USACE has an interagency agreement number with the EPA, number DW96954027, through which the USACE GPI work will be performed. GPI activities will be performed by the A&E (CDM Smith) under contract to the USACE for Architect-Engineering and Surveying Services (Contract Number W9128F-11-D-0023, Task Order 0007) for ongoing removal action support to the EPA Region 8. USACE's Project Manager and Contracting Officer Representative (COR) is Mary Darling.

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

A4.2 Technical Support

A4.2.1 QAPP Development

This QAPP was developed by the A&E at the direction of, and with oversight by, USACE and the EPA. This QAPP contains the required QAPP elements and has been developed in general accordance with the *EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5* (EPA

2001), *EPA Guidance for Quality Assurance Project Plans*, EPA QA/G-5 (EPA 2002), and the *Guidance on Systematic Planning Using the Data Quality Objectives Process*, EPA QA/G4 (EPA 2006).

As required by the EPA, Revision 2 of this QAPP (CDM Smith 2013a) was reviewed by Patti Tyler, EPA Human Subjects Research Officer. That review determined that this project does not meet the regulatory definition of research involving human subjects and therefore is not subject to the Common Rule or to the additional requirements in EPA Regulation 40 CFR 26 (protection of Human Subjects). Collection of data with respect to human subjects has not changed in subsequent revisions of this document. The memorandum documenting this determination is provided in **Appendix E**.

Copies of the QAPP will be distributed by the A&E's Project Manager (or their designee), either in hard copy or in electronic format, as indicated in Section A3. The A&E's Project Manager (or their designee) will distribute updated copies each time a QAPP revision occurs. An electronic copy of the final, signed QAPP (and subsequent revisions) will also be posted to the Libby Field eRoom.

A4.2.2 Field Sampling Activities

The A&E will be responsible for conducting all field investigation activities described in this QAPP. Key A&E personnel that will be involved in this investigation program include:

- Thomas Cook, Project Manager
- Scott Felton, Project Engineer
- Kara McKenzie, Task Leader
- Simon Wilson, Field Team Leader
- Tracy Dodge, Sample Coordinator
- Scott Miller, Field Data Manager
- Karen Repine, Property Operations Coordinator
- Terry Crowell, Quality Assurance Manager
- Damon Repine, Health and Safety (H&S) Manager

A4.2.3 Asbestos Analysis

All samples 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 sample preparation facility (SPF) and analytical laboratory services and providing direction to the entities providing these

services. 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

The project data management processes and reporting requirements, and related contractor responsibilities, are described in the *EPA Data Management Plan for the Libby Asbestos Superfund Site* (EPA 2013). This document is managed by the EPA Data Manager and can be found on the EPA Libby document website (<http://www2.epa.gov/region8/libby-site-documents>).

All sample and location data generated as part of GPIs 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.

CDM Smith's Field Data Manager, Scott Miller, is responsible for overseeing the upload of sample and location 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.).

In addition to sample and location data, GPI property information (e.g., addresses, property identifiers [IDs], geounit IDs, contacts, access and property statuses) will be managed in EPA's Response Manager database. Weston Solutions, Inc. (Weston) is responsible for administering Response Manager, and Brad Morgan is Weston's Response Manager Administrator.

Limited property coordination (i.e., solicitation attempts to contact property owners for GPI participation) and GPI/design process tracking information will be maintained in the project Property Operations Tracking System. This system is integrated with Response Manager for key property and access data, and is administered by Scott Miller (CDM Smith).

Because of the quantity and complexity of the data collected at the Site, the EPA has designated a Libby Data Manager to manage and oversee the various data support contractors. The EPA Region 8 Data Manager for the Libby project is Jeff Mosal.

A4.3 Quality Assurance

There is no individual designated as the EPA Quality Assurance Manager (QAM) for the Libby project. Rather, the Region 8 QA program has delegated authority to the EPA RPMs. This means that the EPA RPMs have the ability to review and approve governing documents

developed by Site contractors. Thus, it is the responsibility of the EPA RPM or their designee for this sampling effort, Dania Zinner, to ensure that this QAPP has been prepared in accordance with the EPA QA guidelines and requirements. Ms. Zinner is independent of the entities planning and obtaining the data described in this QAPP.

For this project, the EPA is supported by the Quality Assurance Technical Support (QATS) contractor, CB&I Federal Services (CB&I). The QATS contractor will evaluate and monitor laboratory QA and quality control (QC) activities and is responsible for performing annual audits of each analytical laboratory. CB&I's QAM for this project is Michael Lenkauskas.

As the project lead on behalf of the EPA, USACE is responsible for overall QA of this investigation program. This includes involvement of USACE QA management and staff, which includes senior-level members that perform duties as QA representatives for the project. These QA representatives are independent of the USACE project team that manage and execute the work (including data collection and use). They are responsible for assuring work is performed in conformance with the QA program and project-specific requirements. The QAM monitors quality through the assigned onsite personnel listed below. If significant issues are encountered, the QAM has stop work authority via the USACE COR, Mary Darling. It is anticipated that David Ray will serve as the USACE QAM for GPIs; however, other staff may ultimately be identified to fill this role. The USACE will notify the EPA of changes in project QA staff.

USACE rotates several personnel to Libby to maintain an onsite presence. Collectively, the onsite personnel are responsible for oversight, coordination of project work/scope objectives, review of property-specific GPI sketches, and contract administration. USACE onsite personnel report to the USACE Project Manager. The following onsite USACE personnel will maintain QA oversight of this investigation program:

- Jeremy Ayala, Project Engineer
- Jeff Hubbard, Backup Project Engineer
- Brian Broekemeier, Onsite QAM and Construction Control Representative

CDM Smith's QA Director, Jo Nell Mullins, implements the CDM Smith QA program. She is independent of project technical staff and reports directly to Gwen Baker, the Federal Services Group President on QA matters. The QA Director has the authority to objectively review projects and identify problems, and the authority to use corporate resources, as necessary, to resolve any quality-related problems. CDM Smith's QAM for this project, Terry Crowell, reports to Ms. Mullins on QA matters. Under Ms. Mullin's oversight, Ms. Crowell is responsible for monitoring and evaluating field quality assurance/quality control (QA/QC), providing oversight of field sampling and data collection activities, and coordinating field QA activities, including identifying qualified, independent staff to conduct assessments of field activities (see Section B5.1.4).

A5. Problem Definition/Background

A5.1 Site Background

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 W.R. Grace and Company from approximately 1963 to 1990. Studies revealed that the vermiculite from the mine contains amphibole-type asbestos, referred to as Libby Amphibole asbestos (LA).

Epidemiological studies revealed that workers at the mine had an increased risk of developing asbestos-related lung disease (McDonald *et al.* 1986, Amandus *et al.* 1987, Amandus and Wheeler 1987, Sullivan 2007; Larson *et al.* 2010, 2012a, 2012b). 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). Although the mine has ceased operations, historic or continuing releases of LA from mine-related materials could be serving as a source of on-going exposure and risk to current and future residents and workers in the area. The Site was listed on the National Priorities List in October 2002.

A5.2 Reasons for this Project

Previous investigations conducted at the Site have demonstrated that LA is present in a variety of media (e.g., soil, bulk materials, dust) from source materials (e.g., vermiculite insulation, vermiculite-containing soils, mine wastes) at properties within operable units (OU) 4 and 7. As a result, individuals may be exposed to LA that is released to air during source disturbance activities. These inhalation exposures may pose a risk of cancer and/or non-cancer effects.

The objectives of the GPI program are twofold:

1. Collect data to confirm the presence/absence of LA and/or LA source materials at residential, commercial, industrial, and public properties within OU4 and OU7, and other OUs as directed by the EPA and USACE.
2. Collect data to evaluate the extent of LA contamination at properties within OU4 and OU7 where previous investigations indicate the presence of LA and/or LA source materials.

Properties within OU4 and OU7 require GPIs to meet these goals.

A5.3 Applicable Criteria and Action Limits

At the Site, the EPA has developed action levels (also referred to as triggers in this document) and cleanup criteria for LA that are applicable to removal actions performed at residential/commercial properties. These are documented in *Libby Asbestos Site*

Residential/Commercial Cleanup Action Level and Clearance Criteria Technical Memorandum (EPA 2003) and its amendments (EPA 2011 and 2014). However, these criteria are not applicable to locations outside of the Site. In addition, final action levels for the Site will not be developed until completion of the remedial investigation/feasibility study and the publication of the record of decision (ROD). Decision rules for specific criteria or action levels that apply to this investigation program are provided in **Table A-3** of **Appendix A**. During review of GPI data, the GPI FTL, or designee, refers to and applies the action levels provided in **Appendix A**.

Personal air monitoring of sampling personnel will be performed in accordance with Occupational Safety and Health Administration (OSHA) requirements, as specified in the Site-specific Accident Prevention Plan (APP) and the *Response Action SAP* (CDM Smith 2011). In accordance with these requirements, samples will be analyzed for asbestos by phase contrast microscopy and compared to the OSHA limits for workplace exposures. The short-term (30-minute) exposure limit is 1.0 fiber per cubic centimeter of air (f/cc), and the long-term time-weighted average exposure limit is 0.1 f/cc.

A6. Project/Task Description

A6.1 Task Summary

GPI is one phase in the ongoing process of identifying and removing contamination at the Site. An overview of this process is presented in **Figure A-2**.

The process begins with the Field Data Manager running remediation status queries (RSQ) to evaluate existing data against the action levels (also referred to as triggers in this document) presented in the *Libby Asbestos Site Residential/Commercial Cleanup Action Level and Clearance Criteria Technical Memorandum* (EPA 2003) and its amendments (EPA 2011 and 2014). Action levels pertaining to flowerpots, as discussed in the flowerpot protocol memorandum (CDM Smith 2013b), will also be considered. Those queries will be used, to the extent possible, to automatically assign a property status indicating whether action is required (additional investigation or removal) at a property. As needed, the Property Operations Coordinator (POC) will conduct manual reviews of existing property data and update property statuses accordingly. The status of a property, in terms of removal actions required, will be based on the decision criteria as presented in **Table A-3** (**Appendix A**). Manual property data review and status verification/updates are documented on the Property Status Evaluation Form (PSEF)¹ completed for each property and retained with the hard copy property folder at the A&E's project office.

For properties requiring an initial screening investigation or additional investigation (where previous data indicates one or more primary triggers are present), property owners will be contacted by Voluntary Recruitment Program (VRP) staff to request permission to conduct the necessary investigation work and obtain a signed Consent for Access to Property form if necessary. Contact attempts, whether successful or unsuccessful, are documented. VRP personnel also work with property owners to schedule investigation activities at the property.

¹ The current version of the PSEF is provided in the Libby Field eRoom.

Scheduled properties are then provided to the GPI FTL to coordinate visits by GPI field teams.

The GPI process is divided into two distinct phases: screening investigation (SI) and detailed investigation (DI). SIs are intended to screen properties for the presence of LA or LA source materials, while DIs are intended to delineate contamination at properties where one or more primary trigger was previously identified. Exceptions to these general guidelines may be made at the direction of the EPA and USACE. The SI and DI phases will generally be performed at separate times.

GPIs include the following basic tasks: scheduling the property visit; reviewing previously collected property data; analyzing archived dust samples; conducting a verbal interview, visually inspecting the property for vermiculite and vermiculite source materials; collecting and analyzing soil samples for asbestos; and preparing a property sketch with visual inspection and analytical results. Each of these tasks is described in greater detail in subsequent sections of this QAPP.

Properties with a completed SI where no primary trigger is present will not progress any further in the process at this time. Properties with a completed DI where no primary trigger is present based on the more comprehensive DI data set will not progress any further in the process at this time. A final determination regarding property status will be made for these properties after the ROD is finalized. Properties where a DI has been completed, and one or more primary triggers are present based on the DI data set, will be added to the queue for design and removal.

Decisions regarding removal are guided by the *Libby Asbestos Site Residential/Commercial Cleanup Action Level and Clearance Criteria Technical Memorandum* (EPA 2003) and its amendments (EPA 2011 and 2014); flowerpot protocols (CDM Smith 2013b); *Libby Asbestos Superfund Site Response Action Work Plan* (PRI-ER 2014) and supported by the DI data gathered in accordance with this QAPP.

A6.2 Work Schedule

CDM Smith's current task order period of performance with the USACE extends from March 30, 2014 through March 29, 2015. GPIs will be conducted within this time frame as weather permits, generally from April through October. Indoor GPIs may be conducted during the off-season. However, the investigation team will need to re-visit the property at a later date to complete the outdoor portion of the investigation.

Exterior field work for the 2014 field season is scheduled for April 14 through October 24, 2014. Field activities may be conducted during the off season if urgent needs arise. GPI work in future field seasons may be conducted under this QAPP. Those schedules will be dependent upon the EPA's annual cleanup goals and will be provided in an annual update to this QAPP.

Approximately 336 SIs and 280 DIs are anticipated to be completed during the 2014 field season. Samples will be submitted for laboratory analyses immediately following each property investigation. Analyses, data receipt, and data review will be ongoing concurrent efforts

throughout the field season. The field effort for SIs and DIs takes approximately 1 and 1.5 days, respectively, to complete for each property. Analytical results are generally received within 30 to 45 business days, or as otherwise specified by the LC based on sample preparation and analytical laboratory capacity. As discussed in Section B5.1.2, QC review is conducted by the GPI FTL, or designee, once the property data is assembled. SIs and DIs are considered complete when the QC review is completed and deficiencies have been addressed. Elapsed time from field work to completed investigation activity (given a 30 to 45-day turnaround time for analytical results) is expected to be approximately twelve weeks. Investigation activities will be ongoing throughout the POP. It is expected that GPI data will collectively be reported in a revision to the OU4 and OU7 Remedial Investigation Report.

A6.3 Locations to be Evaluated

GPI property selection is described in Section B1.1.

A6.4 Resources and Time Constraints

GPI activities may be conducted year-round; however, as noted above, outdoor field work is limited by weather conditions. The number of SIs and DIs performed each year depend upon goals set by the EPA for cleanups and total investigations.

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 investigation 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 QAPP.

A7.2 Performance Criteria

The primary goal of this QAPP is to provide data for the purposes of evaluating the extent of LA contamination at each property selected for investigation at the Site. Therefore the performance criteria and analytical requirements are based on the requirements specified in the *Libby Asbestos Site Residential/Commercial Cleanup Action Level and Clearance Criteria Technical Memorandum* (EPA 2003) and its amendments (EPA 2011 and 2014). These requirements are specified as part of the DQOs (see **Appendix A**). The analytical requirements for LA measurements established in Section B4 provide that results from this study will be directly

comparable to results from historical and planned future sampling efforts.

A7.3 Precision

For dust samples, 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.

For soil samples, field duplicates for both SI and DI soil sampling activities will be collected (see Section B2.4). Analysis of these field duplicates will provide a measure of the precision of the sampling and analysis process.

A7.4 Bias and Representativeness

To the extent feasible, GPI samples will be collected and analyzed in accordance with the procedures set forth in this QAPP, which are consistent with the previous versions of this document. This will ensure that GPI results are representative and appropriate for comparison to other GPI data sets.

A7.5 Completeness

Target completeness for this project is 100%. That is, 100% of samples collected are expected to be analyzed. If any samples are not analyzed, or if LA analysis is not completed successfully, this could result in incomplete property characterization. In this event, additional sampling may be needed to support EPA decision-making.

A7.6 Comparability

The data generated during this study will be obtained using standard or project-specific analytical methods for LA that have been utilized previously in other studies, and will yield data that are comparable to previous analyses of LA in GPI soil, dust, and bulk samples.

A7.7 Method Sensitivity

The method sensitivity (analytical sensitivity) needed for LA analysis of each medium 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 field work, new GPI field team member must complete the following, at a minimum:

Training Requirement	Documentation Specifying Training Requirement Completion
Read and understand the governing APP (CDM Smith 2013c)	APP signature sheet
Attend an orientation session with the field H&S Manager	Orientation session attendance sheet
Complete OSHA 40-Hour Hazardous Waste Operations and Emergency Response (HAZWOPER) and relevant 8-hour refreshers	OSHA training certificates
Hold current 40-hour HAZWOPER medical clearance	Physician letter in the field personnel files
Complete respiratory protection training, as required by 29 Code of Federal Regulations (CFR) 1910.134	Training certificate
Complete asbestos awareness training, as required by 29 CFR 1910.1001	Training certificate

H&S-related training documentation will be stored in the A&E’s Libby project office. It is the responsibility of the field H&S Manager to keep H&S-related training documentation 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
- QA/QC requirements
- H&S requirements

It is the responsibility of each field team member to review and understand applicable governing documents associated with this investigation program, including this QAPP, 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 transmission electron microscope (TEM) and/or analysis of bulk asbestos by polarized light microscopy (PLM). This includes the analysis of NIST/NVLAP standard reference materials, 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 onsite EPA laboratory audit. The details of this EPA audit are discussed in Section B5.3.3. The LC also reserves the right to conduct 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

Initial Mentoring

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 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, United States Geological Survey (USGS) prepared three site-specific reference materials using LA collected at the Libby mine site (EPA 2008a). Upon entry into the Libby program, each laboratory was provided samples of these LA reference materials. Each laboratory analyzed 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 EDS spectra. These laboratory-specific and instrument-specific LA reference spectra (EPA 2008a) 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-VE analysis (EPA 2008b). These reference materials were prepared by adding aliquots of LA spiking material to uncontaminated Libby soils to obtain nominal LA concentrations of approximately 0.2%, 0.5%, 1.0%, and 2.0% (by weight). Each laboratory was provided with samples of these reference materials for use in training PLM 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 PE standards to evaluate PLM laboratory accuracy.

Regular Technical Discussions

On-going training and communication is an essential component of QA for the Libby project. To ensure that all laboratories are aware of 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 aspects of the analytical process, including sample flow, 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 Libby laboratory team has convened on multiple occasions at the ASTM Johnson Conferences in Vermont and at the ASTM Michael E. Beard Asbestos Conferences. These conferences enable the Libby laboratory and technical team members to have an ongoing 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

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 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.

A9. Documentation and Records

A9.1 Field

Field documentation will be collected and stored in order to meet project data reporting requirements, as specified in the *EPA Data Management Plan for the Libby Asbestos Superfund Site* (EPA 2013). Field teams will record information using prescribed electronic technology/systems (e.g., Response Manager), or hard copy forms, as appropriate. Hard copy field documentation will be maintained and archived at the A&E's project office in Libby, MT. Field documentation is discussed in detail in Section B3.1. Field data management, including publishing data to Scribe, is discussed in detail in Section B10.1.

A9.2 Troy Sample Preparation Facility

Prior to asbestos analysis, GPI samples are prepared (dried, sieved, ground) at the Sample Preparation Facility (SPF) in Troy, MT. Troy SPF documentation will be prepared and stored in accordance with project data reporting requirements, as specified in the *EPA Data Management Plan for the Libby Asbestos Superfund Site* (EPA 2013). Troy SPF personnel will record information using available electronic technology/systems, or hard copy forms, as appropriate, and publish required data to Scribe. All log sheets are maintained and archived at the Troy SPF. Scanned copies of log sheets are maintained on the ESAT network drive. These scanned copies are also emailed to the appropriate project Data Manager. Troy SPF data management is discussed in detail in Section B10.2.

A9.3 Laboratory

Analytical laboratory documentation will be prepared and stored in order to meet project data reporting requirements, as specified in the *EPA Data Management Plan for the Libby Asbestos Superfund Site* (EPA 2013). All asbestos analytical (including preparation) data generated in the laboratory will be documented on Site-specific laboratory bench sheets and entered into a database or spreadsheet electronic data deliverable (EDD) for submittal to the ESAT Project Data Manager. Section B4.2 provides detailed information on the requirements for laboratory

documentation and records. Laboratory data management is discussed in detail in Section B10.3.

A9.4 Logbooks and Records of Modification

It is the responsibility of field, Troy SPF, 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 QAPP, or procedures referenced herein governing sample handling, will be discussed with the EPA RPMs (or their designee) and the A&E's Project Manager prior to implementation. Such deviations will be recorded on a Record of Modification (ROM) form². Sections B5.1.3, B5.2.2, and B5.3.2 provide detailed information on the procedures for preparing and submitting ROMs by field, Troy SPF, and analytical laboratory personnel, respectively.

A9.5 QAPP Revision

As described in Sections A9.4, B5.1.3, B5.2.2, and B5.3.2, ROM forms will be used throughout the field season to document significant deviations from, or changes to, this QAPP. At the discretion of USACE in consultation with the EPA, substantive changes may require a QAPP revision instead of a ROM form. USACE and EPA approval of ROM forms is required prior to implementation. Approved ROM forms will be provided to all personnel on the distribution list in Section A3.

At the conclusion of each field season, the USACE Project Manager and the A&E Project Manager will initiate the annual review of the QAPP. This annual QAPP revision will incorporate changes documented on ROM forms throughout the season. Additional changes may be made during the QAPP revision as a result of review comments or at the suggestion of the GPI FTL, USACE, or other project personnel. Approval from all signature authorities for the updated document will be required prior to implementation. The EPA is the approval official for the original and revised QAPP.

B1. Study Design

B1.1 Locations

Candidate properties within OU4 and OU7 for GPIs will be identified by the RSQ and/or POC based on previously collected data and property statuses maintained in Response Manager. Properties will consist of those known to have one or more primary triggers (as defined in EPA's *Libby Asbestos Site Residential/Commercial Cleanup Action Level and Clearance Criteria Technical Memorandum* [EPA 2003] and its amendments [EPA 2011 and 2014] and *Flowerpot Action Level Clarification Memorandum* [CDM Smith 2013b] based on previous investigation

² The current version of the field ROM form is provided in the Libby Field eRoom; current versions of the Troy SPF and laboratory ROM forms are provided in the Libby Lab eRoom.

data), those previously screened using composite samples of fewer than 30 aliquots, and those that have not yet undergone initial screening. To the extent possible, investigations will be geographically clustered. The EPA/USACE will provide specific direction for conducting GPIs at properties in OUs other than OU4 and OU7. **Figure B-1** identifies the location of the Site. **Figure B-2** identifies the OU boundaries for the Site. Approximately 336 SIs and 280 DIs are anticipated to be completed during the 2014 field season.

B1.2 Sampling Design

The following provides an overview of the GPI program that will be conducted. Detailed information on investigation procedures and methods are presented in Section B2.

As previously mentioned, the GPI is designed to screen properties for the presence of LA or LA source materials and evaluate the extent of LA contamination. The overall investigation process is outlined in **Figure B-3**. The sampling program is divided into two stages: SIs and DIs. These are discussed in brief below.

SIs involve:

- Property Selection and Communication
- Verbal Interview
- Interior Inspection
- Exterior Inspection
- Screening Documentation

SI activities will include a full characterization of properties at a screening level intensity. The results of the SI will be evaluated against the current primary triggers. If one or more primary triggers are present, a land survey will be ordered and the property will be placed in the queue for DI. If no primary trigger is present, the property does not require any additional action at this time; however, it may be re-evaluated once the ROD is published.

DIs are performed to capture additional information on a property to support cleanup activities. DIs are completed at properties that have undergone initial screening and one or more primary exterior triggers is present.

DIs involve:

- Property Selection and Communication
- Land Survey (performed by removal contractor)
- Scheduling Investigations
- Review of Previously Collected Data

- Verbal Interview
- Interior Inspection
- Exterior Inspection

Refer to Section B2 for full details on the SI and DI processes.

B1.3 Study Variables

The total number of properties requiring GPI is somewhat variable as geospatial updates are made for the Site, which includes ongoing changes to property boundaries based on land survey data and changes in legal ownership (subdivisions and merges). The geospatial update process is outlined in the *EPA Data Management Plan for the Libby Asbestos Superfund Site* (EPA 2013). Notwithstanding geospatial changes, annual GPI planning and implementation will be based on best available property information, as tracked in Response Manager.

Additionally, the number and types of soil samples to be collected and analyzed for each GPI property can be widely variable depending upon property use and size. The time period required to adequately perform each SI will be estimated by the GPI Field Team Leader (FTL) based primarily on available geospatial information; DIs will be scheduled based on a review of the SI or other screening documentation and the land survey.

As previously mentioned, weather may also be a constraining factor in performing GPIs. It is anticipated that GPIs will be scheduled during the period of the year most conducive to performing both indoor and outdoor investigation (i.e., the field season); however, it is feasible that indoor work continue during periods of inclement weather (i.e., the off-season).

B1.4 Critical Measurements

The primary goal of the GPI effort is to evaluate the nature and extent of LA and LA source materials at OU4 and OU7 properties, and other OUs as directed by the EPA and USACE. This will be accomplished using both visual inspections, sampling for LA in soil and bulk materials, and analysis of interior dust, as needed.

There is an established correlation between visible vermiculite and LA content in Site soils. As such, standardized visual inspection protocol for vermiculite will be employed. The standardized visual inspection protocol, CDM-LIBBY-06 (see **Appendix B**), includes a training component to ensure consistency (to the extent possible) between GPI team members in applying the protocol.

The analysis of LA may be achieved using several different types of methods. For GPI efforts, all soil samples (including field duplicate samples) will be analyzed for asbestos by the PLM visual area estimation method (PLM-VE) and the PLM gravimetric method (PLM-Grav) in accordance with project-specific SOPs SRC-LIBBY-03 and SRC-LIBBY-01, respectively³. To date,

³ The current version of each project-specific analysis SOP is provided in the Libby Lab eRoom.

these methods have proven to be the most appropriate analytical methods to screen and quantify asbestos in Site source materials.

Bulk material samples collected as part of this effort will be analyzed by NIOSH 9002, Issue 2, *Asbestos (bulk) by PLM* (NIOSH 1994b), with subsequent analysis by PLM-PC400 as deemed necessary (refer to **Appendix A** and **Appendix F**), as specified on the COC form.

As appropriate, previously collected dust samples will be obtained from the sample archive and analyzed for LA by transmission electron microscopy (TEM) in accordance with the procedures specified in ASTM D5755-09.

B1.5 Data Reduction and Interpretation

Data collected as part of GPIs are intended to be used to support the OU4 and OU7 remedial investigation and property-specific removal decisions at the Site. See Section B5.1.2 for information regarding the evaluation of data collected under this QAPP as it relates to the DQOs in **Appendix A**.

B2. Investigation Methods

This section summarizes field activities that will be performed in support of GPIs within OU4 and OU7, and other OUs as directed by the EPA and USACE. This section also provides brief summaries of SOPs, including investigation-specific modifications where applicable and investigation-specific details not discussed in the SOPs. As previously mentioned, the GPI is designed to screen properties for the presence of LA or LA source materials and evaluate the extent of LA contamination for subsequent removal. The overall investigation process is outlined in Figure B-3. The sampling program is divided into two key phases: screening investigation (Section B2.2) and detailed investigation (Section B2.3). Specific details on each type of investigation are discussed within this section.

For comprehensive H&S information, field personnel will refer to the SOPs included in **Appendix B**. H&S protocol for GPI activities is provided in the APP (CDM Smith 2013c).

Sampling activities will be performed in accordance with this QAPP. The specific procedures that will be employed during GPIs are located in **Appendix B** and listed below:

- Field Logbook Content and Control (SOP EPA-LIBBY-2012-01)
- Photographic Documentation of Field Activities (SOP EPA-LIBBY-2012-02)
- Control of Measurement and Test Equipment (SOP EPA-LIBBY-2012-03)
- Field Equipment Decontamination (SOP EPA-LIBBY-2012-04)
- Handling Investigation-derived Waste (IDW) (SOP EPA-LIBBY-2012-05)
- Sample Custody (SOP EPA-LIBBY-2012-06)

- Packaging and Shipping Environmental Samples (SOP EPA-LIBBY-2012-07)
- Completion of Field Sample Data Sheets (SOP CDM-LIBBY-03)
- Soil Sample Collection at Residential and Commercial Properties (CDM-LIBBY-05)
- Semi-quantitative Visual Estimation of Vermiculite in Soils at Residential and Commercial Properties (CDM-LIBBY-06)
- Global Positioning System (GPS) Coordinate Collection and File Transfer Process (SOP CDM-LIBBY-09)
- Libby Chain of Custody Documentation (SOP ER8-LIBBY-01)

The following sections summarize field activities that will be performed during the implementation of the investigation efforts described in this QAPP.

Analytical methods for all samples collected in accordance with this QAPP are discussed in Section B.4.

B2.1 Field Preparation

B2.1.1 Field Team Training

Prior to conducting GPI field activities, new field team members must complete the following, at a minimum:

- Read the Site-specific APP (CDM Smith 2013b)
- Attend an orientation session with A&E's onsite H&S officer
- Read and understand all relevant governing documents
- Attain OSHA 40-hour HAZWOPER certification and relevant 8-hour refresher course certifications
- Attain respiratory protection course certification as required by 29 CFR 1910.134
- Attain asbestos awareness course certification as required by 29 CFR 1910.1001
- Complete training on sample collection techniques to the satisfaction of the GPI Task Leader (TL) or FTL
- Complete training on identifying vermiculite and Libby mine-related materials to the satisfaction of the GPI TL or FTL

Documentation of trainings/certifications will be stored in the Libby project files located at the A&E's Libby project office.

B2.1.2 Field Planning Meeting (Readiness Review)

Prior to beginning field activities, a field planning meeting (FPM) will be conducted by the A&E's GPI TL or FTL, which will be attended by the field team members conducting the work, a member of the A&E's QA staff, and a member of the A&E's H&S staff. The agenda, prepared by the GPI TL or FTL, will be reviewed and approved by QA and H&S staff prior to the FPM. A field planning meeting agenda is provided in **Appendix B**. The FPM will briefly address and clarify:

- Documents governing fieldwork that must be in the field
- Changes in the governing documents
- Objectives and scope of the fieldwork
- Equipment and training needs
- Field operating procedures, schedule of events, and individual assignments
- Required quality control (QC) measures
- H&S requirements

During the FPM, copies of the agenda will be distributed and an attendance list will be circulated for signature. The agenda and the completed attendance list will be maintained in the A&E's project files. Additional meetings will be held if major changes to the documents governing fieldwork occur, or the scope of the assignment changes significantly.

Field team members will perform the following activities before and during field activities, as applicable:

- Review and understand applicable governing documents
- Record appropriate levels of documentation regarding activities conducted
- Ensure coordination between key staff, such as the A&E's sample coordinator and the project's removal contractor
- Obtain required sample containers and other supplies
- Obtain, check, and calibrate field sampling equipment
- Obtain and maintain personal protective equipment (PPE)

B2.1.3 Inventory and Procurement of Equipment and Supplies

An inventory of project-procured equipment and supplies will be conducted by the GPI TL or FTL prior to field work. Any additional required equipment or supplies will be procured.

Acceptance of equipment, as pertinent, will be verified according to SOP EPA-LIBBY-2012-03, Control and Measurement and Test Equipment (see **Appendix B**). The following equipment is required for sampling activities conducted under this QAPP:

- Field logbooks
- Indelible ink pens
- Digital camera with memory card, as appropriate
- Sample paperwork and sample labels
- Custody seals
- Plastic zip-top bags
- Soil sampling equipment
- GPS unit(s) (e.g., Trimble® GeoXT or equivalent)
- PPE as required by the Site-specific APP (CDM Smith 2013c)
- Cordless drill and scope
- Ladder(s)
- Standard hand tools (e.g., screwdrivers, hammer, pry-bar)
- Tough tablet/laptop computer equipped with the Response Manager application (for completing forms)
- Measuring wheel/tape
- Land survey (hard copy) and/or aerial photo

B2.2 Screening Investigation

This section describes the sampling methods and procedures that will be used for SIs. The goal of completing an SI is to identify LA and/or potential sources of LA on a given property.

The major phases to an SI include:

- Property Selection and Initial Communication
- Verbal Interview
- Interior Inspection
- Exterior Inspection

- Screening Documentation

SI activities will include a full characterization of properties at a screening level intensity. The results of the SI will be evaluated against the current triggers. If one or more primary triggers are present, a land survey will be ordered and the property will be placed in the queue for DI. If no primary trigger is present, the property does not require any additional action at this time; however, it may be re-evaluated once the ROD is published.

For convenience, the primary interior and exterior triggers are presented in tabular format below. The PLM results shown in the table below and in subsequent sections of this QAPP are defined as follows:

- $\geq 1\%$ - Greater than or equal to 1% LA detected
- $< 1\%$ - Greater than or equal to 0.2% LA and less than 1% LA detected (also referred to as greater than TR)
- TR - Trace ($< 0.2\%$) LA detected
- ND - No LA detected

Table B-1 Primary Exterior Triggers

PLM (VV) Maximum Condition Present	SUAs, CUAs, SBs, and SSs	LUAs
$\geq 1\%$ (Vis+ or Vis-)	<ul style="list-style-type: none"> ▪ If samples are from an SI, perform DI ▪ If samples are from a DI, perform a removal 	If area $< 15,000 \text{ ft}^2$: <ul style="list-style-type: none"> ▪ If samples are from an SI, perform DI ▪ If samples are from a DI, perform a removal If area $\geq 15,000 \text{ ft}^2$: <ul style="list-style-type: none"> ▪ Collect delineation samples and perform removal if delineation samples detect LA at $< 1\%$ or $\geq 1\%$ by PLM-VE
$< 1\%$ (Vis+ or Vis-)	<ul style="list-style-type: none"> ▪ If samples are from an SI, perform DI ▪ If samples are from a DI, perform a removal 	If area $< 15,000 \text{ ft}^2$: <ul style="list-style-type: none"> ▪ If samples are from an SI, perform DI ▪ If samples are from a DI, perform a removal If area $\geq 15,000 \text{ ft}^2$: <ul style="list-style-type: none"> ▪ Collect delineation samples and perform removal if delineation samples detect LA at $< 1\%$ or $\geq 1\%$ by PLM-VE
TR (Vis+)	<u>If area $< 25\%$:</u> <ul style="list-style-type: none"> ▪ No removal required if no primary exterior trigger is present ▪ Remove if primary exterior trigger is present <u>If area $\geq 25\%$:</u> <ul style="list-style-type: none"> ▪ If samples are from an SI, perform DI ▪ If samples are from a DI, perform a removal 	No removal required (see above for evaluation of delineation sample results)

CUA – common use area
 DI – detailed investigation
 LA – Libby amphibole asbestos

SB – secondary building
 SI – screening investigation
 SS – secondary structure

TR – trace ($< 0.2\%$)
 VV – visible vermiculite
 Vis+ – visible vermiculite is observed

PLM (VV) Maximum Condition Present	SUAs, CUAs, SBs, and SSs	LUAs
LUA – limited use area	SUA – specific use area	Vis- – visible vermiculite is not observed

Table B-2 Primary Interior Triggers

Primary Interior Triggers		
Condition Present	PB	SB
Open, uncontained, migrating insulation in: <ul style="list-style-type: none"> ▪ attic ▪ indoor living space ▪ frequently accessed understructure (>12 times per year) 	Perform removal	Perform removal
Dust sample detect >5,000 s/cm ²	Perform removal	Perform removal
≥1% Interior Soil Result (Vis+ or Vis-)	Perform removal	See table above
<1% Interior Soil Result (Vis+ or Vis-)	Perform removal	See table above
TR Interior Soil Result (Vis+ or Vis-)	If frequently accessed (>12 times per year): <ul style="list-style-type: none"> ▪ Perform removal If infrequently accessed (≤12 times per year): <ul style="list-style-type: none"> ▪ No removal required 	See table above
ND Interior Soil Result (Vis+)	If frequently accessed (>12 times per year): <ul style="list-style-type: none"> ▪ Submit sample for re-analysis – removal required if re-analysis detects LA If infrequently accessed (≤12 times per year): <ul style="list-style-type: none"> ▪ No removal required 	No removal required

LA – Libby amphibole asbestos SB – secondary building Vis- – visible vermiculite is not observed
PB – primary building TR – trace (<0.2%)
s/cm² – structures per square centimeter Vis+ – visible vermiculite is observed

B2.2.1 Property Selection and Initial Communication

Property selection and solicitation will be conducted by voluntary recruitment program (VRP) staff in accordance with the Voluntary Recruitment Program Communication and Information Collection Strategy (DEQ 2011).

B2.2.2 Scheduling Screening Investigations

An SI will be scheduled for a time that is convenient for the property owner or tenant to be present and allow access to the interior of each structure/building at the property.

B2.2.3 Verbal Interview

Upon arriving at the property, the investigation team will meet with the property owner or tenants. The investigation team will provide the property owner/tenants with general details on the investigation and removal process, with emphasis that we will not know whether a

removal is required until all investigation is complete. Additionally, the investigation team will note pertinent anecdotal information provided by the property owner for removal planning. This information may include scheduling requests, general property information, details on known contamination present at the property, etc. If the property owner was not solicited through the VRP, the investigation team will complete and obtain property owner signatures on the Consent for Access to Property form that will cover inspections to be performed immediately as well as future investigation activities. Property owners will be notified prior to and may request to be present during future investigation activities. Access information will be tracked in Response Manager in accordance with the EPA project data reporting requirements.

B2.2.4 Interior Screening Inspection

During SI activities, the purpose of the interior inspection is to fully inspect the entire interior of each structure/building on the property to confirm the presence or absence of LA source materials (e.g., raw vermiculite, vermiculite insulation, vermiculite-containing building materials). Inspection information is captured on the Interior Property Inspection Form (IPIF), and associated sketches. Occupant information and history for occupied buildings is captured on the Occupant Information Form (OIF). The interior of each building on the property will be inspected during SI activities, unless inspected during previous investigation (e.g., CSS).

In addition, details collected during the interior inspection will be sufficient to support future removal activities and eliminate the need for subsequent investigation. Interior inspections and sampling will be completed to DI standards, in accordance with Section B2.3.4 of this QAPP.

B2.2.5 Exterior Screening Inspection

Exterior screening inspections will be performed to identify potential LA source materials within the exterior soils of the property. Inspection information is captured on the FSDSs and associated sketches.

If exterior investigation is limited for any reason (e.g., owner request, obstructions, H&S concerns), the field team will contact the FTL immediately. The EPA and/or USACE will be consulted when necessary.

The purpose of the following sections is to detail procedures for conducting exterior inspections, which will be conducted by the A&E and will include the following activities:

- Visual inspection
- Soil sampling
- Exterior inspection documentation

Visual Inspection

Visual inspection of exterior soils will be completed in accordance with CDM-LIBBY-06 with the following exception:

- The number of point inspections to be completed per use area is defined in **Table B-1** (Screening Investigation).
- Areas that have been previously characterized and meet the minimum point inspection/aliquot frequency for SIs, as stated in **Table B-3**, do not require further inspection or sampling. However, areas should be re-sampled if conditions have changed significantly since the initial sample collection (e.g., re-grading, vermiculite present now and not previously documented).

Table B-3 Visual Inspection and Soil Sampling Protocol

Screening Investigation		
Area Type	Visual Inspection Protocol ^a	Soil Sampling Protocol ^{b,c} Maximum Area per Sample
SUA ^d (flowerbed, garden, play area, etc.)	1 PI/1,450 ft ²	1 acre (43,560 ft ²)
SUA ^d (driveway)	1 PI/1,450 ft ²	1 acre (43,560 ft ²)
CUA ^e (yard, etc.)	1 PI/1,450 ft ²	1 acre (43,560 ft ²)
LUA ^{e,f} (field, pasture, etc.)	1 PI/7,260 ft ²	5 acres (217,800 ft ²)
PB (crawlspcace, cellar, etc.)	1 PI/100 ft ²	use area
SB ^g (shed, garage, barn, pump house, etc.)	1 PI/100 ft ²	use area
SS ^h (carport, lean-to, etc.)	1 PI/100 ft ²	use area
NUA ⁱ (wooded area, etc.)	No Inspection	No Sampling
Detailed Investigation		
Area Type	Visual Inspection Protocol ^a	Soil Sampling Protocol ^{b,c} Maximum Area per Sample
SUA ^d (flowerbed, garden, play area, etc.)	1 PI/100 ft ²	1,000 ft ²
SUA ^d (driveway)	1 PI/200 ft ²	6,000 ft ²
CUA ^e (yard, etc.)	1 PI/100 ft ²	3,000 ft ²
LUA ^{e,f} (field, pasture, etc.)	1 PI/7,260 ft ²	5 acres (217,800 ft ²)
PB (crawlspcace, cellar, etc.)	1 PI/100 ft ²	use area
SB ^g (shed, garage, barn, pump house, etc.)	1 PI/100 ft ²	use area
SS ^h (carport, lean-to, etc.)	1 PI/100 ft ²	use area
NUA ^h (wooded area, etc.)	No Inspection	No Sampling

^a A minimum of five points will be inspected per use area regardless of size.

^b All soil samples are 30-point composites.

^c Areas where vermiculite is observed will be segregated and sampled separately.

^d Multiple SUAs of the same type within the same general area may be combined to form one sample area; maximum of six areas can be combined (e.g., flowerbeds may only be combined with other flowerbeds).

^e Multiple CUAs and LUAs of the same type and material within the same general area may be combined to form one sample area; maximum of six areas (e.g., lava rock beds may only be combined with other lava rock beds).

^f If delineation samples are required in LUAs, the maximum area per sample is reduced to 15,000 ft² with 1PI/500ft².

^g Secondary buildings may not be combined with the surrounding area even if the material is the same throughout (e.g., do not sample garage and driveway together even if the same material is present in both areas).

ⁱ Secondary structures may be combined with the surrounding area provided that the material is the same throughout (e.g., if they contain the same material, a carport may be combined with the driveway).

^h If inspection or sampling of NUA is required, use CUA protocol.

SUA – specific-use area
CUA – common-use area
LUA – limited-use area

NUA – non-use area
PB – primary building
SB – secondary building

SS – secondary structure
PI – point inspection
ft² – square feet

Soil Samples

Soil samples will be collected during SIs in order to fully characterize each property. SI samples will only be collected if no primary exterior trigger is present (i.e., an exterior SI will be performed at a property with a primary interior trigger so long as a primary exterior trigger is not also present).

SI soil samples will be collected in accordance with CDM-LIBBY-05 with the following exceptions:

- The maximum area that a single 30-point composite sample may include varies depending on the type of use area. **Table B-3** (Screening Investigation) defines the maximum area per soil sample.
- CUAs may be combined as a sample area if they are proximal and contain the same material. **Table B-3** (Screening Investigation) defines the maximum area per soil sample.
- LUAs may be combined as a sample area if they are proximal and contain the same material. **Table B-3** (Screening Investigation) defines the maximum area per soil sample.
- For landscape features with a solid liner (e.g., no penetrations for plants or loose edges), the soil beneath the liner will not be inspected or sampled. The location will be identified on the SI sketch and a brief explanation provided.
- If soil is not encountered within 6 inches of the surface of decorative material (e.g., mulch, washed rock), the soil beneath the decorative material will not be inspected or sampled. The location will be identified on the SI sketch and a brief explanation provided.
- GPI teams will consider reasonably anticipated future use (RAFU) when assigning location type and will indicate on the FSDS whether location type is based on the current or RAFU.
- Areas that have been previously characterized and meet the minimum point inspection/aliquot frequency for SIs, as stated in **Table B-3**, do not require further inspection or sampling. However, areas should be re-sampled if conditions have changed significantly since the initial sample collection (e.g., re-grading, vermiculite present now and not previously documented).

Each area of the subject property containing visible vermiculite will be sampled separately and in accordance with the sampling guidelines above.

B2.2.6 Screening Documentation

An IPIF will be completed for each primary or secondary building inspected as part of SI activities. An OIF will be completed for each occupied building on the property. Example inspection forms are provided in **Appendix C**, and depict the data to be collected. Inspection forms will be completed electronically in the field using a tough tablet or laptop equipped with the Response Manager software.

For exterior inspections, sample information and visual inspection results will be recorded on a field sketch. Property-specific sketches will be completed on aerial photographs, scaled graph paper, or equivalent. Generally, one sketch is created; however, a property may be broken out into multiple sketches if quality and clarity cannot be maintained on a single sketch.

In addition, investigation teams will review and provide feedback to the POC regarding geounit-to-address relationships for appropriate tracking in Response Manager.

B2.2.7 Investigation Complete Notification

Once all SI data is assembled and has been reviewed, the GPI FTL will notify the POC that the SI is complete. This notification will include the completion date, which is considered the last property visit date. The GPI FTL will also complete a PSEF and submit that along with the hard copy property folder to the POC. The POC will review the PSEF and documentation in the property folder for accuracy and update the property status in RM.

B2.2.8 Letters to Residents

Following the SI complete notification, for properties where no EPA removal action is anticipated based on SI findings, a property status letter will be prepared by a qualified A&E author. The letter will be drafted using the current and appropriate EPA-approved letter template for No Further Action properties⁴, and will be reviewed by a qualified A&E reviewer prior to being mailed to the owner (and tenants, if applicable). A copy of the letter will be maintained in Response Manager in accordance with EPA project document management requirements.

B2.3 Detailed Investigation

This section describes the sampling methods and procedures that will be used to complete DIs. DIs are performed to capture additional information on a property to support cleanup activities. DIs are completed at properties that have undergone initial screening and one or more primary triggers are present.

The following is a summary of field activities that will be performed by the A&E during the DI:

- Property selection and communication
- Land survey

⁴ Current templates are maintained on the A&E's project office server.

- Scheduling investigations
- Review of previously collected data
- Verbal interview
- Interior inspection, as necessary
- Exterior inspection

B2.3.1 Property Selection and Communication

Properties will be selected for DI from candidate properties requiring removal or detailed level investigation as tracked in Response Manager. To the extent possible, investigations will be geographically clustered.

B2.3.2 Land Survey

A land survey will be conducted at each property identified for DI activities. Land surveys will include property boundaries to show the limits of the property for which the detailed investigation is being conducted. Surveys will also include major physical and geographic features of the property (e.g., structures/buildings, trees, individual land use areas). The survey contractor will be a registered and licensed land surveyor in the State of Montana. The survey contractor is currently subcontracted to the project removal contractor.

If a property is known to require removal or detailed level investigation based on previous investigation findings, the GPI FTL will notify the project removal contractor to order a land survey. Once received, a hard copy of the land survey will be used by the investigation team to mark soil sample locations and results, locations of visible vermiculite, and additional inspection information. Specific information to be captured by the investigation team is discussed in the following sections.

B2.3.3 Scheduling Detailed Investigations

If a property has undergone SI and/or partial DI activities, and additional investigation activities are required, the property owner will be notified of the preferred time-frame to conduct these activities. If the property owner has requested to be present during all activities, a DI will be scheduled for a time that is convenient for them.

If interior inspections are required during the DI phase, the DI will be scheduled for a time that is convenient for the property owner or tenant to be present and allow access to the interior of each structure/building on the property.

B2.3.4 Interior Detailed Inspection

Interior detailed inspections will be performed when previous investigation findings (see Section B9) indicate either contamination is present or unknown within buildings (e.g., house, garage, shed, barn) on the property. If previous data indicates that vermiculite insulation is present in the attic, no further attic inspection is required; however, if a plan view and section of the attic are not available from the previous inspection, the GPI field team must sketch a plan view and section of the attic based on features observed from the living space and/or exterior of the house (e.g., size of attic, vertical clearance, location of access, location of vents). To the extent possible, all interior detailed inspections will be completed during the SI phase. GPI interior inspection activities include:

- Attic inspection
- Living space assessment and wall inspection
- Understructure inspection and sampling, as required
- Interior soil samples, as required
- Interior inspection documentation
- Bulk material samples, as required

Interior inspections will be performed to evaluate the location and extent of mine-related materials within a building. Information will also be collected regarding the general construction and condition of the building, and access to mine-related materials. Interior inspections will include attic spaces, living spaces, and understructures (e.g., basement, cellar, crawlspace).

B2.3.4.1 Attic Inspection

Attic inspections will be attempted when the presence/absence of vermiculite could not be confirmed during previous investigations. If previous data indicates that vermiculite insulation is present in the attic, no further attic inspection is required; however, if a plan view and section of the attic are not available from the previous inspection, the GPI field team must sketch a plan view and section of the attic based on features observed from the living space and/or exterior of the house (e.g., size of attic, vertical clearance, location of access, location of vents). Attic inspections will be limited to confirming the presence/absence of vermiculite insulation and collecting sufficient details to support removal activities. Attic spaces will be inspected until either vermiculite insulation is confirmed, or until the entire attic has been inspected and no vermiculite insulation is present.

Once vermiculite insulation is confirmed in an attic space, inspection ceases and general details for the entire attic (including areas that share air space but do not contain vermiculite insulation) will be collected from that location. Detailed information about the attic space will be recorded during the Interior Property Removal Evaluation as discussed in the Response Action Work Plan (PRI-ER 2014).

Attic details will be recorded on the IPIF and associated sketch(es) as discussed in Section B2.3.4.5.

B2.3.4.2 Living Space Assessment and Wall Inspection

Interior living spaces will be inspected to evaluate whether vermiculite materials are present. Vermiculite may appear in living spaces as insulation that is leaking from the attic or walls, or as an additive in building materials. Living space assessments will include inspecting walls, ceiling and wall penetrations (e.g., plumbing, heating, ventilation and air conditioning [HVAC] systems, electrical fixtures, cracks, gaps), and plaster/mortar/chinking materials. If vermiculite is observed during the interior inspection, the team will indicate the location on the interior sketch and record details regarding the type and condition of material. If vermiculite additives are identified within building materials on the interior or exterior, the team will evaluate whether the material is friable (i.e., able to be pulverized by hand) and a note indicating the condition will be included on the interior sketch as well. Friable VCBM will be sampled as described in Section 2.3.4.6.

Based on previous investigation findings, small amounts of vermiculite insulation are likely to be present within wall cavities of structures/buildings that have vermiculite attic insulation. If vermiculite insulation is observed within the attic of a structure/building, it will be assumed that the walls below those attic sections may contain some amount of vermiculite and those walls will not be inspected using methods that may cause a release (e.g., removing outlet covers, drilling/scoping). This will be noted within the interior inspection documentation as detailed in Section B2.3.4.5.

In addition, previous investigations have found vermiculite insulation used as primary insulation within wall cavities. Walls will be inspected to assess whether significant vermiculite insulation is present. At least one location along each exterior and interior wall will be inspected. Non-destructive inspection methods will be utilized when possible. This will include removing electrical outlet and switch covers, and inspecting through other existing wall penetrations. Destructive inspection methods (i.e., drill/scope) will be used only at the discretion of the FTL. If destructive methods are required, care will be taken to minimize damage and inspections will be carried out within inconspicuous areas. The investigation team will seal new penetrations with appropriate patching material.

If interior investigation is limited for any reason (e.g., owner request, obstructions, H&S concerns), the field team will contact the FTL immediately. The EPA and/or USACE will be consulted when necessary. Living space details will be recorded on the IPIF and associated sketch(es) as discussed in Section B2.3.4.5.

B2.3.4.3 Understructure Inspection

Building understructures will be inspected to evaluate whether vermiculite materials are present. Vermiculite may appear in understructures as insulation that is leaking from the attic or walls, as additives in building materials, or as vermiculite in soil floors. Understructure inspections will include inspecting ceiling and wall penetrations (plumbing, HVAC, electrical, cracks, gaps, fixtures, etc.), plaster/mortar materials, and inspecting soil floors. If the building understructure has a soil floor, a visual inspection and soil sampling will be completed per Sections B2.3.6.1 and B2.3.6.2 of this QAPP. In general, only one soil sample will be collected per

understructure. Exceptions may include, but are not limited to, the following:

- Vermiculite is observed in only a portion of the understructure
- Multiple soil types are present in the understructure
- Significant elevation difference (e.g., part basement and part crawlspace)
- Understructure is physically separated into several smaller areas (e.g., crawlspace for original house and addition are separated by foundation wall)

Understructure details will be recorded on the IPIF and associated sketch(es) as discussed in Section B2.3.4.5.

In addition to general details, the investigation team will make a determination as to the frequency the understructure is accessed. Understructures will be categorized as frequently accessed, infrequently accessed, or a combination of the two (for separate areas). In general, understructures will be considered infrequently accessed if they are accessed on average no more than once monthly, and the activities being conducted involve minimal soil disturbance. Understructures that are accessed on average more than once monthly, or if activities during access include significant soil disturbance (e.g., digging), will be considered frequently accessed. Understructure remediation criteria is discussed in the *Final Response Action Work Plan*, Revision 6 (PRI-ER 2014).

B2.3.4.4 Interior Soil Samples

Soil samples will be collected from inside a building if significant soil areas are present (e.g., soil floor, planters). Areas with vermiculite will be sampled separately from areas where vermiculite was not observed. Interior soil samples will be collected in accordance with Section B2.3.6.2 of this QAPP.

B2.3.4.5 Interior Inspection Documentation

An IPIF will be completed for each primary or secondary building inspected as part of this QAPP. IPIFs will be completed electronically in real-time in the field using the Response Manager software. Attic, living space, and understructure sketches will accompany each IPIF as appropriate. Sketches can either be specific to the area inspected, or part of the exterior sketch. Sketches will include the details indicated in **Table B-4**. Sketches will only be prepared for the levels/floors of the structure where LA source materials are observed and/or where samples are collected.

Investigation teams will collect digital photographs in accordance with Section B3.1.4 of this QAPP. Photographs will include access points, interior hazards, pre-existing conditions, vermiculite, and general interior photographs.

B2.3.4.6 Bulk Material Sampling

Bulk material samples may be collected for asbestos analysis from a variety of sources (e.g., log chinking, chimney mortar, plaster, or other building material) where vermiculite additives are visually identified. If the material is friable or deteriorated, or there are plans to demolish or remodel buildings or areas of buildings with VCBM present, sampling of the material will be conducted in general accordance with Administrative Rules of Montana (ARM) 17.74.354(3)(c), Inspection Requirements for Demolition and Renovation Activities. When sampling is deemed necessary, the appropriate number of samples will be collected from each homogenous material as follows:

- 3 samples from each homogeneous material that is 1,000 square feet (ft²) or less
- 5 samples from each homogeneous material that is greater than 1,000 ft² but less than or equal to 5,000 ft²
- 7 samples from each homogeneous material that is greater than 5,000 ft²

Individual samples will be collected in plastic zip-top bags, double bagged, and submitted to the laboratory by the A&E sample coordinator. Bulk material samples will be documented, collected, and submitted using the same documentation and custody processes as all other samples collected as part of this QAPP (see Section B3). Bulk material samples will be analyzed by PLM-9002, with subsequent analysis by PLM-PC400 as deemed necessary (refer to **Appendix A** and **Appendix F**), as specified on the COC form. This overall bulk sampling approach takes into consideration the EPA's Applicability Determination Index Control Number C112: Point Counting, which specifies use of EPA/600/R-93/116 as the accepted verification method. Property-specific removal actions will be directed by the EPA and USACE based on the Decision Tree for Vermiculite-Containing Building Materials (**Appendix F**).

The standard turnaround time for bulk sample results by each PLM method shall be 10 days, unless the COC form accompanying the samples sent to the laboratory indicates otherwise.

Table B-4 Investigation Sketch Details

General Sketch Details To be included on all field sketches		
<ul style="list-style-type: none"> ▪ Property address ▪ AD number ▪ BD number (for all primary and secondary buildings) ▪ Inspection date ▪ Personnel ▪ North arrow ▪ Scale (if applicable) ▪ Sketch description (e.g. attic, first floor, exterior visible and analytical) 		
Interior Inspection Sketch Details		
Attic	Living Space	Understructure
<ul style="list-style-type: none"> ▪ Plan view/layout – including dimensions ▪ Types of insulation ▪ Depth of insulation ▪ Attic accesses – location and size ▪ Vertical clearance – structure cross-section ▪ Hazards (in attic and near access) ▪ Obstacles ▪ Joist – size and spacing ▪ Flooring (above and below joist) 	<ul style="list-style-type: none"> ▪ Floor plan/layout ▪ Location of LA source materials ▪ Soil samples – locations and results ▪ VCBM samples – locations and results 	<ul style="list-style-type: none"> ▪ Soil samples – locations and results ▪ Visual inspection results ▪ Floor types – soil versus solid flooring ▪ Access – location and size ▪ Vertical clearance
Exterior Inspection Sketch Details Visual & Analytical Sketch		
<ul style="list-style-type: none"> ▪ Soil samples – locations and results <ul style="list-style-type: none"> – Sample ID – Location ID ▪ Visual inspection – location and results <ul style="list-style-type: none"> – Visible vermiculite quantity and number of observations – Location ID ▪ Location IDs for all primary and secondary buildings on the property Fence lines Underground utilities – if known Overhead utilities – if not shown on survey 		

B2.3.5 Exterior Detailed Inspection

Exterior detailed inspections will be performed at properties where previously collected data indicates that one or more primary triggers are present. Exterior inspections are performed to further define the location and extent of contaminated material and to adequately characterize the property. Inspection information is captured on the FSDSs and associated sketches.

If exterior investigation is limited for any reason (e.g., owner request, obstructions, H&S concerns), the field team will contact the FTL immediately. The EPA and/or USACE will be consulted when necessary.

A&E exterior inspection activities include:

- Visual inspection

- Soil sampling
- Exterior inspection documentation

B2.3.5.1 Visual Inspection

Visual inspection of exterior soils will be completed in accordance with CDM-LIBBY-06 (see **Appendix B**) with the following exceptions:

- The number of point inspections to be completed per use area is defined in **Table B-3** (Detailed Investigation).
- Areas that have been previously characterized and meet the minimum point inspection/aliquot frequency for DIs, as stated in **Table B-3**, do not require further inspection or sampling. However, areas should be re-sampled if conditions have changed significantly since the initial sample collection (e.g., re-grading, vermiculite present now and not previously documented).
- In general, non-use areas (NUAs) are not inspected as part of this QAPP. However, NUAs will be inspected if: 1) LA source materials are observed in adjacent areas and it appears to continue into the NUA, or 2) if the property owner provides information that indicates LA source materials may be present within a specific portion of the NUA. In this case, the area of concern within the NUA will be inspected as a CUA utilizing the guidelines outlined in **Table B-3**.

B2.3.5.2 Soil Sampling

Existing data will be used in the property investigations as described in Section B9, with additional data collection to augment the data gaps where limitations have been identified. Three types of samples will be collected during DIs: characterization samples, re-characterization samples, and delineation samples. The purpose of these samples is to address the recognized limitations of the existing data, as described below.

Characterization Samples

Previous investigation/screening activities (e.g., CSS, Phase I) focused only on high traffic areas of the property; therefore, some portions of the property may not have been sampled. Characterization samples will be collected to characterize use areas that were not previously sampled.

Re-characterization Samples

Soil samples were collected during previous investigation/screening activities to evaluate the presence or absence of LA within soil. These samples were generally collected as 5-point or 30-point composite samples from relatively large areas. Re-characterization samples will be collected from all previously sampled areas where conditions have changed since the previous sampling event or the field team is unable to identify the bounds of the previously collected sample.

Delineation Samples

Soil samples were collected during previous investigation/screening activities to evaluate the presence/absence of LA within soil, and were collected to characterize relatively large areas. Delineation samples will be collected to further define the extent and boundary of contamination. Delineation samples will be collected from areas where previous sample areas exceed the maximum area per DI soil sample as outlined in **Table B-3**.

Delineation samples are only required in LUAs when the previous result was <1% or ≥1% LA and the area was larger than 15,000 ft². When required, delineation samples of ≤15,000 ft² will be collected. Field teams will consider anecdotal owner information, field observations (e.g., apparent back fill, trenches) when establishing boundaries for delineation samples.

Sample Collection Methods

All DI soil samples will be collected in accordance with SOP CDM-LIBBY-05 (see **Appendix B**) with the following exceptions:

- The maximum area that a single 30-point composite sample may include varies depending on the type of use area. **Table B-3** (Detailed Investigation) defines the maximum area per soil sample.
- CUAs may be combined as a sample area if they are proximal and contain the same material. **Table B-3** (Detailed Investigation) defines the maximum area per soil sample.
- LUAs may be combined as a sample area if they are proximal and contain the same material. **Table B-3** (Detailed Investigation) defines the maximum area per soil sample.
- For landscape features with a solid liner (i.e., no penetrations for plants), the soil beneath the liner will not be inspected or sampled. The team will identify the location on the DI sketch and provide a brief explanation.
- If soil is not encountered within 6 inches of the surface of decorative material (e.g., mulch, washed rock), the soil beneath the decorative material will not be inspected or sampled. The location will be identified on the DI sketch and a brief explanation provided.

- GPI teams will consider RAFU when assigning location type and will indicate on the FSDS whether location type is based on the current or RAFU.
- Areas that have been previously characterized and meet the minimum point inspection/aliquot frequency for DIs, as stated in **Table B-3**, do not require further inspection or sampling. However, areas should be re-sampled if conditions have changed significantly since the initial sample collection (e.g., re-grading, vermiculite present now and not previously documented).

Each area of the subject property containing visible vermiculite will be sampled separately and in accordance with the sampling guidelines above.

B2.3.5.3 Exterior Inspection Documentation

Sample information and visual inspection results will be recorded on a field sketch. If available, a property survey will be utilized as the baseline for these sketches. If a property survey is not available, aerial photographs, scaled graph paper, or an equivalent will be used. Generally, one sketch is created; however, a property may be broken out into multiple sketches if quality and clarity cannot be maintained on a single sketch. Sketches will include the details indicated in **Table B-4**. Field sketches may be used to generate removal plans and as such must be neat and legible.

Investigation teams will collect digital photographs in accordance with Section B3.1.4 of this QAPP. Photographs will include access points, exterior hazards, pre-existing conditions, all areas where LA source materials are observed, and general exterior photographs.

In addition, investigation teams will review and provide feedback to the POC regarding geounit-to-address relationships for appropriate tracking in Response Manager.

B2.4 Field Quality Control Samples

Field QC samples associated with soil samples are field duplicate samples. These samples are discussed in this section and summarized in **Table B-5**.

Field duplicate samples for both SI and DI soil and bulk material sampling activities will be collected at a rate of 1 per 20 field samples collected. Soil and bulk material field duplicate samples will be collected from areas that are being sampled during one of the investigation activities discussed in the previous sections. However, individual composite points for the soil duplicate sample will be collected from different locations within the same use area as the original field sample. Bulk material duplicates will be collected from the same homogenous material and general location as the field sample. Soil field duplicate samples will be collected in accordance with CDM-LIBBY-05 (see **Appendix B**). Dust samples are no longer collected, therefore collection of dust blanks is not required. Sample coordinators will submit dust blanks for a property when the dust samples for that property are requested to be pulled from archive and analyzed. There is currently no acceptance criteria established for soil or bulk material field duplicates. The acceptance criterion for dust blanks is no detectable LA. Field duplicate sample

results may be used preferentially to the field sample results (for the same area) for decision making. Additionally, laboratory QC sample results may also be used preferentially to the field sample results for decision making. The GPI TL or FTL are responsible for maintaining overall GPI program soil and bulk material field duplicate sample collection frequencies.

Table B-5 Summary of Field QC Samples

Sample Type	Associated QC Sample	Collection Frequency	Analysis Frequency	Analysis Request
Soil	field duplicate	1 per 20 field samples	100%	PLM-VE/PLM-Grav
Bulk material	field duplicate	1 per 20 field samples	100%	PLM-9002/PLM-PC400 (as needed)
Dust	field blank	Collection not required (archived blanks will be analyzed when available)	100%	ASTM D5755-09 with project-specific modifications as noted in SRC-LIBBY-05

ASTM D5755 – American Society for Testing and Materials D7555 method
 PLM-9002 – polarized light microscopy NIOSH 9002
 PLM-PC400 – polarized light microscopy by EPA/600/R-93/116 (400 points)
 PLM-Grav – polarized light microscopy gravimetric method
 PLM-VE – polarized light microscopy visual area estimation method

B2.5 General Processes

This section describes the general field processes that will be used to support the sampling described in this QAPP and includes references to the Site-specific SOPs and project-specific procedures when applicable.

B2.5.1 Equipment Decontamination

Decontamination of reusable field equipment will be conducted in accordance with SOP EPA-LIBBY-2012-04, *Field Equipment Decontamination* (see **Appendix B**). Materials used in the decontamination process will be disposed of as IDW as described below.

B2.5.2 Investigation-derived Waste

IDW at each property will consist of excess sample volume, spent decontamination supplies, and PPE. All IDW will be handled in accordance with SOP EPA-LIBBY-2012-05, *Handling Investigation-derived Waste* (see **Appendix B**). In brief, IDW will be double-bagged in clear 6-mil poly bags with 'IDW' written in indelible ink on the outer bag. All IDW generated during GPIs will remain in the custody of the field team, or locked in a storage area, until it can be entered into the waste stream at the local class IV asbestos landfill.

B3. Samples and Locations

B3.1 Field Documentation

In accordance with EPA project records retention requirements, all hard copy and electronic field documentation generated by the A&E as part of GPIs will be retained at the A&E Libby project office until relinquished to the EPA at project closeout.

B3.1.1 Field Sample Data Sheets

As noted previously in Section A9, an FSDS will be completed for each GPI sample and visual inspection in accordance with SOP CDM-LIBBY-03, *Completion of Field Sample Data Sheets* (see **Appendix B**) with the following clarification:

- For SIs without a land survey, the location area must be estimated in the field by the GPI team and recorded on the FSDS. If a land survey is available for an SI, location area must be left null on the FSDS. Location areas for SIs with a land survey will be generated by a drafter and uploaded to Scribe by the Field Data Manager.
- For DIs, the location area must be left null on the FSDS. Location areas for DIs will be generated by a drafter and uploaded to Scribe by the Field Data Manager.

Use of standardized forms ensures consistent documentation across samplers. Current versions of media-specific FSDSs are provided in the Libby Field eRoom. FSDSs are location-specific and allow for the entry of up to three individual samples from the same property 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.

An event ID will be recorded on each FSDS to identify the protocol used for the inspection(s) or sample(s) recorded on that FSDS. For inspections and samples collected under the SI protocol, event ID SI-040014 will be used. For inspections and samples collected under the DI protocol, event ID DI-040014 will be used.

A unique alphanumeric code, or location ID, will identify each location inspected or sampled during GPI activities. The coding system will provide a tracking record to allow retrieval of information about a particular location and to ensure that each is uniquely identified. Location IDs will be sequential and will be recorded on the FSDS. For locations where a sample was collected, both the location ID and sample ID will appear on the FSDS.

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 GPI 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 personnel, the FSDSs are checked for completeness as data are input into the local Scribe field database. Field sample coordination personnel also conduct an independent check of entered data for accuracy and completeness.

If a revision is required to the hard copy FSDS during 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 at the Site, revisions will be made by the GPI FTL, or designee. 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. A&E field administrative staff will manage the hard copy FSDSs in the A&E Libby project office. Original FSDSs will be filed by medium and FSDS number.

B3.1.2 Sample and Location Labeling

A unique alphanumeric sample ID will identify each sample collected during GPIs. Sample IDs provide a tracking record to allow retrieval of information about a particular sample from collection through final archive or disposal. Sample IDs will be sequential and will not be representative of a particular building or equipment. Sample IDs will correlate with location IDs, which will be identified on FSDSs.

The GPI sample labeling scheme is as follows:

4G-XXXXX

Where:

4G identifies that a sample is collected in accordance with GPI protocol, and
XXXXX represents a unique, 5-digit number

Preprinted adhesive sample labels are required to be signed out by sampling personnel. The labels are controlled to prevent duplication in assigning sample IDs. The labels will be affixed to both the inner and outer sample bags for soil and bulk material samples. Sample labels will be used in accordance with SOP EPA-LIBBY-2012-06, *Sample Custody* (see **Appendix B**).

The location labeling scheme for GPIs is as follows:

XX-##### or

BD-#####

Where:

XX identifies sample locations,
BD identifies interior locations, and
represents a 6-digit numeric code

B3.1.3 Field Logbooks

The field logbook is an accounting of GPI activities and will duly note problems or minor deviations from this QAPP. Field logbook entries will be recorded in accordance with SOP EPA-LIBBY-2012-01, *Field Logbook Content and Control* (see **Appendix B**). Sample details will be recorded on an FSDS and FSDS numbers will be recorded in the logbook.

A&E 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 type of activities recorded in each field logbook (i.e., GPIs), and the date when the field logbook was returned. As field logbooks are completed, originals will be catalogued and maintained in the A&E project office. Scanned copies of field logbooks will be maintained on the A&E's project server, which is backed up daily to an offsite location.

B3.1.4 Photographic Documentation

All photographic documentation will be in accordance with SOP EPA-LIBBY-2012-02, *Photographic Documentation of Field Activities* (see **Appendix B**). Captions are not required for photographs taken as part of this QAPP.

Photographs will be taken with a digital camera at places that field personnel deem necessary. Electronic photograph files will be saved each day to the A&E's server located at the Libby project office (backed up daily to an offsite location), and named so that photographs for a particular property or activity can easily be retrieved. The GPI photograph file naming convention is as follows:

45 Montana Ave_DI_092113_001

Where:

45 Montana Ave = the address where GPI activities occurred
DI or SI = the specific activity being documented
092113 = the date the photograph was taken (MMDDYY)
001 = the number of the photograph taken at that property that day

Following completion of GPI activities, all photographic files pertaining to a property will be copied to the A&E server and ultimately copied onto compact disc and filed in Libby along with other property-specific documentation.

B3.1.5 Change Control

Corrections to field documentation, including FSDSs and logbooks, require a single strikeout of the erroneous information, initials, and date. The corrected information will be entered in close proximity to the existing entry. For revisions to FSDSs, it is the responsibility of investigation staff making the revisions to provide the revised originals to the A&E's sample coordinator for updating corresponding electronic data. Updated FSDS data will be published to Scribe by A&E data management staff promptly in order to meet the EPA reporting requirements.

All deviations from the guiding documents will be recorded in the logbooks by the investigation team or on the Record of Modification to Documents Governing Field Activities by the GPI FTL (see Section B5.1.2 for specifics).

B3.1.6 Global Positioning System Coordinate Collection

GPS location coordinates will be collected for inspected or sampled locations in accordance with SOP CDM-LIBBY-09, *GPS Coordinate Collection and Handling* (see **Appendix B**). If a geo-referenced property survey is available (primarily for DIs), location coordinates may be sourced from the geo-referenced property survey by the drafters and provided to the A&E data management team for review and publishing to Scribe. Coordinates for buildings will be collected only if the building does not already have an assigned GPS location.

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 potential data entry errors (e.g., points display on the correct geounit).

B3.1.7 Field Sample Custody

Sample custody and documentation will follow the requirements specified in SOP EPA-LIBBY-2012-06, *Sample Custody* (see **Appendix B**). In general, all teams will ensure that samples, while in their possession, are maintained in a secure manner to prevent tampering, damage, or loss. At the end of each day, investigation teams will relinquish samples directly to sample coordination staff or to a designated secure sample storage location. Relinquishment will be documented in the logbook.

B3.1.8 Chain-of-Custody Requirements

For the Libby project, the chain-of-custody (COC) record is employed as physical evidence of sample custody and condition from the sample coordination team to the receiving facility. A completed COC record is required to accompany each batch of samples, whether it is hand-delivered to the EPA LC or shipped to a processing or analytical facility.

The sample coordination team will produce COC records in accordance with SOP ER8-LIBBY-01, *Libby Chain of Custody Documentation*. Only quality-checked sample information will be used

for COC records. In the event that electronic systems are unavailable (e.g., due to a power outage), hard copy COC records will be employed. Hard copy COC records will be data-entered as soon as electronic systems are back online.

For hand-deliveries, a sample coordinator will relinquish samples and corresponding COC records to the EPA LC under strict custody. During relinquishment, the sample coordinator will complete the following information in the designated spaces at the bottom of the COC record: signature, company name, date, and time. The EPA LC will also complete the required information and will make a note regarding sample condition (e.g., OK – accept). The sample coordinator will retain the bottom copy of the COC record for the A&E’s project record.

B3.1.9 Sample Packaging and Shipping

Samples will be packaged and shipped in accordance with SOP EPA-LIBBY-2012-07, *Packaging and Shipping of Environmental Samples* (see **Appendix B**). Samples will be hand-delivered to the EPA LC, picked up by a delivery service courier, or shipped by a delivery service to the designated facility or laboratory, as applicable. For hand-deliveries, the sample coordinator will package samples 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.

B3.1.10 Field Equipment Maintenance

Field equipment maintenance will be conducted and documented in accordance with SOP EPA-LIBBY-2012-03, *Control of Measurement and Test Equipment* (see **Appendix B**).

B3.2 Holding Times

For the samples specified for collection in this QAPP, no holding time requirements will be employed.

B3.3 Archival and Final Disposition

All samples and grids will be maintained in storage at the Troy SPF, analytical laboratory, or fire cache sample storage facility, unless otherwise directed by the EPA. When authorized by the EPA, the laboratory will be responsible for proper disposal of remaining samples, sample containers, shipping containers, and packing materials in accordance with sound environmental practice, based on the sample analytical results. The laboratory will maintain proper records of waste disposal methods, and will have disposal company contracts on file for inspection.

B4. Analytical Methods and Operations

The EPA will be responsible for all sample analysis, including sample processing prior to analysis. The A&E will be responsible for relinquishing all samples to the EPA LC, or processing facility or laboratory as designated by the EPA LC. The A&E sample coordinator

will also be responsible for communicating with the EPA LC to relay pertinent sample and analysis information including sample quantities; special sample handling requirements, processing, or analysis concerns; and requested turn-around times.

This section discusses the analytical methods, custody and documentation procedures, QA/QC requirements, and data management requirements to be employed by the laboratory in support of property investigation activities.

B4.1 Analytical Methods and Turnaround Times

This section describes the analytical methods used for SI and DI samples.

An analytical requirements summary sheet (see **Appendix D**) specific to sampling activities associated with this QAPP will be distributed by the EPA, and reviewed and approved by all participating laboratories prior to sample handling.

The A&E's sample coordinator will provide the EPA LC with requested turn-around times for all samples relinquished. In general, it is expected that analysis, including soil preparation, for all SI and DI soil samples will be complete within 45 (business) days. Archived dust samples will be complete within five (business) days from the time the laboratory receives them. Analysis of bulk material samples by PLM-9002, and PLM-PC400 if needed, will be complete within 10 (business) days from the time the laboratory receives them.

B4.1.1 PLM-VE/PLM-Grav - Soil Samples

Prior to analysis, all soil samples require processing. Soil samples will be processed using the current version of the Libby soil sample processing SOP ISSI-LIBBY-01 (see **Appendix B**). The A&E will indicate the current version of the soil sample processing SOP in the analysis request section of the COC record. It is the responsibility of the soil preparation facility to specify the appropriate PLM method as it corresponds to the specific sample fraction being submitted for analysis (i.e., fine ground or coarse fraction) on their COC records to the laboratory.

All soil samples collected as part of this effort, including field duplicate samples, will be analyzed for asbestos by PLM-VE and PLM-Grav in accordance with SOPs SRC-LIBBY-03 and SRC-LIBBY-01 (see **Appendix B**), respectively.

B4.1.2 TEM - Dust Samples

Dust samples will not be collected as part of this QAPP. However, archived dust samples collected as part of previous investigations (e.g., CSS) will be analyzed to support the removal decisions and planning. The A&E investigation teams will identify all archived dust samples that require analysis and communicate this to the A&E sample coordinator.

All archived dust samples will be analyzed by TEM in accordance with the project-amended ASTM D5755 method as described in SOP SRC-LIBBY-05 (see **Appendix B**) and Libby

laboratory modification LB-000040.

The laboratory will achieve the target method analytical sensitivity of 1,000 per square centimeter using indirect preparation techniques as described in EPA-LIBBY-08 and Libby laboratory modification LB-000091 (see **Appendix B**).

B4.1.3 PLM – Bulk Material Samples

Bulk material samples collected as part of this effort will be analyzed by NIOSH 9002, Issue 2, *Asbestos (bulk) by PLM* (NIOSH 1994b), with subsequent analysis by PLM-PC400 as deemed necessary (refer to **Appendix A** and **Appendix F**), as specified on the COC form.

Because the level of detection for PLM-9002 is estimated (at <1% asbestos), no specific level of detection has been established for project samples analyzed using this method. The level of detection for PLM-PC400 is 0.25% if 400 points are evaluated during analysis.

B4.1.4 Health and Safety Air Samples

The personal air samples collected for the ongoing health and safety monitoring will be analyzed in accordance with the *Response Action SAP* (CDM Smith 2011). In brief, air samples will be prepared and analyzed by PCM in accordance with NIOSH Method 7400, Issue 2 and the most recent version of Libby Laboratory Modification LB-000015.

B4.2 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 includes a case narrative that briefly describes the analytical methods, deviations from the methods, revisions to data reports, COC discrepancies, etc. The data report also includes copies of the signed COC forms, sample preparation logs, and analytical benchsheets. The data report may also include spectra print outs, grid sketches, instrument preparation logs, instrument print outs, instrument maintenance records, analysis run logs, etc. The laboratory provides an electronic scanned copy of the analytical data report to the LC and others, as directed by the LC.

B4.3 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 for each analytical method and each medium. Since the beginning of the Libby project, each EDD 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 Lab eRoom.

For TEM analyses, detailed raw structure data will be recorded and results will be transmitted using the Libby-specific EDDs for TEM. For PLM analyses, optical property details and results will be recorded on the Libby-specific EDDs for PLM. 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
- 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). Currently, LADT is only utilized by the ESAT laboratory for entry of PLM analytical results. Other labs continue to use Libby-specific EDDs as described above.

B4.4 Custody Procedures

Laboratory custody procedures are provided in the QA management plans for each laboratory. These plans were independently audited and found to be satisfactory by the EPA's laboratory audit team.

The basic laboratory sample custody process is as described herein. Upon receipt at the laboratory, 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 custodian will sign the COC record and maintain a copy for their project files; the original COC record will be appended to the hard copy data report. Next, the sample custodian may assign a unique laboratory number to each sample on receipt. This number will identify the sample through all further handling at the laboratory. It is the laboratory's responsibility to maintain internal logbooks and records throughout sample preparation, analysis, data reporting, and sample archiving.

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 issues/deficiencies associated with field data collection or sample processing are quickly identified and rectified.

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 GPI sampling efforts. Additional information on field training requirements is provided in Section A8.1.

B5.1.2 Document Review

Field personnel submit all records (e.g., FSDS copies, lognotes copies, field sketches) to the GPI FTL, or designee, for review once field activities are complete. The GPI FTL, or designee, is responsible for reviewing these records for completeness and comparing collected data to the DQOs presented in **Appendix A**. This review includes checking:

- FSDS copies against an export of data in Scribe
- that the final sketch reflects the sample areas that were marked on the field sketch
- the entire property has been characterized according to the requirements in this QAPP
- photos for inconsistencies between field conditions and documentation
- IPIF(s) and OIF for completeness

If deficiencies are noted during this review, the field team makes the necessary corrections.

In addition, onsite USACE personnel review documentation for approximately 10% of completed investigations. If comments are generated during USACE review, the GPI FTL is notified and will coordinate with the field team to make corrections. USACE comments will be retained in the hard copy property folder.

B5.1.3 Modification Documentation

All major field deviations from and modifications to this QAPP will be recorded on the Libby field ROM Form. 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. ROMs will not be implemented until approved by USACE and the EPA. See Section A9.5 for details incorporating deviations from ROM forms during QAPP revision.

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 A&E's project QA Coordinator. A ROM tracking log for all field modifications is also maintained by the 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 submitted to the appropriate EPA RPM and USACE Project Manager for review and approval. Approved field ROMs are maintained on the A&E's project server.

B5.1.4 Field Surveillances

Field surveillances consist of periodic observations made to evaluate continued adherence to investigation-specific governing documents. It is not anticipated that field surveillance will be performed for GPI sampling efforts. However, field surveillances may be conducted if field processes are revised or other QA/QC procedures indicate potential deficiencies.

B5.1.5 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. Due to the level of effort for sampling and the duration of the activities discussed in this QAPP, a field audit is anticipated to be scheduled for GPIs annually (see Section C1.1).

B5.1.6 Field QC Samples

Field QC samples are typically collected to help ensure that field samples are not contaminated from exogenous sources during sample collection, and to help evaluate the precision of field sample analytical results. Field QC samples are assigned unique field IDs and are submitted to the analytical laboratory along with the associated field samples. For GPIs, field duplicate soil samples will be collected as described in Section B2.4.

B5.2 Troy SPF

Prior to shipment to a laboratory for analysis, soil samples will be dried 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* (TechLaw, Inc. 2007)⁵, the *SPF HASP*, 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 OSHA 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 an SPF ROM form. The SPF 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.

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 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 significant procedural changes to the soil preparation processes or other SPF governing documents to ensure the new methods are implemented and followed appropriately.

⁵ At the time of this QAPP, this work plan is currently being updated.

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.

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 report is to identify SPF performance problems and isolate the potential causes.

B5.2.4 Preparation QC Samples

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

Sand Blank

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. 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 a 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 in the drying blank, 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 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 any asbestos is detected by PLM-VE in the grinding blank (i.e., result is not Bin A), a qualifier of "GB" is 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 is 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 are 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 evaluated based on a comparison of the reported PLM-VE

bin for the parent field sample and preparation duplicate sample. Because preparation duplicate samples may have inherent small-scale variability that is random and may be either small or large, there is no quantitative requirement for the agreement of preparation duplicates. Rather, results are used to determine the magnitude of this variability to evaluate data usability. The QATS contractor will notify the SPF QAM when preparation duplicate results are different from the parent results 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 performance evaluation (PE) standards to evaluate laboratory accuracy and precision. These PE standards are kept in storage at the Troy SPF and are inserted into the sample train in accordance with SOP ISSI-LIBBY-01, with the following project-specific modification:

- PE standards will not be processed prior to insertion (i.e., no sieving or grinding of the standard will be performed).

PE standards of varying nominal levels will be inserted on a quarterly basis at a rate of at least one PE standard per analytical 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 a 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, and the nominal level of the PE standard 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 will be evaluated based on the nominal concentration of the PE standard. The LC will 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 results reporting. Examples of corrective actions that may be taken include reanalysis and/or re-preparation, 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 investigation program will be provided a copy of and will adhere to the requirements of this QAPP. Samples collected under this 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 analytical guidance documents, or this QAPP, will be recorded on the Request for Modification to Laboratory Activities or Request for Modification to Soil Sample Preparation Activities form as appropriate. Deviations that impact, or have the potential to impact, investigation objectives will be discussed with the OU4 EPA Remedial Project Manager and A&E FTL prior to implementation. In addition, the appropriate record of modification form will be used to document information of interest as requested by the EPA. As modifications are approved by the EPA and implemented, the EPA LC will communicate the changes to the EPA laboratories. Sample results data will be delivered to the EPA in accordance with the *EPA Data Management Plan for the Libby Asbestos Superfund Site* (EPA 2013).

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 Site. They also provide information on the availability of sufficient laboratory capacity to meet potential testing needs associated with the Site.

External Audits

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 report 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 will 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

There are two different types of microscopy techniques that may be utilized to analyze GPI samples for asbestos –TEM and PLM. The most recent versions of all referenced analysis methods and SOPs are available in the Libby Lab eRoom.

The following sections summarize project-specific QA/QC requirements. The analytical methods should be consulted for detailed descriptions of method-required QA/QC measures.

B5.3.4.1 Laboratory QC for TEM

General Procedures

Laboratory-based QA/QC for TEM is based on satisfactory performance covered by the requirements in the National Institute of Standards and Technology (NIST) National Voluntary Laboratory Accreditation Program (NVLAP). This laboratory accreditation signifies the competency of a laboratory to provide testing services. The third-party accreditation complies with the standards published by ISO and the International Electro-technical Commission (IEC), specifically ISO/IEC 17025.

The NVLAP program reviews management and technical requirements pertaining to quality systems, personnel, facilities, test and calibration methods, equipment, measurement traceability, sampling, handling of test and calibration items, and reporting (NIST 2006a,b). Laboratories are required to pass TEM NVLAP accreditation every 2 years. In addition, TEM laboratories are required to participate in proficiency testing every 6 months. Unsatisfactory performance due to non-participation in regularly scheduled proficiency test rounds or unresolved technical nonconformities can subject a laboratory to denial or suspension of their accreditation and subsequent suspension on the Libby project. It is the responsibility of the TEM laboratory manager to provide current copies of NVLAP certifications to the LC for the contract files.

Site-Specific Procedures

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 reparations. 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 is completed. Inter-laboratory analyses for TEM will be selected post hoc by the QATS contractor or their designate 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.

B5.3.4.2 Laboratory QC for PLM-9002

General Procedures

Laboratory-based QA/QC for PLM is maintained by following the requirements specified in NIOSH Method 9002 and through satisfactory completion of the requirements specified by NVLAP for PLM. Laboratories are required to pass PLM NVLAP accreditation every 2 years. In addition, PLM laboratories are required to participate in proficiency testing every 6 months. Unsatisfactory performance due to non-participation in regularly scheduled proficiency test

rounds or unresolved technical nonconformities can subject a laboratory to denial or suspension of their accreditation and subsequent suspension on the Libby project. It is the responsibility of the PLM laboratory manager to provide current copies of NVLAP certifications to the LC for the contract files.

NVLAP requirements include monthly checks of the refractive index liquids, daily microscope adjustments, USGS standards, and evaluations of various blanks to check for contamination. Overall QC analysis should be at a rate of at least 10%, including inter- and intra-analyst laboratory duplicates, laboratory blanks, and inter-laboratory analyses. It is the responsibility of the laboratory QAM to ensure that these QA/QC assessments are conducted at the specified frequency and that results are recorded in the laboratory logbooks. Copies of all QA/QC assessment results are submitted directly to the LC by the laboratory manager. Results for laboratory QC analyses should also be included (and clearly identified as QC) in the electronic deliverable for each laboratory job. It is the responsibility of the LC to notify the QATS contractor of any issues identified during these QA/QC assessments.

Site-specific Procedures

Additional Site-specific QA/QC requirements for NIOSH Method 9002 have not been established.

B5.3.4.3 Laboratory QC for PLM-VE and PLM-Grav

Laboratory QA/QC for PLM-Grav is ensured through compliance with laboratory-based QA/QC requirements for the NIOSH Method 9002, as specified by NVLAP. No additional project-specific QA/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 be 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., sampling shovels, ladders, GPS units) will be maintained in basic accordance with manufacturer specifications. Maintenance and calibration of equipment shall be done in accordance with EPA-LIBBY-2012-03 and/or CDM-LIBBY-09 as included in **Appendix B**. 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 FTL 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 allow timely repair of “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. Specifics regarding maintenance and calibration of equipment are detailed in ISSI-LIBBY-01, EPA-LIBBY-08, SRC-LIBBY-01, SRC-LIBBY-03, and SRC-LIBBY-05, as provided in **Appendix B**. 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 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 GPI TL or FTL will check the field equipment/supply inventory and procure additional equipment and supplies that are needed. The GPI TL or FTL will also check that in-house measurement and test equipment used to collect data/samples as part of this QAPP is in good, working order, and procured equipment is acceptance tested prior to use (according to SOP EPA-LIBBY-2012-03, *Control and Measurement and Test Equipment*, **Appendix B**). Items that the GPI TL or FTL deems unacceptable will be removed from inventory and repaired or replaced as necessary. The inventory and procurement of equipment and supplies is discussed in detail in Section B2.1.3.

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 blank samples, as described in Section B5.

B9. Non-direct Measurements

As mentioned previously, the EPA has conducted previous investigations at the Site to evaluate the nature and extent of LA and LA source materials at OU4 properties. As part of these studies, LA has been measured in dust and soil. The dust and soil sample results from the GPI program may be compared to existing and future Libby data sets for these environmental media, as discussed within this section.

The Field Data Manager will run RSQ to evaluate existing data against the action levels (also referred to as triggers in this document) presented in the *Libby Asbestos Site Residential/Commercial Cleanup Action Level and Clearance Criteria Technical Memorandum* (EPA 2003) and its amendments (EPA 2011 and 2014). Action levels pertaining to flowerpots, as discussed in the flowerpot protocol memorandum (CDM Smith 2013b), will also be considered. Those queries will be used, to the extent possible, to automatically assign a property status indicating whether action is required (additional investigation or removal) at a property. As needed, the POC will conduct manual reviews of existing property data and update property statuses accordingly. The status of a property, in terms of removal actions required, will be based on the decision criteria as presented in **Table A-3 (Appendix A)**. Manual property data review and status verification/updates are documented on the PSEF completed for each property and retained with the hard copy property folder at the A&E's project office. If one or more primary triggers are present at the property, that property information is transmitted to the GPI FTL to prepare for the DI.

Prior to sending an investigation team to a property, the GPI FTL, or designee, will review all previously collected data to assess whether that data can be used to meet the GPI objectives described in **Appendix A**. For the purposes of this QAPP, only data that is both relevant (i.e., still physically present at the property) and has not been superseded (e.g., CSS results will not be considered if SI or DI results are available for that area) will be evaluated. Sample-by-sample relevancy and adequacy data will be accessed by the data reviewer via Scribe reporting (see Sections B10.4 and B10.5 below). Previously collected data may include Phase 1, Contaminant Screening Study (CSS), Pre-Design Inspection (PDI) and/or SI data (including bulk material, soil, and dust sample results), and data collected during an Environmental Resource Specialist initial property visit. All previously collected data were collected under the following EPA-approved SAPs/QAPPs:

- Phase 1 Sampling and Quality Assurance Project Plan, Revision 1 (EPA 2000)
- Final Sampling and Analysis Plan, Remedial Investigation Contaminant Screening Study (CDM Smith 2002)
- Final Sampling and Analysis Plan, Remedial Investigation Contaminant Screening Study, Revision 1 (CDM Smith 2003a)
- Final Sampling and Analysis Plan, Remedial Investigation (CDM Smith 2003b)
- Draft Final Pre-design Inspection Activities Work Plan (CDM Smith 2003c)

Below is a list of previously collected data items and the conditions under which investigation-specific data may be used to meet GPI objectives. Supporting documentation (e.g., logbook notes, FSDSs, photos, field sketches) is also reviewed, but not used independently of the data discussed below.

- Analytical data report:
 - Soil sample results for areas that have been previously characterized and meet the minimum point inspection/aliquot frequency, as stated in **Table B-3**, for the type of investigation they are undergoing (SI or DI) may be used (however, areas should be re-sampled if conditions have changed significantly since the initial sample collection)
 - Results for previously collected dust samples will be used as long as the target analytical sensitivity was achieved
- Primary Structure and Property Assessment Information Field Form:
 - Interior living space and attic inspection information can be used as long there is no contradictory information in other documentation (e.g., IFF says no vermiculite in the attic, logbook says vermiculite was observed in the attic)
 - Understructure inspection information is insufficient for earthen understructures as samples were not collected (if no exposed soil, IFF documentation is sufficient)
 - Attic and understructure inspection information is insufficient if IFF documents no access to these areas when there is currently means to access them
- Exterior Inspection Checklist (EIC):
 - All data will be used unless exterior property conditions have changed
- Supplemental Exterior Inspection Checklist and Visual Vermiculite Estimation Form:
 - All data will be used unless exterior property conditions have changed
 - Visible vermiculite data may only be used when paired with a sample result that meets the criterion above.
- Supplemental Interior Inspection Checklist (SIIC):
 - All data will be used unless building conditions have changed

Section B2.3.6 provides guidance on which areas require sampling or re-sampling as a result of data gaps in, or non-achievement of DQOs for, previously collected data.

In many cases, dust samples collected during previous investigations were not analyzed but were archived for potential future analysis. The investigation team will coordinate with the

A&E sample coordinator to ensure that any necessary dust samples are retrieved from archive and analyzed.

If an OIF has not yet been completed with the property owner, the DI team will conduct a verbal interview during the property visit and complete the OIF in accordance with Section B2.2.3.

Data users will utilize the appropriate project databases to access data for evaluation. See Sections B10.4 and B10.5 for additional information on project databases and data reporting. Only those data that have undergone data verification and validation (see Section D2) and been evaluated with regard to data usability (see Section D3) will be utilized for the purposes of making comparisons.

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 2013).

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&E 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 the A&E and resides on the local server in the project 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. 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 sample preparation information. Soil 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 soil 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 (Libby-specific EDD and LADT) developed specifically for the Libby project to ensure consistency between laboratories in the presentation and submittal of analytical data. Once the analytical laboratory has generated an EDD with results, the spreadsheet(s) are uploaded to a local FTP site maintained by the ESAT project data manager.

Additionally, EDDs may be transmitted to email recipients as specified by the ESAT LC.

The ESAT Project Data Manager utilizes a local analytical Scribe project database (i.e., LibbyLab2014.mdb) to maintain analytical results information by calendar year. The EDDs are uploaded directly into the analytical Scribe project database. 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) and an analytical results Scribe project (i.e., LibbyLab2014.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 website⁶. The data user must then

⁶ http://www.ertsupport.org/scribe_home.htm

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 field and lab projects 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” 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 data queries and standard reports to ensure that they are accurate, complete, and representative. If issues are identified by the data user, they will be reported to the EPA Region 8 Data Manager or their designate for resolution using Google Docs. 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.

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

Performance assessments are quantitative checks on the quality of a measurement system and are appropriate to analytical work. Performance assessments for the laboratories may be accomplished by submitting blind reference material (i.e., performance evaluation samples). These assessment samples are samples with known concentrations that are submitted to the laboratories without identifying them as such to the laboratories. Performance assessments will be coordinated by the EPA or the QATS contractor.

System assessments (e.g., audits, surveillances) are qualitative checks of different aspects of project work for the use of appropriate QC measures, compliance with specified procedures, and the general function of the QA system. USACE is not required to conduct a formal system assessment for the GPI program. Rather, USACE QA will focus on field oversight and quality control checks of specific work products generated by the A&E, as deemed necessary by the USACE Project Manager or onsite USACE representative. Minor issues noted by the USACE during field oversight or work product review will be reported real-time to the GPI FTL, who is responsible for resolving the issue and/or recommending/implementing process improvements to prevent the issue from recurring. Major issues will be reported to the EPA and the A&E's Project Manager immediately, and a plan for resolution put in place.

Due to the level of effort and the duration of the activities discussed in this QAPP, the A&E will conduct an internal field audit annually for GPI work. In addition, an internal project office audit was conducted in September 2013 to assess compliance of the A&E's project filing system with this QAPP and contract requirements. No deficiencies were noted and the audit report was provided to the USACE Project Manager. An office audit will not be performed during the 2014 field season. Field audits will be performed under the direction of the A&E's QAM, with support from the A&E's QA Coordinator.

During an audit, the auditor will examine activities and documentation to assess whether activities are in conformance with the appropriate the QAPP, work plan, and other governing documents. The auditor will document all audit results, and will maintain a list of personnel contacted during the audit. At the completion of the audit, the auditor will hold a conference to present the preliminary results of the audit and to encourage rapid correction of any deficiencies. The audit report will detail both proficiencies and deficiencies, and will include any corrective action (and supporting documentation) that was taken to correct the problem.

Because field audits may identify deficiencies that impact (or have the potential to impact) project or data quality objectives, field audits are performed early in the field season. It is anticipated that the following field activities will be assessed during the 2014 field audit: field measurements; visual vermiculite inspections; sample collection, handling, and shipping; field documentation; equipment decontamination; and maintenance of applicable governing documents in the field. A copy of the audit report will be available upon request.

The internal office systems audit, which involves an assessment of the project file for compliance with the EPA Data Management Plan (EPA 2013), this QAPP, and the *CDM Federal Programs Corporation Quality Assurance Manual* (CDM Smith 2012a) specified in CDM Smith's USACE contract, was conducted in September 2013. An office audit will not be conducted during the 2014 field season.

Project technical/performance assessments are discussed in Sections A8.2, B5.2.3, B5.2.5, and B5.3.3.

C1.2 Response Actions

Corrective actions will be required if there are any unresolved deficiencies found in conformance with the QAPP and other governing documents. In this case, the auditor, through a corrective action request (CAR), will request the audited party to take corrective action. When evidence is received that acceptable corrective action has been completed, the A&E's QAM will issue an audit completion notice to formally close out the audit. The audit completion notice will be distributed to the recipients of the audit report. The A&E's QAM will identify the person responsible for implementing corrective action (often the Project Manager), set a date on which the response is due, and distribute the CAR. The QAM will review the CAR response to evaluate the adequacy of the corrective action. If the stated corrective action appears appropriate, the A&E's QAM will examine the objective evidence that the corrective action has been completed. If the evidence provided to the QAM is acceptable, the QAM will sign and date the form. Corrective actions will be implemented on a case-by-case basis to address quality problems. The A&E Project Manager will notify the USACE of corrective actions taken to address deficiencies noted by A&E personnel. This notification will be included in the monthly Task Order Report provided to the USACE, as mentioned in Section C2 below.

As lead QA role, USACE performs informal and/or formal assessments of GPI. For instance, major deficiencies noted during routine oversight result in appropriate corrective action. If contractual or QAPP deficiencies are identified by onsite USACE personnel, these will be reported to the USACE COR immediately. The USACE COR, with assistance from the USACE QAM, may generate a Non-Conformance Report and Corrective Action Report requiring the A&E Project Manager to take action to address the non-conformance.

C2. Reports to Management

No regularly-scheduled written QA reports to management are planned as part of this sampling program. However, QA reports will be provided by the A&E's QA Coordinator to the A&E's Program Manager and Project Manager for routine audits and whenever quality problems are encountered. USACE will be notified in the weekly work in advance of the audit and audit report writing. The A&E's Program or Project Manager may provide these reports to, or discuss the quality issues with, the EPA RPM and/or USACE Project Manager. Weekly reports and change request forms are not required for work performed under this QAPP. The A&E Project Manager includes a brief summary of GPI progress in the monthly Task Order Report provided to the USACE Project Manager. This monthly report is provided to the EPA by USACE.

Facilitated by shared workspace, there is constant real-time discussion about GPI activities between the GPI FTL, GPI TL, and USACE onsite personnel. This verbal reporting includes progress updates and minor issue resolution. Onsite USACE personnel document pertinent information from those discussions in the Daily Significant Report to the USACE Project Manager, the EPA RPM, and the EPA Onsite RPM/FTL.

D1. Data Review, Verification and Validation

D1.1 Data Review

Data review of 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 issues or discrepancies are found in asbestos data, the data reviewer will contact the EPA Region 8 Data Manager (Jeffrey Mosal), who will then notify the appropriate party in order to correct the issue.

D2. Verification and Validation Methods

D2.1 Data Verification

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 database. The goal of data verification is to identify and correct data reporting errors.

For analytical laboratories that utilize the Libby-specific EDD spreadsheets for asbestos data reporting, 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 non-investigative Libby samples (i.e., samples that are not directly used in the risk assessment to make risk assessment decisions). This data verification process utilizes Site-specific SOPs⁷ developed to ensure analytical results and field sample information in the project database is accurate and reliable.

The data verification review ensures that data reporting issues are identified and rectified to limit the 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 A&E staff 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. It is the responsibility of the project database 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 database manager is also responsible for electronically tracking in the project database which data have been verified, who performed the verification, and when.

⁷ Site-specific field sample information and data review/data entry verification SOPs are available on the EPA Libby document website (<http://www2.epa.gov/region8/libby-site-documents>).

D2.2 Data Validation

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 for asbestos in air and soil will be validated by the QATS contractor (CB&I) in accordance with the applicable method, investigation-specific Analytical Requirements Summaries, laboratory ROMs, and Libby-specific data validation SOPs developed by CB&I, which include SOP QATS-70-094 (*Validation of PLM Data Deliverables*), SOP QATS-70-095 (*Validation of Libby TEM Data Deliverables*), and SOP QATS-70-096 (*Validation of PCM Data Deliverables*). Criteria that will be evaluated include sample receipt, sample preparation, microscope alignment, instrument calibrations, stopping rules, structure recording and identification, blank analysis (if applicable), recount/repreparation analysis (if applicable), and overall assessment of data. A total of 5% of sample results are selected annually by CB&I for validation by randomly choosing sample results to be representative of each laboratory, analytical method, and media type. A comprehensive data validation effort will be completed annually by the QATS contractor and results will be reported in a yearly data validation report. This report shall detail the validation procedures performed and provide a narrative on the quality assessment for all analytical methods, including a summary of any data qualifiers that are to be added to the project database to denote when results do not meet project-specific acceptance criteria, and shall detail any deficiencies and required corrective actions stemming from the data validation review. Results of the data validation will be summarized in an addendum to the *Quality Assurance and Quality Control Summary Report for the Libby Asbestos Superfund Site* (CDM Smith 2012b; 2014). This addendum will also include recommendations for Site QA/QC program changes to address any data quality issues. For OU4 data reviews, provide a summary of the records that have been validated (AnalysisID and SampNo), the date they were validated, any recommended data qualifiers, and their associated reason codes to the ESAT Region 8 Data Manager. 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.

D3. Reconciliation with User Requirements

Once all samples from a specific property have been collected and analytical data has been generated, data will be reviewed to evaluate whether investigation objectives were achieved. This is typically performed by the A&E's FTL (or other designated investigation staff) whose responsibility it is ensure reported investigation results are adequate and appropriate for their intended use. To the extent possible, this data usability assessment will utilize results of any data verification and data validation efforts to provide information on overall data quality specific to each investigation.

The data usability assessment will evaluate results with regard to several data usability indicators, including precision, accuracy/ bias, representativeness, comparability, completeness, and whether specified analytic requirements (e.g., sensitivity) were achieved.

Table D-1 provides detailed information for how each of these indicators may be evaluated for the reported asbestos data. The data usability assessment results and conclusions will 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.

Table D-1: General Evaluation Methods for Assessing Asbestos Data Usability

Data Usability Indicator	General Evaluation Method
Precision	<p><u>Sampling</u> – Review results for co-located samples and field duplicates to provide information on variability arising from medium spatial heterogeneity and sampling and analysis methods.</p> <p><u>Analysis</u> – Review results for PLM laboratory duplicates, TEM recounts, and TEM reparations 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.</p>
Accuracy/Bias	<p><u>TEM</u> – Calculate the background filter loading rate and use results to assign detect/non-detect in basic accordance with ASTM 6620-00.</p> <p><u>PLM</u> – 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.</p>
Representativeness	Review relevant audit report findings and any 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 (e.g., 99 of 100 samples, 99%).
Sensitivity	<u>TEM</u> – 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

SOP - standard operating procedure

ROM - record of modification

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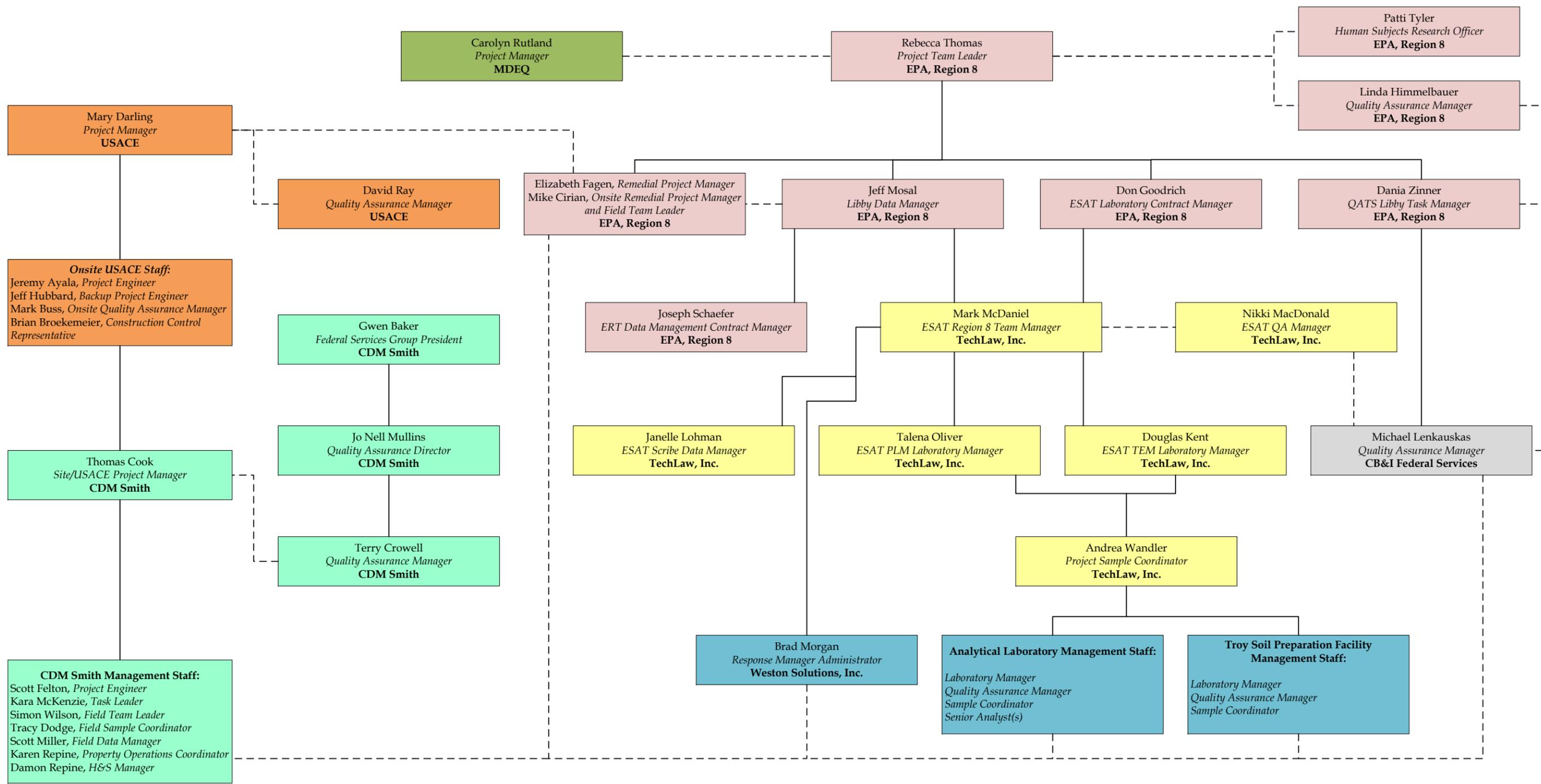
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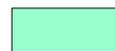
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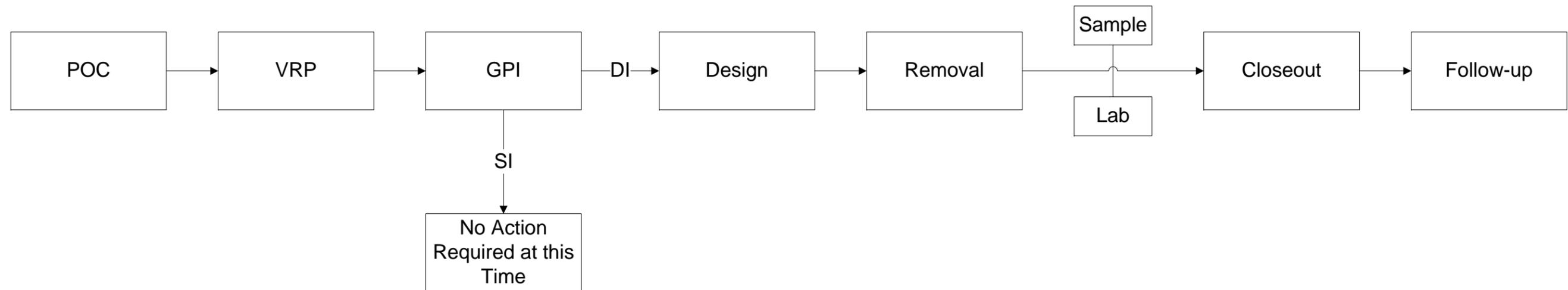
 EPA Region 8 Staff
 USACE Staff
 MDEQ Staff

 CDM Smith Staff
 TechLaw Staff
 TechLaw Subcontractors

 CB&I Staff
 Lines of authority
 Lines of communication

Figure A-1
 Organizational Chart for GPI
 Libby Asbestos Site
 Lincoln County, Montana


Revised 03/27/14



Acronyms:

DI detailed investigation
 GPI general property investigation
 POC Property Operations Coordinator
 SI screening investigation
 VRP Voluntary Recruitment Program

Figure A-2
Process Overview Libby Asbestos Site Lincoln County, Montana
CDM Smith

Figure B-1
Site Location Map
Libby Asbestos Site
Lincoln County, Montana

 Approximate Site Boundary

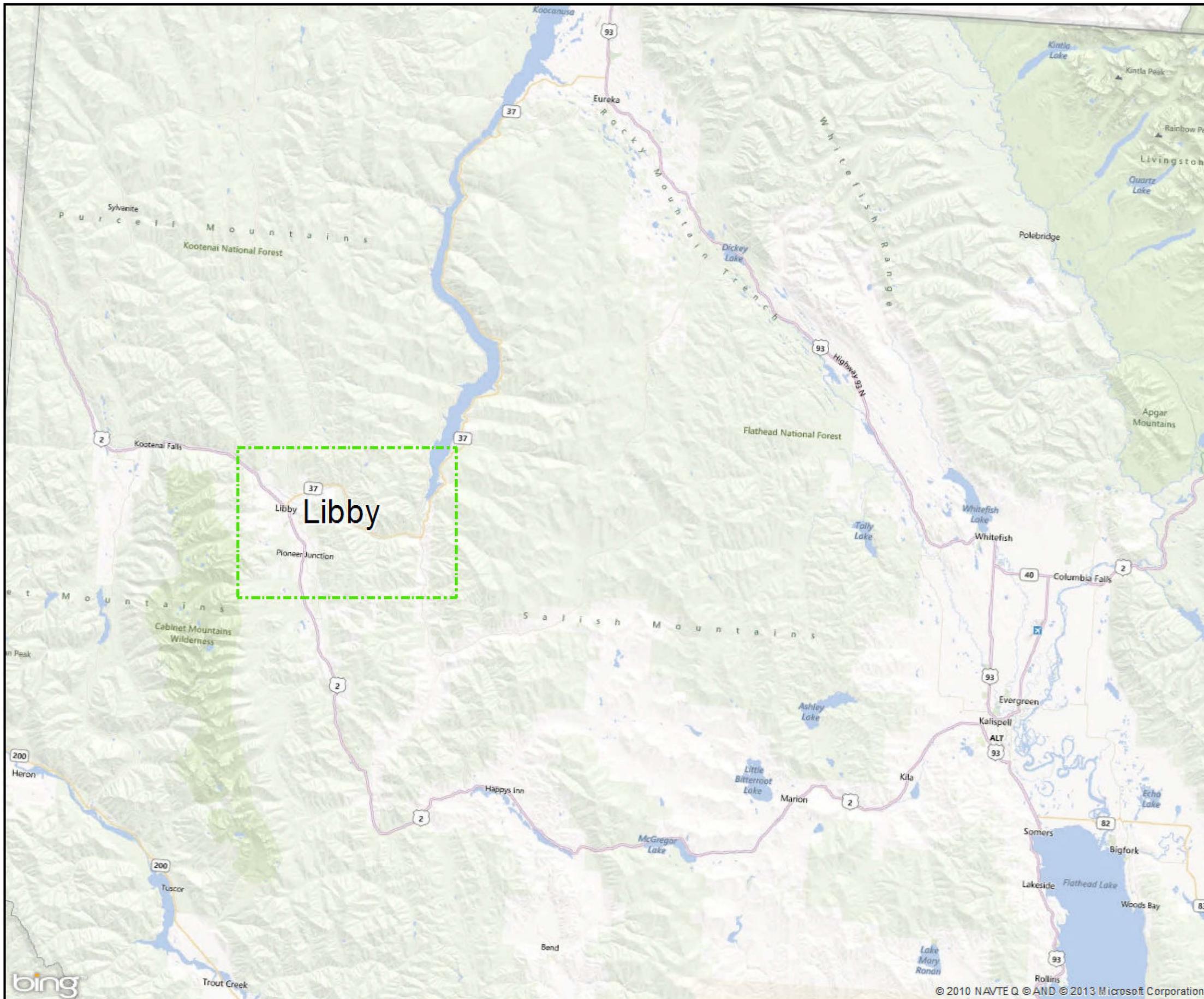
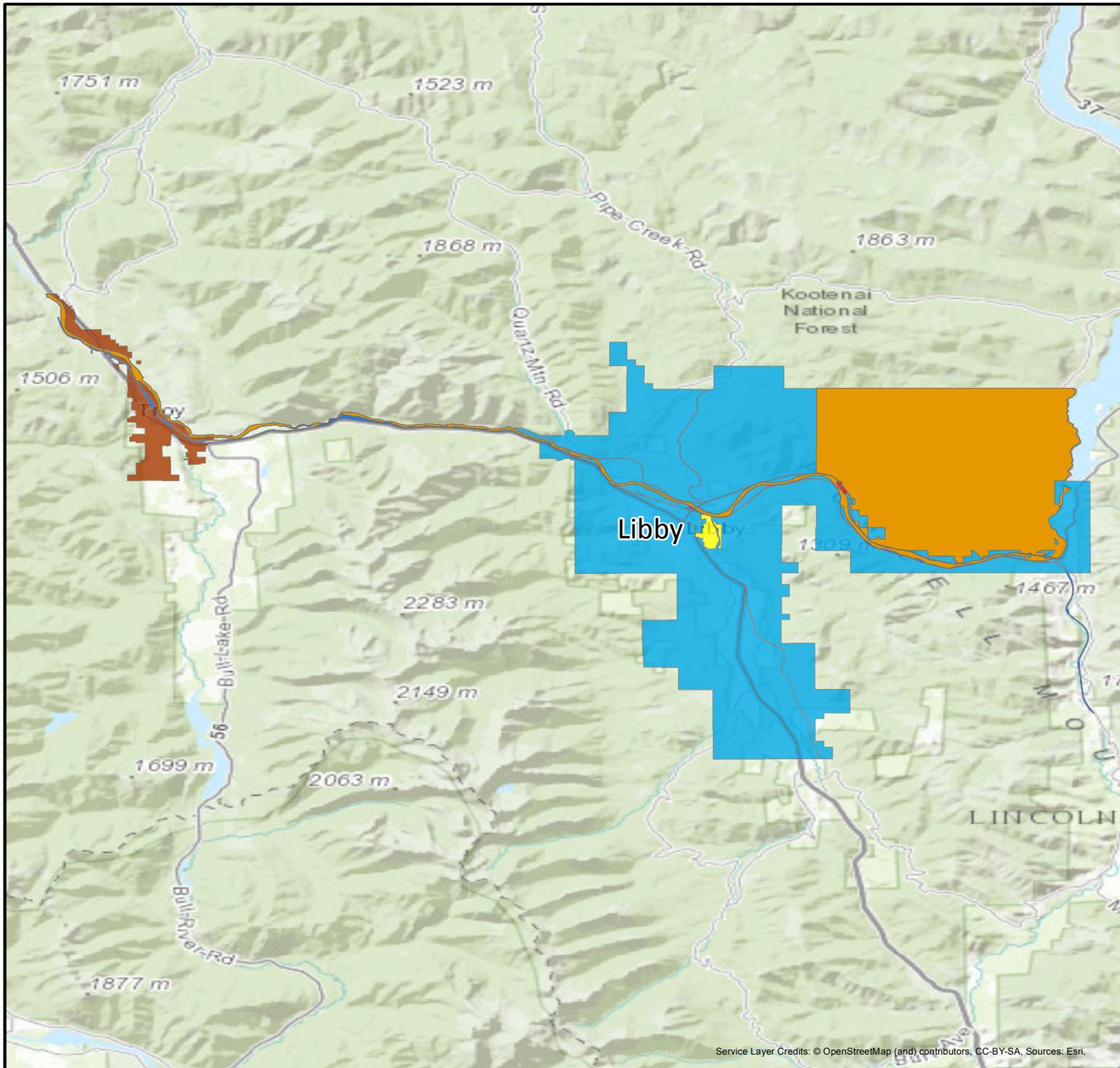


Figure B-2
 Operable Unit Map
 Libby Asbestos Site
 Lincoln County, Montana

- OU 1 - Former Export Plant
- OU 2 - Former Screening Plant
- OU 3 - Mine and Kootenai River
- OU 4 - Libby
- OU 5 - Former Stimson Lumber
- OU 6 - BNSF Rail Corridor
- OU 7 - Troy
- OU 8 - State Highway Corridors



Service Layer Credits: © OpenStreetMap (and) contributors, CC-BY-SA, Sources: Esri,

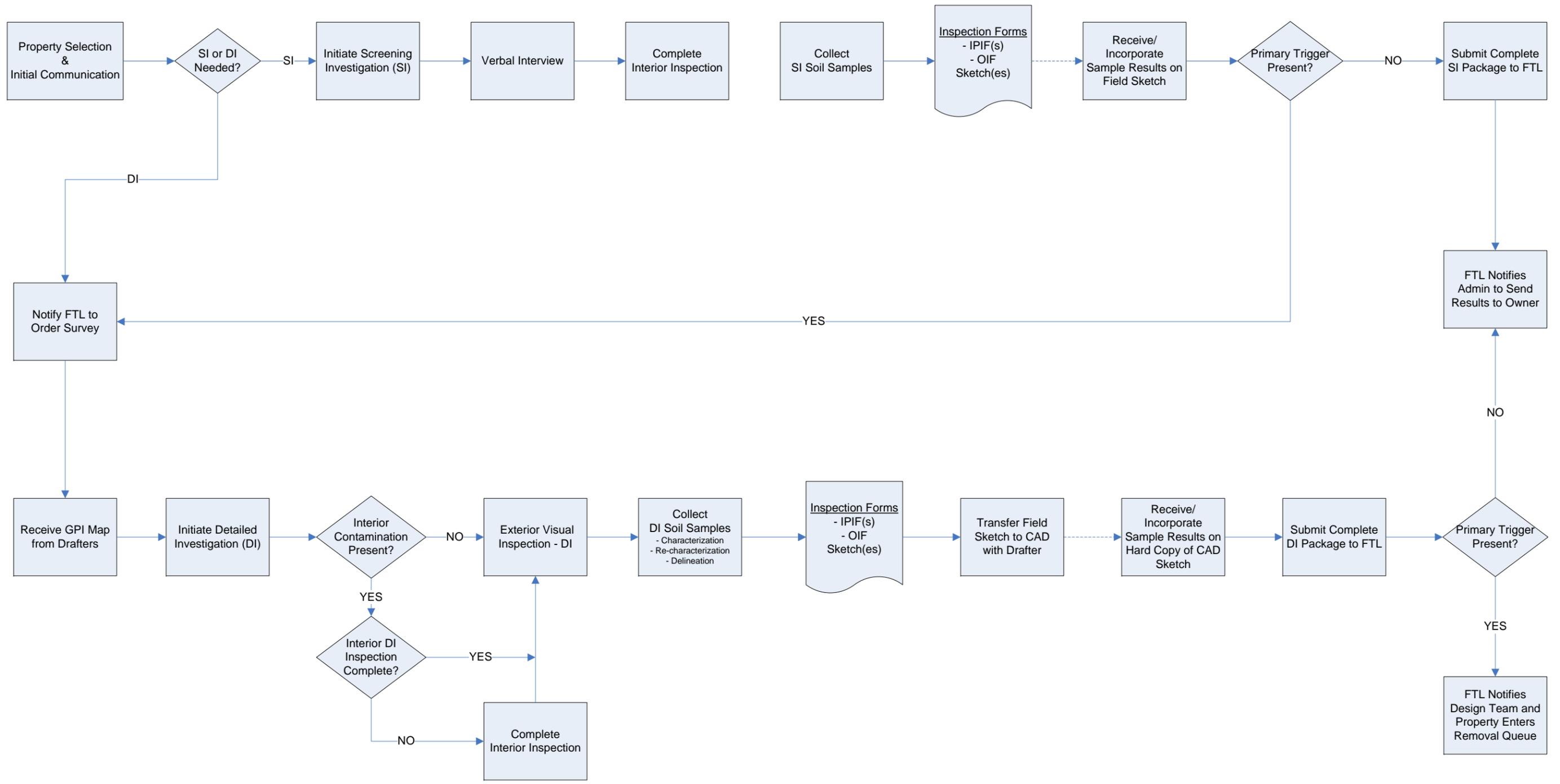


Figure B-3
 Property Investigation Process
 Libby Asbestos Site
 Lincoln County, Montana

APPENDIX A
Detailed Data Quality Objectives

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Appendix A

Data Quality Objectives for General Property Investigation

The DQO process, based on scientific methods, is a series of planning steps that are designed to ensure that the type, quantity, and quality of environmental data used in decision-making are appropriate for the intended purpose. The DQOs presented in this section were developed in accordance with EPA guidance (EPA 2006).

The DQO process specifies project decisions, the data quality required to support those decisions, specific data types needed, data collection requirements, and analytical techniques necessary to generate the specified data quality. The process also ensures that the resources required to generate the data are justified. The DQO process consists of seven steps; output from each step influences the choices that will be made later in the process. These steps include:

1. State the problem
2. Identify the decision
3. Identify the inputs to the decision
4. Define the study boundaries
5. Develop a decision rule
6. Specify tolerable limits on decision errors
7. Optimize the design

A.1 Step 1 - State the Problem

The purpose of this step is to describe the problem to be studied so that the focus of the investigation will be unambiguous.

Several property investigation field efforts have been completed for the Site, including the Phase 1 and Phase 2 investigations and the CSS. A number of properties were not inspected during these investigations due to various circumstances including refusal of access, inability to contact property owners, and incomplete county parcel information. Consequently, there are a number of properties where it is unknown whether LA contamination exists. In addition, some historical investigations were performed using techniques that differ from current site investigation protocols (e.g., 5-point composite sampling, high-traffic area visual inspections, semi-quantitative estimation of vermiculite).

This GPI QAPP was developed to reflect current investigation and sampling methods. The GPI process is divided into two distinct phases; SI and DI. SIs are intended to screen properties for

the presence of LA or LA source materials, while DIs are intended to delineate contamination at properties where one or more primary trigger was previously identified. Exceptions to these general guidelines may be made at the direction of EPA and USACE. The SI and DI phases will be performed at separate times. The overall GPI sampling program described in this QAPP is designed to:

- Evaluate whether LA and/or LA source materials are present at residential, commercial, industrial, or public properties within OU4 and OU7.
- Evaluate the extent of LA contamination on each property if LA or LA source materials are present.

Section B.2 of this QAPP describes the sampling and inspection procedures that will be used to collect data of sufficient quality and representativeness to evaluate each of these items.

In all subsequent sections, common use, specific use, secondary structures, and secondary buildings are collectively referred to as the outdoor living area. All principal study questions will be evaluated on a per property (residence) basis.

A.2 Step 2 - Identify the Decision

This step identifies what questions the investigation will attempt to resolve and what actions may result. The principal study questions and possible alternative actions are as follows:

Table A-1 Decision Statements

Response Item Evaluated	Principal Study Question	Alternative Actions
<i>Screening Investigation</i>		
Evaluate whether LA or LA source materials are present on individual properties.	Is open, non-contained, or migrating vermiculite containing material present in buildings?	<ul style="list-style-type: none"> ▪ Sketch and document location and extent of vermiculite for removal action ▪ Take no action
	Is LA detected at concentrations $\geq 5,000$ s/cm ² in indoor dust from any one previously collected dust sample?	<ul style="list-style-type: none"> ▪ Clearly note which building(s) contains the elevated dust concentration(s) on the exterior field sketch ▪ Take no action
	Is LA detected at levels >TR in any surface soil samples?	<ul style="list-style-type: none"> ▪ Document location of LA-contaminated surface soils and prepare for DI phase ▪ Take no action
	Is TR LA detected in areas with VV covering >25% of the outdoor living area?	<ul style="list-style-type: none"> ▪ Document location of co-located LA- and VV-contaminated surface soils and prepare for DI phase ▪ Take no action
	Is LA detected in friable VCBM (e.g., plaster)?	<ul style="list-style-type: none"> ▪ Sketch and document location and extent of LA-contaminated friable building materials for possible removal if action is required at the property based on a primary trigger ▪ Take no action

Response Item Evaluated	Principal Study Question	Alternative Actions
Detailed Investigation Evaluate the extent of potential LA contamination on individual properties if LA or LA source materials are present.	Is open, non-contained, or migrating vermiculite containing material present in buildings?	<ul style="list-style-type: none"> ▪ Sketch and document location and extent of vermiculite for removal action ▪ Take no action
	Is LA detected at concentrations $\geq 5,000$ s/cm ² in indoor dust from any one previously collected dust sample?	<ul style="list-style-type: none"> ▪ Clearly note which building(s) contains the elevated dust concentration(s) on the exterior field sketch ▪ Take no action
	Is TR LA detected in areas with VV covering >25% of the outdoor living area?	<ul style="list-style-type: none"> ▪ Collect soil samples, sketch and document location and extent of LA- and VV-contaminated surface soil for removal action ▪ Take no action
	Is LA detected in friable VCBM (e.g., plaster)?	<ul style="list-style-type: none"> ▪ Sketch and document location and extent of LA-contaminated friable building materials for possible removal if action is required at the property based on a primary trigger ▪ Take no action

CUA – common-use area

DI – detailed investigation

LA – Libby Amphibole asbestos

LUA – limited-use area

VV – visible vermiculite

s/cm² – structures per square centimeter

SI – screening investigation

SUA – specific-use area

TR – Trace (<0.2%)

VCBM – vermiculite-containing building materials

> – greater than

\geq – greater than or equal to

% – percent

A.3 Step 3 – Identify the Inputs to the Decision

The purpose of this step is to identify the information and measurements that need to be obtained to resolve the decision statements. The information needed to resolve the principal study questions are summarized in Table A-2.

A.4 Step 4 – Define the Boundaries of the Study

This step specifies the spatial and temporal boundaries of this investigation.

A.4.1 Spatial Bounds

The information gathered to answer the objectives will be collected from properties within the boundaries of OU4 and OU7, and other OUs as directed by the EPA and USACE (Figure B-2). The vertical spatial boundaries extend from the highest point at a property, approximately two stories, to the depth of soil samples collected, approximately 6 inches below ground surface.

A.4.2 Temporal Bounds

For each property, the temporal boundaries of this investigation include the time from when an SI begins to the time analytical results indicate LA is not detected above action levels on the property or when a DI is complete.

A.5 Step 5 – Develop Decision Rules

The purpose of this step is to describe the method that the EPA will use to assess whether the data collected indicate acceptance and the resulting decision applied when acceptance is not obtained. The principal study question, inputs to resolve study questions, action levels, and decision rules are summarized in Table A-3.

A.6 Step 6 – Specify Tolerable Limits on Decision Errors

The tolerable limits on decision errors, used to establish performance goals for the data collection design, are specified in this step.

Specific to performing SIs and DIs, two types of decision errors are possible:

- A Type I (false negative) decision error would occur if a risk manager decides that an inspection/sample does not contain vermiculite/LA above a level of concern, when in fact it is of concern.
- A Type II (false positive) decision error would occur if a risk manager decides that an inspection/sample does contain vermiculite/levels of LA above a level of concern, when in fact it does not.

The EPA is most concerned about guarding against the occurrence of Type I errors, since an error of this type may leave humans exposed to unacceptable levels of LA.

The EPA is also concerned with the probability of making Type II decision errors. Although this type of decision error does not result in unacceptable human exposure, it may result in unnecessary expenditure of resources. Generally, the EPA allows for a 20 percent false positive rate.

For the purposes of completing all seven steps of the DQO process, the null hypotheses and consequences of making an incorrect decision are summarized in Table A-4. However, the gray region and tolerable limits on decision errors are not proposed because they are not applicable in this case.

Typically, Step 6 of the DQO process is useful to encourage careful design of decision rules by defining and integrating the errors that are acceptable based upon a myriad of integrated project management decisions such as reduction in risk to human health, implementability/practicability, and cost. As stated in the guidance document for development of DQOs: QA/G-4 (EPA 2006), solely statistically generated tolerable limits on decision errors are not necessary in certain cases provided that a line of reasoning (scientific justification) is presented that adequately defines acceptable limits or decision errors. This particular effort was put forth in the Action Level/Clearance Criteria Technical Memorandum (EPA 2003) and its amendments (EPA 2011 and 2014) for DQOs for the following sampling and inspection: (1) vermiculite in property structures/buildings; (2) surface soil samples; and (3) indoor dust samples.

A.7 Step 7 – Optimize the Design for Obtaining Data

This step identifies a resource-effective data collection design for generating data that are expected to satisfy the DQOs. The data collection design is described in detail in the remaining sections of this QAPP and other site documents referenced in Section B.

Referencing the *Action Level/Clearance Criteria Technical Memorandum* (EPA 2003) and its amendments (EPA 2011 and 2014) and data previously generated for the site, the DQOs have been designed to support the proposed SI and DI activities and represent the best possible project planning effort. However, in implementing the requirements contained in this QAPP, unforeseen situations may arise or team members may find more efficient means to carry out some of the day-to-day activities. Therefore, team members are always afforded the opportunity to recommend optimization of the data gathering design. Recommendations must come through proper channels (i.e., through the TL or FTL) and documented using either a Record of Modification to Documents Governing Field Activities form or an addendum to this QAPP. All modifications or addendums must be approved prior to making the proposed changes.

Table A-2 Summary of Inputs to Resolve Study Questions and Use of Information Acquired from Inputs

Principal Study Question	Input to Resolve Question	Use of Input to Resolve Question
Screening Investigation		
Is open, non-contained, or migrating vermiculite containing material present in buildings?	Visual Inspection	For each property undergoing a screening investigation, a visual inspection will be performed within each building on the property that has not undergone an interior inspection to current protocol. The results of the visual inspection will be used to evaluate the extent of LA source materials for removal planning.
Is LA detected at concentrations $\geq 5,000$ s/cm ² in indoor dust from any one previously collected dust sample?	Dust Samples	For each property where dust samples were collected during previous investigations, analytical results will be reviewed to evaluate whether LA contamination is present in indoor dust at individual properties for removal planning. Dust samples will not be collected as part of this investigation.
Is LA detected at levels >TR in any surface soil samples?	Soil Samples	For each property undergoing an SI, surface soil samples will be collected from use areas. The results of the surface soil samples will be used to evaluate whether LA contamination is present at individual properties and to assess whether DI activities are required.
Is TR LA detected in areas with VV covering >25% of the outdoor living area?	Visual Inspection / Soil Samples	For each property undergoing an SI, use areas will be visually inspected and soil sampled. The results of the visual inspection and surface soil samples will be used to evaluate whether LA and VV contamination is present at individual properties and to evaluate whether DI activities are required.
Is LA detected in friable VCBM (e.g., plaster)?	Bulk Material Samples	For each property undergoing a screening investigation, a visual inspection will be performed within each building on the property that has not undergone an interior inspection to current protocol. If friable VCBM is observed, bulk material samples will be collected and analyzed. The bulk material results will be used to aid in removal planning if action is required at the property based on a primary trigger.
Detailed Investigation		
Is open, non-contained, or migrating vermiculite containing material present in buildings?	Visual Inspection	For each property undergoing a detailed investigation, a visual inspection will be performed within each building on the property that has not undergone an interior inspection to current protocol. The results of the visual inspection will be used to evaluate the extent of LA source materials for removal planning.

Principal Study Question	Input to Resolve Question	Use of Input to Resolve Question
Is LA detected at concentrations $\geq 5,000$ s/cm ² in indoor dust from any one previously collected dust sample?	Dust Samples	For each property where dust samples were collected during previous investigations, analytical results will be reviewed to evaluate whether LA contamination is present in indoor dust at individual properties for removal planning. Dust samples will not be collected as part of this investigation.
Is LA detected at levels >TR in any surface soil samples?	Soil Samples	For each property undergoing an SI, surface soil samples will be collected from use areas. The results of the surface soil samples will be used to evaluate the extent of LA contamination at individual properties and to assess whether removal is required.
Is TR LA detected in areas with VV covering >25% of the outdoor living area?	Visual Inspection / Soil Samples	For each property undergoing a DI, use areas will be visually inspected and soil sampled if previous inspection and sampling do not meet DI requirements. The results of the visual inspection and surface soil samples will be used to evaluate the extent of LA and VV contamination at individual properties and to assess whether removal is required.
Is LA detected in friable vermiculite-containing building materials (e.g., plaster)?	Bulk Material Samples	For each property undergoing a screening investigation, a visual inspection will be performed within each building on the property that has not undergone an interior inspection to current protocol. If friable vermiculite-containing building materials are observed, bulk material samples will be collected and analyzed. The bulk material results will be used to aid in removal planning if action is required at the property based on a primary trigger.

CUA – common-use area
DI – detailed investigation
LA – Libby Amphibole asbestos

LUA – limited-use area
SI – screening investigation
SUA – specific-use area

TR – Trace (<0.2%)
VV – visible vermiculite
VCBM – vermiculite-containing building materials

Table A-3 Decision Rules

Principal Study Question	Input to Resolve Question	Input Requirements	Action Level	Decision Rule
Screening Investigation				
Is open, non-contained, or migrating vermiculite containing material present in buildings?	Visual Inspection	Presence or absence of vermiculite via visual inspection	Presence of vermiculite	If open, non-contained, or migrating vermiculite is observed, the location will be documented for subsequent removal action. If contained vermiculite is observed, the location will be documented. If vermiculite is not observed, take no action.
Is LA detected in indoor dust from previously collected individual dust samples?	Dust Samples	Analysis: TEM by ASTM D5755 with project-specific modifications Reported Result: s/cm ² AS: 1,000 per cm ²	5,000 s/cm ²	If LA is detected $\geq 5,000$ s/cm ² in any dust sample, the building that the dust sample represents will be clearly identified on the exterior field sketch for subsequent removal action. If LA is detected at levels <5,000 s/cm ² in any dust sample, take no action.

Table A-3 Decision Rules

Principal Study Question	Input to Resolve Question	Input Requirements	Action Level	Decision Rule
Is LA detected at levels >TR in any surface soil samples?	Soil Samples	Analysis: PLM-VE and PLM-Grav with project-specific modifications Reported Result: % LA AS: 0.2%	> TR LA	If levels of LA > TR are detected in surface soil samples, preparation for a DI will begin. If levels of LA ≤TR are detected in surface soil samples, take no action.
Is TR LA detected in areas with VV covering >25% of the outdoor living area?	Visual Inspection / Soil Samples	Presence or absence of VV via visual inspection Analysis: PLM-VE and PLM-Grav with project-specific modifications Reported Result: % LA AS: 0.2%	Presence of VV co-located with TR LA >25% of the outdoor living area	If VV is observed in an area that also has a paired (VV observation and soil sample collection occurred simultaneously) TR LA soil sample result and areas meeting this condition account for 25% of the outdoor living area, preparation for a DI will begin. If VV is absent, take no action. If LA is not detected, take no action. If co-located VV and TR LA account for <25% of the outdoor living area, take no action.
Is LA detected in friable vermiculite-containing building materials (e.g., plaster)?	Bulk Material Samples	Analysis: PLM by NIOSH 9002 Reported Result: % LA AS: Method defined as 1%, but qualitative estimates of LA present below 1% reported as <1% or ND	Any detectable LA	Refer to the Decision Tree for Vermiculite-Containing Building Materials (Appendix F of RA QAPP). If a primary trigger is present at the property, and: <ul style="list-style-type: none"> ▪ If LA is detected in one or more representative bulk material samples at a level of ≥1%, remove friable homogenous VCBM. ▪ If LA is not detected in any representative bulk material sample, take no action. ▪ If LA is detected in one or more representative bulk material samples at a level of <1%, verify results using PLM-PC400. See Verification Analysis input requirements below.
		Verification Analysis: PLM by EPA/600/R-93/116 (400 points) (PLM-PC400) AS: .25%	Any detectable LA	If a primary trigger is present at the property, and: <ul style="list-style-type: none"> ▪ If LA is detected at any level in one or more representative bulk material samples, remove friable homogenous VCBM. ▪ If LA is not detected in any representative bulk material sample, take no action.

Table A-3 Decision Rules

Principal Study Question	Input to Resolve Question	Input Requirements	Action Level	Decision Rule
<i>Detailed Investigation</i>				
Is open, non-contained, or migrating vermiculite containing material present in buildings?	Visual Inspection	Presence or absence of vermiculite insulation via visual inspection	Presence of vermiculite	<p>If open, non-contained, or migrating vermiculite is observed, the location will be documented for subsequent removal action.</p> <p>If contained vermiculite is observed, the location will be documented.</p> <p>If vermiculite is not observed, take no action.</p>
Is LA detected in indoor dust from previously collected individual dust samples?	Dust Samples	<p>Analysis: TEM by ASTM D5755 with project-specific modifications</p> <p>Reported Result: s/cm²</p> <p>AS: 1,000 per cm²</p>	5,000 s/cm ²	<p>If LA is detected $\geq 5,000$ s/cm² in any dust sample, the building that the dust sample represents will be clearly identified on the exterior field sketch for subsequent removal action.</p> <p>If LA is detected at levels $< 5,000$ s/cm² in any dust sample, take no action.</p>
Is LA detected at levels >TR in any surface soil samples?	Soil Samples	<p>Analysis: PLM-VE and PLM-Grav with project-specific modifications</p> <p>Reported Result: % LA</p> <p>AS: 0.2%</p>	> TR LA	<p>If levels of LA > TR are detected in surface soil samples, the location will be documented for subsequent removal action.</p> <p>If levels of LA \leq TR are detected in surface soil samples, take no action.</p>
Is TR LA detected in areas with VV covering > 25% of the outdoor living area?	Visual Inspection / Soil Samples	<p>Presence or absence of VV via visual inspection</p> <p>Analysis: PLM-VE and PLM-Grav with project-specific modifications</p> <p>Reported Result: % LA</p> <p>AS: 0.2%</p>	Presence of VV co-located with TR LA >25% of the outdoor living area	<p>If VV is observed in an area that also has a paired (VV observation and soil sample collection occurred simultaneously) TR LA soil sample result and areas meeting this condition account for 25% of the outdoor living area, the location will be documented for subsequent removal action.</p> <p>If VV is absent, take no action.</p> <p>If LA is not detected, take no action.</p> <p>If co-located VV and TR LA account for <25% of the outdoor living area and no primary exterior trigger is present, take no action.</p> <p>If co-located VV and TR LA account for <25% of the outdoor living area and a primary exterior trigger is present, the location will be documented for subsequent removal action.</p>

Table A-3 Decision Rules

Principal Study Question	Input to Resolve Question	Input Requirements	Action Level	Decision Rule
Is LA detected in friable vermiculite-containing building materials (e.g., plaster)?	Visual Inspection	Analysis: PLM by NIOSH 9002 Reported Result: % LA AS: Method defined as 1%, but qualitative estimates of LA present below 1% reported as <1% or ND	Any detectable LA	Refer to the Decision Tree for Vermiculite-Containing Building Materials (Appendix F of RA QAPP). If a primary trigger is present at the property, and: <ul style="list-style-type: none"> ▪ If LA is detected in one or more representative bulk material samples at a level of ≥1%, remove friable homogenous VCBM. ▪ If LA is not detected in any representative bulk material sample, take no action. If LA is detected in one or more representative bulk material samples at a level of <1%, verify results using PLM-PC400. See Verification Analysis input requirements below.
		Verification Analysis: PLM by EPA/600/R-93/116 (400 points) (PLM-PC400) AS: .25%	Any detectable LA	If a primary trigger is present at the property, and: <ul style="list-style-type: none"> ▪ If LA is detected at any level in one or more representative bulk material samples, remove friable homogenous VCBM. ▪ If LA is not detected in any representative bulk material sample, take no action.

AS – analytical sensitivity
 ASTM – American Society for Testing and Materials
 DI – detailed investigation
 LA – Libby Amphibole asbestos
 ND – none detected

PLM – polarized light microscopy
 s/cm² – structures per square centimeter
 SI – screening investigation
 SUA – specific-use area
 TEM – transmission electron microscopy

TR – Trace (<0.2%)
 VCBM – vermiculite-containing building materials
 VV – visible vermiculite
 % – percent

Table A-4 Limits on Decision Errors

Principal Study Question	Null Hypothesis	Type I Error Will Result in:	Type II Error Will Result in:
Screening Investigation			
Is open, non-contained, or migrating vermiculite containing material present in buildings?	Vermiculite is present in property buildings.	Determining that property buildings do not contain vermiculite when they actually do. This would result in no subsequent removal action and in turn, an increased risk to human health.	Determining that property buildings contain vermiculite when actually they do not. This would result in unnecessarily performing removal action planning and adds to investigation costs.
Is LA detected in indoor dust from previously collected dust samples?	Indoor dust is contaminated with LA.	Determining that indoor dust is not contaminated with LA when it actually is. The LA-contaminated living space would not be included in the removal action and in turn, pose an increased risk to human health.	Determining that indoor dust is contaminated with LA when it actually is not. This would result in unnecessarily including an interior cleaning in the removal action and adds unnecessary costs to the removal.
Is LA detected at levels >TR in any surface soil samples collected from individual properties?	Surface soils are contaminated with LA at levels >TR	Determining that surface soils are not contaminated with LA at levels >TR when they actually are. This may result in no subsequent exterior DI and in turn, an increased risk to human health.	Determining that surface soils are contaminated with LA at levels >TR when they actually are not. This would result in unnecessarily performing an exterior DI and adds to investigation costs.
Is TR LA detected in areas with VV covering >25% of the outdoor living area?	Surface soils are contaminated with TR LA co-located with VV.	Determining that surface soils are not contaminated with co-located TR LA and VV when they actually are. This may result in no subsequent exterior DI and in turn, an increased risk to human health.	Determining that surface soils are contaminated with co-located TR LA and VV when they actually are not. This may result in unnecessarily performing an exterior DI and adds to investigation costs.
Is LA detected in friable vermiculite-containing building materials (e.g., plaster)?	Friable building materials contain LA.	Determining that friable building materials do not contain LA when they actually do. This would result in no subsequent removal action and in turn, a possible increased risk to human health.	Determining that friable building materials contain LA when they actually do not. This may result in unnecessarily removing building materials during a removal action and adds unnecessary costs to the removal.
Detailed Investigation			
Is open, non-contained, or migrating vermiculite containing material present in buildings?	Vermiculite insulation is present in property buildings.	Determining that property buildings do not contain vermiculite insulation when they actually do. This would result in no subsequent removal action and in turn, an increased risk to human health.	Determining that property buildings contain vermiculite insulation when they actually do not. This would result in unnecessarily performing removal action planning and adds to investigation costs.

Principal Study Question	Null Hypothesis	Type I Error Will Result in:	Type II Error Will Result in:
Is LA detected in indoor dust from previously collected dust samples?	Indoor dust is contaminated with LA.	Determining that indoor dust is not contaminated with LA when it actually is. The LA-contaminated living space would not be included in the removal action and in turn, pose an increased risk to human health.	Determining that indoor dust that contains is contaminated with LA when it actually is not. This would result in unnecessarily including an interior cleaning in the removal action and adds unnecessary costs to the removal.
Is LA detected at levels >TR in any surface soil samples collected from individual properties?	Surface soils are contaminated with LA at levels >TR	Determining that surface soils are not contaminated with LA at levels >TR when they actually are. This may result in no subsequent exterior removal and in turn, an increased risk to human health.	Determining that surface soils are contaminated with LA at levels >TR when they actually are not. This would result in unnecessarily including exterior excavation in the removal action and adds unnecessary costs to the removal.
Is TR LA detected in areas with VV covering >25% of the outdoor living area?	Surface soils are contaminated with TR LA co-located with VV.	Determining that surface soils are not contaminated with co-located TR LA and VV when they actually are. This may result in no subsequent exterior removal and in turn, an increased risk to human health.	Determining that surface soils are contaminated with co-located TR LA and VV when they actually are not. This may result in unnecessarily including exterior excavation in the removal action and adds unnecessary costs to the removal.
Is LA detected in friable vermiculite-containing building materials (e.g., plaster)?	Friable building materials contain LA.	Determining that friable building materials do not contain LA when they actually do. This would result in no subsequent removal action and in turn, a possible increased risk to human health.	Determining that friable building materials contain LA when they actually do not. This may result in unnecessarily removing building materials during a removal action and adds unnecessary costs to the removal.

DI – detailed investigation

LA – Libby Amphibole asbestos

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APPENDIX B

Standard Operating Procedures (SOPs)

Panel A: Field SOPs

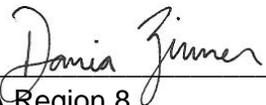
SOP ID	SOP Description
EPA-LIBBY-2012-01	Field Logbook Content and Control
EPA-LIBBY-2012-02	Photographic Documentation of Field Activities
EPA-LIBBY-2012-03	Control of Measurement and Test Equipment
EPA-LIBBY-2012-04	Field Equipment Decontamination
EPA-LIBBY-2012-05	Handling Investigation-derived Waste
EPA-LIBBY-2012-06	Sample Custody
EPA-LIBBY-2012-07	Packaging and Shipping Environmental Samples
CDM-LIBBY-03	Completion of Field Sample Data Sheets
CDM-LIBBY-05	Soil Sample Collection at Residential and Commercial Properties
CDM-LIBBY-06	Semi-Quantitative Visual Estimation of Vermiculite in Soils at Residential and Commercial Properties
CDM-LIBBY-09	GPS Coordinate Collection and File Transfer Process

Panel B: Laboratory SOPs

SOP ID	SOP Description
ISSI-LIBBY-01	Soil Sample Preparation
ER8-LIBBY-01	Libby Chain of Custody Documentation
EPA-LIBBY-08	Indirect Preparation of Samples for TEM Analysis
SRC-LIBBY-01	Analysis of Asbestos in Soil by PLM-Grav
SRC-LIBBY-03	Analysis of Asbestos in Soil by PLM-VE
SRC-LIBBY-05	Collection and Analysis of Asbestos in Indoor Dust

Libby Asbestos Superfund Site Standard Operating Procedure Field Logbook Content and Control

Prepared by:  Date: 7/23/12
CDM Smith

Approved by:  Date: 7/23/12
EPA Region 8

Revision No.	Date	Reason for Revision
0	4/12/12	--
1	7/23/12	To maintain consistency with requirements for completing other field documentation (e.g., field sample data sheets), eliminated the requirement to strike through, initial, and date any self-adhesive labels placed in the logbook.

1.0 Objective

Logbooks are an essential tool to document field activities conducted by the U.S. Environmental Protection Agency or its contractors in support of the Libby Asbestos Superfund Site (Libby Site). The objective of this standard operating procedure (SOP) is to establish baseline requirements, procedures, and responsibilities for the content and control of Libby Site field logbooks. Additions or modifications to this SOP may be detailed in governing documents referencing this SOP.

2.0 Background

2.1 Definitions

Libby Asbestos Superfund Site (Libby Site) – All buildings and land within the boundaries of the EPA's designated operable units (OUs), as illustrated on the most recent version of the OU boundary map.

Ruler or similar scale – Used with a property-specific drawing or plan to measure distance and sizes of objects, buildings, and zones.

Site – All buildings (if applicable) and land within the boundaries of the EPA's designated geounits, which may represent individual properties within the Libby Site, a collection of properties, or a larger geographical area.

2.2 Discussion

Field logbooks are an accounting of observations and/or activities occurring at or associated with the Libby Site. Field logbooks are also used to duly document changes to or deviations from governing documents referencing this SOP. Information recorded in field logbooks includes date/time, site personnel, observations, calculations, weather, locations of field activities, and a description of the field activity, methods, instruments, and results. Additionally, the logbook may contain descriptions of waste, biota, geologic material, and site features including sketches, maps, or drawings as appropriate.

3.0 Responsibilities

Successful execution of this SOP requires a clear hierarchy of assigned roles with different sets of responsibilities associated with each role. All staff responsible for documenting activities in field logbooks will understand and implement the requirements contained herein, as well as any additional requirements stated in governing documents referencing this SOP.

Team Leader (TL) – The TL is responsible for ensuring that the format and content of data entries are in accordance with this procedure. It is also the responsibility of the TL to communicate the need for any changes to/deviations from the SOP with the appropriate personnel, and document the change/deviation using a Libby Field Record of Modification Form.

Field Team Members – Field team members who make entries in field logbooks are required to read this procedure before engaging in this activity. Field team members will be assigned a field logbook prior to field activities and will be responsible for the care and maintenance of the logbook. Field team members will return field logbooks to the project file at the end of the assignment.

4.0 Equipment

The following is required for the proper completion of field logbooks:

- Logbook
- Indelible black or blue ink pen
- Ruler or similar scale

5.0 Procedures

5.1 Preparation

Commercially available, bound field logbooks with waterproof paper and lined, consecutively numbered pages will be used. Separate field logbooks will be kept for each field activity and the cover (some items may be recorded on the inside cover) of each field logbook shall clearly indicate:

- Field logbook sequence number
- Start date and end date of entries
- Title of document governing field activities
- Activity (if the logbook is to be activity-specific), site name, and location
- Contact name and phone number (typically the Project Manager)

For ongoing field activities that may span months or years, designated staff (e.g., field administrative staff) shall manage the field logbooks by tracking to whom and the date each field logbook was assigned, the general activities recorded in each field logbook, and the date the field logbook was returned to the project file.

The first two pages of the logbook will be reserved for a table of contents (TOC), and the third page will be reserved for abbreviations, acronyms, and definitions.

5.2 Operation

The following general requirements will apply when completing logbook entries for the Libby Site:

- Record equipment calibrations, work, observations, and quantities of materials, calculations, drawings, and related information directly in the logbook. If data collection forms are required by the governing document referencing this SOP, the information collected on the form does not need to be duplicated in the logbook. However, any forms used to record site information must be referenced in the logbook.
- Correct erroneous information recorded in a field logbook with a single line strikeout, initial, and date. The correct information will be entered in close proximity to the erroneous entry.
- Do not start a new page until the previous one is full or has been marked with a single diagonal line so that additional entries cannot be made. Use both sides of each page.
- Do not remove any pages from the logbook.
- Document relinquishment of the logbook from one author to another (both parties must sign and date the transfer).
- Sign and date the final entry each day.
- When columns are used to organize information recorded on laboratory documents, the information recorded in the columns shall be identified in a column heading.

Entries into the field logbook shall be preceded with the time (written in military units) of the observation. The time should be recorded frequently and at the point of events or measurements that are critical to the activity being logged. All measurements made and samples collected must be recorded unless they are documented by automatic methods (e.g., data logger) or on a separate form required by an operating procedure. In these cases, the logbook must reference the automatic data record or form.

At each location where a sample is collected or an observation or measurement made, a detailed description of the location is required and a sketch of the location may be warranted. All maps or sketches made in the logbook should have descriptions of the features shown and a direction indicator. It is preferred that maps and sketches be oriented so that north is toward the top of the page. Any maps, sketches, figures, or data that will not fit on a logbook page, or any separate forms or drawings (e.g., FSDS sheets, drawing markups) required by the governing document referencing this SOP should be referenced in the logbook.

Other events and observations that should be recorded include:

- Changes in weather or site conditions that impact field activities or have the potential to impact data collection (e.g., rain impacting air samples, upwind disturbances)
- Deviations from procedures outlined in any governing documents referencing this SOP, including the rationale and authorization for the deviation as appropriate
- Problems, downtime, or delays
- Visitors to the site

5.3 Post-operation

To guard against loss of data as a result of damage or disappearance of logbooks, completed pages and any supporting attachments shall be periodically photocopied (weekly, at a minimum) and maintained in the project file.

At the conclusion of each field activity or phase of site work, the individual responsible for the logbook will ensure that all entries have been appropriately signed and dated, that corrections were made properly, and that the cover information and TOC are complete. As field logbooks are completed, electronic copies may need to be posted to a project eRoom – refer to the governing document referencing this SOP for requirements. All original logbooks will be catalogued and maintained in the project file.

6.0 Restrictions/Limitations

Field logbooks constitute the official record of onsite technical work, investigations, and data collection activities. Their use, control, and ownership are restricted to activities pertaining to specific field operations carried out by governing agency personnel and their subcontractors. They are documents that may be used in court to indicate dates, personnel, procedures, and techniques employed during site activities. Entries made in these logbooks should be factual, clear, precise, and non-subjective. Field logbooks, and entries within, are not intended for personal use.

7.0 Quality Assurance/Quality Control

Quality assurance/quality control (QA/QC) for activities described in this SOP will be attained through a variety of processes, including, at a minimum, the items discussed below. Additional QA/QC requirements, such as audits or field assessments, will be addressed in the governing document referencing this SOP.

7.1 Training

Every effort will be made to ensure consistency in recording information in field logbooks for Libby Site activities. Consistency will be achieved to the extent possible through proper training, use of designated field staff, and provision of TL oversight. Any deficiencies or inconsistencies in implementing this SOP noted by the TL will require re-training of the field team members.

7.2 Field Checks

Field logbooks may be checked for completeness and adherence to SOP requirements on a daily basis by the TL for the first week of each field activity. These checks can be extended to once per month as field activities continue, and any errors noticed during the checks will be discussed with the author and corrected. If field activities continue beyond six months, the frequency of assessing field logbook entries will be established by the field Quality Assurance Manager.

8.0 References

Adapted from CDM Smith Technical Standard Operating Procedure 4-1, Field Logbook Content and Control, January 2012.

Libby Asbestos Superfund Site Standard Operating Procedure Photographic Documentation of Field Activities

Prepared by: *Lee Howell* Date: 4/12/12
CDM Smith

Approved by: *Dania Zimmer* Date: 4/12/12
EPA Region 8

Revision No.	Date	Reason for Revision
0	4/12/12	--

1.0 Objective

Photographic documentation, which includes still and digital photography and videotape or digital versatile/video disc (DVD) recordings, is an essential tool to document field activities conducted by the U.S. Environmental Protection Agency or its contractors in support of the Libby Asbestos Superfund Site (Libby Site). The objective of this standard operating procedure (SOP) is to establish baseline requirements, procedures, and responsibilities for photographic documentation. Additions or modifications to this SOP may be detailed in governing documents referencing this SOP.

2.0 Background

2.1 Definitions

Arrows and Pointers – Used to indicate and/or draw attention to a special feature within the photograph.

Contrasting Backgrounds – Backdrops used to lay soil samples, cores, or other objects on for clearer viewing and to delineate features.

Data Recording Camera Back – A camera attachment or built-in feature that will record, at the very least, frame numbers and dates directly on the film. Digital cameras and recorders may also be equipped with a date stamping feature.

Identifier Component – Visual components used within a photograph such as visual slates, reference markers, and pointers.

Libby Asbestos Superfund Site (Libby Site) – All buildings and land within the boundaries of the EPA's designated operable units (OUs), as illustrated on the most recent version of the OU boundary map.

Photographer – The camera operator (professional or amateur) for still photography, including digital photography, or videotape or DVD recording, whose primary function with regard to this SOP is to produce documentary or data-oriented visual media.

Reference Marker – A reference marker used to indicate a feature size in the photograph and is a standard length of measure, such as a ruler, meter stick, etc. In limited instances, if a ruled

marker is not available or its use is not feasible, it can be a common object of known size placed within the visual field and used for scale.

Site – All buildings (if applicable) and land within the boundaries of the EPA's designated geounits, which may represent individual properties within the Libby Site, a collection of properties, or a larger geographical area.

Slates – Blank white index cards, paper, or a dry-erase board used to present information pertaining to the subject/procedure being photographed. Letters and numbers on the slate will be bold and written with black indelible marking pens.

2.2 Discussion

Photographs and videotape or DVD recordings made during field activities are used as an aid in documenting and describing site features, sample collection activities, equipment used, and conditions during the field activity being performed. This SOP is designed to illustrate the format and desired placement of identifier components, such as visual slates, standard reference markers, and pointers. These items shall become an integral part of the "visual media" that, for the purpose of this document, shall encompass still photographs, digital photographs, videotape recordings (or video footage), and recordings on DVDs. The use of a photographic logbook and standardized entry procedures are also outlined. These procedures and guidelines will minimize potential ambiguities that may arise when viewing the visual media and ensure the representative nature of the photographic documentation.

3.0 Responsibilities

Successful execution of this SOP requires a clear hierarchy of assigned roles with different sets of responsibilities associated with each role. All staff responsible for photographic documentation will understand and implement the requirements contained herein, as well as any additional requirements stated in governing documents referencing this SOP.

Team Leader (TL) – The TL is responsible for ensuring that the format and content of photographic documentation are in accordance with this procedure. The TL is responsible for directing the photographer to specific situations, site features, or operations that the photographer will be responsible for documenting.

Photographer – The photographer shall seek direction from the TL and regularly discuss the visual documentation requirements and schedule. The photographer may be responsible for maintaining a logbook or itemization of photos/recordings or providing captions. Specific requirements will be defined in the governing document referencing this SOP.

4.0 Equipment

The following equipment may be used for photographic documentation:

- 35-millimeter (mm) camera and appropriate film (e.g., medium speed or multi-purpose fine-grain color)
- Disposable, single-use camera (35mm or panoramic use)
- Digital camera
- Video camera and appropriate storage media (e.g., videotapes, DVDs)
- Extra batteries
- Standard reference markers
- Slates

- Arrows or pointers
- Contrasting backgrounds
- Logbook
- Data recording camera back (if available)
- Indelible black or blue ink pen
- Storage medium for digital camera

5.0 Procedures

5.1 Preparation

In addition to this SOP, photographers must be familiar with all procedures applicable to the field activity being performed. These procedures should be consulted as necessary to obtain specific information about equipment and supplies, health and safety (including requirements for personal protective equipment at a site), sample collection, equipment and personnel decontamination, documentation, etc. These procedures should be maintained on site by field staff at all times for easy reference.

The photographer should also be aware of any potential physical hazards while photographing the subject (e.g., traffic, operating equipment, low overhead hazard, edge of excavation area).

If required, a commercially available, bound logbook will be used to log and document photographic activities. Alternatively, a portion of the field logbook may be designated as the photographic log and documentation section.

Because digital cameras and DVD recorders have multiple photographic quality settings, if not specified in the governing document referencing this SOP, the TL shall specify the resolution (quality) at which photographic documentation should be collected. It should be noted that a camera or DVD recorder that obtains a higher resolution (quality) has a higher number of pixels and will store a fewer number of photographs per digital storage medium.

5.2 Operation

The following sections provide general guidelines that should be followed to visually document field activities and site features using still/digital cameras and video equipment. Slate and caption information will not be required at the Libby Site unless specified in the governing document referencing this SOP.

5.2.1 Still Photography

Slate Information

Each new roll of film or digital storage medium will contain on the first usable frame (for film) a slate with consecutively assigned control numbers (a unique, consecutive number that is assigned by the photographer).

Caption Information

Still photographs will have a full caption permanently attached to the back or permanently attached to a photo log sheet. Digital photographs should have a caption added after the photographs are downloaded. Unless modified by the governing document referencing this SOP, captions should contain the following information:

- Film roll control number (if required) and photograph sequence number
- Site name or location

- Description of activity/item shown
- Date and time
- Direction (if applicable)
- Photographer

Close-up and Feature Photography

Close-up photographs should include a standard reference marker of appropriate size as an indication of the feature size.

Feature samples, core pieces, and other lithologic media should be photographed as soon as possible after they have been removed from their *in situ* locations to enable a more accurate record of their initial condition and color for formal lithologic observations and interpretations.

Site Photography

Site photography, in general, consists predominantly of medium- and wide-angle shots. A standard reference marker should be placed adjacent to the feature or, when this is not possible, within the same focal plane. While it is encouraged that a standard reference marker and caption/slate be included in the scene, it is understood that situations will arise that preclude their inclusion within the scene. This will be especially true of wide-angle shots. In such a case, the logbook (field or photographic), photographic caption, or digital file name shall specify all information pertinent to the scene.

5.2.2 Photographic Documentation Using Video Cameras

As a reminder, it is not within the scope of this document to set appropriate guidelines for presentation or “show” videotape or DVD recording. The following guidelines are set for documentary videotape or DVD recordings only and should be implemented at the discretion of the site personnel.

Documentary videotape or DVD recordings of field activities may include an audio slate for all scenes, as directed by the governing document referencing this SOP. At the beginning of each video session, an announcer will recite the following information: date, time (in military units), photographer, site ID number, and site location. This oral account may include any additional information clarifying the subject matter being recorded.

A standard reference marker may be used when taking close-up shots of site features with a video camera. The scene may also include a caption/slate. It should be placed adjacent and parallel to the feature being photographed.

A standard reference marker and caption/slate may be included in all scenes, as directed by the governing document referencing this SOP. The caption information is vital to the value of the documentary visual media and should be included. If it is not included within the scene, it should be placed before the scene.

Original video recordings will not be edited. This will maintain the integrity of the information contained on the videotape or DVD. If editing is desired, a working copy of the original video recording can be made.

A label should be placed on the videotape or DVD with the appropriate identifying information (project name, project number, date, location, etc.).

5.2.3 Photographic Logs

Photographic activities shall be documented in a photographic log or in a section of the field logbook, as directed by the governing document referencing this SOP. The photographer will be responsible for making proper entries.

The following information shall be maintained in the appropriate logbook:

- Photographer name
- Roll/tape/DVD control number (as appropriate)
- Sequential tracking number for each photograph taken (for digital cameras, the camera-generated number may be used)
- Date and time (military time)
- Location
- Description of the activity/item photographed
- Description of the general setup, including approximate distance between the camera and the subject
- Other pertinent information to assist in the identification of the subject matter

5.3 Post-operation

5.3.1 Processing

All film will be sent for development and printing to a photographic laboratory (to be determined by the photographer). The photographer will be responsible for arranging transport of the film from the field to the photographic laboratory. The photographer will also be responsible for arranging delivery of the negatives and photographs, digital storage medium, or videotape or DVD to the TL to be placed in the project file.

Digital media should be downloaded daily to a personal computer or secure server; the files should be in either "JPEG" or "TIFF" format. Files should be renamed at the time of download in accordance with any file-naming conventions required by the governing document referencing this SOP, or to correspond to the logbook. At a minimum, the file name should include the corresponding sampling location and/or sample number and the photograph date (e.g., "123 Elm St_2-15-2011", "AA-12345_3-18-2009").

5.3.2 Documentation

At the end of each day's photographic session, the photographer(s) will ensure that all photographic documentation has been maintained in accordance with this SOP.

5.3.2 Archive

Unless otherwise specified in Libby Site data management requirements or the governing document referencing this SOP, digital photographs will be stored on a secure server (with a nightly backup) or posted to a web-based location (e.g., an eRoom or SharePoint portal). These files will be archived until project closeout, at which time project management will determine a long-term electronic file storage system.

6.0 Restrictions/Limitations

This document is designed to provide a set of guidelines for the field personnel to ensure that an effective and standardized program of visual documentation is maintained.

The procedures outlined herein are general by nature. The photographer is responsible for specific operational activity or procedure. Questions concerning specific procedures or requirements should be directed to the TL.

7.0 Quality Assurance/Quality Control

Quality assurance/quality control (QA/QC) for activities described in this SOP will be attained through a variety of processes, including, at a minimum, the items discussed below. Additional QA/QC requirements, such as audits or field assessments, will be addressed in the governing document referencing this SOP.

7.1 Training

Every effort will be made to ensure quality photographic documentation is gathered to support site activities. Consistency will be achieved to the extent possible through proper training, use of designated field staff, and provision of TL oversight. Any deficiencies or inconsistencies in implementing this SOP noted by the TL will require re-training of the field team members.

7.2 Field Checks

Photographic documentation processes may be checked for completeness and adherence to SOP requirements on a daily basis by the TL for the first week of each field activity. These checks can be extended to once per month as field activities continue, and any errors noticed during the checks will be discussed with the photographer and corrected. If field activities continue beyond six months, the frequency of assessing photographic documentation will be established by the Quality Assurance Manager.

8.0 References

Adapted from CDM Smith Technical Standard Operating Procedure 4-2, Photographic Documentation of Field Activities, January 2012.

Libby Asbestos Superfund Site Standard Operating Procedure Control of Measurement and Test Equipment

Prepared by: Sean Connell Date: 4/12/12
CDM Smith

Approved by: Donna Zimmer Date: 4/12/12
EPA Region 8

Revision No.	Date	Reason for Revision
0	4/12/12	--

1.0 Objective

The objective of this standard operating procedure (SOP) is to establish baseline requirements, procedures, and responsibilities for the control of measurement and test equipment (M&TE) used by the U.S. Environmental Protection Agency or its contractors in support of the Libby Asbestos Superfund Site (Libby Site). Additions or modifications to this SOP may be detailed in governing documents referencing this SOP.

2.0 Background

2.1 Definitions

Libby Asbestos Superfund Site (Libby Site) – All buildings and land within the boundaries of the EPA's designated operable units (OUs), as illustrated on the most recent version of the OU boundary map.

Traceability – The ability to trace the history, application, or location of an item and like items or activities by means of recorded identification.

2.2 Discussion

M&TE may be government furnished (GF), rented or leased from an outside vendor, or purchased. It is essential that measurements and tests resulting from the use of equipment be of the highest accountability and integrity. To facilitate that, the equipment shall be used in full understanding and compliance with the instructions and specifications included in the manufacturer's operations and maintenance and calibration procedures, and in accordance with any other related requirements specified in the governing document referencing this SOP.

3.0 Responsibilities

All staff with responsibility for the direct control and/or use of M&TE is responsible for being knowledgeable of, and understanding and implementing the requirements contained herein, as well as any additional related requirements.

Team Leader (TL) – Responsible for identifying the technical specifications (e.g., precision, accuracy) for M&TE needed to meet project data collection objectives, and determining any

additional applicable Libby Site-specific requirements (e.g., periodic calibration of primary calibration sources) for M&TE.

Requisitioner – Responsible for ensuring M&TE is obtained or procured that meets the technical specifications identified by the TL, and facilitates obtaining the manufacturer's operations and maintenance and calibration procedures prior to field work.

Receiver – Responsible for receipt and/or unpackaging of M&TE and notifying the TL that the item has been received.

User – Responsible for the proper preparation and use of M&TE to collect the quality and quantity of data needed to meet project objectives. Users are typically field team members.

4.0 Equipment

Required M&TE will be specified in the governing document referencing this SOP.

5.0 Procedures

The following general requirements apply to M&TE at the Libby Site. Additional details and responsibilities are described later in this section.

- Manufacturer maintenance and calibration procedures must be followed when using M&TE
- Obtain the maintenance and calibration procedures if they are missing or incomplete
- Attach or include the maintenance and calibration procedures with the M&TE
- Prepare and record maintenance and calibration in an equipment or field log according to requirements stated in the governing document referencing this SOP
- Maintain M&TE records
- Label M&TE requiring routine or scheduled calibration (when required)
- Perform maintenance and calibration using the appropriate procedure and calibration standards
- Identify and take action on nonconforming M&TE

5.1 Preparation

5.1.1 Obtain the Operating, Maintenance, and Calibration Documents

For Procured M&TE

Requisitioner – Specify that the maintenance and calibration procedures be included.

For GF M&TE Acquired as a Result of Property Transfer

TL – Inspect the M&TE to determine whether maintenance and calibration procedures are included with the item. If missing or incomplete, obtain the appropriate documentation from the manufacturer.

For Rented or Leased M&TE

Requisitioner – Specify that the maintenance and calibration procedures, the latest calibration record, and the calibration standards certification be included. If this information is not delivered with the M&TE, request it from the vendor.

5.1.2 Prepare and Record Maintenance and Calibration Records

For All M&TE

Receiver – Upon receipt of an item of M&TE, notify the TL for the overall property control of the equipment.

TL and User – Record all maintenance and calibration events in an equipment or field log. The log must have sequentially-numbered pages.

5.2 Operation

TL and User – Operate, maintain, and calibrate M&TE in accordance with the maintenance and calibration procedures. Record maintenance and calibration actions in the equipment log or field log.

5.2.2 Traceability of Calibration Standards

For All M&TE

TL and User –

- When ordering calibration standards, request nationally recognized standards as specified or required. Request commercially available standards when not otherwise specified or required. Or, request standards in accordance with other related project-specific requirements.
- Require certifications for standards that clearly state the traceability.
- Require Material Safety Data Sheets to be provided with standards.
- Note standards that are perishable and consume or dispose of them on or before the expiration date.

5.2.3 M&TE That Fails Calibration

For any M&TE item that cannot be calibrated or adjusted to perform accurately:

User – Immediately discontinue use and segregate the item from other equipment.

TL – Review the current and previous maintenance and calibration records to determine if the validity of current or previous measurement and test results could have been affected and notify the appropriate authorities (typically the Project Manager) of the results. Any test results that are known to impact or have the potential to impact project data will be documented using a Libby Field Record of Modification Form.

5.3 Post-operation

M&TE shall be promptly returned to the owner at the end of field activities. All operations, maintenance, and calibration procedures shall be retained with the M&TE. Project M&TE records (e.g., equipment logs) will be retained in the project file.

6.0 Restrictions/Limitations

On an item-by-item basis, exemptions from the requirements of this SOP may be granted by the Health and Safety Manager and/or Quality Assurance Manager. All exemptions shall be documented by the grantor and included in the equipment records as appropriate.

7.0 Quality Assurance/Quality Control

Quality assurance/quality control (QA/QC) for activities described in this SOP will be attained through a variety of processes. Every effort will be made to ensure the appropriate and functional M&TE are used to support site activities. This will be achieved to the extent possible through proper training, use of qualified procurement and designated field staff, and provision of TL oversight. Any deficiencies or inconsistencies in implementing this SOP noted by the TL will require discussion with appropriate management and, as appropriate, re-training of the field team members. Additional QA/QC requirements, such as audits or field assessments, will be addressed in the governing document referencing this SOP.

8.0 References

Adapted from CDM Smith Technical Standard Operating Procedure 5-1, Control of Measurement and Test Equipment, January 2012.

Libby Asbestos Superfund Site Standard Operating Procedure Field Equipment Decontamination

Prepared by: *Leah Powell* Date: 4/12/12
CDM Smith

Approved by: *Dania Zimmer* Date: 4/12/12
EPA Region 8

Revision No.	Date	Reason for Revision
0	4/12/12	--

1.0 Objective

Decontamination of field equipment is necessary to ensure acceptable quality of samples by preventing cross contamination. Further, decontamination reduces health hazards and prevents the spread of contaminants off site. The objective of this standard operating procedure (SOP) is to establish baseline requirements, procedures, and responsibilities for the decontamination of field equipment used by the U.S. Environmental Protection Agency or its contractors in support of the Libby Asbestos Superfund Site (Libby Site). Additions or modifications to this SOP may be detailed in governing documents referencing this SOP.

2.0 Definitions

Clean – Free of contamination and when decontamination has been completed in accordance with this SOP.

Cross contamination – The transfer of contaminants through equipment or personnel from the contamination source to less contaminated or non-contaminated samples or areas.

Decontamination – The process of rinsing or otherwise cleaning the surfaces of equipment to rid them of contaminants and to minimize the potential for cross contamination of samples or exposure of personnel.

De-mineralized water – Water that has had most to all minerals removed from it. De-mineralized water shall only be stored in clean glass, stainless steel, or plastic containers that can be closed when not in use.

Libby Asbestos Superfund Site (Libby Site) – All buildings and land within the boundaries of the EPA's designated operable units (OUs), as illustrated on the most recent version of the OU boundary map.

Material Safety Data Sheet (MSDS) – Document that discusses the proper storage and physical and toxicological characteristics of a particular substance used during field operations. MSDSs are to be maintained on site at all times during field operations.

Potable water – Tap water may be obtained from any municipal system. Chemical analysis of the water source may be required before it is used.

Sampling equipment – Equipment that comes into direct contact with the sample media. Such equipment includes split spoon samplers, well casing and screens, and trowels or bowls used to collect and/or homogenize samples.

Soap – Low-sudsing, non-phosphate detergent (e.g., Liquinox®).

Solvent rinse – Pesticide-grade (or better) isopropanol, acetone, or methanol.

3.0 Responsibilities

Successful execution of this SOP requires a clear hierarchy of assigned roles with different sets of responsibilities associated with each role. All staff responsible for field equipment decontamination will understand and implement the requirements contained herein, as well as any additional requirements stated in governing documents referencing this SOP.

Team Leader - The TL is responsible for ensuring that field personnel are properly trained and that decontamination is conducted in accordance with this procedure and any other pertinent Libby Site decontamination processes cited in the governing document referencing this SOP.

Field Team Members – Field team members performing operations on the Libby Site are responsible for adhering to the procedures contained in this SOP and any other decontamination processes specified in the governing document referencing this SOP. If required, field team members will collect and document rinsate samples (also known as equipment blanks) to provide quantitative verification that these procedures have been correctly implemented. Field team members are also responsible for communicating any problems pertaining to the decontamination of field equipment to the TL.

4.0 Equipment

The following equipment may be employed wholly or in part during use of this SOP (refer to the governing document referencing this SOP for detailed requirements):

- Stiff-bristle scrub brushes
- Plastic buckets, scoops, trowels, and troughs
- Soap
- Nalgene® or Teflon® sprayers or wash bottles or 2- to 5-gallon, manual-pump sprayers (pump sprayer material must be compatible with the solution used)
- Plastic sheeting, plastic bags, and/or aluminum foil to keep decontaminated equipment clean between uses
- Disposable wipes, rags, or paper towels
- Potable water (potable water may be required to be tested for contaminants before use)
- De-mineralized water
- Gloves, safety glasses, and other protective clothing as specified in the health and safety plan
- High-pressure pump with soap dispenser or steam-spray unit (for large equipment only)
- Appropriate decontamination solutions pesticide grade or better and traceable to a source

- Tools for equipment assembly and disassembly
- 55-gallon drums or tanks for temporary storage of decontamination water
- Pallets for drums or tanks holding decontamination water

5.0 Procedures

All reusable equipment (non-dedicated) used to collect, handle, or measure samples shall be decontaminated before coming into contact with any sample media or personnel using the equipment. Decontamination of equipment shall occur either at a specified location, central decontamination station or at portable decontamination stations set up at the sampling location, drill site, or monitoring well location. The centrally-located decontamination area may include an appropriately-sized bermed and lined area on which equipment decontamination occurs and equipped with a collection system and/or storage vessels. In certain circumstances, berming may not be necessary when small quantities of water are being generated and for some short duration field activities. Equipment shall be transported to and from the decontamination area in a manner to prevent cross contamination of equipment and/or the area.

Typically at the Libby Site, decontamination water will not be captured and will be discharged to the ground at the site. However, the exact procedure for decontamination waste disposal may be discussed in the governing document referencing this SOP. Also, solvent rinse fluids may need to be segregated from other investigation-derived waste (IDW).

All items that come into contact with potentially contaminated media shall be decontaminated before use, between sampling locations (does not need to be performed between aliquots of an individual sample) and/or drilling locations, and after use. All decontamination procedures for the equipment being used are provided in the following sections.

General Guidelines

- Potable or de-mineralized water shall be free of all contaminants of concern. Depending upon the governing document referencing this SOP, analytical data from the water source may be required to ensure it is clean.
- Sampling equipment that has come into contact with oil and grease shall be cleaned with methanol or other approved alternative to remove the oily material. This may be followed by a hexane rinse and then another methanol rinse. Regulatory or Libby Site-specific requirements regarding solvent use shall be stated in the governing document referencing this SOP.
- All solvents¹ shall be pesticide-grade or better and traceable to a source. The corresponding lot numbers shall be recorded in the appropriate field logbook.
- Decontaminated equipment shall be allowed to air dry before being used.
- Documentation of all equipment, including type of equipment, date, time, method of decontamination, and any associated field quality control sampling, shall be recorded in the field logbook.

¹Solvents are potentially hazardous materials and must be handled, stored, and transported accordingly. Solvents shall never be used in a closed building. See the investigation-specific health and safety plan and/or the chemical's MSDS for specific information regarding the safe use of the chemical.

- Gloves, boots, safety glasses, and any other personnel protective clothing and equipment shall be used as specified in the governing document referencing this SOP and/or health and safety plan.

5.1 Heavy Equipment Decontamination

Heavy equipment typically used at the Libby Site includes drilling rigs, trucks, and excavators. For any heavy equipment used during EPA response actions, the equipment decontamination procedures provided in the current version of the Libby Asbestos Site Response Action Work Plan shall apply. For all other field activities, follow these steps when decontaminating heavy equipment:

1. Establish a bermed decontamination area that is large enough to fully contain the equipment to be cleaned. If available, an existing wash pad or appropriate paved and bermed area may be used; otherwise, use one or more layers of heavy plastic sheeting to cover the ground surface and berms. All decontamination pads shall be upwind of the investigation area(s).
2. With the heavy equipment in place, spray areas (rear of rig or backhoe) exposed to contaminated media by pressurized means. Be sure to spray down all surfaces, including the undercarriage.
3. Use brushes, soap, and appropriate decontamination water to remove dirt whenever necessary.
4. Remove equipment from the decontamination pad.
5. After decontamination activities are completed, collect all plastic sheeting, and disposable gloves, boots, and clothing in containers or receptacles. All receptacles containing contaminated items must be properly labeled for disposal as detailed in the governing document referencing this SOP.

5.2 Downhole Equipment Decontamination

Downhole equipment includes hollow-stem augers, drill pipes, rods, and stems. Follow these steps when decontaminating this equipment:

1. Set up a centralized decontamination area, if possible. This area shall be set up to collect contaminated rinse waters and to minimize the spread of airborne spray.
2. Set up a "clean" area upwind of the decontamination area to receive cleaned equipment for air-drying. At a minimum, clean plastic sheeting must be used to cover the ground, tables, or other surfaces on which decontaminated equipment is to be placed. All decontamination areas shall be upwind of any areas under investigation.
3. Using soap and appropriate water with pressurization (e.g., Hudson[®] sprayer), spray the contaminated equipment. Aim downward to avoid spraying outside the decontamination area. Be sure to spray inside corners and gaps especially well. Use a brush, if necessary, to dislodge dirt.
4. If using soapy water, rinse the equipment using clean appropriate water with pressurization.
5. Remove the equipment from the decontamination area and place in a clean area upwind to air dry.
6. After decontamination activities are completed, collect all plastic sheeting, and disposable gloves, boots, and clothing in containers or receptacles. All receptacles containing

contaminated items must be properly labeled for disposal as detailed in the governing document referencing this SOP.

5.3 Sampling Equipment Decontamination

Follow these steps when decontaminating sampling equipment:

1. Set up a decontamination line. The decontamination line shall progress from "dirty" to "clean." A clean area shall be established upwind of the decontamination wash/rinse activities to dry the equipment.
2. Disassemble any items that may trap contaminants internally. Do not reassemble the items until decontamination and air drying are complete.
3. Wash the items with appropriate water and soap using a stiff brush as necessary to remove particulate matter and surface films. With the exception of polyvinyl chloride or plastic items, the items may be steam-cleaned using soap and hot water as an alternative to brushing. Items that have come into contact with concentrated and/or oily contaminants may need to be rinsed with a solvent such as hexane and allowed to air dry prior to this washing step.
4. Thoroughly rinse the items with potable water.
5. If sampling for organic compounds, thoroughly rinse the items with solvent (e.g., isopropanol) followed by a rinse using de-mineralized water. The specific chemicals used for the solvent rinse phase shall be specified in the work plan. Solvents are potentially hazardous materials and care must be exercised when using these chemicals to prevent adverse health effects. Appropriate personal protective equipment (PPE) must be worn when using these chemicals. These chemicals (including spent rinsate) must be managed and stored appropriately. Special measures such as proper labels, paperwork, notification, etc. may be required when transporting or shipping solvent chemicals.
6. Rinse the items thoroughly using de-mineralized water.
7. Allow the items to air dry completely.
8. After decontamination activities are completed, collect all plastic sheeting, and disposable PPE. Place the contaminated items in properly labeled bags or containers for disposal. Refer to the governing document referencing this SOP for labeling and waste management requirements.

5.4 Pump Decontamination

Follow the manufacturer's recommendation for specified pump decontamination procedures. At a minimum, follow these steps when decontaminating pumps:

1. Set up the decontamination area and separate "clean" storage area using plastic sheeting to cover the ground, tables, and other surfaces. Set up three containers: the first container shall contain dilute (non-foaming) soapy water; the second container shall contain potable water; and the third container shall contain de-mineralized water.
2. The pump shall be set up in the same configuration as for sampling. Submerge the pump intake (or the pump, if submersible) and all downhole-wetted parts (tubing, piping, foot valve) in the soapy water of the first container. Pump soapy water through the pump assembly. Scrub the outside of the pump and other wetted parts with a metal brush.

3. Move the pump assembly to the potable water container while leaving discharge outlet in the waste container. All downhole-wetted parts must be immersed in the potable water rinse. Pump potable water through the pump assembly until it runs clear.
4. Move the pump intake to the de-mineralized water container. Pump the water through the pump assembly. Pump the volume of water through the pump specified in the field plan. Usually, three pump-and-line-assembly volumes shall be required.
5. Remove the decontaminated pump assembly to the clean area and allow it to air dry upwind of the decontamination area. Intake and outlet orifices shall be covered to prevent the entry of airborne contaminants and particles.

5.5 Instrument Probe Decontamination

Instrument probes used for field measurements (e.g., pH meters, conductivity meters) shall be decontaminated between samples and after use with de-mineralized water. At no time shall a sample probe be placed in contact with water within a sample container.

5.6 Waste Disposal

Waste disposal should follow the requirements listed in Libby project-specific SOP for handling investigation-derived waste (IDW) and the governing document referencing this SOP. The following are guidelines for disposing of waste:

- Decontamination water will typically not be captured, packaged, labeled, or stored as IDW at the site. Decontamination water will be discharged to the ground at the work site. Other materials used in the decontamination process will be disposed of as IDW.
- Small quantities of decontamination solutions may be allowed to evaporate to dryness.
- If large quantities of used decontamination solutions shall be generated, each type of waste shall be segregated in separate containers.
- Plastic sheeting and disposable protective clothing will be treated and disposed of as asbestos-containing materials.

6.0 Restrictions/Limitations

If the field equipment is not thoroughly rinsed and allowed to completely air dry before use, volatile organic residue, which interferes with the analysis, may be detected in the samples. The occurrence of residual organic solvents is often dependent on the time of year sampling is conducted. In the summer, volatilization is rapid, and in the winter, volatilization is slow. Check with EPA Region 8 and the State of Montana for approved decontamination solvents.

7.0 Quality Assurance/Quality Control

Quality assurance/quality control (QA/QC) for activities described in this SOP will be attained through a variety of processes, including, at a minimum, the items discussed below. Additional QA/QC requirements, such as audits or field assessments, will be addressed in the governing document referencing this SOP.

7.1 Training

Every effort will be made to ensure proper field equipment decontamination, which will be achieved to the extent possible through proper training, use of designated field staff, and

provision of TL oversight. Any deficiencies or inconsistencies in implementing this SOP noted by the TL will require staff re-training.

7.2 Field Checks

Adherence to field equipment decontamination requirements may be checked on a daily basis by the TL for the first week of each field activity. These checks can be extended to once per month as field activities continue, and any non-compliance discussed with the field team member. If field activities continue beyond six months, the frequency of assessing field equipment decontamination will be established by the field Quality Assurance Manager.

8.0 References

Adapted from CDM Smith Technical Standard Operating Procedure 4-5, Field Equipment Decontamination, January 2012.

Libby Asbestos Superfund Site Standard Operating Procedure Handling Investigation-derived Waste

Prepared by: *Scott Powell* Date: 4/12/12
CDM Smith

Approved by: *Dominia Zimmer* Date: 4/12/12
EPA Region 8

Revision No.	Date	Reason for Revision
0	4/12/12	--

1.0 Objective

The objective of this standard operating procedure (SOP) is to establish baseline requirements, procedures, and responsibilities for handling investigation-derived waste (IDW) resulting from work performed by the U.S. Environmental Protection Agency or its contractors in support of the Libby Asbestos Superfund Site (Libby Site). Additions or modifications to this SOP may be detailed in governing documents referencing this SOP.

2.0 Background

2.1 Definitions

Hazardous Waste – Discarded material that is regulated listed waste, or waste that exhibits ignitability, corrosivity, reactivity, or toxicity as defined in 40 CFR 261.3 or state regulations.

Investigation-derived Waste (IDW) – Discarded materials resulting from field activities such as sampling, surveying, drilling, excavation, and decontamination processes that, in present form, possess no inherent value or additional usefulness without treatment.

Libby Asbestos Superfund Site (Libby Site) – All buildings and land within the boundaries of the EPA's designated operable units (OUs), as illustrated on the most recent version of the OU boundary map.

Site – All buildings (if applicable) and land within the boundaries of the EPA's designated geounits, which may represent individual properties within the Libby Site, a collection of properties, or a larger geographical area.

Treatment, Storage, and Disposal Facility (TSDF) – Permitted facilities that accept hazardous waste shipments for further treatment, storage, and/or disposal. These facilities must be permitted by the EPA and appropriate state and local agencies.

2.2 Discussion

At the Libby Site, field investigation and response action activities may result in the generation of IDW. IDW may include soil and cuttings from test pits or well installation; soil and other materials from the collection of samples; personal protective equipment (PPE); and other wastes or supplies used during the sampling and testing of potentially hazardous materials.

The vast majority of Libby Site IDW is expected to relate to the contaminant of concern – Libby amphibole asbestos. The overall management of IDW must comply with applicable regulatory requirements.

3.0 Responsibilities

Successful execution of this SOP requires a clear hierarchy of assigned roles with different sets of responsibilities associated with each role. All staff responsible for handling IDW will understand and implement the requirements contained herein, as well as any additional requirements stated in governing documents referencing this SOP.

Team Leader (TL) – The TL is responsible for identifying Libby Site-specific requirements for the disposal of IDW in accordance with federal, state, and/or facility requirements, and ensuring that all IDW procedures are conducted in accordance with this SOP. The TL will communicate with the field team members regarding the specific objectives and anticipated situations that require deviation from this SOP.

Field Team Members – Field team members are responsible for adhering to the procedures contained in this SOP, and communicating any unusual or unplanned condition to the TL.

4.0 Equipment

Equipment required for IDW containment may vary according to field activity requirements. Management decisions concerning the necessary equipment required shall consider containment method, sampling, labeling, maneuvering, and storage (if applicable). Equipment must be onsite and inspected before commencing work.

4.1 IDW Containment Devices

The appropriate containment device (e.g., bags, drums, tanks, etc.) and the ultimate disposition of the IDW shall be specified in the governing document referencing this SOP. Typical IDW containment devices include:

- Plastic sheeting (polyethylene) with a minimum thickness of 6 mil
- U.S. Department of Transportation (DOT)-approved steel containers
- Polyethylene or steel bulk storage tanks

The volume of the appropriate containment device shall be specified in the governing document referencing this SOP.

4.2 IDW Container Labeling

A “Waste Container” or “IDW Container” label or indelible marking shall be applied to each container. Labeling or marking requirements for onsite IDW not expected to be transported offsite are as detailed below.

- Labels and markings must contain the following information: project name, generation date, location of waste origin, container identification number, sample number (if applicable), and contents.
- Each label or marking will be applied to the upper one-third of the container at least twice, on opposite sides.

- Containers that are 5 gallons or less may only require one label or set of markings.
- Labels or markings will be positioned on a smooth part of the container. The label must not be affixed across container bungs, seams, ridges, or dents.
- Labels must be constructed of a weather-resistive material with markings made with a permanent marker or paint pen and capable of enduring the expected weather conditions. If markings are used, the color must be easily distinguishable from the container color.
- Labels will be secured in a manner to ensure that they remain affixed to the container.

Labeling or marking requirements for IDW expected to be transported off of the work site must be in accordance with the requirements of 29 CFR 1926.1101.

4.3 IDW Container Movement

Staging areas for IDW containers shall be predetermined and in accordance with investigation-specific requirements. Arrangements shall be made before field mobilization as to the methods and personnel required to safely transport IDW containers to the staging area. Transportation of IDW containers offsite via a public roadway is prohibited unless 49 CFR 172 requirements are met.

4.4 IDW Container Storage

Containerized IDW awaiting results of pending chemical analysis or further onsite treatment shall be staged on site. Staging areas and bulk storage procedures are to be determined according to investigation-specific requirements. Containers are to be stored in such a fashion that the labels can be easily read. A secondary/spill container must be provided for liquid IDW storage and as appropriate for solid IDW storage (e.g., steel drums shall not be stored in direct contact with the ground).

5.0 Procedures

The three general options for managing IDW are: 1) collection and onsite disposal; 2) collection for offsite disposal; and 3) collection and interim management. The option selected shall take into account the following factors:

- Type (soil, sludge, liquid, debris), quantity, and source of IDW
- Risk posed by managing the IDW onsite
- Compliance with regulatory requirements
- IDW minimization and consistency with the Libby Site remedy

5.1 Collection and Onsite Disposal

5.1.1 Soil/Sludge/Sediment

Unless otherwise specified in the governing document referencing this SOP, when handling soil/sludge/sediment IDW at the Libby Site, the following will apply:

- Return IDW to boring, pit, or source immediately after generation as long as returning the media to these areas will not increase site risks (i.e., the contaminated soil will not be in a different area or at a different depth than from where it was originally obtained).

5.1.2 Aqueous Liquids

Unless otherwise specified in the governing document referencing this SOP, options for handling aqueous liquid IDW at the Libby Site are listed below. These options may require results of laboratory analysis to obtain client and/or regulatory approval.

- Discharge to ground surface close to the well from which it was extracted, only if soil contaminants will not be mobilized in the process and the action will not contaminate clean areas. If IDW from the sampling of background up-gradient wells is not a community concern or associated with soil contamination, this presumably uncontaminated IDW may be released on the ground around the well.
- When small amounts (i.e., less than 5 gallons) of used decontamination fluids are generated during site characterization activities (e.g., during soil sampling), the fluids may be discharged to the ground surface within the sampling area or allowed to evaporate from an open bucket.

5.1.3 Disposable PPE

Disposable PPE IDW (not including excess soil volume) for the Libby Site will be collected in garbage bags and marked "IDW" with an indelible ink marker. These bags will be deposited into the asbestos-containing material (ACM) waste stream for appropriate disposal at the local Class IV asbestos landfill. Excess soil volume will be returned to the area from where it was collected.

5.2 Collection and Interim Management

Collection and interim management options that may be employed for Libby Site IDW are provided herein.

Storing IDW onsite until the final action may be practical in the following situations:

- Returning wastes (especially sludges and soils) to their onsite source area would require re-excavation for disposal as determined for the final site remedy.
- Interim storage in containers may be necessary to provide adequate protection to human health and the environment.
- Storing IDW until the final disposal of all wastes from the site will eliminate the need to address this issue more than once.
- Interim storage may be necessary to provide time for sampling and analysis.

6.0 Restrictions/Limitations

Managers of the site shall determine the most appropriate disposal option for IDW on an activity-specific basis. Parameters to consider, especially when determining the level of protection, include: the volume of IDW and the nature of contaminants present in the site soil. Special disposal/handling may be needed for drilling fluids because they may contain significant solid components and therefore may need to be handled, treated, and disposed as non-liquid waste. Disposable sampling materials, disposable PPE, decontamination fluids, etc. will always be

managed on a site-specific basis. Under no circumstances shall these types of materials be stored in a site office, facility, or warehouse.

7.0 Quality Assurance/Quality Control

Quality assurance/quality control (QA/QC) for activities described in this SOP will be attained through a variety of processes, including, at a minimum, the items discussed below. Additional QA/QC requirements, such as audits or field assessments, will be addressed in the governing document referencing this SOP.

7.1 Training

Every effort will be made to ensure proper handling of IDW, which will be achieved to the extent possible through proper training, use of designated field staff, and provision of TL oversight. Any deficiencies or inconsistencies in implementing this SOP noted by the TL will require staff re-training.

7.2 Field Checks

Adherence to requirements for handling IDW may be checked on a daily basis by the TL (or their designate) for the first week of each field activity. These checks can be extended to once per month as field activities continue. Any deficiencies or inconsistencies in implementing this SOP noted by the TL will require field team member re-training. If field activities continue beyond six months, the frequency of assessing field logbook entries will be established by the field Quality Assurance Manager or their designate.

8.0 References

Adapted from CDM Smith Technical Standard Operating Procedure 2-2, Guide to Handling Investigation-derived Waste, January 2012.

Libby Asbestos Superfund Site Standard Operating Procedure Sample Custody

Prepared by: Lee Howell Date: 4/12/12
CDM Smith

Approved by: Dania Zimmer Date: 4/12/12
EPA Region 8

Revision No.	Date	Reason for Revision
0	4/12/12	--

1.0 Objective

Sample custody procedures are integral to maintaining and documenting the possession of environmental samples collected by the U.S. Environmental Protection Agency or its contractors in support of the Libby Asbestos Superfund Site (Libby Site). The objective of this standard operating procedure (SOP) is to establish baseline requirements, procedures, and responsibilities for sample custody for the Libby Site. Additions or modifications to this SOP may be detailed in governing documents referencing this SOP.

2.0 Background

2.1 Definitions

Chain-of-custody record (COC) – Used to document the custody, control, transfer, analysis, and disposition of samples.

Custody seal – An adhesive-backed seal that is applied to an individual sample or sample container to demonstrate that sample integrity has not been compromised during sample transfer.

Facility – A designated sample processing facility, analytical laboratory, or long-term storage area, for Libby Site samples.

Field sample data sheet (FSDS) – A controlled document used to record sample information.

Libby Asbestos Superfund Site (Libby Site) – All buildings and land within the boundaries of the EPA's designated operable units (OUs), as illustrated on the most recent version of the OU boundary map.

Sample – Material to be analyzed that is contained in single or multiple containers representing a unique sample number.

Sample custody – The possession or safe-keeping of samples in such a manner that prevents tampering, damage, or loss.

Sample labels – Adhesive-backed labels that contain, at a minimum, the unique sample number/identifier. Sample labels are typically used on field documentation, sample cassettes, and containers, and may be pre-printed to minimize sequencing or transcription errors.

2.2 Discussion

Because of the evidentiary nature of samples collected during environmental investigations, possession must be traceable from the time the samples are collected until their derived data are introduced as evidence in legal proceedings. To maintain and document sample possession, sample custody procedures must be followed.

3.0 Responsibilities

Successful execution of this SOP requires a clear hierarchy of assigned roles with different sets of responsibilities associated with each role. All staff responsible for the custody of samples will understand and implement the requirements contained herein, as well as any additional requirements stated in governing documents referencing this SOP.

Team Leader (TL) – Responsible for ensuring that strict chain-of-custody procedures are maintained during all sampling events.

Sampler – Responsible for the care and custody of samples from the time of collection until they are transferred.

Field Sample Coordinator (FSC) – Responsible for accepting samples into their custody from the sampler(s), producing COCs, and relinquishing or shipping samples to the appropriate facility.

Laboratory Coordinator (LC) – Responsible for coordinating the preparation and/or analysis of Libby Site samples with project facilities in order to achieve requested turnaround times for analytical data.

4.0 Equipment

Depending upon staff responsibility, the following equipment will be employed during use of this SOP:

- Field logbook
- FSDSs
- Indelible blue or black ink pens
- Sample labels
- Zip-top plastic bags
- Custody seals
- Container(s) in which to keep/protect samples

5.0 Procedures

5.1 Preparation

Communications between the TL, sampler(s), the FSC, the LC are critical to ensure the efficient throughput of samples to meet project data objectives. As such, an FSC will attend all field planning meetings to gather information about sampling events (e.g., sample quantities, special sample handling, processing, or analysis concerns, and requested turnaround times). For long-term field programs, sampling staff will notify the FSC daily of the estimated number and type of samples to be collected. In either case, the FSC will relay the pertinent investigation-specific information to the LC, who will, in turn, coordinate preparation and/or analysis with project facilities. On an as-needed basis (typically daily during the field season), the FSC will schedule meetings in which to relinquish samples to the LC.

5.2 Operation

A sample is under custody if it is: 1) in your possession, 2) in your view after being in your possession, 3) in your possession and you locked it up, or 4) in a designated secure area. The following procedures detail the process used to maintain the custody of each Libby Site sample. Note that if at any point samples are left unattended or receipt of samples is refused, this must be documented in the field logbook or on the COC, as appropriate.

5.2.1 Sampler Custody

Sample custody begins at the time of sample collection and will be maintained using a field logbook and FSDSs to document pertinent sample-related information. Samples will be placed in safe areas where they are protected from tampering, damage, or loss. Following sample collection, custody seals will be used as an indicator of tampering. Samples will remain in the sampler's possession, within sight, or in a secure area (e.g., locked vehicle) until the sample is relinquished.

For samples collected using zip-top bags as the primary container, all samples will be double-bagged and custody sealed on the outer bag by the sampler. For samples collected using cassettes, the cassette will be custody sealed so that both end caps of the sampling cassette are covered but sample labels or identifiers are not obstructed. The cassette will then be placed in a zip-top bag.

Sampler(s) may be required to transfer custody of samples directly to an FSC or a designated secure sample storage location, or to hand deliver or ship samples to a facility – refer to the governing document referencing this SOP for specifics. Project-specific SOP EPA-LIBBY-2012-07, *Packaging and Shipping Environmental Samples*, will be followed for samples that are required to be shipped.

If relinquishing to an FSC or secure storage area, the sampler will note in the field logbook the time of transfer, and the name and company affiliation of the receiver or dedicated storage location. Completed and quality-checked FSDSs will accompany the samples.

5.2.2 FSC Custody

Upon receipt of samples and accompany FSDSs, the FSC will verify that:

- Each FSDS is complete
- Each sample is accounted for
- Soil samples are double-bagged
- Each cassette is sealed in its own zip-top bag and caps on cassettes are in place
- Sample containers (e.g., bags, bottles) are tightly sealed
- Custody seals are correctly and securely placed on each sample
- Samples appear to be in an acceptable condition (i.e., cassettes are not cracked; sample containers are not leaking, etc.).
- No information is provided on the sample or sample container that would disclose the origin of the sample to the facility

The FSC will immediately contact the sampler if any acceptance issues are encountered. Once accepted, the FSC will prepare a COC using EPA-specified data management tools (e.g., Data Entry Tool, Scribe). An investigation-specific Analytical Summary Sheet (available in the SAP or Libby Field eRoom) will be attached to the COC. The FSC will group or batch the appropriate number of individual samples on a COC to facilitate data reporting, or as otherwise requested by the LC.

The following general batching guidelines will be used for commonly sampled Libby Site media:

- 10 or fewer non-clearance air samples on one COC
- one set of five clearance air samples and two corresponding field blanks on one COC
- 20 or fewer soil or soil-like (e.g., duff, wood chip) samples on one COC
- 10 or fewer dust samples on one COC

Following coordination with the LC, the FSC will hand deliver or ship samples (following project-specific SOP EPA-LIBBY-2012-07, *Packaging and Shipping Environmental Samples*) to the designated facility. All samples will be maintained in a secure location by the FSC until they are relinquished to another party.

5.3 Post-operation

Sample documentation (logbooks, FSDSs, field copy of the COC, etc.) will be maintained in accordance with Libby Site data management requirements and any special requirements stated in the governing document referencing this SOP (e.g., posting to an eRoom).

6.0 Restrictions/Limitations

For EPA Contract Laboratory Program sampling events, combined chain-of-custody/traffic report forms generated with Scribe or other EPA-specific records may be used. Refer to EPA regional guidelines for completing these forms. Scribe software may be used to customize sample labels and custody records when directed by the client.

7.0 Quality Assurance/Quality Control

Quality assurance/quality control (QA/QC) for activities described in this SOP will be attained through a variety of processes, including, at a minimum, the items discussed below. Additional QA/QC requirements, such as audits or field assessments, will be addressed in the governing document referencing this SOP.

7.1 Training

Every effort will be made to ensure proper sample custody from the point of collection to final disposition. Sample custody will be maintained to the extent possible through proper training, use of designated field staff, and provision of TL oversight. Any deficiencies or inconsistencies in implementing this SOP noted by the TL will require staff re-training.

7.2 Field Checks

Field checks for adherence to this SOP may be performed on a daily basis by the TL for the first week of each field activity. These checks can be extended to once per month as field activities continue. Any non-compliance issues will be discussed with field personnel and corrected. If field activities continue beyond six months, the frequency of assessing sample custody procedures will be established by the field Quality Assurance Manager.

8.0 References

Adapted from CDM Smith Technical Standard Operating Procedure 1-2, Sample Custody, January 2012.

Libby Asbestos Superfund Site Standard Operating Procedure Packaging and Shipping Environmental Samples

Prepared by: *Leif Crowell* Date: 4/12/12
CDM Smith

Approved by: *Dania Zimmer* Date: 4/12/12
EPA Region 8

Revision No.	Date	Reason for Revision
0	4/12/12	--

1.0 Objective

The objective of this standard operating procedure (SOP) is to establish baseline requirements, procedures, and responsibilities for the packaging and shipping of environmental samples collected by the U.S. Environmental Protection Agency or its contractors in support of the Libby Asbestos Superfund Site (Libby Site). Sections 2.0 through 7.0 of this SOP outline requirements for the packaging and shipping of regulated environmental samples under the U.S. Department of Transportation (DOT) Hazardous Materials Regulations, the International Air Transportation Association (IATA), and International Civil Aviation Organization (ICAO) Dangerous Goods Regulations (for shipment by air) and applies only to domestic shipments.

This SOP does not cover the requirements for packaging and shipment of equipment or bulk chemicals that are regulated under the DOT, IATA, and ICAO, nor does it address shipment of hazardous materials. Hazardous material will not be shipped unless personnel have received training that meets the requirements of the governing agency and the DOT.

Additions or modifications to this SOP may be detailed in governing documents referencing this SOP.

2.0 Background

2.1 Definitions

Bottle ware – Plastic or glass bottles or jars used to contain sampled material. Their purpose is to keep sampled material from mixing with the ambient environment.

Chain-of-custody record (COC) – Used to document the custody, control, transfer, analysis, and disposition of samples.

Custody seal – An adhesive-backed seal that is applied to an individual sample or sample container to demonstrate that sample integrity has not been compromised during sample transfer.

Environmental sample – An aliquot of air, water, plant material, sediment, or soil that represents potential contaminant levels at a site. This procedure applies only to environmental samples that

contain less than reportable quantities for any foreseeable hazardous constituents according to DOT regulations promulgated in 49 CFR - Part 172.101 Appendix A.

Facility – A sample processing facility, analytical laboratory, or long-term storage area that serves as the receiver for Libby Site samples.

Excepted quantity – Excepted quantities are limits to the mass or volume of a hazardous material in the sample containers below which DOT, IATA, ICAO regulations do not apply. The excepted quantity limits are very low. Most regulated shipments will be made under limited quantity.

Libby Asbestos Superfund Site (Libby Site) – All buildings and land within the boundaries of the EPA's designated operable units (OUs), as illustrated on the most recent version of the OU boundary map.

Limited quantity – Limited quantity is the maximum amount of a hazardous material below which there are specific labeling or packaging exceptions.

Performance testing – Performance testing is the required testing of outer packaging. These tests include drop and stacking tests.

Qualified Shipper – A qualified shipper is a person who has been adequately trained to perform the functions of shipping hazardous materials.

Site – All buildings (if applicable) and land within the boundaries of the EPA's designated geounits, which may represent individual properties within the Libby Site, a collection of properties, or a larger geographical area.

2.2 Discussion

Proper packaging and shipping is necessary to ensure the integrity of environmental samples during transport. These shipments are potentially subject to regulations published by DOT, IATA, or ICAO. Failure to abide by these rules places both the governing agency and the individual employee at risk of serious fines.

3.0 Responsibilities

Successful execution of this SOP requires a clear definition of assigned roles and responsibilities. All staff responsible for packaging or shipping Libby Site environmental samples will understand and implement the requirements contained herein, as well as any additional requirements stated in governing documents referencing this SOP.

Team Leader (TL) – Responsible for overseeing sample packaging and shipping processes as described in this SOP.

Packer/Shipper – Party (typically the Field Sample Coordinator or Sampler) responsible for properly packaging and shipping samples to the designated project facility.

Qualified Shipper – Responsible for ensuring that samples undergoing shipment contain no other contaminant that meets the definition of "hazardous material" as defined by DOT, and for determining the amount of preservative in each sample so that accurate determination of quantities can be made.

4.0 Equipment

4.1 Environmental Samples without Preservatives

The following equipment will be used when packaging and shipping Libby Site samples:

- Shipping containers (e.g., insulated cooler for limited quantities, a sturdy box for air samples)
- Bubble wrap or other space filler
- Heavy-duty plastic garbage bags
- Plastic zip-top bags
- Custody seals
- Clear packaging tape
- Completed chain-of-custody record
- Duct tape
- Completed shipping label
- Completed return address label (for return of coolers)

Vermiculite, shredded paper, expanded polystyrene, or other absorbent material will not be used for packaging or shipping Libby Site samples. Plastic bubble wrap and ice (as required) is acceptable packing material.

4.2 Environmental Samples with Preservatives

In addition to the equipment listed in Section 4.1, the following additional equipment is required when packaging samples containing preservatives:

- Sample containers
- Insulated coolers
- ice packs/bags or “blue ice”
- Sample labels
- Nitrile gloves

5.0 Procedures

5.1.1 Preparation

Considerations that must be made prior to shipping samples include selecting the appropriate shipping option (e.g., overnight delivery) so that analytical holding times for the samples are not exceeded; packaging samples in time to meet courier or shipping service pick-up times; and making arrangements with the project facility regarding Saturday receipt of samples.

5.2 Operation

5.2.1 Solid Media Samples without Preservatives

The following processes will be employed by the Packager/Shipper for non-preserved, solid media samples (soil, duff, bark, bulk material), and samples collected on cassettes (air, dust). Section 5.2.2 provides procedures for packaging and shipping aqueous samples (groundwater, surface water), or samples with aqueous content (sediment, sludge). Due to the potential for cross contamination, samples collected on cassettes must not be shipped in the same container as solid media samples. Refer to the guidance document referencing this SOP for temperature control requirements (ice).

1. Verify the samples undergoing shipment meet the definition of an “environmental sample” and are not a hazardous material as defined by DOT. Professional judgment and/or consultation with qualified persons such as the Health and Safety Manager shall be observed.
2. Select a sturdy shipping container. Ensure that coolers are in good repair. Air and dust samples must be shipped in separate containers from solid media samples.
3. Place samples into the shipping container. During placement, ensure custody seals are securely in place and verify the contents of the shipping cooler against the COC. The COC shall reflect only those samples within the shipping container.
4. Fill all remaining space with bubble wrap or other appropriate space filler, to prevent the sample(s) from being jostled.
5. After the COC has been signed and dated (time included), retain the field copy of the COC. If using a cooler, place the following items into a zip-top plastic bag for inclusion in the cooler: the top two copies of the COC, an analytical parameters table (if applicable), a copy of the investigation-specific analytical requirements summary sheet (applicable to any asbestos analysis), a completed return shipping label for return of the cooler, and any additional contact, results distribution, or billing information. Tape the sealed zip-top bag to the inside of the cooler lid and securely close. If using a box, include all aforementioned documentation inside the box along with the samples.
6. Attach a completed custody seal across the opening of the shipping container on opposite sides. If using a cooler, the cooler lid shall be secured with tape by wrapping each end of the cooler a minimum of two times. The tape shall be affixed to the cooler so that only half of the custody seal is covered, preventing the cooler from being opened without breaking the seal.
7. Secure the completed shipping form to the shipping container. Schedule the container for pickup or drop off at shipper.
8. Once the container is shipped, notify the laboratory of the shipment number and anticipated arrival date/time.

5.2.2 Aqueous or Aqueous-content Samples without Preservatives

This process below will be employed by the Packager/Shipper for non-preserved, aqueous (or aqueous content) samples collected in bottle ware (water, sediment, sludge). Refer to the guidance document referencing this SOP for temperature control requirements (ice).

1. Verify the samples undergoing shipment meet the definition of an “environmental sample” and are not a hazardous material as defined by DOT. Professional judgment and/or consultation with qualified persons such as the Health and Safety Manager shall be observed.
2. Be sure the caps on all bottles are tightened to prevent leaking. Ensure custody seals are securely in place.
3. For glass containers, wrap each container in bubble wrap and secure with waterproof tape to prevent breakage.
4. Place each plastic or bubble-wrapped glass container into a zip-top bag. Smaller glass containers, such as 40-milliliter vials, may be wrapped together for the same sample.
5. Remove as much trapped air when sealing the bag.

6. Select a sturdy cooler in good repair. To control contents: duct tape closed any interior drain plugs from the inside; duct tape closed any exterior drain plugs from the outside; and line the cooler with two large heavy-duty plastic garbage bags.
7. Place the samples into the cooler with sufficient space to allow for the addition of packing material between the samples. It is preferable to place glass sample bottles and jars into the cooler vertically (glass containers are less likely to break when packed vertically rather than horizontally). During placement, verify the contents of the shipping cooler against the COC. The COC shall reflect only those samples within the cooler.
8. Fill all remaining space with bubble wrap or other appropriate space filler to prevent the sample(s) from being jostled.
9. After the COC has been signed and dated (time included), retain the field copy of the COC. Place the following items into a zip-top plastic bag for inclusion in the cooler: the top two copies of the COC, an analytical parameters table (if applicable), a copy of the Analytical Summary Sheet as provided in the governing document referencing this SOP (only applicable to asbestos analysis), a completed return shipping label for return of the cooler, and any additional contact, results distribution, or billing information. Tape the sealed zip-top bag to the inside of the cooler lid and securely close.
10. Fill all remaining space between the samples with packing material. Remove excess air from garbage bags and seal each bag by securely taping the opening closed and then applying a custody seal on the outermost bag.
11. Attach a completed custody seal across the opening of the cooler on opposite sides. The cooler lid shall be secured with tape by wrapping each end of the cooler a minimum of two times. The tape shall be affixed to the cooler so that only half of the custody seal is covered, preventing the cooler from being opened without breaking the seal.
12. Secure the completed shipping form to the shipping container. Schedule the container for pickup or drop off at shipper.
13. Once the container is shipped, notify the laboratory of the shipment number and anticipated arrival date/time.

5.2.3 Samples Requiring Temperature Controls

If temperature controls (i.e., ice) are required (refer to the guidance document referencing this SOP), in addition to the procedures listed in Section 5.2.1 (for solid media samples) or Section 5.2.2 (for aqueous samples), the Packager/Shipper will:

1. Duct tape closed any drain plugs (inside and outside) and line the cooler with two large heavy-duty plastic garbage bags. (This step will already have been performed for aqueous/aqueous-content samples.)
2. Place ice in one-gallon plastic zip-top bags and properly seal the bags.
3. Place bags of ice on top of and between the samples to ensure adequate temperature controls during transport.
4. Ensure a temperature blank is secured inside the cooler.

5.2.4 All Samples with Preservatives

Prior to shipping samples with preservatives, the Qualified Shipper will determine the amount of preservative in each sample. Excepted quantities of preservatives are provided in the following table:

Excepted Quantities of Preservatives

Preservative		Desired in Final Sample		Quantity of Preservative (ml) for Specified Container				
		pH	Conc.	40 ml	125 ml	250 ml	500 ml	1 L
5 drops = 1 ml								
NaOH	30%	>12	0.08%	--	0.25	0.5	1	2
HCl	2N	<1.96	0.04%	0.2	0.5	1	--	--
HNO ₃	6N	<1.62	0.15%	--	2	4	5	8
H ₂ SO ₄	37N	<1.15	0.35%	0.1	0.25	0.5	1	2

Conc. = concentration
ml = milliliters
% = percent
L = liter

NaOH = sodium hydroxide
HCl = hydrochloric acid
HNO₃ = nitric acid
H₂SO₄ = sulfuric acid

In addition to the steps outlined in the appropriate section above for the specific media sampled, these additional steps are to be followed when packaging limited-quantity sample shipments:

1. Nitrile gloves are to be worn by anyone handling the sampling containers.
2. All sample containers will be labeled with the sample number and what preservative is being used. Protect the labels with waterproof tape. At a minimum the sample label must contain:
 - Sample number
 - Project or Case number
 - Date and time of sample collection
 - Preservative
 - Analysis

The FSDS will be used to collect all other sample information.

3. The Packager/Shipper will ensure a trip blank(s) is secured inside the cooler(s).
4. The maximum weight of the cooler shall not exceed 30 kg (66 lbs) for any limited-quantity shipment of dangerous goods.

5.3 Post-operation

Shipping documentation will be maintained by the Packager/Shipper to confirm that shipments have been delivered and accepted by the receiver.

6.0 Quality Assurance/Quality Control

Quality assurance/quality control (QA/QC) for activities described in this SOP will be attained through a variety of processes, including, at a minimum, the items discussed below. Additional QA/QC requirements, such as audits or field assessments, will be addressed in the governing document referencing this SOP.

6.1 Training

Every effort will be made to ensure proper sample custody from the point of collection to final disposition. Sample custody will be maintained to the extent possible through proper training, using designated field staff, and providing TL oversight. Any deficiencies or inconsistencies in implementing this SOP noted by the TL will require staff re-training.

6.2 Field Checks

Field checks for adherence to this SOP may be performed on a daily basis by the TL (or their designate) for the first week of each investigation. These checks can be extended to once per month as investigation activities continue, and any errors noticed during the checks will be discussed with field personnel and corrected. If investigation activities continue beyond six months, the frequency of assessing sample packaging and shipping procedures will be established by the field Quality Assurance Manager or their designate.

7.0 References

Adapted from CDM Smith Technical Standard Operating Procedure 2-1, Packaging and Shipping Environmental Samples, January 2012.

Libby Asbestos Superfund Site Site-specific Procedure Completion of Field Sample Data Sheets

Prepared by: Tracy Dodge Date: 3/28/14
Tracy Dodge, CDM Smith

Reviewed by: Diane M. Rode Date: 3/27/14
Diane Rode, CDM Smith Technical Reviewer

Reviewed by: Terry Crowell Date: 3/27/14
Terry Crowell, CDM Smith Quality Assurance Reviewer

Revision No.	Date	Reason for Revision
0	5/8/02	--
1	5/16/03	Annual update to align guidance with current versions of FSDSs
2	--	Not finalized/approved
3	4/12/06	Annual update to align guidance with current versions of FSDSs
4	4/13/09	Annual update to align guidance with current versions of FSDSs
5	5/26/09	Minor administrative changes to address FSDS changes
6	4/18/12	Annual update to align guidance with current versions of FSDSs
7	3/27/14	Annual update to align guidance with current versions of FSDSs

1.0 Objective

The objective of this site-specific procedure is to establish baseline requirements, procedures, and responsibilities for the completion of field sample data sheets (FSDSs) by the U.S. Environmental Protection Agency (EPA) or its contractors in support of the Libby Asbestos Superfund Site (Libby Site). Additions or modifications to this procedure may be detailed in governing documents referencing this procedure.

2.0 Definitions

Data Entry Tool (DET) – A local MS Access® tool used to enter information from the FSDS and used to temporarily store information until it is published to Scribe.

Field sample data sheet (FSDS) – The hard copy form on which sample and location information is recorded.

Libby Asbestos Superfund Site (Libby Site) – All buildings and land within the boundaries of the EPA’s designated operable units (OUs), as illustrated on the most recent version of the OU boundary map. Note that the Libby Site is organized into 8 formal OUs numbered 1 through 8; OU99 is used to identify properties that lie outside the EPA National Priorities List Site boundary

and properties that do not require investigation and response action for Libby amphibole contamination.

Response Manager – An EPA data management system used to manage Libby Site property information.

Scribe – An EPA data management system used to manage location, sample, and analytical data.

3.0 Responsibilities

Team Leader (TL) – Responsible for ensuring that FSDSs are completed in accordance with this procedure and any additional FSDS requirements stated in the governing document referencing this procedure.

Sampler – Responsible for completing FSDSs in accordance with this procedure and any additional FSDS requirements stated in the governing document referencing this procedure.

Field Sample Coordinator (FSC) – Staff member to whom samples and FSDSs are relinquished; responsible for preparing chain-of-custody forms (COCs) and submitting samples to the appropriate project facility.

Office Administrator – Responsible for preparing sample number and location identification (ID) logs and labels, and preparing unique and sequentially numbered FSDSs for completion in the field.

4.0 Operation

4.1 Recording Information for All Sample Media

This section provides background information, as well as descriptions and instructions for completing FSDS data items common to all sample media. Data items specific to certain media are discussed in Section 4.2.

Some FSDS data items are required to be completed to be in compliance with EPA data reporting requirements or the governing document referencing this procedure, or to track other critical field information. These data items will be referred to as “required” throughout this procedure. Required data items are indicated on FSDSs with an asterisk (*). A required data item must be populated with an appropriate valid value. Note that “NA” (not applicable) may be a valid value.

Other data items may be required conditionally. These will be referred to as “conditional” throughout this procedure and these fields will not be asterisked on the FSDS. Conditional data items and any corresponding valid values may be specified in EPA data reporting requirements or the governing document referencing this procedure.

Data items that are not required or conditional may be left blank. Information recorded on the FSDS is entered into the DET.

Field team members are not required to line out any labels, initial, or date them, unless they are making a revision. To revise a data item on an FSDS, line through the incorrect data (single line), record the correct data in close proximity to the erroneous data, and date and initial the change.

Sheet No.: A pre-assigned unique, sequential sheet number assigned by an Office Administrator, in the format: \$\$-##### or \$-#####, where \$ refers to the media being sampled and ##### refers to the sequential number.

Event ID: An identifier for a specific data collection effort, most commonly a combination of the event-specific sample number prefix and the approved date of the document governing the event. These Event IDs use the format: \$-##### or \$\$-#####, where \$ or \$\$ is a one- or two-digit set of characters, as specified in the governing document referencing this procedure, and ##### refers to the governing document date in MMDDYY format.

Address: The concatenated address (as it appears in Response Manager) of the property being investigated and/or sampled.

Date: The date of sample collection in the format MM/DD/YY. For air samples collected over more than one day using the same cassette, the end date (i.e., date the sample period concludes) will be recorded.

Property ID: For non-OU7 properties, a unique identifier assigned to each property in the format: AD-#####, where ##### is a unique number. OU7 and some OU99 Property IDs are in the format: AD-2#####. Property IDs should be verified using Response Manager before being transcribed to the FSDS. Property IDs may be used as Location IDs in appropriate circumstances.

Field Logbook No.: The number of the logbook being used to record information specific to the samples on the FSDS.

Page No.: The page number(s) in the logbook being used to record information specific to the samples on the FSDS.

Sampler(s): The first initial and full last name of all members of the field team. For data entry, the FSC will select only one of the field team members listed. The company affiliation of the field team member(s) need only be listed after their name if they work for a company other than "CDM Smith".

Location ID: A unique number assigned to each location representing the investigated and/or sampled area specific to the information on the FSDS. Previously assigned Location IDs should be verified using Scribe before being transcribed to the FSDS, whenever possible. Contact a member of the onsite data management team for assistance with verification.

Location IDs in the format BD-##### will be assigned to (or used for, in the case of previously assigned Building Location IDs) habitable, fully enclosed primary or secondary buildings, including buildings that may have broken windows and/or missing doors. All primary and secondary buildings will be assigned a BD-##### number.

Location IDs in the format XX-##### will be assigned to secondary structures (e.g., open structures, 3-sided structures, carports, and lean-tos).

Location IDs in the format XX-##### will be assigned to outdoor investigation areas and may be used for any GPI soil samples collected, including samples collected within primary and secondary buildings and secondary structures with prior approval by the TL. XX-##### Location IDs will not be used during removal soil confirmation sampling.

Location IDs in the format SP-##### will be assigned to excavated soil areas (including areas with open structures) during removal soil confirmation sampling. SP-##### Location IDs will be used for air and water monitoring events and fill material sampling as specified in the governing document referencing this procedure.

For personal and stationary air samples, a previously assigned Property ID or Building Location ID will be used in most cases. If a new Location ID is assigned, the Location portion of the Soil-like and Location FSDS must be completed in addition to the Air FSDS.

For lot blanks, "AD-OU4NA" is used for the Property ID and Location ID.

For field blanks, generally, the Property ID where field samples are being collected is used for outdoor sampling, while the Building Location ID is used if sampling occurs indoors. For air and dust field blanks specifically, the Location ID should be used that corresponds to the air space where the field blank is exposed (i.e., Property ID for field blanks exposed in outdoor spaces; Building Location ID for field blanks exposed in indoor living spaces).

Sample ID: Unique number assigned to each sample in the format \$-##### or \$\$-#####, where \$ or \$\$ is a one- or two-digit set of characters indicating the governing document referencing this procedure, and ##### is a 5-digit sequential number.

For Field Team Completion, Completed by: Initials of the field team member, verifying that required data items on the FSDS have been completed correctly.

For Field Team Completion, Quality Checked (QC) by: Initials of the second field team member (independent of the member completing the FSDS) or other trained reviewer, verifying that required data items on the FSDS have been completed correctly.

For Data Entry, Entered by: Initials of the FSC or data entry staff performing data entry of FSDS information into the DET.

For Data Entry, QC by: Initials of the FSC or other trained reviewer verifying FSDS data entered into DET is complete and accurate.

4.2 Recording Location Information

The following sections provide instructions for recording location information on FSDSs. Note that new locations for air sampling locations must be recorded on a Soil-like Sample & Location FSDS.

Is this a new Location?: Indicate "Yes" when assigning a new Location ID, indicate "No" when a Location ID has previously been assigned, and indicate "Revised" when revising previously collected location data. If the response is "No", "Z" through the rest of the location section. Data for new locations will be imported to Scribe. Revised location data will be manually edited in Scribe.

Location Type: Record the location type of the area being investigated and/or sampled.

For removal confirmation soil samples, record "EA" for excavation area. For perimeter or clearance air samples, or water samples, record "NA".

For General Property Investigation (GPI) locations/samples, select from the following values (abbreviations may be used):

SUA – specific-use area	CUA – common-use area	LUA – limited-use area
NUA – non-use area	PB – primary building	SB – secondary building
SS – secondary structure		

Location Description: Record the description of the area being investigated and/or sampled. Select from the values listed below (do not abbreviate). Additional values may be added with prior approval by the TL and FSC.

alley	flowerbed	road (paved)
animal pen	former house foundation	road (unpaved)
apartment	garage	root zone
barn	garden	shed
borrow source	greenhouse	shop
brush	house	shrub bed
building	lean-to	stockpile
burnpile	NA	underneath porches/decks
carport	outhouse	underneath secondary building
corral	park	undeveloped area
decorative gravel/rock	parking lot (paved)	verge
driveway (paved)	parking lot (unpaved)	walkway (paved)
driveway (unpaved)	planter	walkway (unpaved)
field (maintained)	play area	wooded area
field (unmaintained)	property	yard
firepit	pumphouse	

Location Area (ft²): Record the square footage of the area to which the FSDS pertains. This data item may be left blank if not specified in the governing document referencing this procedure.

Location Comment: For GPIs, describe the restoration type applicable to a location. This data item may be left blank if not required by the governing document referencing this procedure.

building	pea gravel	topsoil
chipped rock	potting soil	topsoil w/liner
common fill	sand	washed rock
grass	structural fill	wood chips
landscape rock	tall grass	wooded area

Location Comment 2: Record the detailed description of the location that may not be reflected in the Location Comment. This data item may be left blank if not specified in the governing document referencing this procedure.

4.3 Recording Media-specific Information

The following sections provide instructions for recording media-specific information on FSDSs. FSDS may be customized to accommodate event-specific data requirements (e.g., matrix, if other than soil); however, the TL will consult with the FSC prior to any field work to prepare the customized FSDS.

4.3.1 Soil-Like Material

Use based on: To distinguish whether location information is assigned based on current use or reasonably anticipated future use (RAFU), check the appropriate box. If “Current Use” is selected, or the data item is not applicable (i.e., for non-GPI samples), no data will be entered. If marked “RAFU”, the acronym will be appended to the Location Comment 2 information by the FSC.

Location Zone: Record the location zone if required by the governing document referencing this procedure. This data item may be left blank if not specified in the governing document referencing this procedure.

Visible Vermiculite: Record the total number of visual inspection points of no (N), low (L), intermediate (M), or high (H) levels of vermiculite observed during the semi-quantitative visual inspection for vermiculite. For visible vermiculite observations corresponding to a sample, the sum of these fields must equal the number of sample aliquots (e.g., 30). Values for visual inspection point observations (N, L, M, or H) must be provided; record “0” to indicate no observations were required/made.

Soil Depth Top: Record the top depth of the sample/visual inspection observation, recorded in inches, in relation to ground surface. For samples collected below ground surface, record a positive, whole number. For samples collected above ground surface (e.g., vegetative samples), record a negative, whole number.

Soil Depth Bottom: Record the bottom depth of the sample/visual inspection observation, recorded in inches, in relation to ground surface. For samples collected below ground surface, record a positive, whole number. For samples collected above ground surface (e.g., vegetative samples), record a negative, whole number.

VV Sub Location: For exterior samples, record “property (exterior)”. For GPI interior locations, select from the list below. If “other interior soil” is selected, record details in the visible vermiculite comments. This data item may be left blank if not specified in the governing document referencing this procedure.

property (exterior)	crawlspace	soil floor
basement	cellar	interior planter
other interior soil		

Visible Vermiculite Comments: Record any comments pertaining to the visual inspection observation. This data item may be left blank if not specified in the governing document referencing this procedure.

Sample ID: Record the unique sample number assigned to each sample, as designated by the governing document referencing this procedure.

Sample Time: Record the time (in military units) the sample was collected.

ABS: Record whether the sample was collected as part of an activity-based sampling program.

Sample Venue: Record whether the sample was collected indoors or outdoors. Record “NA” for field blanks.

Sample PrePostClear: For removal confirmation soil samples, circle the appropriate clearance sequence. For all other samples, circle “NA” unless otherwise specified in the governing document referencing this procedure.

Sample Type: Circle “FS” for a field sample, “FD” for a field duplicate, or write in an alternative sample type if specified in the governing document referencing this procedure.

Delineation sample?: This question is not a required database item, rather a cue for the sampler to record the parent sample ID the next field. Circle “No” or “Yes”.

Sample Parent ID: For field QC samples (e.g., field duplicates), record the Sample ID of the parent field sample. Refer to the governing document referencing this procedure for field QC sample requirements. For other requirements using Sample Parent ID (e.g., delineation samples), refer to the governing document referencing this procedure.

Composite: Indicate if the sample collected is a composite of multiple aliquots. Circle “N” if the sample is a grab sample.

Sample/Inspection Aliquots: For 30-point composite samples, circle “30”, or indicate the number of aliquots collected/inspected in the space provided. If a grab sample was collected, circle “0”.

Sample Location Description: For exterior removal confirmation soil samples, provide the sampling area designation(s) corresponding to the draft redline sketch. For interior removal confirmation soil samples, record the building description and the sampling area designation(s) corresponding to the draft redline sketch location of where the sample was collected (e.g., Area 1 – greenhouse; Area 12 – pumphouse; Area 3 – crawlspace). For GPI and other sampling programs, provide any detailed location information that may not be reflected in the general Location Description, such as specific location within the building that was sampled (e.g., middle of barn, SW corner of crawlspace.) The square footage of the sampled area inside a building may be recorded here.

Sample Field Comments: Record any additional information that may be important to data users. Refer to the governing document referencing this procedure for any specific requirements.

4.3.2 Stationary Air

As mentioned in Section 4.1, a previously assigned Property ID or Building Location ID will be used on the FSDS for stationary air samples in most cases. Property IDs are used for stationary air samples collected outside buildings, while Building Location IDs are used for samples collected inside buildings. If a new Location ID is assigned, the Location portion of the Soil-like and Location FSDS must be completed in addition to the Air FSDS.

Sample ID: A unique sample number assigned to each sample, as designated by the governing document referencing this procedure.

ABS: Record whether the sample was collected as part of an activity-based sampling program.

Sample Venue: Record whether the sample was collected indoors, outdoors, both, or NA. The Sample Venue for field blanks should be recorded as "NA". For samples collected inside a vehicle with the windows closed, circle "Indoor". For samples collected inside a vehicle with the windows open, circle "Both".

Sample PrePostClear: For removal clearance air samples, circle the appropriate clearance sequence. For all other samples, including field blanks, circle "NA" unless otherwise specified in the governing document referencing this procedure.

Sample Type: Circle "FS" for a field sample, "FD" for a field duplicate, "LB" for lot blank, "DB" for drying blank, or write in an alternative sample type as specified in the governing document referencing this procedure.

Sample Parent ID: Applicable to the high volume sample, when co-located high- and low-volume samples are collected. For the high-volume sample, record the low-volume Sample ID as the Sample Parent ID. For the low-volume sample, the Sample Parent ID is left blank.

Sample Location Description: Provide a detailed description of the indoor or outdoor sample location. Record "Blank" for field blanks. Refer to the governing document referencing this procedure for any additional requirements.

Sample Air Type: Circle the appropriate stationary air type (Ambient or Perimeter). The Sample Air Type for blanks should be recorded as "NA".

Sample Air Volume Type: When co-located high- and low-volume samples are collected, record "LV" for low-volume or "HV" for high-volume samples. Record "NA" for all other samples.

Flow Meter Type: Circle the applicable flow meter used. Circle "NA" for all types of blank samples.

Cassette Lot Number: Record the cassette lot number of the sample cassettes being used.

Flow Meter ID Number: Record the identification number of the flow meter used. If more than one flow meter is used, use Sample Field Comments to record the additional Flow Meter ID(s).

Pump ID Number: Record the ID of the pump used. If more than one pump is used, use Sample Field Comments to record the additional pump ID(s), and provide the reason for use of multiple pumps. For all types of blank samples, "Z" out the data items from "Pump ID" to "Sample Air Stop Flow".

Sample Air Start Date: Record the start date in the format MM/DD/YY. Note that multiple start and stop dates/times, as well as start and stop flow rates, may need to be recorded for samples collected over multiple days using the same cassette. Refer to the governing document referencing this procedure for additional requirements.

Start Time: Record the starting time (in military units) of each air sample aliquot.

Start Flow: Record the starting pump flow rate, in liters per minute (L/min) for the air sample collected.

Stop Date: Record the stop date in the format MM/DD/YY.

Stop Time: Record the stopping time (in military units) of each air sample aliquot.

Stop Flow: Record the stopping pump flow rate (in L/min) for the air sample collected. If a flow rate is recorded while the pump is running, the stop time and next recorded start time will be the same.

Pump Fault: Circle "Y" or "N" to indicate a pump fault. For all types of blank samples, circle "NA". Use Sample Field Comments to note if a pump faulted during air sample collection, as determined by an unacceptable flow rate deviation (refer to the governing document referencing this procedure for flow rate requirements), or due to a mechanical fault (pump shut-off).

Sample Total Time (min): Sample Total Time is the total sample collection period in minutes (min). TLs will provide direction on calculating sample times. Generally, removal-related air sample total times will be calculated by the FSC, while other programs (e.g., ABS) will call for samplers to calculate total times.

Sample Quantity (L): The sample quantity represents the total volume in liters (L) of the sample collected. TLs will provide direction on calculating sample quantities. Generally, removal-related air sample quantities will be calculated by the FSC, while other programs (e.g., ABS) will call for samplers to calculate sample quantities.

Sample Field Comments: Record any additional information that may be important to data users. Refer to the governing document referencing this procedure for any specific requirements.

Filter Diameter: For all standard Libby Site air sampling, sample cassettes with a 25-millimeter filter diameter will be used. This data item is pre-printed on the Air FSDS.

Pore Size: For standard Libby Site air sampling, sample cassettes with a 0.8-micron filter pore size will be used. This data item is pre-printed on the Air FSDS.

4.3.3 Personal Air

Complete Personal Air FSDSs as for Stationary Air, with the following adjustments:

Sample PrePostClear: For all personal air samples and blanks, circle "NA" unless otherwise specified by the governing document referencing this procedure.

Sample Air Type: Circle one of the following personal air types:

- TWA – Time-weighted average sample, collected over an 8-hour period (may be composited with other personal air samples to represent an average work day)
- EXC – Excursion sample, collected over a 30-minute period (time may be approximate)

- ABS – Sample collected during activity-based sampling (not health and safety related)
- NA – Use for all types of blank samples, or as otherwise specified in the governing document referencing this procedure

Personnel ID: Record the company-assigned ID of the worker being monitored.

Name: Record the first and last name of the worker being monitored.

Personnel Task: For health and safety-related samples, select from the list below. For samples collected as part of ABS, refer to the governing document referencing this procedure for requirements.

bulk removal	investigation (Level D)	removal oversight (Level D)
demolition	laborer	support personnel
detailing attic	operator	truck driver (Level C)
excavator operator	other	truck driver (Level D)
investigation (Level C)	removal oversight (Level C)	wet wipe/HEPA vac living space

For samples collected at Rainy Creek Rd or Lincoln County Landfill, select the most appropriate value from the list above, and then provide additional information in Sample Field Comments from the list below:

upper dozer	laborer - PAPR
water truck driver – PAPR	equipment operator - PAPR
truck driver – PAPR	truck driver – Level C and Level D

4.3.4 Bulk-Like Material

Sample Time: Record the time (in military units) the sample was collected.

ABS: Record whether the sample was collected as part of an activity-based sampling program.

Matrix if other than Bulk: Record tissue, ash, or other bulk-like material here.

Sample Venue: Record whether the sample was collected indoors or outdoors. Record “NA” for field blanks.

Sample PrePostClear: For removal-related samples, circle the appropriate clearance sequence. For all other samples, circle “NA” unless otherwise specified in the governing document referencing this procedure.

Sample Type: Circle “FS” for a field sample, “FD” for a field duplicate, or write in an alternative sample type if specified in the governing document referencing this procedure.

Sample Parent ID: For field QC samples (e.g., field duplicates), record the Sample ID of the parent field sample. Refer to the governing document referencing this procedure for field QC sample requirements.

Composite: Indicate if the sample collected is a composite of multiple aliquots. Circle "N" if the sample is a grab sample.

Sample/Inspection Aliquots: For 30-point composite samples, circle "30", or indicate the number of aliquots inspected/collected in the space provided. If a grab sample was collected, circle "0".

Sample Location Description: Record any detailed location information that may not be reflected in the general Location Description, such as specific location within the building that was sampled (e.g., chimney; chinking SW wall). Refer to the governing document referencing this procedure for any specific requirements.

Sample Field Comments: Record any additional information that may be important to data users. Refer to the governing document referencing this procedure for any specific requirements.

4.3.5 Water

Sample Time: Record the time (in military units) the sample was collected.

ABS: Record whether the sample was collected as part of an activity-based sampling program.

Sample Venue: Record whether the sample was collected indoors or outdoors. Record "NA" for field blanks.

Sample PrePostClear: Circle "NA" unless otherwise specified in the governing document referencing this procedure.

Sample Type: Circle "FS" for a field sample, "FD" for a field duplicate, or write in an alternative sample type if specified in the governing document referencing this procedure.

Sample Parent ID: For field QC samples (e.g., field duplicates), record the Sample ID of the parent field sample. Refer to the governing document referencing this procedure for field QC sample requirements.

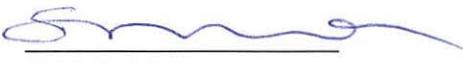
Composite: Indicate if the sample collected is a composite of multiple aliquots. Circle "N" if the sample is a grab sample.

Sample/Inspection Aliquots: For 30-point composite samples, circle "30", or indicate the number of aliquots inspected and/or collected in the space provided. If a grab sample was collected, circle "0".

Sample Location Description: Record any detailed location information that may not be reflected in the general Location Description. Refer to the governing document referencing this procedure for any specific requirements.

Sample Field Comments: Record any additional information that may be important to data users. Refer to the governing document referencing this procedure for any specific requirements.

Libby Asbestos Superfund Site Site-specific Procedure 30-point Composite Sampling of Surface Soil for Asbestos

Prepared by:  Date: 4-1-14
CDM Smith

Reviewed by:  Date: 4/1/14
CDM Smith Technical Reviewer

Reviewed by:  Date: 4/1/14
CDM Smith Quality Assurance Reviewer

Revision No.	Date	Reason for Revision
0	5/7/02	--
1	5/17/03	<ul style="list-style-type: none"> • Administrative updates • Updated land use area designations • Updated sampling approach to collect samples in large land use areas (driveways and yards) where vermiculite is observed
2	5/10/07	<ul style="list-style-type: none"> • Administrative updates • Addition of Responsibilities and Sample Custody sections • Separate QA/QC requirements into new section • Updated sampling approach and collection requirements, including: <ul style="list-style-type: none"> – subsample requirements changed from 5-point to 30-point – refinement of property zone definitions and sizes – updated land use area designations – changes in sample depth increments for use areas – use of formalized procedure for the semi-quantitative estimation of visible vermiculite in soil
3	5/1/12	<ul style="list-style-type: none"> • Administrative updates • Eliminate the use of bowls used to homogenize soil samples • Eliminate the use of aluminum foil for wrapping re-usable sampling equipment during transport • Addition of reference to Libby Site-specific standard Operating Procedures throughout • Change in composited soil sample size from 2,000 – 2,500 grams to 750 – 1,000 grams
4	2/12/13	<ul style="list-style-type: none"> • Clarify the definition of visible vermiculite
5	4/1/14	<ul style="list-style-type: none"> • Administrative updates • Addition of Secondary Structure to use areas

1.0 Objective

The objective of this site-specific procedure is to establish baseline requirements, procedures, and responsibilities for the collection of 30-point composite surface soil samples by the U.S. Environmental Protection Agency (EPA) or its contractors related to investigations conducted at the Libby Asbestos Superfund Site (Libby Site). This procedure describes the equipment and operations to be used for sampling surface soils for the analysis of Libby amphibole asbestos. Additions or modifications to this procedure may be detailed in governing documents referencing this procedure.

2.0 Definitions

Composite sampling – A sampling approach in which multiple sample points are compiled together and submitted for analysis as a single sample.

Field sample data sheet (FSDS) – The controlled (i.e., pre-numbered and tracked) hard copy form on which sample and location information, and any visible vermiculite observations, is recorded.

Land use area – A portion of a property segregated according to how the property owner uses the area.

Libby Asbestos Superfund Site (Libby Site) – All buildings and land within the boundaries of the EPA's designated operable units (OUs), as illustrated on the current version of the OU boundary map.

Point inspection (PI) – A PI is an intrusive visual inspection of the top portions of the soil at a randomly selected point within a land use zone. A PI consists of the active displacement of the surface soil with a small shovel and visual inspection of the displaced soil and surface soil within an approximate 2-foot radius of the displaced soil (i.e., immediate field of view) for visible vermiculite (VV). If VV is observed during the PI, the location and a semi-quantitative estimate of VV will be recorded.

Subsample – The portion of a composite sample representing a discreet location within the sampled area.

Visible Vermiculite – Exfoliated and/or unexfoliated vermiculite, amphibole asbestiform minerals, and mine tailings present in soils as part of response actions – herein collectively referred to as visible vermiculite (VV).

3.0 Responsibilities

Successful execution of this procedure requires a clear hierarchy of assigned roles with different sets of responsibilities associated with each role. All staff responsible for collecting soil samples using this procedure will understand and implement the requirements contained herein, as well as any additional requirements stated in governing documents referencing this procedure.

Team Leader (TL) – The TL is responsible for overseeing the sample collection process outlined in this procedure, and for checking and verifying that the work performed satisfies the objectives of the governing document referencing this procedure. The TL will communicate with the field team members regarding specific collection objectives, and will communicate the need for any deviations from this procedure with the appropriate client personnel, and document the deviations using a Libby Field Record of Modification Form, as provided in the governing document referencing this procedure.

Field Team Members - Field team members performing the sampling described in this procedure are responsible for adhering to the tasks specified herein. The field team members should have limited discretion with regard to collection procedures but should exercise judgment regarding the exact location of sample points, within the boundaries outlined by the TL.

4.0 Equipment

The following equipment will be used during implementation of this procedure:

- Measuring tape or wheel – Used to estimate the square footage of each land use area.
- Pin flags – Used to identify subsample points within each sampling area.
- Trowel or push probe
- Shovel
- Gallon-sized plastic zip-top bags – Used to homogenize soil subsamples following collection (two bags per sample).
- Personal protective equipment (PPE) – For personal protection and to prevent cross-contamination of samples (e.g., disposable, powderless plastic or latex gloves).
- Field sprayers – Used to suppress dust during sample collection and to decontaminate non-disposable sampling equipment between samples.
- De-mineralized water – Used in field sprayers to suppress dust and to clean and decontaminate sampling equipment.
- Plastic bristle brush – Used to clean and decontaminate sampling equipment.
- Alconox – Used to clean and decontaminate sampling equipment weekly.
- Paper towels – Used to dry decontaminated sampling equipment.
- 6-mil poly bag – Used to store and dispose of investigation-derived waste (IDW).
- Trash bag – Used to store and dispose of general trash.
- Indelible ink pen (blue or black ink only)
- Field logbook – Used to record progress of sampling effort and record any problems and field observations.
- Blank FSDSs

- Sample Identification (ID) Labels – Pre-printed self-adhesive stickers used to label sample containers and on field documentation (e.g., FSDSs).
- Cooler or other rigid container – Used to store samples while in the field.
- Custody Seals – Self-adhesive seals applied to an individual sample or sample container to demonstrate that sample integrity has not been compromised during sample transfer.

5.0 Sampling Approach

Upon arrival at each property, the field team will locate all parcels requiring sample collection depending on the investigation-specific objectives detailed in governing guidance documents. Parcels on a property will be sectioned into zones that share a similar land use. Zones established by land use areas may be subdivided based on site conditions (e.g., access, construction setup considerations, etc.). Use areas include:

- Specific-use area (SUA): flowerbed, garden, stockpile, play area, dog pen, driveway (non-paved), parking lot (non-paved), road (non-paved), alley (non-paved), fire pit/burn pile
- Common-use area (CUA): yard, former garden, former flowerbed, walkway, maintained/mowed field
- Limited-use area (LUA): pasture, un-maintained field, overgrown areas with trails/footpaths, overgrown areas in between SUAs/CUAs
- Non-use area (NUA): wooded lot, NUAs will be identified but will not be sampled because they are not presently considered a complete exposure pathway. However, to the extent that NUAs may become a complete exposure pathway in the future, they may be revisited.
- Primary building (PB): crawlspace, earthen basement
- Secondary building (SB): soil floor of garage, pumphouse, shed, greenhouse, etc.
- Secondary structure (SS): lean-to, barn

After areas have been designated as zones (i.e., SUA zones, CUA zones, LUA zones, etc.), the field team will measure the zones with a measuring wheel and label the zone type and approximate square footage on the field sketch and/or design drawings. This procedure does not specify a minimum or maximum square footage restriction on any zone; however, the governing document referencing this procedure may specify zone size.

In establishing zones at the property, no area type may be combined with any other area type. For example, driveways and flowerbeds are both SUAs but will be separated into unique zones for soil sampling. Similarly, large CUAs such as yards may be subdivided into front yard, side yard, and back yard zones dependent on site conditions. Sectioning properties into additional zones will be at the discretion of the TL but consistent among the teams. Conversely, not all land use areas previously mentioned will be applicable at every property.

It is anticipated that SUA, SS, PB, and SB zones will generally tend to be smaller areas. Combining small, proximal SUAs of similar type into one zone will be at the discretion of the TL but consistent

among teams (e.g., two separate flowerbeds). With the exception of proximal SUAs, all other land use areas will be contiguous when establishing zones at each property.

Composite sampling requires soil collection from multiple (subsample) points. Composite samples will be collected from similar land use areas (i.e., SUA, CUA, etc.) and will not be combined with any other use area.

For SUAs (e.g., driveway, garden, flowerbed), composite samples will be collected from the 0- to 6-inch depth interval. If a depth of 6 inches cannot be attained given the varying levels of compaction in driveways, roads, etc. the maximum depth attainable will be documented on the FSDS. For non-SUAs (e.g., yard, former flowerbed, crawlspace, etc.), composite samples will be collected from the 0- to 3-inch depth interval. All composite soil samples will have 30 subsamples (i.e., 30-point composite sample) of approximately equal size for a final sample volume between 750 and 1,000 grams. Table 1 lists the sample depth for each type of land use area.

Table 1. Sampling Area and Depth

Land Use Area	Sampling Depth Increment (inches)
Specific-use Area (SUA)	0 – 6
Common-use Areas (CUA)	0 – 3
Limited-use Area (LUA)	0 – 3
Non-use Area (NUA)	Not Sampled
Secondary structure (SS)	0 – 3
Secondary Building (SB)	0 – 3
Primary Building (PB)	0 – 3

In cases where an SS or SB is used in the same manner as an SUA (e.g., a greenhouse where part or all of the soil floor is used as a garden), the sampling team shall use the more conservative (i.e., deeper) sampling depth.

As each subsample is collected, the soil will be inspected for VV and the location and semi-quantitative estimates of VV will be recorded on the FSDS in accordance with the current version of CDM-LIBBY-06 (Semi-Quantitative Visual Estimation of Vermiculite in Soil). Areas with VV will not be sampled with areas that do not have VV. However, if an SUA is less than 1,000 square feet (ft²), it is not necessary to split it into samples with and without VV.

6.0 Sample Collection

Don the appropriate PPE as specified in the governing health and safety plan and/or governing document referencing this procedure. A new pair of disposable gloves will be worn for each sample collected. Segregate land use areas on the property into zones as described in Section 5.0. To reduce dust generation during sampling, use a sprayer with de-mineralized water to wet each subsample location prior to collection. Use the trowel to check beneath the surface soil layer, but do not advance more than 6 inches. If VV is observed, record the information on the field sketch or design drawing.

Within each zone, select 30 subsample locations equidistant from each other. These 30 subsample locations will comprise the 30-point composite sample for that zone. All composite subsamples will originate from the same land use area – do not mix subsamples from one land use area with subsamples from a different land use area.

Clean the subsample locations of twigs, leaves, and other vegetative material that can be easily removed by hand. Using the trowel or push probe, excavate a hole in the soil approximately 2 inches in diameter and 6 inches deep for SUAs, or 3 inches deep for non-SUAs. Conduct PI and place the material into the zip-top plastic bag. Repeat this step for each subsequent subsample until the appropriate number of composite subsamples has been collected. VV observations associated with a sample will be recorded on the FSDS as described in the current version of CDM-LIBBY-06 (Semi-Quantitative Visual Estimation of Vermiculite in Soil).

Homogenize the sample as required by the governing document referencing this procedure. Once the sample is homogenized, fill the zip-top plastic bag approximately a quarter full (750 – 1,000 grams of material). Affix the sample ID label to the inside of the bag and write the sample ID number on the outside of the bag, or affix an additional label using clear packing tape. The sample ID number format will be specified in the governing document referencing this procedure. Double bag the sample and repeat the labeling process for the outer bag.

Decontaminate equipment between composite samples (not between subsamples of one sample), as discussed in Section 7.2 below.

Repeat steps outlined above until all samples from a property have been collected. Refer to Section 8.2 for field quality control (QC) sample requirements.

7.0 Associated Procedures

7.1 Field Documentation

Field documentation for samples collected using this procedure will follow the current versions of CDM-LIBBY-03 (Completion of Field Sample Data Sheets) and EPA-LIBBY-2012-01 (Field Logbook Content and Control) unless otherwise specified in the governing document referencing this procedure.

7.2 Field Equipment Decontamination

All reusable sampling equipment must be decontaminated between composite samples in accordance with EPA-LIBBY-2012-04 (Field Equipment Decontamination) unless otherwise specified in the governing document referencing this procedure.

7.3 IDW

IDW will be managed as described in EPA-LIBBY-2012-05 (Handling IDW) and any other applicable governing documents. In general, replace the soil plug with excess sample volume. The soil should be placed back into the hole and tamped down lightly. If sandy areas such as playgrounds are sampled, refilling the soil plug is not necessary. Rinse water, the roots of vegetation removed during sampling, and any excess soil volume may be returned to the sampled area.

Spent wipes, gloves, and PPE must be disposed of or stored properly as IDW in accordance with EPA-LIBBY-2012-05 (Handling IDW) unless otherwise specified in the governing document referencing this procedure.

7.4 Sample Custody, Packaging, and Shipping

Sample custody requirements for samples collected using this procedure will follow the current version of EPA-LIBBY-2012-06 (Sample Custody), unless otherwise specified in the governing document referencing this procedure.

As may be applicable, sample packaging and shipping will follow the procedures outlined in EPA-LIBBY-2012-07, unless otherwise specified in the governing document referencing this procedure.

8.0 Quality Assurance/Quality Control

Quality assurance/quality control (QA/QC) for activities described in this procedure will be attained through a variety of processes, including, at a minimum, the items discussed below. Additional QA/QC requirements, such as audits or field assessments, will be addressed in the governing document referencing this procedure.

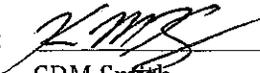
8.1 Training

Every effort will be made to ensure consistency in collecting surface soil samples in support of the Libby Site. Consistency will be achieved to the extent possible through proper training, using designated field staff, and providing TL oversight. Any deficiencies or inconsistencies in implementing this procedure noted by the TL will require re-training of field team members.

8.2 Field Quality Control Samples

Soil field duplicate samples will be collected at the rate specified in the governing document referencing this procedure. Field duplicate samples will be collected as co-located samples in the same zone as the parent sample. The duplicate will be collected from the same number of subsamples as the parent sample, but the subsample locations of the duplicate sample will be randomly located in the zone. The inspection for VV at each subsample location will follow the same protocol as referenced above. These samples will be independently collected with separate sampling equipment or with the original sampling equipment after it has been properly decontaminated. For tracking purposes, the parent/duplicate sample relationship will be recorded in accordance with sample documentation requirements stated in the governing document referencing this procedure. These samples will be used to determine the variability of sample results in a given land use area, but will not be used to determine variability in sampling technique.

Libby Asbestos Superfund Site Site-specific Procedure Semi-Quantitative Visual Estimation of Vermiculite in Soils

Prepared by: 
CDM Smith

Date: 3/20/13

Reviewed by: 
CDM Smith Technical Reviewer

Date: 3/20/13

Reviewed by: 
CDM Smith Quality Assurance Reviewer

Date: 3/20/13

Revision No.	Date	Reason for Revision
0	10/12/06	--
1	5/10/07	<ul style="list-style-type: none">• To refine the process after initial trial phase.
2	03/20/13	<ul style="list-style-type: none">• To reflect current documentation procedures and clarify the definition of visible vermiculite.• To align with current procedure format.

1.0 Objective

The EPA will identify and delineate the extent of any visible vermiculite, amphibole asbestiform minerals, and mine tailings suspected to be sourced from the former W.R. Grace mine – herein collectively referred to as visible vermiculite (VV) – present in soils as part of investigations conducted at the Libby Asbestos Superfund Site (Site) and specified in governing documents referencing this procedure. The goal of this standardized procedure is to provide a consistent approach to identify and characterize any VV present in soils.

The semi-quantitative approach presented in this procedure for visually estimating VV in soil will be revised as required to optimize data collection as the sampling teams gain experience. This will be accomplished by expanding and/or improving this procedure, supporting pictorial standards, and additional electronic data acquisition efforts, as necessary.

2.0 Definitions

Specific-use Area (SUA) – Discrete exterior parcels on a property with a designated specific use. Due to the nature of activities typically carried out in SUAs, residents may be especially vulnerable to exposures when Libby amphibole asbestos (LA)-contaminated soil becomes airborne. SUAs may be bare or covered with varying amounts of vegetation. SUAs include, but are not limited to:

- Flowerbeds
- Gardens
- Stockpiles

- Play Areas
- Dog Pens
- Driveways (non-paved)¹
- Parking Lots (non-paved)
- Roads (non-paved)
- Alleys (non-paved)

Common-use Area (CUA) – Exterior parcels on a property with varied or generic use. CUAs may be bare or covered with varying amounts of vegetation. CUAs include, but are not limited to:

- Walkways
- Yards (front, back, side, etc.)
- Former Gardens
- Former Flowerbeds

Limited-use Area (LUA) – Exterior parcels on a property that are accessed, utilized, and maintained on a very limited basis. LUAs may be bare or covered with varying amounts of vegetation. LUAs include, but are not limited to:

- Pastures
- Maintained/Mowed Fields
- Area underneath porches/decks²
- Overgrown Areas (with trails/footpaths, or between SUAs/CUAs)

Primary Building (PB) – Has four walls and a roof, a fully-enclosed design, and is intended for residential or commercial occupancy. PBs include, but are not limited to:

- Houses (including understructures)
- Some shops (when the shop is the primary occupied building on the property)
- Warehouses

Secondary Building (SB) – Has four walls and a roof, a fully-enclosed design, and is large enough for human entry. SBs include, but are not limited to:

- Garages
- Some shops (when another primary building is present on the property)
- Barns
- Sheds

¹ Non-paved driveways considered an SUA starting 2007 – previously considered a CUA.

² The soils underneath porches and decks will be classified as CUAs or LUAs depending on ground clearance and accessibility to homeowners and pets. If these areas are not accessible, they will be classified as NUAs.

- Enclosed lean-tos
- Some pump houses
- Larger animal houses

Secondary Structure (SS) – Designed to be open on at least one side and/or is mobile. Some SSs may be enclosed similar to a SB, but are too small or are not intended for human occupancy (e.g., pump or dog houses). SSs include, but are not limited to:

- Carports
- Open lean-tos
- Some pump houses
- Dog houses
- Other small animal housing

Non-use Area (NUA) – Exterior parcels on a property with no current use (e.g., areas that are unmaintained and not accessed). NUAs may be bare or covered with varying amounts of vegetation. NUAs include:

- Wooded lots
- Un-maintained fields
- Inaccessible areas below porches/decks

Because NUAs are not currently accessed, they are not presently considered a complete exposure pathway. As such, semi-quantitative visual estimates of vermiculite in soil will not be captured at this time. However, to the extent that NUAs may become a complete exposure pathway in the future at a property, the EPA may revisit NUAs at a later date or include NUAs as part of long-term site operations and maintenance.

Zone³ – Parcels on a property that share a similar land use or subdivisions of a land use area based on site conditions (e.g., access, construction setup considerations) or sampling requirements. Within a zone, no area type may be combined with any other area type. For example, driveways and flowerbeds are both SUAs but will be separated into unique zones for visual inspection. Similarly, large CUAs such as yards may be subdivided into front yard, side yard, and back yard zones depending on site conditions. Sectioning properties into additional zones will be at the discretion of the field team leader (FTL) but consistent among the teams using this procedure as specified in the governing document referencing this procedure.

It is anticipated that SUA, PB, SB, and SS zones will generally tend to be smaller parcels. Combining small, proximal SUAs into one zone will be at the discretion of the FTL but consistent among the teams. In addition, combining small, proximal CUAs into one zone will be at the discretion of the

³ The restriction on the maximum square footage of SUA zones (1,000 ft²) and non-SUA zones (2, 500 ft²) was eliminated from a previous iteration of this SOP after the data were reviewed by the EPA and determined to sufficiently characterize the presence of VV regardless of zone square footage. Additionally, this will allow the flexibility necessary for field teams to identify areas of zones most cost effectively for removal purposes.

FTL but consistent among teams. No PB, SB, or SS will be combined with any other PB, SB, or SS for visual inspection. There is not a maximum square footage restriction for any zone.

Point Inspection (PI) – Used in SUA, CUA, LUA, PB, SB, and SS zones. A PI is an intrusive visual inspection of the top portions of the soil at a randomly selected point within a zone. A PI consists of the active displacement of the surface soil with a small shovel and visual inspection of the displaced soil to determine if VV is present. If VV is observed during the PI, the location and a semi-quantitative estimate of VV contamination will be recorded.

Libby Asbestos Superfund Site (site) - All buildings and land within the boundaries of each operable unit (OU) of the site as illustrated on the most recent version of the OU boundary map.

3.0 Responsibilities

Field Team Leader – The FTL is responsible for overseeing the visual inspection process for their field teams, ensuring field team members are adequately trained in this procedure, and checking for consistency among their field teams.

Field team members – Field team members are responsible for conducting visual inspection, as specified in this procedure and the governing document referencing this procedure, and reporting any irregular observations or issues to the FTL.

4.0 Applicability

This procedure applies to properties within the site that will undergo screening and detailed investigations and, as applicable, certain risk-based investigations. Investigation-specific modifications to this procedure shall be outlined in the investigation-specific guidance document. The following locations on a property will be evaluated for the presence/absence of VV:

- All zones where soil samples are being collected.
- All zones requiring visual inspection per the requirements in the governing document referencing this procedure.

5.0 Procedure

Figure 1 illustrates the procedures and decision rules for this procedure. The three primary procedural steps are listed below:

- Establish preliminary zones
- Perform PI
- Perform semi-quantification of VV

Each is described in the following subsections.

5.1 Establish Preliminary Zones

Upon arrival at the property, the field team will locate all areas requiring visual inspection. Parcels will be identified as SUA zones, CUA zones, LUA zones, PB zones, SB zones, SS zones, or NUA zones.

Zones will be assigned according to the definitions provided above. Zone boundaries may be updated on the field sketch based on visual inspection results.

5.2 Point Inspections⁴

As defined above, a PI is an intrusive visual inspection performed at randomly selected points across the entire surface of a zone. Professional judgment may be used to determine the exact location of PIs; however, the following guidelines will be implemented to maintain consistency.

A minimum of 30 PIs will be evaluated per zone if sampling is required within that zone. If soil sampling is not required, a minimum of five PIs will be evaluated within each zone. Zones larger than 500 square feet (ft²) will require evaluation at a minimum of 1 PI per 100 ft² (10 foot by 10 foot area). The PI locations will be randomly selected and will be spatially representative of the entire zone. Locations of the PIs and semi-quantitative estimates of VV (i.e., low [L], intermediate [M] or high [H]) will be recorded on the field sketch for each PI. While a minimum of five PIs will be conducted per zone, there is no set maximum. Rather, the maximum number of PIs is variable, dependent upon the total area of the zone and achieving the minimum required frequency of one PI per 100 ft².

The following sections outline procedures for inspecting each use area (e.g., SUA, CUA, LUA, PB, SB, SS). The procedure for semi-quantification of VV is provided in the next section.

SUA Zone:

- Use a spade or trowel to remove any cover material, including excess debris (e.g., mulch, rock) and organic material, from the surface of the soil. Remove and visually inspect soil to a depth of 0-6 inches below ground surface⁵.
- If a depth of 6 inches cannot be attained given the varying levels of compaction in driveways, roads, etc. the maximum depth attainable will be documented in the field logbook and on the field sample data sheet (FSDS).
- Record semi-quantitative estimate of VV observed as described in Section 5.3.
- Replace soil and cover material.
- Repeat as necessary employing procedure outlined above.

CUA and LUA Zones:

- Using a spade or trowel, carefully removing organic material, including grass, from the surface of the soil. Remove and visually inspect soil to a depth of 0-3 inches below ground surface⁶.
- Record semi-quantitative estimate of VV observed as described in Section 5.3.

⁴ Surface Inspections- The non-intrusive visual inspection of the immediate surface of a zone was eliminated from a previous iteration of this SOP after their data were reviewed and determined by the EPA to provide no additional information over that gained through Point Inspections.

⁵ A soil depth of 6 inches for SUAs was chosen to approximate the depths to which digging would be expected during typical activities occurring in these SUA zones (e.g., gardening, child digging in dirt).

⁶ A soil depth of 3 inches was chosen to approximate the depths to which soil disturbance would be most likely during typical activities occurring in these CUA and LUA zones (e.g., lawn mowing).

- Carefully replace all soil and organic material.
- Repeat as necessary employing procedure outlined above.

PB, SB, and SS Zones:

- Move items as necessary to access the soil surface.
- Using a spade or trowel, remove and visually inspect soil to a depth of 0-3 inches below ground surface⁷.
- Record semi-quantitative estimate of VV observed as described in Section 5.3.
- Repeat as necessary employing procedure outlined above.

If during the PI, VV is observed to be localized within a zone, the portion with vermiculite will be denoted on the field sketch. If additional PIs are necessary to determine the boundaries of the area, approximately 10 to 20% additional PIs will be evaluated to determine the extent of localized vermiculite.

5.3 Semi-Quantification of Visual Vermiculite

During a PI, the field team will estimate the quantity of vermiculite observed. Each PI location for all zones will be assigned a semi-quantitative estimate of visible vermiculite content using a 4-point scale: none (blank), L, M, and H⁸. For PI locations where VV is observed, semi-quantitative estimates (e.g., L, M, or H) will be recorded on the field sketch. PI locations where VV is not observed will not be recorded on the field sketch. Photographs illustrating these quantities are attached to this procedure as Figure 2.

6.0 Health & Safety/Engineering Controls

All personnel will carry out visual inspections in accordance with proper personal protective equipment (PPE) and other monitoring/governing requirements outlined in the current version of the Accident Prevention Plan governing the work being conducted.

All visual inspections will employ appropriate engineering controls to minimize dust (e.g., wetting soil during inspection) as prescribed in the current version of CDM-LIBBY-05 (30-point Composite Sampling of Surface Soil for Asbestos).

7.0 Equipment Decontamination

Equipment decontamination is not required between each PI from the same zone, but is required before moving to another inspection zone. Decontamination of equipment will be conducted as required by the governing document referencing this procedure.

⁷ A soil depth of 3 inches was chosen to approximate the depths to which soil disturbance would be most likely during typical activities occurring in these PB, SB, and SS zones (e.g., entering crawlspace, retrieving items from shed).

⁸ Based on the EPA's review of previous data, the 5-level scale VV identification scheme was not meaningful and has been reduced to a 4-level scale. As such, the semi-quantitative estimation "Gross" VV in a previous iteration of this procedure was combined with "High" estimations. Previously collected data of Gross VV should be considered analogous to High VV under this revised procedure.

8.0 Documentation

As noted above, information about the presence of vermiculite will be recorded on the field sketch for the property under investigation. Each zone will be marked with:

- Zone type (i.e., SUA, CUA, LUA, PB, SB, SS, or NUA)
- Semi-quantitative estimate of VV content for each PI (i.e., L, M, H)

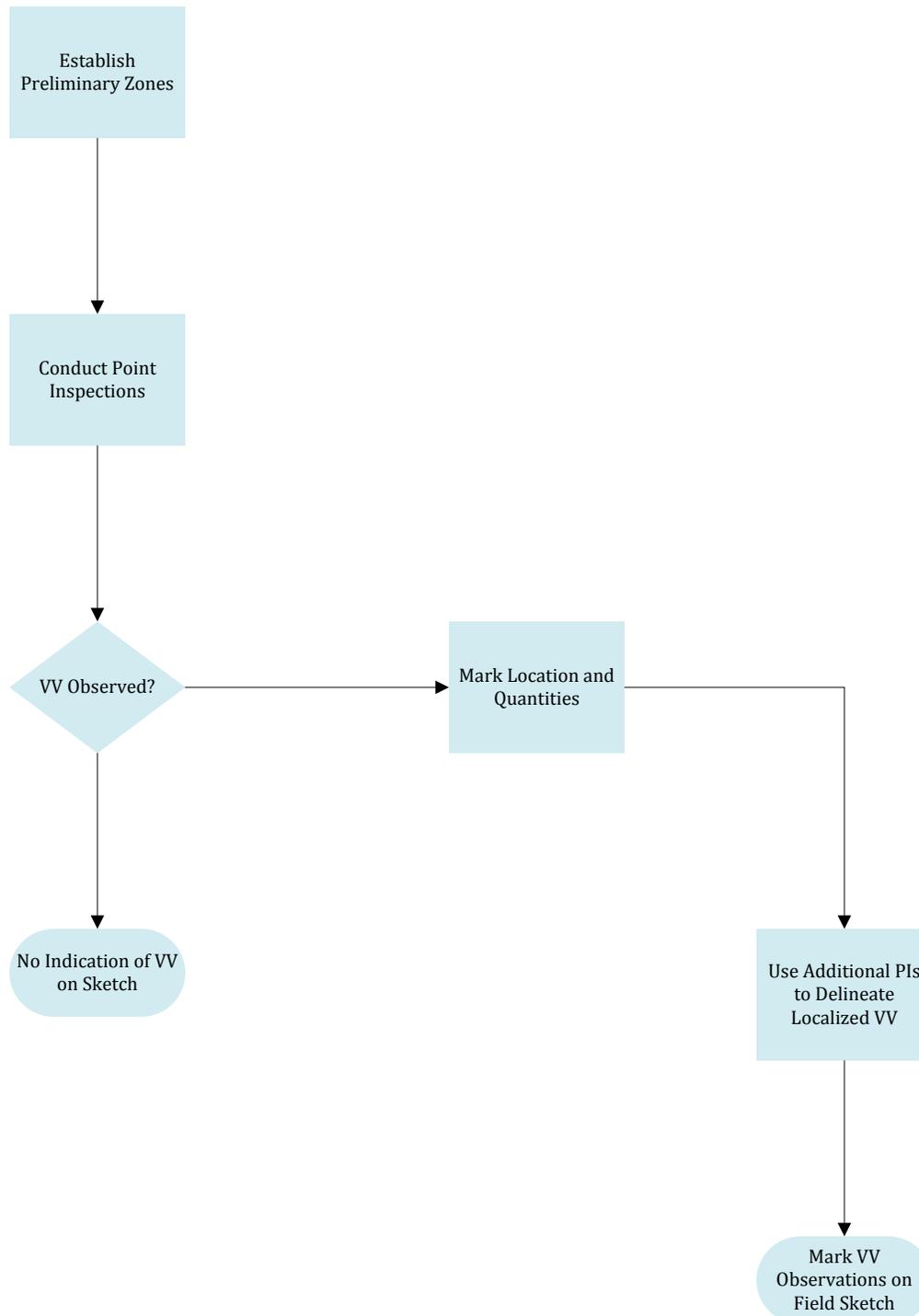
In addition to field sketch documentation, each field team will document the semi-quantitative visual estimates of VV on the FSDS for each zone. If material other than vermiculite was observed during the inspection (e.g., tremolite, mine tailings, micaceous minerals), the specific location and material observed should be noted on the field sketch and recorded in the visible vermiculite comment field on the FSDS. The FSDS will be managed according to governing guidance documents.

9.0 Quality Assurance/Quality Control

Every effort will be made to maintain consistency in the semi-quantitative evaluation of VV in soil. Quality assurance/quality control processes and measures will include training; use of specimen examples (e.g., jars/photographs of low, intermediate, and high quantities of vermiculite); use of designated field staff; and oversight by the FTL. Figures illustrating none, low, intermediate, and high quantities of vermiculite are attached to this procedure for reference (Figure 2).

To maintain consistency over time, the FTL will verify semi-quantitative assignments at a rate of one zone per team per week, or at the rate specified in the governing document referencing this procedure. The FTL will sign off on those field sketches that were verified. If inconsistencies are noted, the FTL will hold re-training with all teams participating simultaneously. Updates to this procedure and its attached specimen examples will occur as necessary and the EPA will be notified when these updates are recommended by FTL or field investigation manager.

Figure 1
Inspection Process



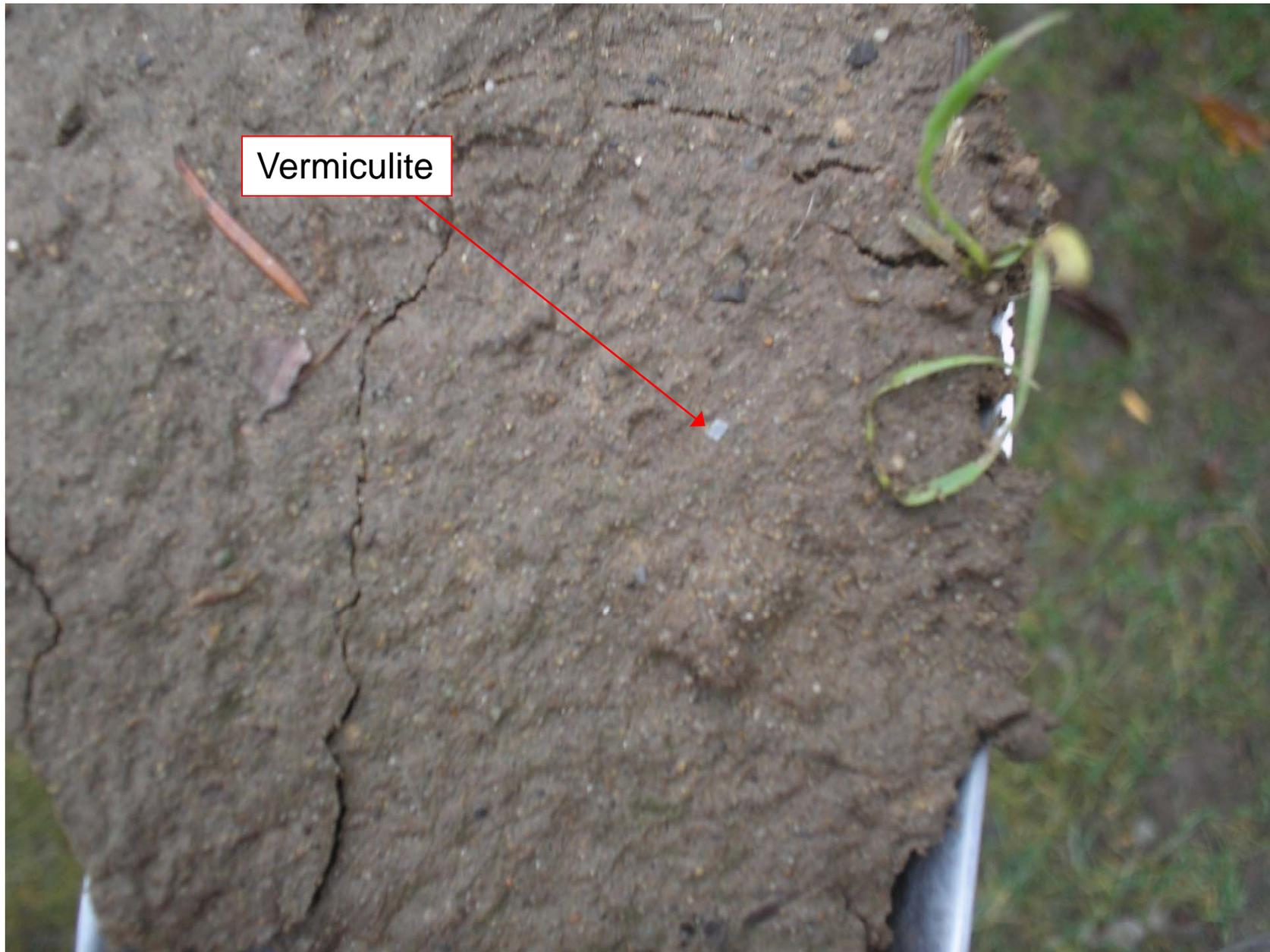


Figure 2a: *Low Visible Vermiculite* – A maximum of a few flakes of vermiculite observed within a given visual inspection point



Figure 2b: *Medium Visible Vermiculite* – Vermiculite easily observed throughout visual inspection point, including the surface.



Figure 2c: *High Visible Vermiculite* – Visual inspection point contains Approximately 50% (or greater) vermiculite by volume

Libby Asbestos Superfund Site Site-specific Procedure GPS Coordinate Collection and Handling

Prepared by:  Date: 03/24/14
CDM Smith

Reviewed by:  Date: 3/24/14
CDM Smith Technical Reviewer

Reviewed by:  Date: 3/24/14
CDM Smith Quality Assurance Reviewer

Revision No.	Date	Reason for Revision
0	5/21/07	--
1	--	Not finalized/approved
2	7/27/09	Updated to align processes with current GPS collection equipment, and data management processes and requirements
3	4/24/12	Updated to align processes with current GPS collection equipment, and data management processes and requirements
4	8/14/13	Updated to align processes with current GPS collection equipment, and data management processes and requirements
5	03/24/14	Updated to align processes with current location coordinate sourcing from geo-referenced surveys

1.0 Objective

The objective of this site-specific procedure is to establish baseline requirements, procedures, and responsibilities for collecting and handling global positioning system (GPS) data by the U.S. Environmental Protection Agency (EPA) or its contractors related to investigations conducted at the Libby Asbestos Superfund Site (Libby Site). This procedure describes the equipment and operations to be used for collection of location coordinate data. Additions or modifications to this procedure may be detailed in governing documents referencing this procedure.

2.0 Background

2.1 Definitions

Libby Asbestos Superfund Site (Libby Site) – All buildings and land within the boundaries of the EPA’s designated operable units (OUs), as illustrated on the most recent version of the OU boundary map.

Libby YYMMDD.ddf Data Dictionary (Libby data dictionary) – The filename for the Libby data dictionary contains the date of the latest revision in the format YYMMDD. All Trimble® handheld units

used by CDM Smith at the Libby Site should be pre-loaded with a generic data dictionary that handles collection of lines, points, and areas. In addition, the Trimble® units will be uploaded with the Libby data dictionary by designated onsite data management staff.

Scribe – An EPA data management system used to manage location, sample, and analytical data.

2.2 Discussion

The Libby data dictionary is set up to meet the location coordinate requirements discussed in Appendix C of the *Libby Asbestos Site Data Management Plan* (EPA 2013). For all locations assigned by CDM Smith during investigation and soil excavation activities, a latitude and longitude coordinate representing the location will be collected; additional specifics for coordinate collection will be included in the governing document referencing this procedure. All personnel required to collect GPS data will be familiar with the contents of the Libby data dictionary.

The following attributes are required to be collected, as indicated in **Table 1**, for each feature type when a GPS coordinate is collected:

Table 1. Attributes Collected in the Libby_YYMMDD Data Dictionary

Feature	Attributes Collected
Any Location	LocationID
BD Location	LocationID
SP Location	LocationID
XX Location	LocationID

These features are discussed in detail in Section 4.0 of this document. Instructions for loading a data dictionary onto a datalogger are discussed in Section 4.3.

3.0 Responsibilities

Team Leader (TL) – The TL is responsible for overseeing the GPS point collection process for their field teams, ensuring field team members are adequately trained and coordinating with onsite data management staff to ensure the completeness of the GPS dataset required to be collected (as specified in the governing document referencing this procedure).

Field sample data sheet (FSDS) – The hard copy form on which sample and location information is recorded.

Field team members – Field team members are responsible for collecting GPS data, as specified in the governing document referencing this procedure, and reporting any data collection issues to the TL. For readability, field team members are also referred to as Trimble® unit “operators” throughout this procedure.

Onsite data management staff – This staff is responsible for publishing finalized GPS data to Scribe. This staff will also support the TL to ensure the required GPS dataset for each field activity is complete.

4.0 Equipment, Software, and Configuration

Software can vary with rental equipment; however, the preferred software for transfer and processing of GPS data is GPS Pathfinder Office and TerraSync. **Table 2** contains guidelines for configuration settings (based on TSC1 5.27 software), that should be implemented for GPS point collection. Configuration settings for TerraSync are outlined immediately following Table 2. Note that some GPS Pathfinder Office and TerraSync settings can be changed to accommodate data collection needs.

Table 2. Configuration Settings for Trimble® ProXRS

GPS Rover Options - Logging Options		
Logging Intervals	Point feature	1 s
	Line / area	3 s
	Not in feature	none
	Velocity	none
Confirm end feature	no	
Minimum Positions	30	
Carrier phase	Carrier mode	off
	Minimum time	10mins
GPS Rover Options – Position Filters		
Position mode	Manual 3D	
Elevation mask	15 degrees	
SNR mask	6.0	
DOP type	PDOP	
PDOP mask	6.0	
PDOP switch	4.0	
GPS Rover Options – Real-time input		
Preferred correction source	use uncorrected GPS	
GPS Rover Options – General real-time settings		
Correction age limit	10s	
GPS Rover Options – Antenna options		
Height	Set according to model	
Measure	Vertical	
Confirm	Never	
Type	auto-filled when part number is entered	
Part number	get part number off of antenna	
GPS Rover Options – Initial Position		
North	USft	
East	USft	
GPS Rover Options – 2D altitude		
Altitude(MSL)	USft	
Computed at	time	
Computed at	date	
GPS Base Station Options – Logging Options		
Logging Intervals	Measurements	5s
	Positions	30s
Audible Click	Yes	
Log DOP data	Yes	
GPS Base Station Options – Position Filters		
Position mode	Manual 3D	
Elevation mask	15 degrees	
SNR mask	4.0	
PDOP mask	6.0	

PDOP switch	4.0	
GPS Base Station Options – Real-time output options		
Real-time output mode	off	
Radio type	Custom	
Baud rate	9600	
Data bits	8	
Stop bits	1	
Parity	Odd	
RTCM options	Station	1
	Message type	Type 1
	Message interval	5s
	Message suffix	None
	CTS flow control	Off
	CTS xmit delay	0ms
	RTS mode	High
	RTS edge delay	0ms
GPS Base Station Options – Reference position		
Datum	WSG 1984	
Zone	11 North	
NMEA/TSIP Output options		
Output	TSIP	
Baud rate	38400	
Coordinate System	Latitude/Longitude	
Map display options	All show with no background	
Units and Display		
Units	Distance(2D)	US Survey Ft
	Area	Square feet
	Velocity	Miles/Hour
	Angle format	DDMMSSss
	Order	North/East
	North reference	True
	Magnetic declination	Auto
	Null string	
	Language	English
	Time and Date	24 hour clock
Time		##:##:##
Date format		MM/DD/YYYY
Date		MM/DD/YY weekday
Quickmarks	Attributes	Repeat
	Confirm	No

TerraSync (v4.15) Setup

The following configuration settings should be employed:

Logging Settings: Antenna Height

GPS Settings: PDOP Settings are determined on basis of Productivity versus Precision. Slide the bar to obtain the highest precision for a given location.

Real-time Settings: Use Uncorrected GPS

Coordinate System Settings: Coordinate System: Latitude/Longitude; Datum: WGS84

Units: Distance Units: US Survey Feet; Area Units: Square Feet; Angle Units: Degrees; Lat/Long Format: DD°MM'SS.ss; Offset Format: Horizontal/Vertical; North Reference: True; Magnetic Delineation: Auto(15.2°E);

External Sensors: None

5.0 Procedures

The following sections describe how GPS points are collected and handled for features commonly used at the Libby Site.

5.1 Selecting Locations

All features collected at the Libby Site are point features. Any location feature will allow the entry of any 9-digit text value, which will correspond to the Location ID assigned on the field sample data sheet (FSDS). For ease and accuracy of data entry of location values, three additional location features are available for which the Location ID attribute defaults to the values "BD-", "SP-", or "XX-" accordingly. The prefix code values are specific to the field event and defined in the governing document referencing this procedure.

Building Locations

For building locations, a GPS point is collected near the front door or main entrance of the building. Refer to the governing document for details regarding building location types.

Locations Where No Sample is Collected

For investigation locations where a sample is not collected, a GPS point is collected at the approximate center of each location area, or as specified in the governing document referencing this procedure.

Soil Sample Locations

For grab sample locations, a GPS point is collected at the exact sampling location.

For composite sample locations, a GPS point is collected at the approximate center of the sample area. In the case of an irregular-shaped sample area or sample area that is non-continuous (e.g., a flowerbed that wraps around a house), a GPS point is collected at the center of the largest continuous sample area.

A GPS point is collected once per unique sample location. All subsequent samples taken at that location (including field duplicate samples) will use the previously assigned Location ID and corresponding coordinates.

Outdoor Stationary Air and Dustfall (Settled Dust) Samples

For permanent outdoor stationary air and dustfall sample locations (i.e., those representing a consistent monitoring zone or area, and are collected on a routine schedule), a GPS point is collected once per unique sample location. All subsequent samples taken at that location use the previously assigned Location ID and corresponding coordinates.

Interest Point, Interest Area

GPS points for interest point and interest area features are not routinely collected at the Libby Site. However, they are included in the Libby data dictionary in the event that a GPS point or a series of points is collected to document the perimeter of an interest area or sample area or other point that does not correspond to a location in the Scribe database.

Pre-determined Sample Areas

For pre-determined sample areas (e.g., gridded) where waypoints are available, the Trimble® units may be pre-loaded with waypoint files to guide samplers to sampling locations. Pre-loading of coordinates is typically performed by onsite data management staff. It should be noted that, in order to ensure GPS coordinate data are included in the project database, *GPS points will also be collected at the time of sampling for sample locations located using waypoint files.*

Features Not Requiring GPS Points

GPS points are not collected for the following features, unless otherwise specified in the governing document referencing this procedure:

- Stationary air, dust, and soil samples collected inside or beneath buildings (these locations are associated with the coordinates of the building where the sample was collected)
- Stationary air samples, with the exception of permanent monitoring locations as designated in site-specific removal work plans or Response Action Work Plan Addenda
- Duplicate or replicate air or dust samples (which are assigned the same Location ID and coordinates as the parent sample)
- Soil samples taken at depth from the same sample area as a previously collected sample (the at-depth soil sample will be assigned the same Location ID as the shallower sample in order to relate both samples to the same coordinates)
- Duplicate or split soil samples (which are assigned the same Location ID and coordinates as the parent sample)
- Personal air samples (locations are associated with the coordinates of the building (i.e., BD Location ID) or property (i.e., AD Location ID) where the sample was collected)

5.2 Operation of GPS Handheld Units

GPS points at the Libby Site will be collected using Trimble® GPS handheld units, or equivalent equipment that meets the EPA's accuracy standards for geospatial data. Operators must be standing at the sample location before the unit starts to collect positions. Once the unit has started collecting positions, the operator must remain standing at the sample location until the minimum required positions have been collected. A minimum of 30 positions will be collected for each GPS location point. More positions may be required in circumstances where the GPS collection parameters are excessive due to poor satellite position. GPS target parameters should be consistent with those listed in **Table 2** (Configuration Settings for Trimble® ProXRS). These parameters should be emulated as closely as possible if using other GPS unit models.

Accuracy Criteria

Due to GPS unit availability from third-party vendors, various Trimble® models may be used at the Libby Site. However it is imperative the model's performance rating not exceed accuracy exceptions greater than 5 meters, in order to comply with EPA Policy CIO 2131.0 *National Geospatial Data Policy, Tier 2* standards (EPA 2005). EPA verification of these standards is built into post-processing logarithms. Data verification in the upload process will check for a horizontal precision of less than 5 meters and that a minimum of 30 positions were compiled for each point (see Section 6.0 for more detail).

Record-keeping Requirements

Serial numbers of the Trimble® datalogger, receiver, and antenna or beacon will be recorded in a field logbook. GPS filenames will be recorded in the logbook. Recording GPS filenames on FSDSs is not required.

Upgrades to GPS Equipment and Software

GPS unit equipment and software is subject to change according to availability. The TL or designee is responsible for contacting the technical support of the vendor if there are any questions regarding setup, operation, or data transfer of models not previously used at the Libby Site.

5.3 GPS Data Transfer from Handheld Units to Lbysvr1

Most Trimble® units connect to a personal computer (PC) through the charger unit using a universal serial bus (USB) cable (type A to type B), and Microsoft Active Sync software. Note that there are Active Sync connection settings to enable or disable once the device is connected to the PC: from the Active Sync menu, select Tools, select Options. These connect the Trimble® to other Windows applications on the PC (e.g., email, task managers, etc.). The main reason to disable these settings at the CDM Smith Libby project office is that the Trimble® units are shared and it does not make sense to activate them.

1. Turn on the Trimble® unit
2. Open Terrasync
3. Select Data
4. At the bottom of list, select File Manager
5. Open Pathfinder
6. Select Utilities
7. Select Data Transfer. The receive tab should be active.
8. From the Device list, select GIS Datalogger on Windows CE
9. Click on the connect icon (the button with the checkmark circled in green). A picture on the right will indicate the connection status.
10. Select Add

11. Select Open (make sure all files are highlighted)

12. Select Transfer All

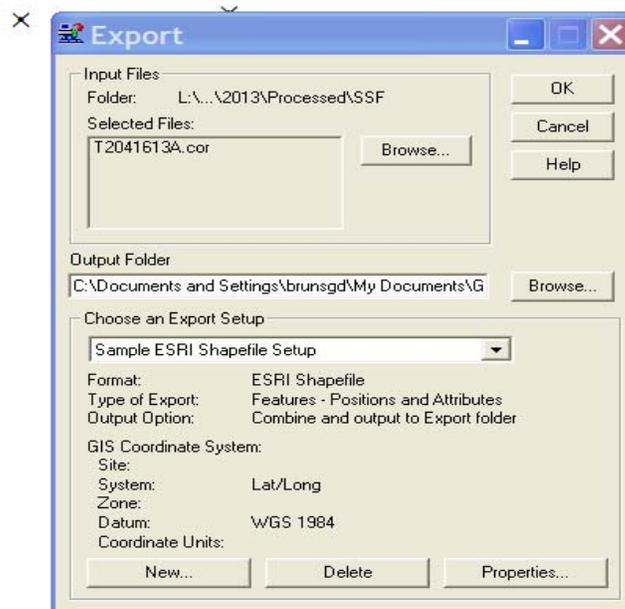
Note: To load a data dictionary onto the datalogger, from Step 7, select the Send tab. When adding the file, navigate to the file you wish to load onto the datalogger. Make sure the file is highlighted before selecting transfer all.

5.4 GPS Data Processing

Following download, the Trimble® files are stored on the CDM Smith Libby project server in the \\Lbysvr1\Projects\Data Management\Pfdata\Libby folder. The files, denoted by their .ssf extension, are differentially corrected and coordinate data for each unique location is uploaded to the Scribe location table using the procedures below:

1. Open GPS Pathfinder Office. Establish default folder and differential correction settings as shown in Table 3. Drag the .ssf files of interest to the Pathfinder map window. From the Pathfinder main menu options select Utilities. Select Differential Correction. A .cor file will be generated with a filename that corresponds with the .ssf filename. The .ssf files and the .cor files will be filed within the \\Lbysvr1\Projects\Data Management\Pfdata\Libby folder.
2. In order to prepare files for updating Scribe and to produce maps for a quality control (QC) verification of the points, select Utilities from the Pathfinder menu. Select Export. Review the selected file and output folder shown in the Export window. Choose an Export Setup of “Sample ESRI Shapefile Setup” (**Figure 1**).

Figure 1



9. Click on Select a File. Select one of the unprocessed files and click on Open. The Select File Location window will disappear.
10. Click on Run Process. Data will be queued for updating location coordinates in the Scribe location table.
11. Repeat steps 10 and 11 until all the unprocessed files are queued. Once this occurs, click on Update Scribe which updates the Scribe location table.
12. To review any location coordinates that have not been successfully updated in Scribe, click “See Points w Issues.” Make revisions/corrections to this table as needed and update Scribe as needed.

Table 3. Pathfinder Office Settings

Pathfinder Differential Correction
Processing Type - Automatic Carrier and Code Processing
H-Star Processing - Use a Single Base Provider
o Correct Settings
▪ Output corrected and uncorrected positions
▪ Smart automatic rover filtering
▪ Re-correct real-time positions
o Base Data – Bonners Ferry or other nearest
o Folder Search – set to default
o Reference Position - Bonners Ferry or other nearest
o Output folder - set to directory of input file
o Output filename - Use original filename, overwriting any existing .cor file
Pathfinder Export
Input files - .cor file
Output Folder – set to default
Choose an Export Setup - Sample ESRI Shapefile Setup
Properties
Coordinate System
o Use Current Display Coordinate System
▪ Export Coordinates As XY
▪ Projection File – set to default
o ESRI Shapefile
▪ Export Tracking Themes
▪ Track ID Attribute Name
o Position Filter
▪ GPS Position Info
▪ Minimum Satellites – 2D (3 or more SVs)
▪ Maximum PDOP – Any
▪ Minimum HDOP - Any
o Include Positions That Are
▪ All options other than Uncorrected
▪ Options other than Filter By Precision (68% confidence)
▪ Include Non-GPS Positions
o Data
▪ Features – Position and Attributes. Export All Features
▪ Output - Combine all input and output to export folder under Output Files
▪ DOS Files under System File Format
o Attributes
▪ Attribute Value under Export Menu Attributes As

<ul style="list-style-type: none"> ▪ Generated Attributes, all options for All Feature Types and Point Features ▪ No selections for Line Features or Area Features
<ul style="list-style-type: none"> ○ Units - Use Current Display Units <ul style="list-style-type: none"> ▪ Distance Units: US Survey Feet ▪ Area Units: Square Feet ▪ Velocity Units: Feet Per Second
<ul style="list-style-type: none"> ○ Decimal Places <ul style="list-style-type: none"> ▪ Lat/Long: 9 ▪ North/East: 3 ▪ Precision: 1 Time: 0 ▪ All other selections: 3
Pathfinder Options
<ul style="list-style-type: none"> ○ Units <ul style="list-style-type: none"> ▪ Distance – US Survey Feet ▪ Area – Square Feet ▪ Velocity – Feet per second ▪ Offsets – US Survey Feet ▪ Offset Distance Format: Horizontal and Vertical ▪ Precisions – US Survey Feet ▪ Confidence – 68% Precisions ▪ North Reference: True ○ Coordinate System <ul style="list-style-type: none"> ▪ Coordinate System and Zone ▪ System – Lat / Long ▪ Datum WGS 1984 ▪ Altitude Measured: MSL ▪ Altitude Units - Meters

5.6 Digitized Coordinates

For situations where GPS points are not collected using a GPS unit (i.e., detailed investigation [DI] portion of the General Property Investigation and planned exterior soil removals), location coordinates will be digitized by the drafting team using the property-specific land survey provided by a certified surveyor contracted to the RC. The CAD drawing is composed by the drafting team using the survey and the coordinates provided in the land survey. The CAD drawing is geo-referenced with the survey coordinates provided by the surveyor; therefore, these coordinates meet the standard of the survey-grade GPS unit used for survey, which are well within EPA’s Tier 2 standards.

Afterwards, the desired points are digitized using the DI sketch (for GPIs) or the draft redline drawing (for planned exterior soil removals). A Microsoft Excel export file containing the location coordinates is then emailed to CDM Smith. Data is imported into the DataConsolidation MS Access database where updates are made to the Scribe location table.

6.0 Quality Assurance/Quality Control (QA/QC)

All GPS data are visually reviewed and verified after processing.

Visual review involves using the shapefile exported in Step 5 in Section 5.4, in a geographic information system (e.g., ArcView), by the TL. Mapped points are viewed to ensure they represent the expected area at the expected property. Points with obvious errors are omitted and/or recollected.

Verification involves comparing data attributes against EPA-established accuracy criteria, which is performed by onsite data management staff during the “Run” process (Step 11 in Section 5.4). Any point location not within 5 meters of “Horz_Prec” (horizontal precision) or collected using less than 30 positions is flagged in the DataConsolidation MS Access database. Additionally, the following formula is applied to each point to evaluate the point’s accuracy: $[\text{Horz_Prec}] + (1.645 \times [\text{Std_Dev}]) = X$, where X must be less than 5 to ensure the point falls within 5 meters of the intended target with 95% confidence. Any point exceeding a 5-meter calculated position is flagged. These flagged points will be reviewed a minimum of once per month by the CDM Smith onsite data manager.

7.0 References

EPA. 2005. CIO Policy Transmittal 05-002, *National Geospatial Data Policy*. August 24. [<http://www.epa.gov/irmpoli8/policies/21310.pdf>].

EPA. 2013. *EPA Data Management Plan, Libby Asbestos Superfund Site, Version 2013.1*. July 3. [[https://team.cdm.com/erom/R8-RAC/libby/Libby Data Management Plan](https://team.cdm.com/erom/R8-RAC/libby/Libby%20Data%20Management%20Plan)]

LIBBY ASBESTOS SUPERFUND SITE STANDARD OPERATING PROCEDURE
APPROVED FOR USE AT THE LIBBY ASBESTOS SUPERFUND SITE ONLY

SOIL SAMPLE PREPARATION

Date: December 5, 2012

SOP No.: ISSI-LIBBY-01 (Revision 11)

SOIL SAMPLE PREPARATION

SYNOPSIS: This is a standardized method for the preparation of fine and coarse-grain soil samples collected at the Libby Asbestos Superfund Site for asbestos analysis at an approved laboratory.

APPROVALS:

USEPA Region 8

Dania Zinner
Signature

12-6-12
Date

Dania Zinner
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LIBBY ASBESTOS SUPERFUND SITE STANDARD OPERATING PROCEDURE
APPROVED FOR USE AT THE LIBBY ASBESTOS SUPERFUND SITE ONLY

SOIL SAMPLE PREPARATION

Date: December 5, 2012

SOP No.: ISSI-LIBBY-01 (Revision 11)

RECORD OF CHANGES

Revision	Date	Principal Changes and Author
0	Unknown	This SOP was originally prepared by ISSI Consulting Group. ISSI is no longer in existence, and finalization of the SOP was performed by William Brattin at Syracuse Research Corporation (SRC).
1	01/07/2000	Incorporated sieving to the sample preparation
2	07/12/2000	Revision in sieve size and other minor edits
3	05/07/2002	Incorporated minor edits
4	08/01/2002	Modified sieving procedure and added grinding step
5	03/06/2003	Incorporated modifications to the procedure and documentation requirements
6	03/24/2003	Incorporated modifications to the log sheets to conform with electronic data storage requirements and added grinder blank requirements
7	08/05/2003	Incorporated modifications to drying and sample storage procedures
8	05/04/2004	Incorporated modifications to drying batch size and recording of preparation information
9	05/14/2007	Incorporated modifications so as to expand use to other Operable Units (removed references to OU4/CSF, changed Index ID to Sample ID). Repaired formatting. Removed reference to missing Figure 1. Added optional use of electronic logs. Oven temperature set to 90 ± 10 degrees C. Lowered inventory batch size from ~120 to ~50 samples so that one inventory batch can fit in one tub. Designated drying batch as one batch per oven (~20 samples). Allowed for optional use of disposable drying pans. Removed direction to NOT move grinding plates during decontamination (new BICO design allows plates to be separated for decontamination without adjusting gap). Ovens will be calibrated daily. <i>[Note: Revision 9 was as unsigned version that reflects changes made at the Troy Sample Preparation Facility. Some of the changes in Revision 9 are retained in Revision 10, below].</i>
10	12/06/2007	Incorporated modifications so as to expand use to other Operable Units. Designated drying batch as ~20 samples. Allowed for optional use of disposable drying pans. Allowed alternative methods for decontamination of plate grinder. Clarified and modified QC requirements. General editing for clarity.
11	07/27/2012, 11/15/2012, 12/05/2012	Entire SOP review and update provided by ESAT Region 8, which includes procedures for equipment calibration.

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LIST OF ATTACHMENTS

Attachment 1: Sample Drying Bench Sheet

Attachment 2: Sample Preparation Bench Sheet

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1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to standardize the methods used to prepare soil samples collected at the Libby Asbestos Superfund Site (referred to as the Libby Site from this point forward) for the analysis of asbestos content by an approved laboratory. This SOP is specifically intended for application at the Libby Site and focuses on homogenizing soils and soil-like materials in order to produce equal fractions of the original sample with reproducible results.

2.0 SCOPE AND APPLICATION

Soil samples from the Libby Site are processed at the Environmental Services Assistance Team (ESAT) Region 8 Troy Sample Preparation Facility (SPF) before submittal to the laboratory for analysis. This process separates the coarse fraction of the soil from the fine fraction. The fine fraction constitutes all material passing through a ¼-inch sieve. The fine fraction is homogenized and ground to a maximum particle size of approximately 250 microns (µm). This fine fraction is further sub-divided into four fractions using a riffle splitter. One or more of these fractions is then submitted to an approved and accredited laboratory for polarized light microscopy (PLM) analysis.

3.0 RESPONSIBILITIES

- 3.1 It is the responsibility of the SPF supervisor to ensure that all preparation, quality assurance (QA) and quality control (QC) procedures are performed in accordance with this SOP and to identify and take appropriate corrective action to address any deviations that may occur during sample preparation.
- 3.2 The ESAT Team Manager, QA Coordinator (QAC), and/or SPF Lead will communicate with project managers at the United States Environmental Protection Agency (EPA; also referred to as the client), or their designate, any situations where a modification to or deviation from the SOP may be useful or necessary. ESAT must receive approval from the EPA for any deviation or modification from the SOP before incorporating any such deviation or modification into the sample preparation process (refer to Section 8.2).
- 3.3 All SPF personnel are responsible for reading and understanding the SPF-specific Health and Safety Plan (HASP) and performing all tasks in accordance with the requirements of the HASP.

4.0 METHOD DESCRIPTION

Soil samples received at the SPF are dried in a laboratory oven, and then split into a preparation sample and an archive sample. The preparation sample is sieved to separate coarse material (>¼-inch) from fine material (<¼-inch). The fine material is ground to a particle size of less than 250 µm, and this fine ground material is split into several aliquots. This grinding step is needed to achieve a reasonable degree of homogeneity in the sample, and to allow for preparation of slides for PLM analysis. The coarse fraction (if any) and one aliquot of the fine ground material are then sent to an approved analytical laboratory for PLM analysis. Fine-ground samples are analyzed according to the current version of SOP SRC-LIBBY-03, *Analysis of Asbestos Fibers in Fine Soil*

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by *Polarized Light Microscopy*, and coarse samples are analyzed according to the current version of SOP SRC-LIBBY-01, *Qualitative Estimation of Asbestos in Coarse Soil by Visual Examination Using Stereomicroscopy and Polarized Light Microscopy*. Sample fractions that are not sent to an analytical lab for analysis are stored in an archive facility currently maintained by SPF personnel. Fractions are tracked both on paper and electronically.

5.0 ACRONYMS

ACM	Asbestos Containing Material
EPA	United States Environmental Protection Agency
ESAT	Environmental Services Assistance Team
HASP	Health and Safety Plan
HAZWOPER	Hazardous Waste Operations and Emergency Response
HEPA	High Efficiency Particulate Air
LA	Libby Amphibole
NIST	National Institute of Standards and Technology
OSHA	Occupational Safety and Health Administration
PE	Performance Evaluation
PLM	Polarized Light Microscopy
PPE	Personal Protective Equipment
QA	Quality Assurance
QAC	Quality Assurance Coordinator
QC	Quality Control
QMP	Quality Management Plan
SOP	Standard Operating Procedure
SPF	Troy Sample Preparation Facility
SRC	Syracuse Research Corporation
USGS	United States Geological Survey

6.0 HEALTH AND SAFETY

- 6.1 Follow general laboratory health and safety policies and regulations in the HASP, Chemical Hygiene Plan, or equivalent.
- 6.2 All sample handling and preparation activities (drying, splitting, sieving, grinding, etc.) are performed within the SPF Work Zone, which is fitted with a negative pressure, ventilated hood with an operating High Efficiency Particulate Air (HEPA) filtration system. A sample container should only be open within the Work Zone at the following times: when the sample is inside of the sample preparation hood, when transferring a sample from the ventilation hood to the oven, or when transferring a sample from the oven to the grinding hood. Appropriate personal protective equipment (PPE) must be worn at all times within the Work Zone.

7.0 CAUTIONS

After processing each sample, thoroughly decontaminate all equipment and work surfaces in order to prevent cross-contamination between samples.

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8.0 GENERAL LABORATORY PRACTICES

8.1 QA Program

- 8.1.1 The SPF operates under a QA program appropriate to the type, range, and volume of work it performs.
- 8.1.2 It is the responsibility of SPF personnel to read, understand, and follow the ESAT Region 8 Quality Management Plan (QMP). Additional QA/QC requirements specific to the SPF are described in Section 18.0.
- 8.1.3 All work is performed at a permanent location. The SPF is able to carry out all preparation, calibration, and daily QA/QC activities independently, and at one location. There are no remote or sub-facilities where preparation work is performed.

8.2 Documenting SOP Modifications

- 8.2.1 Any deviation from the SOP shall be documented in a laboratory modification form. Additionally, when there is reason to suspect a departure from the SOP has affected the result or validity of data provided to the client, the client must be notified of the nature of the departure from the SOP and informed about the possible effect on the result or validity of the analysis. The course of action taken to keep the departure from recurring must also be discussed with the client.

9.0 PERSONNEL QUALIFICATIONS

- 9.1 Personnel performing sample preparation activities must read and understand the HASP and all associated SOPs. In addition, personnel must complete the 40-hour Occupational Safety and Health Administration (OSHA) Hazardous Waste Operations and Emergency Response (HAZWOPER) training and the required OSHA 8-hour Refresher courses. Additional training may be identified prior to project implementation and will be administered prior to any individual beginning work at the SPF.
- 9.2 The health and safety records for all personnel, including HAZWOPER 40-hour and 8-hour certificates must be kept in a central location and available at all times.

10.0 EQUIPMENT AND SUPPLIES

The SPF must be equipped with the following instrumentation, hardware, software, and all other materials and supplies required to perform this SOP. All equipment must be properly maintained and calibrated (as appropriate) prior to use.

- General purpose laboratory oven capable of maintaining a constant temperature of approximately 90°C
- Analytical balance capable of measuring in a range of 0.1 g to at least 2,000 g
- Weight set, traceable to National Institute Standards and Technology (NIST)
- Riffle splitter with ¾-inch chutes
- Plate grinder with plates adjustable from ¼-inch to approximately 250 µm

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- Wet/dry vacuum with HEPA filtration
- Compressed air
- Metal scoop or spoon (plastic scoops or spoons are not acceptable)
- ¼-inch metal sieve and catch pan (plastic sieves and pans are not acceptable)
- Shims used for grinder plate calibration
- NIST traceable certified micrometer
- 60-mesh (250 µm) sieve and 200-mesh (74 µm) sieve
- Asbestos-free quartz sand
- Drying pans (disposable aluminum baking pans)
- HEPA-filtered hood, a class 1 biohazard hood, or glove box with continuous airflow (negative pressure)
- Vaneometer
- Re-sealable Poly Bags, 4-mil – sizes 4x6 inch and 10x13 inch (these two sizes are standard; however, a larger 4-mil poly bag may be used if a larger master sample bag is needed)
- Disposable, powder-free examination gloves (nitrile or latex)
- Half-face respirator with disposable P100 cartridges
- Safety glasses or goggles (Z-87 rated) with side shields
- Tyvek coveralls with attached hood/boots
- Additional PPE required by the SPF-specific HASP
- Sample Drying bench sheets (provided in Attachment 1)
- Sample Preparation bench sheets (provided in Attachment 2)
- Equipment maintenance/calibration logbooks, document controlled
- Self-adhesive sample labels
- Asbestos containing material (ACM) waste bags
- Indelible marking pen
- Water in spray bottles and paper towels (for wet wiping)

11.0 SOIL STORAGE

Upon receipt at the SPF, samples are grouped into an inventory batch and assigned an inventory batch number. This number is an identifier in the following format: 12-1014, where 12 = two-digit calendar year (as in 2012) and 1014 = four-digit consecutive number, starting with 0001. Whenever soil samples are not being processed, they are stored in plastic bins or shipping boxes/coolers. The samples do not require refrigeration but must be kept in an orderly, clean fashion. All bins will be assigned a bin identification number, or Bin ID, which is a four-digit consecutive number starting with 0001. The Bin ID is displayed on a prominent hanging tag. Bins will be arranged on labeled shelves by the Bin ID for easy retrieval. All bins will also be labeled with one or more inventory batch numbers. Bin information is tracked by the sample coordinator in an Excel file, which indicates the Bin ID, bin contents, and its physical location within the SPF.

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12.0 BULK SOIL DRYING

12.1 Equipment Verification Checks

- 12.1.1 Samples will be weighed prior to and following drying activities. A verification check of the analytical balance is performed each day when samples are loaded into or unloaded from the oven (note that a verification check is performed on the balance for other soil processing purposed as well, even if samples are not being dried on a particular day). Before weighing samples, perform the verification check using Class 6 weights (equivalent to Class S-1 weights) and record the results, any required maintenance, and the balance number in the Analytical Balance Verification and Maintenance Logbook.
- 12.1.2 All drying activities will be performed in a HEPA-filtered hood, a class 1 biohazard hood, or glove box with continuous airflow (negative pressure). Prior to loading the oven, use a vaneometer to verify that the hood's ventilation system is operating properly, and record the results and any required maintenance in the Ventilation Hood Verification and Maintenance Logbook. Verification of the hood's ventilation system is performed daily.
- 12.1.3 A HEPA vacuum will be used to decontaminate the oven following the removal of dried samples. A verification check is performed on the HEPA vacuum daily prior to drying activities. All system checks, required maintenance and the vacuum number will be recorded in the HEPA Vacuum Verification and Maintenance Logbook.
- 12.1.4 An oven temperature verification check will be performed daily during periods of operation. Oven temperature verification checks and any required maintenance will be documented in the Oven Temperature Verification and Maintenance Logbook.

12.2 Drying Procedure

- 12.2.1 Group samples into drying batches of approximately 20 samples per batch. Assign each batch a drying batch number, and record this number on the Sample Drying bench sheet, as well as the number of the oven used to dry the samples.
- 12.2.2 Include one preparation blank per oven. See Section 18.1 for more details regarding preparation blanks.
- 12.2.3 Set the oven temperature to 90°C ($\pm 10^\circ\text{C}$). For every drying batch, check the oven temperature to verify that proper temperature has been reached and document the start date/time and temperature on the Sample Drying bench sheet.
- 12.2.4 Transfer each sample to be dried from its 4-mil poly bag into a clean drying pan.
 - 12.2.4.1 Prior to drying each sample, record the starting sample mass to the nearest 0.1 g on the Sample Drying bench sheet (Attachment 1).
 - 12.2.4.1.1 Place the empty drying pan on the analytical balance and tare the balance to zero. Then, pour the sample into the drying pan and record the weight to the nearest 0.1 g. By taring the balance to zero, the recorded weight is only that of the sample and not the sample plus the drying pan.
 - 12.2.4.2 Each sample must be transferred to its respective drying pan under the HEPA-filtered hood.

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- 12.2.4.3 Label each drying pan with the Sample ID of the sample. Place each sample in the oven.
- 12.2.5 Leave the samples in the oven until completely dry (potentially 24-48 hours). Verify that each sample is dry by squeezing a portion of the soil with a freshly gloved thumb and forefinger to test the cohesiveness.
- 12.2.6 Turn off the oven and allow the samples to cool in the oven.
- 12.2.7 Once the samples are cooled, unload each sample and transfer to a clean, disposable aluminum pan labeled with the Sample ID.
 - 12.2.7.1 The weight of each dried sample must be recorded to the nearest 0.1 g on the Sample Drying bench sheet (Attachment 1).
 - 12.2.7.1.1 Place the empty aluminum pan on the analytical balance and tare the balance to zero. Then, transfer the sample into the new aluminum pan and record the weight to the nearest 0.1 g. By taring the balance to zero, the recorded weight is only that of the sample and not the sample plus the aluminum pan.
 - 12.2.7.2 All samples transfers should be made in the HEPA-filtered hood to prevent potential exposure to asbestos fibers that might be released from the sample.
- 12.2.8 Once all information on the Sample Drying bench sheet is completed (including the technician's initials and the date) a second technician must perform a QC check of the information, and initial and date the bench sheet.
- 12.2.9 Then, each sample can either be poured into a clean 4x6 inch 4-mil poly bag (identified with the Sample ID) or personnel can proceed directly with sample processing (see Sections 13 through 16).

12.3 Decontamination

Decontaminate all equipment used (drying pans, the inside of the hood, and the inside of the drying oven) using the HEPA vacuum and wet wiping all surfaces before loading a new batch for drying.

13.0 DIVISION OF ARCHIVE AND PREPARATION SAMPLES

All dried samples are mixed and split into two portions: one portion is held in archive, and the second portion is prepared for shipment to an approved analytical laboratory for asbestos analysis. Additional splitting may be necessary to reduce the portion size to a size appropriate for grinding. The sections below describe the sample splitting procedure.

13.1 Equipment Calibration

All splitting, sieving, and grinding activities will be performed in a HEPA-filtered hood, a class 1 biohazard hood, or glove box with continuous airflow (negative pressure). Prior to any splitting, sieving, or grinding activities, use a vaneometer to verify that the hood's ventilation system is operating properly, and record the results and any required maintenance in the Ventilation Hood Verification and Maintenance Logbook.

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13.2 Procedure for Sample Splitting

Splitting must be performed in the HEPA-filtered hood to prevent potential exposure to asbestos fibers that might be released from the sample. Samples will be divided using the following procedures:

- 13.2.1 Place the cooled, re-bagged samples in the hood, and knead the contents of the bag to break up any soil clumps.
- 13.2.2 Place one collection pan on each side of the riffle splitter. Pour the sample through the splitter in order to divide the sample into two equal sub-parts.
- 13.2.3 After splitting, set aside one portion for sample preparation (preparation procedures described below). If the mass of the portion for preparation is larger than about 200 grams, split the preparation sample again so that $\frac{3}{4}$ of the original sample will be archived and $\frac{1}{4}$ will be set aside for processing.
- 13.2.4 Place the remaining portion(s) into a clean, 4-mil poly bag, re-bag the sample in another clean, 4-mil poly bag, and store as an archive sample in the event additional analyses are required in the future. Identify the archive sample with the Sample ID and the suffix "A" (for archive fraction). Record the technician's initials and date in the Sample Preparation bench sheet (Attachment 2). Store the archive portion in the numbered inventory box noted in the Sample Preparation bench.

13.3 Preparation Duplicate Samples

One preparation duplicate sample will be prepared for every 20 field samples (or 5%) processed. A preparation duplicate is generated by using the riffle splitter to divide the preparation fraction into two equivalent portions ("parent" and "duplicate"). The duplicate portion is assigned an independent Sample ID, and both the parent sample and the duplicate sample are then processed in an identical fashion. Each sample is submitted to the laboratory blind, meaning that the laboratory is not made aware of which sample is the parent or the duplicate. For further information on preparation and processing of preparation duplicates, refer to Section 18.4.

13.4 Performance Evaluation Samples

Performance Evaluation (PE) samples are used to assess the accuracy of the analytical laboratory and to check for any potential contamination or loss of asbestos during processing. For further information on preparation and processing of PE samples, refer to Section 18.3.

13.5 Decontamination

13.5.1 The splitter need not be decontaminated following this step if the next use of the splitter will be the division of the fine-ground fraction of the same samples into four fractions (see Section 16.0). If for any reason the next use of the splitter is division of material from a different sample, the riffle splitter must be decontaminated as follows.

13.5.2 Use the HEPA vacuum and compressed air to decontaminate the splitter and

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brush or wipe off any visible material that is not removed by the air blast. The splitter is now ready to process the next sample.

14.0 SIEVING THE PREPARATION SAMPLE

All preparation samples are sieved prior to grinding to separate the coarse fraction from the fine fractions. The sample sieving procedure is described in the sections below.

14.1 Equipment Calibration

14.1.1 All sieving activities will take place in the HEPA-filtered hood. Prior to any splitting, sieving, or grinding activities, use a vaneometer to verify that the hood's ventilation system is operating properly, and record the results and any required maintenance in the Ventilation Hood Verification and Maintenance Logbook.

14.1.2 Samples are weighed during sieving activities. A verification check of the analytical balance is performed each day when samples are sieved. Before weighing samples, perform the verification check using Class 6 weights (equivalent to Class S-1 weights) and record the results, any required maintenance, and the balance number in the Analytical Balance Verification and Maintenance Logbook.

14.2 Sample Sieving Procedure

14.2.1 Pour the sample onto a clean ¼-inch stainless-steel sieve with a clean pre-weighed catch pan. Shake the screen until all particles <¼-inch in size have passed through the screen into the pan. When needed, a pestle may be used to gently break up any remaining soil clumps to ensure all particles <¼-inch in size pass through the screen.

14.2.2 Pour all material which does not pass through the screen (>¼-inch) into a clean 4x6 inch 4-mil poly bag. This is the Coarse Fraction sample.

14.2.2.1 The weight of the coarse fraction must be recorded to the nearest 0.1 g on the Sample Preparation bench sheet (Attachment 2).

14.2.2.1.1 Place the clean poly bag on the analytical balance and tare the balance to zero. Then, transfer the coarse fraction into the poly bag and record the weight to the nearest 0.1 g. By taring the balance to zero, the recorded weight is only that of the coarse fraction and not the coarse fraction plus the poly bag.

14.2.3 If all of the material passes through the screen, such that there is no coarse fraction, record a mass of zero for the coarse fraction on the Sample Preparation bench sheet.

14.2.4 Double-bag the coarse fraction sample in a 4x6 inch 4-mil poly bag, and identify the sample with the Sample ID and "C" suffix on the sample bag. Coarse fraction samples are now ready to be packaged for shipment to the analytical laboratory or archived as directed.

14.2.5 All material that passes through the ¼-inch screen is the Fine Fraction. Weigh and record the mass of the fine fraction to the nearest 0.1 g on the Sample Preparation Log bench sheet.

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- 14.2.5.1 The weight of the fine fraction must be recorded to the nearest 0.1 g on the Sample Preparation bench sheet.
 - 14.2.5.1.1 Place the clean poly bag on the analytical balance and tare the balance to zero. Then, transfer the fine fraction into the poly bag and record the weight to the nearest 0.1 g. By taring the balance to zero, the recorded weight is only that of the fine fraction and not the fine fraction plus the poly bag.
- 14.2.6 Whenever possible, immediately process the fine fraction material according to the procedures described in Sections 15.0 and 16.0. If processing cannot occur immediately, pour the fine fraction material into a new 4-mil poly bag and identify the fine sample material with the Sample ID and the suffix "F" (for "fine fraction"). Double-bag the sample and identify the sample with the Sample ID and suffix on the outside of the bag.

14.3 Decontamination

All non-disposable pans and sieves must be decontaminated between samples. Decontaminate sieves and pans (and the pestle, if used) in the HEPA-filtered hood using compressed air. Wipe or brush off any visible material that is not removed from the air blast. A HEPA vacuum may also be used to remove any residual material.

15.0 GRINDING THE FINE-FRACTION SOIL SAMPLES

The fine fraction of each preparation sample will be ground to produce a material of approximately 250 μm ¹. The procedure for grinding the fine fraction is outlined below.

15.1 Equipment Calibration

- 15.1.1 All grinding activities must take place in the HEPA-filtered hood. Prior to any splitting, sieving, or grinding activities, use a vaneometer to verify that the hood's ventilation system is operating properly, and record the results and any required maintenance in the Ventilation Hood Verification and Maintenance Logbook.
- 15.1.2 A plate grinder will be used to grind fine-fraction samples to the particle size appropriate for asbestos analysis. Verification checks of the plate grinder will be performed weekly to verify proper particle size (approximately 250 μm) and to demonstrate that samples are not being over-processed. A traceable certified micrometer will be used as a standard to perform the weekly verification checks that the shim being used for calibration is within tolerance (+/-5%). Eventually, shims will fail due to wear, bends, cracks, etc., at which time they will be replaced with a new shim that meets the requirements. The micrometer will be calibrated annually by a third party.
- 15.1.3 If the required particle size cannot be achieved even after plate adjustment, other grinder maintenance such as plate replacement may be required. Grinding of field samples cannot resume until the desired particle size is achieved. Document the

¹ Note that the particle size is cited as "approximately 250 μm ". This is due to the nature of grinding asbestos material. Some material that is longer than 250 μm may pass through the grinder if its longest side is parallel with the vertical grinder plates. The material that comes in contact more nearly perpendicular to the vertical grinder plates will be ground to <250 μm .

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grinder number, verification of acceptable adjustment, and any observations in the Grinder Verification and Maintenance Logbook.

15.1.4 Samples will be weighed following grinding activities. A verification check of the analytical balance is performed each day when samples grinding activities take place. Before grinding samples, perform the verification check using Class 6 weights (equivalent to Class S-1 weights) and record the results, any required maintenance, and the balance number in the Analytical Balance Verification and Maintenance Logbook.

15.2 Grinding Blanks

One grinding blank per grinder will be prepared daily, and will be associated with all samples prepared by that grinder on that day. For further information on grinding blanks refer to Section 18.2.

15.3 Grinding Fine-Fraction Soil Samples

15.3.1 The sample portion that was sieved to <1/4-inch will be ground to a particle size of approximately 250 µm. Set up a catch pan under the grinder to collect all the ground material. Take the fine-fraction preparation sample set aside in Section 13.2, load the grinder hopper, and allow the fine-fraction sample to pass through the plate grinder into the catch pan. Note the technician's initials, date of grinding, and grinder number on the Sample Preparation bench sheet.

15.3.2 The net recovery of fine ground material must not be less than 90% of the mass of fine material placed into the grinder. If recovery is less than 90%, soil grinding must be stopped and the grinder re-adjusted until the mass recovery of test sand and/or soil samples exceeds 90%.

15.4 Decontamination of the Plate Grinder

15.4.1 If the plate grinder can be readily disassembled for cleaning without altering its grinding properties, disassemble the grinder and clean the chutes and plates with the HEPA vacuum and compressed air. Then, if needed, wet wipe to ensure decontamination. If wet wiping is used, the plates and chutes must be thoroughly dried before reassembly.

15.4.2 If the grinder is not easily disassembled, clean the grinder with the HEPA vacuum and several blasts of compressed air, paying special attention to areas where dust from the grinding process is known to accumulate (e.g., between the plates and areas adjacent to the catch pan clamps).

15.4.3 Pass an aliquot of approximately 20 g of asbestos-free quartz sand through the grinder to clean out any residual soil.

15.4.4 Discard the quartz sand and re-clean the grinder with the vacuum and another round of high pressure air blasts. After this decontamination procedure, the grinder is ready to process the next sample.

15.4.5 In general, all soil containers, hoppers and catch pans associated with use of the grinder should be decontaminated using the HEPA vacuum and/or wet wiping, followed by a blast of high pressure air.

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15.5 Decontamination of the Calibration Sieves

- 15.5.1 The stacked sieves used to calibrate the plate grinder will be decontaminated using the HEPA vacuum and compressed air between calibration uses.

16.0 SPLITTING FINE-GROUND SOIL SAMPLES

Fine-ground soil samples should be distributed into four approximately equal sub-samples using a splitter. All splitting activities will be performed in the HEPA-filtered hood. Prior to any splitting activities, use a vaneometer to verify that the hood's ventilation system is operating properly, and record the results and any required maintenance in the Ventilation Hood Verification and Maintenance Logbook.

16.1 Splitting Procedure for Fine-Ground Sample

The following method for splitting a soil sample was adapted from EPA Method 540-R-97-028 (EPA, 1997).

- 16.1.1 Set up receiving pans on each side of the splitter. Load the soil from the grinder catch pan into the splitter, collecting the sample in two receiving pans.
- 16.1.2 Tap the catch pan vigorously several times to free any remaining material. Tap the splitter to facilitate the flow of all material through the chutes into the receiving pans.
- 16.1.3 Empty one receiving pan into the grinder catch pan and the other receiving pan into the sieve catch pan. Set the sieve catch pan aside; this portion of fine ground sample will be split again later.
- 16.1.4 Replace the receiving pans under the splitter. Take the grinder catch pan, containing half of the fine ground sample, and re-load the contents into the splitter as detailed above.
- 16.1.5 Repeat the process of dispersing the sample material by shaking the catch pan and tapping the splitter to uniformly distribute the sample. The resulting splits are the "FG1" and "FG2" portions on the Sample Preparation bench sheet.
- 16.1.6 Take these two portions and carefully transfer each into a clean 4x6 inch 4-mil poly bag labeled with the Sample ID.
- 16.1.6.1 The weight of each fine-ground sample portion must be recorded to the nearest 0.1 g on the Sample Preparation bench sheet (Attachment 2).
- 16.1.6.2 Place the clean poly bag on the analytical balance and tare the balance to zero. Then, transfer the sample into the poly bag and record the weight to the nearest 0.1 g. By taring the balance to zero, the recorded weight is only that of the fine-ground sample portion and not the sample plus the clean 4x6 inch 4-mil poly bag.
- 16.1.7 Re-bag each fine-ground sample portion in another clean, 4x6 inch 4-mil poly bag. Identify each fine-ground sample with the Sample ID, the suffix "FG" (for "fine fraction, ground"), and the fraction number (e.g., CS-12345-FG1 for fine ground fraction #1). Set aside the FG1 and FG2 fractions of the sample.
- 16.1.8 Place the two empty receiving pans from the FG1 and FG2 fractions next to the splitter. Repeat the splitting procedure using the other fine ground portion set aside in the sieve pan and split the remaining sample material to create the "FG3"

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and "FG4" portions.

- 16.1.9 Take the remaining "FG3" and "FG4" portions and repeat the procedures described in Sections 16.1.6 and 16.1.7.
- 16.1.10 Combine all of the bagged coarse and fine portions of the sample into one large clean, 4-mil poly bag (10x13 inch).
- 16.1.11 Coarse and fine-ground samples are now ready to be packaged for shipment to the analytical laboratory or archived as directed. When samples are requested for shipment, randomly select one of the fine-ground fractions to send to the analytical laboratory. If further analyses are required of the fine-ground fractions, the remaining fractions will be double bagged and sent. All archived fine-ground fractions will be filed in the appropriate inventory archive box noted on the Sample Preparation bench sheet.

16.2 Decontamination

The splitter must be decontaminated between each sample. Use the HEPA vacuum and/or wet wiping followed by a blast of compressed air to decontaminate the splitter, and brush or wipe off any visible material that is not removed by the vacuum or air blast. The splitter is now ready to process the next sample.

17.0 DOCUMENTATION

- 17.1 Sample ID numbers are recorded on the Sample Drying bench sheet, Sample Preparation bench sheet, and on all sample containers. Once all information on the Sample Drying bench sheet and Sample Preparation bench sheet is completed, a second technician must perform a QC check of the information, and initial and date the bench sheet. Sample Drying bench sheets and Sample Preparation bench sheets will be filed or archived according to their associated dry batch and preparation batch number. If revisions to the Sample Drying bench sheets and/or Sample Preparation bench sheets are necessary, the appropriate parties will be notified of the changes; however, these changes will not necessitate revision to the current SOP. Instead, a modification form will be filled out to document the revisions.
- 17.2 Equipment verification and maintenance logbooks are completed each day equipment is used.
 - 17.2.1 An additional logbook must be completed by SPF personnel each day sample preparation activities take place (Daily Activities Logbook). The Daily Activities Logbook must contain the following information:
 - Date
 - Time
 - Personnel initials who worked that day
 - PPE used
 - SOP and any other laboratory-specific governing plan being followed
 - Descriptions of any deviations from the SOP, including the reason for the deviation, and/or any modification forms being followed

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- Summary of the daily activities (including number of samples prepared, and equipment used)
- Any additional comments

17.3 A single-line strikeout (initialed and dated) is required for any corrections/changes made to bench sheets, logbooks, or any other form used at the SPF.

18.0 QUALITY CONTROL

QC samples are inserted into the sample processing train to monitor for potential contamination introduced during the preparation process and to assess accuracy of analyses that may be affected by the preparation procedures. If sample results indicate the occurrence of contamination or inconsistent results, the ESAT Team Manager, QAC, and SPF Lead will be notified. The ESAT Team Manager or QAC will then notify the client in order to review laboratory procedures and identify any changes in the SOP that may be necessary. Any such reviews and resultant changes will be documented accordingly by the SPF Lead.

18.1 Preparation Blanks

- 18.1.1 A preparation blank is a sample of approximately 200g of asbestos-free quartz sand that is treated identically to a field soil sample. That is, the preparation sample is dried in the oven along with the field soil samples, split into archive and preparation fractions using a riffle splitter, screened through a ¼-inch sieve (even though there are no particles larger than ¼-inch), and ground by passing through the plate grinder. This type of sample is intended to detect contamination that may occur at any stage of the soil preparation procedure.
- 18.1.2 At least one preparation blank will be processed with each drying batch of 20 field samples. Preparation blanks will be assigned a random, unique Sample ID and will be submitted to the laboratory blind. The Sample ID assigned to each preparation blank must follow the numbering system specified in the program-specific project plan.
- 18.1.3 Detection of asbestos fibers in any preparation blanks at a level greater than non-detect by PLM-Visual Estimation should be taken as a sign of potential cross-contamination, and all field samples associated with the preparation batch for the preparation blank having detectable asbestos will be reviewed and qualified appropriately if detectable levels of asbestos are also found in any of the corresponding field samples. If the overall fraction of preparation blanks that contains detectable asbestos exceeds 1%, a review of laboratory procedures should be undertaken to identify and address the source of the contamination.

18.2 Grinding Blanks

- 18.2.1 A grinding blank consists of 100-200 grams of asbestos-free quartz sand that is passed through the plate grinder. The purpose of this type of sample is to evaluate the effectiveness of decontamination procedures for the plate grinder.
- 18.2.2 One grinding blank per grinder will be prepared for each day that field samples are being ground. Each grinder used in the laboratory will be assigned a number and all samples processed will be associated with the grinder used for preparation.

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The grinder number used for each sample will be recorded on the Sample Preparation bench sheet. Grinding blanks will not be dried, split for archive, or sieved. Rather, a grinding blank will only be ground and split into four fine-ground samples. The grinding blank is assigned a random and unique Sample ID and is submitted to the laboratory blind. The Sample ID assigned to each grinding blank must follow the numbering system specified in the program-specific project plan.

- 18.2.3 Detection of asbestos fibers in any grinding blank at a level greater than non-detect should be taken as a sign of potential cross-contamination, and all field samples associated with the grinding blank that reports detectable asbestos will be reviewed and qualified appropriately if detectable levels of asbestos are also found in any of the corresponding field samples. If the overall fraction of grinding blanks that contains detectable asbestos in a soil preparation facility exceeds 1%, steps should be taken to develop an improved method for grinder decontamination.

18.3 PE Samples

- 18.3.1 PE samples consist of asbestos-free soil that is spiked with a known quantity of Libby Amphibole (LA) asbestos. These samples were created by the United States Geological Survey (USGS) for use at the Libby Site by spiking uncontaminated soil from Libby with a known mass of LA collected at the mine site, and then grinding the sample to a particle size of $\leq 250 \mu\text{m}$ as described above. Several different concentration values of PE samples were prepared, ranging from $< 0.1\%$ to 2% .
- 18.3.2 PE samples will be utilized in the two following ways:
- 18.3.2.1 First, the SPF will insert unprocessed PE samples into the sample processing train of samples being sent to the laboratory for PLM analysis. This type of PE sample is intended to evaluate the performance of the analytical laboratory (rather than the SPF).
- 18.3.2.2 Second, the soil preparation laboratory will process PE samples in the same way that field soil samples are processed, with the exception of splitting the samples due to the limited quantity of each PE sample. This type of PE sample is intended to determine if there is any loss of asbestos during sample processing. In addition, considered in conjunction with a grinding blank that is passed through the decontaminated grinder immediately following the PE sample, the PE sample will also be used to facilitate assessment of grinder decontamination procedures.
- 18.3.3 The frequency of PE samples will be determined by the client (typically one per quarter). The asbestos concentration of the PE sample to be used, the type of PE sample (processed or unprocessed), and the analytical laboratory that will receive the PE sample will also be at the direction of the client.
- 18.3.4 Results of PE samples processed by the SPF are evaluated by the client by comparing the reported results for LA to the nominal values. Deviations from nominal values may be the result of variations either in soil processing procedures and/or in the analytical procedure. If the frequency of strongly discordant results exceeds 10%, then the source of the inconstancy should be investigated and remedied.

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18.4 Preparation Duplicates

- 18.4.1 A preparation duplicate is prepared by using a riffle splitter to divide a field soil sample into two approximately equal portions, creating a parent and duplicate sample. Both samples are then processed in the same fashion. The preparation duplicate is assigned a unique Sample ID, and is submitted to the laboratory blind. The Sample ID assigned to each preparation duplicate must follow the numbering system specified in the program-specific project plan.
- 18.4.2 Preparation duplicate samples will be processed at a rate of 5% of the field samples processed (approximately one preparation duplicate for every 20 field samples prepared). Results from duplicate samples serve to evaluate the precision of the combined sample preparation process and the laboratory analysis. Inconsistent results between parent and duplicate samples may be due to variability in sample preparation, sample analysis, and/or to small scale variability in the sample that is not fully controlled by mixing and splitting. If the overall frequency of strongly discordant results (i.e., the results for the parent sample and duplicate are different by more than one bin category according to the current version of Libby-specific SOP SRC-LIBBY-03) is greater than 10%, steps should be taken to identify and address the source of the variability in the sample preparation procedure.
- 18.4.3 The rate of preparation duplicate samples should be tracked in a spreadsheet to ensure the rate is 5% of field samples processed.

18.5 Additional QA/QC

- 18.5.1 Periodic (at least annually) audits of the SPF will be conducted to ensure this SOP is being implemented and to identify any corrective actions or changes that need to be made to the SOP. Audits may be conducted internally by ESAT personnel, TechLaw, Inc., or by the client.

19.0 DECONTAMINATION

- 19.1 All non-disposable equipment used during soil sample preparation must be decontaminated prior to use. Scoops, spoons, splitters, sieves, and drying pans that are re-used must be decontaminated with the HEPA vacuum, compressed air, wet-wiping, and/or by brushing off any residual material.
- 19.2 The walls and counter top of the grinding station hood and any other area of sample handling and preparation, including floors, must be wet-wiped and HEPA-vacuumed at the end of each work day.
- 19.3 The sample drying oven will be HEPA-vacuumed and wet-wiped after each batch of samples.
- 19.4 If soil particles are visible on any of the equipment, repeat the decontamination procedure until the equipment is clean.

Note: To reduce the potential for human exposure to asbestos in the SPF, compressed air

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should be used carefully according to the manufacturer's instructions and only in the HEPA-filtered hood.

20.0 REFERENCES

- 20.1 American Society for Testing and Materials. 1998. Standard Practice for Reducing Samples of Aggregate to Testing Size. ASTM Designation: C 702 - 98, 4 p.
- 20.2 United States Environmental Protection Agency. 1997. Superfund Method for the Determination of Releasable Asbestos in Soils and Bulk Materials. Method 540-R-97-028.

LIBBY ASBESTOS SUPERFUND SITE STANDARD OPERATING PROCEDURE
APPROVED FOR USE AT THE LIBBY ASBESTOS SUPERFUND SITE ONLY

SOIL SAMPLE PREPARATION

Date: December 5, 2012

SOP No.: ISST-LIBBY-01 (Revision 11)

ATTACHMENT 1

Libby Asbestos Superfund Site Sample Drying Bench Sheet

Sample Drying Bench Sheet

TechLaw, Inc. ESAT Region 8
Troy Sample Preparation Facility

SOP: ISSI-LIBBY-01, Rev. 11
Oven No.: _____

Batch ID: (YY-####)		COC:		Due Date:	
Begin Date: (MM/DD/YY)		Begin Time: (HH:MM)		Begin Temp: C°	
End Date: (MM/DD/YY)		End Time: (HH:MM)		End Temp: C°	
Beginning Technician(s):			Ending Technician(s):		
Sample ID:	Wet Weight (g)	Dry Weight (g)	Drying Comments		
1.					
2.					
3.					
4.					
5.					
6.					
7.					
8.					
9.					
10.					
11.					
12.					
13.					
14.					
15.					
16.					
17.					
18.					
19.					
20.					

* Each dried sample will be temporarily stored in a plastic tote identified by a Batch ID in the format "YY-# # # #" (e.g., 10-1000)

Comments: _____

QC: _____ CK COC: _____ Date: _____ Loaded: _____ Shipped: _____

LIBBY ASBESTOS SUPERFUND SITE STANDARD OPERATING PROCEDURE
APPROVED FOR USE AT THE LIBBY ASBESTOS SUPERFUND SITE ONLY

SOIL SAMPLE PREPARATION

Date: December 5, 2012

SOP No.: ISST-LIBBY-01 (Revision 11)

ATTACHMENT 2

Libby Asbestos Superfund Site Sample Preparation Bench Sheet

LIBBY ASBESTOS SUPERFUND SITE STANDARD OPERATING PROCEDURE
APPROVED FOR USE AT THE LIBBY ASBESTOS SUPERFUND SITE ONLY

LIBBY CHAIN OF CUSTODY DOCUMENTATION

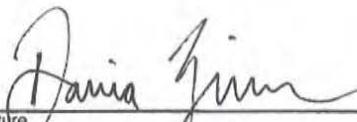
Effective Date: August 7, 2013

SOP No.: ER8-LIBBY-01 (Revision 0)

LIBBY CHAIN OF CUSTODY DOCUMENTATION

APPROVALS:

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Signature
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8/5/13
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Title

8/6/13
Date

Revision	Date	Principal Changes and Author
0	TBD	Initial Author: Talena Oliver, ESAT Region 8

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Attachment 1: Example corrected COC

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide a traceable and consistent process for the handling of Chain of Custody (COC) forms. Information from these COC forms is managed in Scribe, an online software program accessible by personnel within the United States Environmental Protection Agency (EPA), as well as approved EPA contractors. Scribe houses data in multiple databases from all aspects of the Libby Asbestos Superfund Site (referred to as the Libby Site from this point forward), and it is necessary that the information on a COC form matches the information in Scribe. This SOP focuses on how to properly document changes or corrections made to a COC or record additional information on a COC.

2.0 SCOPE AND APPLICABILITY

This SOP is specifically intended for application at the Libby Site by sampling agencies, preparation facilities and analytical laboratories associated with the Libby Site. This SOP is applicable during the entire life-span of a sample. The procedures for handling, relinquishing, receiving, and storing COCs for the Libby Site are described.

3.0 SUMMARY OF PROCEDURE

Sample collection agencies and the Sample Preparation Facility (SPF) create an electronic field sample data sheet (eFSDS), an electronic COC (eCOC) and an original hardcopy COC (referred to as the original COC), of which the original COC will accompany the specified samples for the remainder of their life-span. Data from these COCs is maintained in Scribe, which is updated throughout a samples life-span with additional information. Any modification to samples or their COC must be properly documented by hand on the original COC, and this information must be communicated to the appropriate agencies to ensure that the modification is made in all applicable databases.

4.0 ACRONYMS

COC	Chain of Custody
eCOC	Electronic Chain of Custody
EDD	Electronic Data Deliverable
eFSDS	Electronic Field Sample Data Sheet
EPA	United States Environmental Protection Agency
LIMS	Laboratory Information Management System
SOP	Standard Operating Procedure
SPF	Sample Preparation Facility
TAT	Turnaround Time
TEM	Transmission Electron Microscopy

5.0 GENERAL COC INFORMATION

- 5.1 A COC establishes a traceable, legal record of the possession of samples from the moment the original COC is generated until the samples are disposed of. For this reason, the original COC must accompany the samples at all times. All actions involving a sample

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- (e.g. removal from one COC to another, re-labeling or re-numbering of a sample) must be recorded on the original COC.
- 5.2 The Libby Site is an ongoing project that is constantly evolving and changing. Thousands of samples have been collected and are retained indefinitely until their disposal is approved by the EPA. As a result, it is vital that the samples are tracked properly, beginning with the original COC.
- 5.3 Sampling agencies maintain applicable field databases from which they create original COCs. Some of these samples require preparation to create new samples for analysis. In these cases, the original COC accompanies the original field samples, along with an eFSDS, to the SPF. An eFSDS contains information necessary to create new COCs in Scribe. Once processed, the SPF will create new COC forms from the SPF database. These SPF COC forms are the original COC for these new samples.
- 5.4 An eCOC is exported from the field or SPF database and is delivered via email to the receiving agency (with the exception of samples that need preparation at the SPF). An eCOC contains all of the sample-specific data that is found within the main body of the original COC.
- 5.4.1 The purpose of the eCOC is two-fold: to expedite the sample receipt process and to reduce the frequency of data entry errors.
- 5.4.2 An eCOC is used in addition to the original COC; it does not replace the original COC.
- 5.4.3 Analytical laboratories are not required to use eCOCs; however, if they wish to use them, they must be provided with the samples.
- 5.4.4 If an error is found within an eCOC, and that error is not on the original COC, this error must be communicated to the agency that created it to allow the error to be corrected in the applicable database from which it came (this communication may be written or verbal; written communication is not required since the original COC is correct, and the only correction would be to the database from which the eCOC was generated).
- 5.5 There may be instances when the original COC contains an error or discrepancy that is discovered at some point after the samples are relinquished from the sampling agencies or SPF.
- 5.5.1 In order to prevent the propagation of the error into the sample analytical results and Scribe, and to rectify the discrepancy, both errors and discrepancies must be communicated between the relinquishing and receiving agencies.
- 5.5.2 All changes to a COC must be communicated in writing (see Section 8.0).
- 5.6 Information collected during the entire life-span of a sample is maintained in applicable Scribe databases, and it is important that these databases match the data contained within the original COC.
- 5.6.1 Most errors on a COC originate in the database from which they are created, and these electronic discrepancies must be addressed as soon as possible.

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5.6.2 If the discrepancies are not addressed electronically when they are first discovered, they will need to be addressed at some point in the future, which may cause Electronic Data Deliverable (EDD) and/or Analytical Test Report corrections to be issued (potentially from multiple analytical laboratories), and multiple databases may need to be updated by multiple agencies.

5.7 Analytical laboratories should not use their internal COCs when relinquishing samples to another agency (for exceptions, see Section 7.4). All of the data on each COC is tracked in Scribe, and when a laboratory uses an internal COC, there is no record of that COC in Scribe, and no correlating location for the analytical results within the applicable Scribe databases. When the databases are compared, this lack of correlation will cause problems for the data user.

6.0 RECEIVING A COC

6.1 Upon receipt of samples, the receiving agency will perform, at a minimum, the following sample receiving procedures.

6.1.1 Verify the integrity of the shipping container. If it was damaged during transit and is not the original container, this must be noted on the COC (this may be evident by the taping of the original shipping label to the new container, or a new label that is not generated by the relinquisher).

6.1.2 If the samples have not yet been analyzed, verify that a custody seal is present either on each sample or on the master COC bag, and that the seal is not broken. The presence of an unbroken custody seal indicates that the sample contents have not been modified or tampered with.

6.1.2.1 If the seal is broken, it must be noted on the COC, added to the Analytical Test Report Case Narrative and added to the analysis comment field(s) in the EDD.

6.1.2.2 If samples were mistakenly sent to the wrong analytical laboratory, it is possible that the custody seal on the master bag was broken in order to review the COC. If this is the case, it is not necessary to make a notation on the COC; however, the laboratory should attach a new custody seal to the master bag, and then forward the samples to the correct laboratory.

6.1.3 Verify that the sample numbers and tags written on the individual sample bags match those listed on the COC. If the labels on the inner sample bag match the COC, but those on the outer sample bag do not, it is sufficient to correct by hand the outer sample bag label without making a notation of it on the COC. However, if the inner sample bag does not match the COC, then procedures in Sections 8.0 and 9.0 must be followed.

6.1.4 Verify that the Analyses and Turnaround Time (TAT) requested on the COC are what was expected before the samples were shipped. If the TAT is not what was agreed upon before the shipment was made, contact the Sample Coordinator at the SPF for further instructions.

6.1.5 Verify that samples were received on the expected date. If samples are received on a date later than expected, notify the Sample Coordinator at the SPF so that shipping costs may be adjusted/refunded between the SPF and freight agency. The SPF Sample Coordinator must be notified regardless of which agency the

samples are received from.

- 6.2 All discrepancies must be recorded on the original COC with the initials and date of the individual making the notation. Also, all discrepancies must be communicated either verbally or in writing to the relinquishing agency. If it is determined that there is an error on the COC or sample bags that needs to be corrected, the relinquishing agency must communicate that change in writing to the receiving agency (see Section 8.0).
 - 6.2.1 For procedures on documenting changes to a COC, see Section 9.0.
 - 6.2.2 Once the discrepancies are resolved, it is important that the resolution be communicated in writing to all agencies involved with the samples up to that point to ensure that the data is consistent within all applicable Scribe databases, as well as the analysis paper trail.
 - 6.2.3 It is important that the eCOC be modified as well if it will be used during sample receipt procedures. However, if samples have already been received and the data entered into a Laboratory Information Management System (LIMS), it is not necessary to update the eCOC unless it is being retained by the analytical laboratory.
- 6.3 Once all the information on the COC forms and samples has been verified, and all discrepancies noted, the individual receiving the samples must complete the bottom section of the COC form with their signature, their agency/lab ID (e.g., ESATR8), date and time of sample receipt, and condition of samples upon receipt. All written requests for changes to the COC must be received before the samples are processed any further.
- 6.4 For all other sample receiving procedures not specified in this SOP, laboratories should reference their internal SOPs.

7.0 RELINQUISHING A COC

- 7.1 Each agency is responsible for ensuring the accuracy of the COC and the associated samples in their custody. The agency with custody is always required to confirm that the samples within their custody match the COC. This must be completed prior to relinquishing or immediately after receiving a COC regardless of how many times a COC has been relinquished or received previously.
- 7.2 Typically, the agency receiving samples will make a copy (electronic and/or hardcopy) of the COC for their records. This may be done after the COC is signed as received, after any changes are made to the COC, and again after the COC is signed as relinquished.
 - 7.2.1 Any copy that is made must be marked "Copy of COC" at the top middle of the copy.
 - 7.2.2 Each agency should reference their own internal SOPs for direction on when to make copies of COCs, as well as procedures to follow when relinquishing samples.
- 7.3 When a COC and its associated samples are relinquished to another location, only the original COC should accompany the samples (see Section 9.4 for exceptions).

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- 7.3.1 In the case of a carbon copy COC, the top white paper is the original that stays with the samples, and all others are the copies which do not accompany the samples.
- 7.3.2 Corrected COC copies emailed to the relinquishing agency, or any copies made while in the relinquishing agency's custody, are to be retained by that agency and should not be shipped with the samples.
- 7.4 For Transmission Electron Microscopy (TEM) interlaboratory analyses, new COCs will be created and emailed to the relinquishing laboratory for the specific interlaboratory samples only. Only the grid boxes for those specific samples will be relinquished and sent to the analytical laboratory.
 - 7.4.1 After the interlaboratory analysis is complete, the interlaboratory COC and its associated grids will be relinquished and returned to the originating laboratory.
 - 7.4.2 Once received at the original location, the grids from the interlaboratory COC will be returned to the original COC sample grid box.
 - 7.4.3 When the original COC and samples are shipped to another location, the grids will be retained by the analytical laboratory along with the interlaboratory COC. A copy of the interlaboratory COC should not accompany the original COC.

8.0 REQUESTING CHANGES TO A COC

- 8.1 When a change needs to be made to a COC, the request for the change must be made in writing. This written request of a change must be received before samples move beyond initial sample receipt procedures (e.g. before samples are logged into a LIMS, before sample analysis commences).
- 8.2 The most efficient written communication is for the relinquishing agency to correct, by hand, their copy of the COC, and email this to the receiving agency. This is the only COC that should be emailed to the receiving agency for the correction. Once the database from which the COC was created is updated, a new, corrected COC should not be emailed to the receiving agency. For procedures on documenting changes to a COC, see Section 9.0.
 - 8.2.1 While emailing a corrected COC copy is the most efficient way to communicate the request, it is not required. The relinquishing agency may choose to write out the request in the body of an email, and this may serve as the written request.
 - 8.2.2 If an email is written to request a change, this email must follow the procedures outlined in the remainder of this section.
- 8.3 If the SPF initially received the samples and then shipped them to an analytical laboratory, the SPF will make the correction on their COC copy, as well as forward the email to the appropriate laboratory so that the correction may be transferred to the original COC.
- 8.4 Once emailed, the receiving agency will transfer these changes by hand to the original COC (see Section 9.0). The received written request must be printed to hardcopy, initialed and dated to acknowledge it was read and received, labeled with the applicable laboratory

job number, and included in the Analytical Test Report behind the original COC.

- 8.5 The written request must be filed and saved with the other records retained for that specific COC for an indefinite amount of time, until otherwise directed by the EPA. These records must be made available to any other agency that requests them.

9.0 DOCUMENTING CHANGES TO A COC

- 9.1 Any time a change needs to be made to a COC, specific procedures must be followed to ensure that all information regarding the change is recorded and consistent. Standard laboratory practices regarding errors and corrections have been modified to accommodate the frequent change of hands for a given COC.
- 9.2 When information presented on a COC is incorrect, a single line is drawn through it, and the correct information is hand-written next to it, followed by the first initial, last name and agency of the individual requesting the change (if relevant), then the initials and date of the individual hand-writing the change (e.g., ~~2D-00111~~ 2D-00222 per A. Wandler, ESATR8, DK 03/12/13).
- 9.2.1 If the individual recording the change on a COC is the same individual requesting the change, it is not necessary for that individual to record their first initial, last name and agency. They are only required to record the change with their initials and date.
- 9.2.2 For corrected copy COC written requests, the individual requesting the change is the individual whose initials and date are recorded on the COC copy. This may be different than the individual that actually emails the COC copy as a written request to the receiving laboratory.
- 9.2.3 For written requests in the body of an email, the individual requesting the change is the individual that sent the email.
- 9.3 When information needs to be added to a COC, the same procedures in Section 9.2 are followed, except that nothing needs to be crossed out. If the additional information applies all pages of a COC (e.g., master bag custody seal is not present), it should be added to the last page only (the last page typically has more free space than the preceding pages). If the additional information applies to a specific sample, it should be added to the page containing that sample only.
- 9.3.1 Additional information on a COC should be recorded in open or blank areas, such as a Comments field, below the complete list of samples, or at the bottom of the COC below the signature fields.
- 9.3.2 If information is added near the margins of a COC, any copies or scans made must clearly show this information.
- 9.4 When specific samples need to be removed from one COC and added to another COC, the following procedures must be followed.
- 9.4.1 If the samples being removed from a COC will be shipped to another location before being added to another COC, a copy of the original COC must accompany

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them.

9.4.1.1 Make a copy of the original COC before recording any comments and before relinquishment. At the top middle of the COC, write "Copy of COC".

9.4.1.2 On the copy COC, circle the sample numbers being removed. Record a comment indicating that the samples are being removed from the COC and shipped to another location, the new COC number to which the samples are being added (if known), the first initial, last name and agency of the individual making the request (if different than the individual recording the change), and the recorders initials and date (e.g., "Samples shipped to Troy SPF, removed from this COC and transferred to COC XX-XXXX per A. Wandler, ESATR8, NM 03/27/13").

9.4.1.3 At the bottom of the copy COC, indicate the number of samples being shipped and relinquish the COC with a signature and date.

9.4.1.4 On the original COC (which is retained by the relinquishing agency), cross out with a single line the sample numbers being removed. Record a comment indicating that the samples are being removed from the COC, the new COC number to which the samples are being added (if known), the first initial, last name and agency of the individual making the request (if different than the individual recording the change), and the recorders initials and date.

9.4.2 If the samples being removed from one COC are added to another COC before shipment, only the COC they are added to must accompany them.

9.4.2.1 The original COC should have the sample numbers being removed crossed out with a single line.

9.4.2.2 Record a comment indicating that the samples are being removed from the COC, the new COC number to which the samples are being added (if known), the first initial, last name and agency of the individual making the request (if different than the individual recording the change), and the recorders initials and date.

9.4.3 If the samples are being added to an existing COC, the sample information should be recorded on the COC as additional information (see Section 9.3).

9.4.4 The COC (either new or existing) the samples are added to does not require any notations referring to the sample's original COC; notations will already be made on the original COC, as well as in Scribe.

10.0 COC ARCHIVE AND STORAGE

10.1 Samples that are processed at the SPF are split into multiple fractions, and the fractions that are not shipped to an analytical laboratory for analysis are stored in an EPA-approved archive facility.

10.1.1 These fractions are assigned to an archive COC, which is printed and kept with the samples in storage bins. This COC is not signed as relinquished or received because the samples are not relinquished to another agency. However, an initial and date of the individual verifying the COC and its contents must be recorded in

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the bottom right corner of the archive COC.

- 10.1.2 If a sample on an archive COC needs to be shipped to an analytical laboratory, it will be removed from the archive COC with a notation stating what sample is being removed, which COC that sample is being transferred to and the recorder's initials and date (see Section 9.4).
- 10.2 When samples are shipped to an EPA-approved archive facility for storage, samples will be received following procedures described in Section 6.0. The original COC will remain with the samples in the storage bin, and a copy of the COC will be maintained by the SPF Sample Coordinator.
- 10.3 For proper legal management of samples, it is important that the original COC be well documented (as necessary) and that the original COC remain the only copy that stays with the samples.
- 10.4 Prior to the implementation of this SOP, a COC may have been copied multiple times, each copy containing different added information, and all copies kept with the samples.
 - 10.4.1 These COC copies will remain with the samples, be organized in descending order with the most recent copy placed on top, and stapled together.
 - 10.4.2 If one of these COC needs to be relinquished or modified, only the top (most recent) copy will be signed or modified, but the stack will remain intact unless otherwise directed by the EPA.

11.0 SUPPORTING DOCUMENTATION

The following is a list of documents that agencies should refer to in conjunction with this SOP:

Data Management Plan (posted on Libby e-Room)
Site-Wide Quality Assurance Reference Document (posted on Libby e-Room)
Agency-specific SOPs regarding sample receipt procedures

12.0 REFERENCES

There are no references for this SOP. The information contained within this SOP is based on verbal and written communication between the EPA and Libby contractors.

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ATTACHMENT 1

Example Corrected COC

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Date: 1/23/07

SOP No. EPA-LIBBY-08

Title: INDIRECT PREPARATION OF AIR AND DUST SAMPLES FOR TEM ANALYSIS

Author: Ron Mahoney, Ed Cahill

EMSL Analytical, Inc.

SYNOPSIS: A standardized method is presented for indirect preparation of air and dust samples for analysis by TEM.

Received by QA Unit:

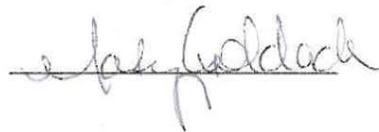
APPROVALS:

TEAM MEMBER

SIGNATURE/TITLE

DATE

EPA Region 8



1/23/07

REVISION LOG

Revision	Date	Reason
0	11/28/06	--
1	1/23/07	Clarification of filter configuration, secondary and tertiary dilution procedures.

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1.0 PURPOSE

Some air samples collected at the Libby Superfund site are overloaded with debris and/or have obvious non-uniform loading, so analysis for asbestos by Transmission Electron Microscopy (TEM) requires an indirect preparation of the sample. All dust samples collected at the Libby Superfund site are prepared for TEM analysis using an indirect preparation. The purpose of this SOP is to provide a standardized procedure for the indirect preparation of air and dust samples that minimizes the loss of sensitivity. In addition, this SOP allows for the retention of a portion of the original air sample filter for archive whenever possible.

2.0 RESPONSIBILITIES

The Laboratory Director is responsible for ensuring that all laboratories participating in the analysis of air samples at the Libby site are aware of this SOP and that all analysts follow this SOP. Laboratory managers and analysts are responsible for communicating to the Libby laboratory coordinator (CDM), Volpe Center and appropriate USEPA Region 8 Remedial Project Manager or Regional Chemist any recommended changes or proposed improvements to the SOP.

3.0 EQUIPMENT

Equipment needed to perform indirect preparations of air samples includes the following:

- Transmission electron microscope (NVLAP compliant)
- Energy dispersive X-ray system (NVLAP compliant)
- High vacuum carbon evaporator with rotating stage
- HEPA hood (NVLAP compliant)
- Exhaust or fume hood
- Particle-free water
- Glass container for ashing
- Disposable single use containers of at least 100 ml capacity
- Waterproof marker
- Forceps
- Ultrasonic bath
- Appropriate disposable glass or variable pipets with disposable tips
- Disposable 25 mm filter funnels
- Side arm filter flask
- Cellulose support pad, 25 mm diameter
- MCE filters, 25 mm diameter, $\leq 0.22 \mu\text{m}$ and $5.0 \mu\text{m}$ pore size
- Storage container for 25 mm filter
- Glass slides, approximately 25 x 76 mm in size
- Scalpel blades, # 10 or equivalent and handle
- Desiccator or low temperature drying oven
- Acetone, reagent grade
- Glacial acetic acid

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- Plasma asher, low temperature
- pH paper
- Tygon tubing, or equivalent
- Small vacuum pump for filtration
- Glass petri dishes
- Jaffe washer
- Carbon evaporator rods
- Wash bottles, plastic
- Reagent alcohol

4.0 METHOD SUMMARY

Figure 1 presents a simplified overview of the TEM indirect preparation procedure for overloaded air samples and dust samples. As seen, there are two general indirect preparation procedures, one that includes ashing of the primary filter and one that does not include ashing of the primary filter.

Laboratory modification LB-000053 provides a list of which sample prefix codes shall be prepared using an ashing procedure and which should not be prepared using an ashing procedure. In cases where there is a conflict regarding sample type between the sample prefix as defined by the most recent version of LB-000053 and the chain of custody instructions, the chain of custody instructions take precedent. Additionally, once sample preparations have begun, there may be cases where the analyst determines that ashing is necessary to obtain acceptable filter loading. Samples for which ashing may be warranted include indoor air or dust samples collected from properties with elevated levels of organic particulates (e.g., due to cigarette smoke or use of a wood-burning stove). In these samples, ashing may further reduce particulate loading, thus allowing for an improved analytical sensitivity.

The sections below present the detailed steps associated with each procedure. For all indirect preparations, specimen preparation should be performed in a clean facility that is separate from both bulk and air preparation areas and preparation shall take place in a negative flow HEPA hood to prevent any possible contamination of the laboratory or personnel.

4.1 PROCEDURE 1: Indirect Preparation with Ashing

This procedure should be followed for air and dust samples where LB-000053 or the chain of custody form indicates that ashing should be performed. For the purpose of the Libby Superfund Site, air samples are defined as overloaded if there is >25% obscuration on the majority of the grid openings.

If there is no loose material present in the air cassette or adhering to the cowl, this procedure is generally similar to the indirect preparation method specified in ISO 13794, but has been modified to increase the total solution volume from 40 ml to 100 ml and to retain a portion of the original filter. The use of a 100 ml final volume is selected because it allows for preparation of a

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series of indirect samples with volumes that are sufficiently large that secondary dilution is not needed to ensure uniform deposition on the filter.

If there is loose material present in the air cassette or adhering to the cowl, or if the sample is a dust sample, a portion of the original filter is not retained for archive, since it is assumed that there will be uneven loading on the filter. Because of this, an archived portion of the original filter is unlikely to be representative. In this case, the indirect preparation procedure is similar to the method specified in ASTM D-5755, but has been modified to include an ashing of the primary filter.

- 4.1.1 Carefully wet-wipe the exterior of the cassettes to remove any possible contamination prior to taking the cassettes into the clean preparation area.
- 4.1.2 Within a safety hood, carefully open the cassette and verify if there is any loose material in the cassette or adhering to the cowl. **If this is an air sample and there is no visible loose material present, proceed to Step 4.1.6.**
- 4.1.3 Any loose material that is present in the cassette should be poured into a disposable 50 ml glass beaker or similar container.
- 4.1.4 Using freshly cleaned forceps, remove the sample collection filter from the sampling cassette and place it in the same disposable 50 ml glass beaker or similar container with the side containing the sample facing down.
- 4.1.5 Using a 50/50 alcohol/particle-free water solution, rinse any material adhering to the cowl into a new 25 mm diameter disposable filtration funnels. If the filtration unit does not come pre-assembled with the necessary components (e.g. contains a glass fiber filter instead of the required MCE filter), it will be necessary to disassemble the stock cassette as it comes from Whatman and discard the glass-fiber filter. Rinse the filter unit thoroughly with particle free water and reassemble the filter unit using a cellulose support pad (Pall 66238), a 5.0µm pore size MCE diffuser filter (Enviro-pore FILA500A025A), and a 0.2 µm pore size MCE final filter (Enviro-pore FILA020A025A). Apply vacuum. When all solution has passed through, rinse sides of filter funnel with a stream of particle free water to dislodge any particulate that might be adhering to the sides of the filter funnel. Once filtration is complete turn off vacuum, remove filter from unit and dry. Once the filter is dry, place it in the container with the original filter and **proceed to Step 4.1.8.**
- 4.1.6 Using freshly cleaned forceps, remove the sample collection filter from the sampling cassette and place it on a clean glass microscope slide that will be used as a cutting surface. Using a freshly cleaned curved scalpel blade, cut off ½ of the filter (estimate the ½ as precisely as possible as this affects the final concentration) with a rocking motion.
- 4.1.7 Place the remaining portion of the original filter in archive. (Note: In cases where an initial direct preparation of an air sample was attempted and found to be overloaded, this archive portion will be approximately ¼ of the original filter.) Place ½ of the primary filter

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in a clean, single use disposable glass container with the side containing the sample facing down.

- 4.1.8 Cover the container with aluminum foil, forming a tight seal around the mouth.
- 4.1.9 Perforate the foil in 15-20 places with a syringe needle to allow for gas exchange during plasma ashing.
- 4.1.10 Place the sample container in the plasma asher chamber. Depending on the size of the plasma asher chamber, several samples may be ashed simultaneously.
- 4.1.11 Operate the plasma asher using the minimum power at which a glow-discharge is observed, until the filter appears to be completely ashed. Loss of particulate and fibers from the container will occur if the plasma asher is operated at excessive radio-frequency power. During ashing of mixed cellulose ester (MCE) filters, a critical point is reached during the oxidation at which a sudden, violent ignition may occur if the radio-frequency power is excessive. This may result in a loss of fibers from the container, contamination of the interior of the chamber, and possible cross-contamination of the samples. For this reason, ashing of the blank should be observed closely during the early stages of oxidation, in order to ensure that the radio-frequency power setting is such that sudden ignition does not occur.
- 4.1.12 After 100% ashing is complete based on visual observation, increase the plasma asher power to maximum and ash for a period of one additional hour.
- 4.1.13
While final ashing is in progress, set up the filtration system to be used. In order to minimize the chances of contamination, only 25 mm diameter disposable filtration funnels shall be used. If the filtration unit does not come pre-assembled with the necessary components (e.g. contains a glass fiber filter instead of the required MCE filter), it will be necessary to disassemble the stock cassette as it comes from Whatman and discard the glass-fiber filter. Rinse the filter unit thoroughly with particle free water and reassemble the filter unit using a cellulose support pad (Pall 66238), a 5.0 μ m pore size MCE diffuser filter (Enviro-pore FILA500A025A), and a 0.2 μ m pore size MCE final filter (Enviro-pore FILA020A025A). Filter as usual, using restraint with the amount of vacuum applied to avoid uneven loading. Add 20 ml of particle-free water to the filtration apparatus prior to applying vacuum and introduction of the sample suspension. When seating the filters in the filtration unit, it is essential that the vacuum be evenly applied to help ensure an even distribution of particulate on the filter. There should be no air bubbles or surface abnormalities anywhere in the filter assemblage. This is accomplished through wetting each successive filter as it is placed in the filtration unit and applying a light vacuum. This will ensure that the filters are flat and that there are no air bubbles.
- 4.1.14 After ashing is complete, admit air slowly to the chamber and remove the samples from the plasma asher chamber and place back into a safety hood.

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- 4.1.15 Remove the aluminum foil from the top of the sample container.
- 4.1.16 Using particle free water in a squirt bottle, carefully rinse the ashed residue from the ashing container into a clean disposable sample container of at least 100 ml with a watertight lid, such as a sealed specimen cup. Rinse the residue into the 100 ml container to an initial volume of approximately 90 ml. Adjust pH to approximately 3-4 using a 10% solution of glacial acetic acid, and checking with pH paper. Bring the final volume to 100 ml and cap tightly.
- 4.1.17 Briefly hand shake (3 seconds) the capped container containing the sample suspension.
- 4.1.18 Place the container in a calibrated tabletop ultrasonic bath and sonicate at 50 - 100 nW/ml for three minutes. The liquid level in the bath should be $\frac{1}{2}$ to $\frac{3}{4}$ the height of the sample containers. Wipe the outside of the sample containers dry when removing them from the bath.
- 4.1.19 After sonication, lightly hand shake the suspension for 3 seconds, and allow it to stand undisturbed for 2 minutes to allow large particles to settle to the bottom or float to the top.
- 4.1.20 For each sample, prepare three secondary filters by applying volumes of 50 ml, 25 ml, and 10 ml. For air samples where the direct preparation proves to be overloaded, it is acceptable to filter aliquot volumes other than the usual 10 ml, 25 ml, and 50 ml series, either a greater or lesser volume, in order to produce a sample with the highest possible f-factor without violating the overload criteria. Draw each aliquot to be filtered with the same pipette and dispense into the appropriate filter funnel. Avoid pipetting any large settled or floating particles. Apply vacuum to the filtration apparatus to draw each volume through the filter. For samples where the 10 ml aliquot filter is obviously overloaded and a secondary dilution will be required (see 4.1.21), it is not necessary to attempt to filter the 25 ml and 50 ml aliquots through 25 mm filter units.
- 4.1.21 If a preliminary observation of the 10 ml secondary filter appears overloaded take 10 ml of the remaining volume and dilute to 100 ml. From this secondary dilution, prepare a second series of filters using 50 ml, 25 ml, and 10 ml (corresponding to 5 ml, 2.5 ml, and 1 ml of the original suspension). Based on the original 10 ml aliquot filter loading, it is acceptable to filter aliquot volumes other than the usual 10 ml, 25 ml, and 50 ml series in order to produce a sample with the highest possible f-factor without violating the overload criteria. In some instances, it may be necessary to perform a tertiary serial dilution, taking 10 ml of the secondary dilution, adding it to 90 ml of particle free water, and filtering another series of aliquots of 10 ml, 25 ml, and 50 ml.
- 4.1.22 Disassemble the filtration units. Carefully remove the filters from the filtration apparatus using fine forceps, being careful to only touch the inactive rim of the filter that has not been exposed to the sample. Place each filter in a labeled petri dish or other similar container, active side up and dry.

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- 4.1.23 Select the secondary filter from the dilution series yielding the largest possible f-factor (highest possible volume) which does not violate the criteria for an overloaded sample. Experience has shown that a light staining of the filter will yield a suitable preparation for analysis.
- 4.1.24 Perform a standard TEM sample preparation procedure.
- 4.1.25 If TEM examination of the lowest volume aliquot filtered is deemed overloaded (>25% particulate), consult with the Libby laboratory coordinator (CDM) to select the most appropriate next step.
- 4.1.26 Carefully label and place each of the unused secondary filters and the remaining portion of the selected secondary filter in archive.
- 4.1.27 Place any remaining sample solution in a graduated cylinder or pipet. The largest known quantity of the remaining solution should be filtered through a 25 mm disposable filtration unit with a $\leq 0.22 \mu\text{m}/5.0 \mu\text{m}$ pore size MCE filter set in conjunction with a cellulose support pad and dried after removal from the filtration unit. A larger diameter (e.g. 47 mm) filtration unit with the same filter configuration may be used as needed to avoid situations where a 25 mm diameter filter may become obstructed with material. The dried filter shall be placed in an appropriate container, and labeled with the sample number, filter type, and volume applied to the filter. This filter will then be archived with the other archived filters from the sample.
- 4.1.28 Discard the remaining portion of the sample solution using standard laboratory protocols.

4.2 PROCEDURE 2: Indirect Preparation without Ashing

This procedure should be followed for air and dust samples where LB-000053 or the chain of custody form indicates that ashing should not be performed. For the purpose of the Libby Superfund Site, samples are defined as overloaded if there is >25% obscuration on the majority of the grid openings.

If there is no loose material present in the air cassette or adhering to the cowl, this procedure is generally similar to the indirect preparation method specified in ASTM D-5755, but has been modified to allow for an archive of the original filter.

If there is loose material present in the air cassette or adhering to the cowl, or if the sample is a dust sample, a portion of the original filter is not retained for archive, since it is assumed that there will be uneven loading on the filter. Because of this, an archived portion of the original filter is unlikely to be representative. In this case, the indirect preparation procedure is equivalent to the method specified in ASTM D-5755.

- 4.2.1 Carefully wet-wipe the exterior of the cassettes to remove any possible contamination prior to taking the cassettes into the clean preparation area.

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- 4.2.2 Carefully open the cassette and verify if there is any loose material in the cassette or adhering to the cowl. **If this is an air sample and there is no visible loose material present, proceed to Step 4.2.5.**
- 4.2.3 Using a 50/50 alcohol/particle-free water solution, rinse any material adhering to the cowl down onto the sample collection filter (still inside the sampling cassette).
- 4.2.4 Using freshly cleaned forceps, remove the sample collection filter from the sampling cassette and place it into a clean disposable sample container of at least 100 ml with a watertight lid, such as a sealed specimen cup. **Proceed to Step 4.2.7.**
- 4.2.5 Using freshly cleaned forceps, remove the sample collection filter from the sampling cassette and place it on a clean glass microscope slide that will be used as a cutting surface. Using a freshly cleaned curved scalpel blade, cut off ½ of the filter (estimate the ½ as precisely as possible as this affects the final concentration) with a rocking motion.
- 4.2.6 Place the remaining portion of the original filter in archive. (Note: In cases where an initial direct preparation of an air sample was attempted and found to be overloaded, this archive portion will be approximately ¼ of the original filter.) Place ½ of the primary filter in a clean disposable sample container of at least 100 ml with a watertight lid, such as a sealed specimen cup.
- 4.2.7 Bring the total volume of the suspension up to approximately 90 ml using particle-free water only.
- 4.2.8 Adjust the suspension to a pH of 3-4 using a 10 % solution of acetic acid. Use pH paper to test.
- 4.2.9 Bring the total volume up to 100 ml using particle-free water and cap tightly.
- 4.2.10 Set up the filtration system to be used. In order to minimize the chances of contamination, only 25 mm disposable filtration funnels (such as Whatman cat. #:1922-1820) shall be used. If the filtration unit does not come pre-assembled with the necessary components (e.g. contains a glass fiber filter instead of the required MCE filter), it will be necessary to disassemble the stock cassette as it comes from Whatman and discard the glass-fiber filter. Rinse the filter unit thoroughly with particle free water and reassemble the filter unit using a cellulose support pad (Pall 66238), a 5.0µm pore size MCE diffuser filter (Enviro-pore FILA500A025A), and a 0.2 µm pore size MCE final filter (Enviro-pore FILA020A025A). Filter as usual, using restraint with the amount of vacuum applied to avoid uneven loading. Add 20 ml of particle-free water to the filtration apparatus, prior to applying vacuum and introduction of the sample suspension. When seating the filters in the filtration unit, it is essential that the vacuum be evenly applied resulting in even distribution. There should be no air bubbles or surface abnormalities anywhere in the filter assemblage. This is accomplished through wetting each successive filter as it is placed in the filtration unit and

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applying a light vacuum. This will assure that the filters are flat and that there are no air bubbles. Ensure that suspension is filtered within 24 hours to avoid problems associated with bacterial and fungal growth.

- 4.2.11 Briefly hand shake (3 seconds) the capped container containing the sample suspension.
- 4.2.12 Place the container in a calibrated tabletop ultrasonic bath and sonicate at 50 - 100 nW/ml for three minutes.
- 4.2.13 After sonication, lightly hand shake the suspension for 3 seconds, and allow it to stand undisturbed for 2 minutes to allow large particles to settle to the bottom or float to the top.
- 4.2.14 For each sample, prepare three secondary filters by drawing aliquots of 50 ml, 25 ml, and 10 ml. For air samples where the direct preparation is overloaded, it is acceptable to filter aliquot volumes other than the usual 10 ml, 25 ml, and 50 ml series (either greater or lesser volumes) in order to produce a sample with the highest possible f-factor without violating the overload criteria. Draw each aliquot to be filtered with the same pipette and dispense into the appropriate filter funnel. Avoid pipetting any large settled or floating particles. Apply vacuum to the filtration apparatus to draw each volume through the filter. For samples where the 10 ml aliquot filter is obviously overloaded and a secondary dilution will be required (see 4.2.15), it is not necessary to attempt to filter the 25 ml and 50 ml aliquots through 25 mm filter units.
- 4.2.15 If a preliminary observation of the 10 ml secondary filter appears overloaded take 10 ml of the remaining volume and dilute to 100 ml. From this secondary dilution, prepare a second series of filters using 50 ml, 25 ml, and 10 ml (corresponding to 5 ml, 2.5 ml, and 1 ml of the original suspension). Based on the original 10 ml aliquot filter loading, it is acceptable to filter aliquot volumes other than the usual 10 ml, 25 ml, and 50 ml series (either greater or lesser volumes) in order to produce a sample with the highest possible f-factor without violating the overload criteria. In some instances, it may be necessary to perform a tertiary serial dilution, taking 10 ml of the secondary dilution, adding it to 90 ml of particle free water, and filtering another series of aliquots of 10 ml, 25 ml, and 50 ml.
- 4.2.16 Disassemble the filtration unit. Carefully remove the filter from the filtration apparatus using fine forceps, being careful to only touch the inactive rim of the filter that has not been exposed to the sample. Place the filter in a labeled petri dish or other similar container, active side up and dry.
- 4.2.17 Select the secondary filter from the dilution yielding the largest possible f-factor (highest volume) which does not violate the criteria for an overloaded sample. Experience has shown that a light staining of the filter will yield a suitable preparation for analysis.
- 4.2.18 Perform a standard TEM sample preparation procedure.

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- 4.2.19 If TEM examination of the lowest volume aliquot filtered is deemed overloaded, consult with the Libby laboratory coordinator (CDM) to select the most appropriate next step.
- 4.2.20 Place each of the unused secondary filters and the remaining portion of the selected secondary filter in archive.
- 4.2.21 Place any remaining sample solution in a graduated cylinder or pipet and add to a prepared 25 mm filtration unit containing a $\leq 0.22 \mu\text{m}/5.0 \mu\text{m}$ pore size filter set with a cellulose support pad in a disposable filtration unit with a small volume of particle free water to facilitate the production of a homogeneous solution and record the volume of sample solution added. A larger diameter (e.g. 47 mm) filtration unit with the same filter configuration may be used as needed to avoid situations where a 25 mm diameter filter may become obstructed with material. Add 10 ml particle free water to the sample container containing the residual filter and sonicate for three minutes. Add this solution to the filtration unit for the corresponding filtration unit for each sample as described in the first part of this paragraph. Do not include this 10 ml in the volume calculation of the sample solution added. This solution should then be filtered through the filtration unit and dried after removal from the filtration unit. The dried filter shall be placed in an appropriate container, and labeled with the sample number, filter type, and volume applied to the filter. This filter will then be archived with the other archived filters from the sample.
- 4.2.22 Discard the remaining portion of the sample solution using standard laboratory protocols.

5.0 DOCUMENTATION AND ARCHIVE STORAGE

Project-specific Index IDs are recorded on all air samples. During each indirect preparation step, this Index ID is noted on the sample-specific beakers, containers, and filtration units.

In those cases where no loose material is present in the cassette or adhering to the cowl, the remaining portion of the original primary filter is placed in a suitable container and clearly labeled with the sample number and indicated that it is the original primary filter. In those cases where secondary or tertiary filters are prepared, all filters or remnants of filters will be archived into suitable containers, and clearly labeled with the sample number and the volume of the aliquot applied to each filter.

Analysis-specific details about the indirect preparation will be recorded in the sample TEM electronic data deliverable (EDD) spreadsheet. In the TEM EDD, if the sample is prepared using Procedure 1 (see Section 4.1) the preparation method should be identified as “IA – Indirect, ashed” and the appropriate inputs should be recorded in the fields provided. If the sample is prepared using Procedure 2 (see Section 4.2), the preparation method should be identified as “I – Indirect” and the appropriate inputs should be recorded in the fields provided. The spreadsheet is designed to automatically calculate the dilution factor, or f-factor, which is used in the calculation the sample air or dust concentration.

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6.0 QUALITY ASSURANCE

All quality control sample results will be monitored for potential contamination. If sample results indicate cross-contamination, the laboratory will identify the affected samples and notify the USEPA Regional Chemist and project laboratory coordinator (CDM). Laboratory procedures will be re-assessed and appropriate changes will be made and documented accordingly by the project laboratory coordinator.

6.1 Lot Blanks

All cassettes utilized in the Libby project are screened for contamination by either TEM analysis or a combination of TEM and PCM analysis. One lot blank is prepared and analyzed from each carton of cassettes prior to using the lot of cassettes for sampling. The entire carton of cassettes will be rejected if any asbestos fiber is detected on the lot blank.

6.2 Filter blanks

Prior to filtration of the sample aliquot, 100ml particle-free water should be filtered. Acceptance criteria for filter blanks are as specified for laboratory blanks in the latest version of laboratory modification of LB-000029.

6.3 Plasma asher blanks

To ensure that contamination is not introduced during the ashing process, a container with an unused filter should be run as a blank with each batch of samples ashed. This sample will be prepared using the standard TEM sample preparation procedure and examined as per the established QC sequence. Acceptance criteria for plasma asher blanks are as specified for laboratory blanks in the latest version of laboratory modification of LB-000029.

7.0 DECONTAMINATION

All non-disposable equipment used during sample preparation must be decontaminated prior to use. Because the prescribed filtration units used to prepare the secondary filters are disposable, decontamination of filtration units is not required.

8.0 GLOSSARY

EDD - Electronic Data Deliverable. A Libby-specific spreadsheet designed to capture the detailed analysis and raw structure data generated during a TEM analysis. Contact the project laboratory coordinator (CDM) for the current TEM spreadsheet version.

HEPA - High Efficiency Particulate Air

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MCE - Mixed Cellulose Ester

TEM - Transmission Electron Microscopy

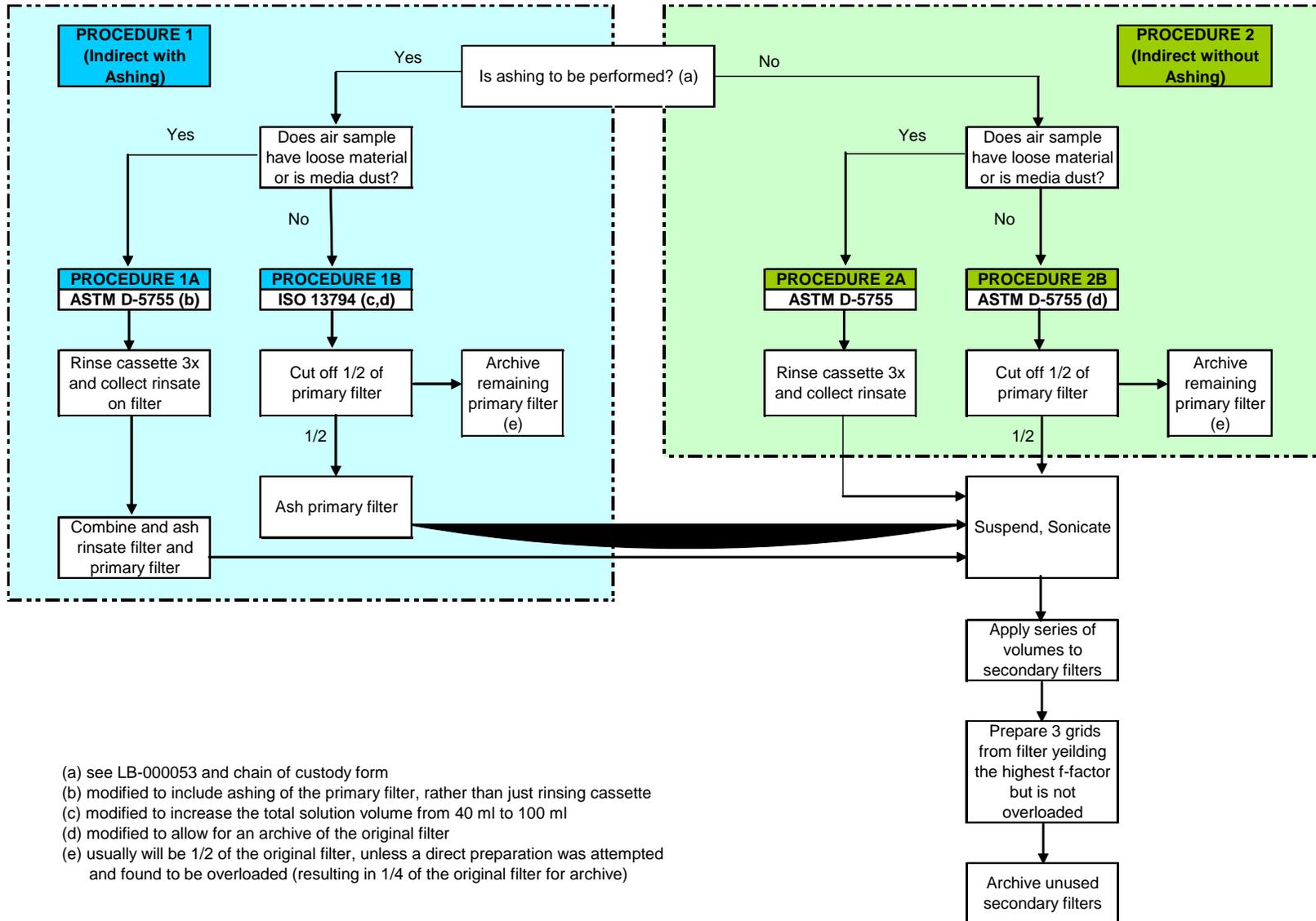
9.0 REFERENCES

ISO 13794. Ambient air - Determination of asbestos fibres - Indirect-transfer transmission electron microscopy method. International Organization for Standardization (ISO) 13794:1999. November 15, 1999.

ASTM D-5755. Test Method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Structure Number Surface Loading. ASTM D 5755-03. October 2003.

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FIGURE 1. INDIRECT PREPARATION OF OVERLOADED AIR SAMPLES AND DUST SAMPLES FOR TEM ANALYSIS



LIBBY ASBESTOS SUPERFUND SITE STANDARD OPERATING PROCEDURE
 APPROVED FOR USE AT THE LIBBY ASBESTOS SUPERFUND SITE ONLY

QUALITATIVE ESTIMATION OF ASBESTOS IN COARSE SOIL BY VISUAL EXAMINATION USING
 STEREO MICROSCOPY AND POLARIZED LIGHT MICROSCOPY

Date: September 19, 2012

SOP No.: SRC-LIBBY-01 (Revision 3)

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 USING STEREO MICROSCOPY AND POLARIZED LIGHT MICROSCOPY

SYNOPSIS: A standardized method for identifying and quantifying (in mass percent) asbestos fibers in coarse soil (>¼-inch particles) using stereomicroscopy and polarized light microscopy is provided. This method is based on NIOSH Method 9002 and EPA Method 600/R-93/116, with project specific modifications intended for application at the Libby Asbestos Superfund Site. Sampling and plan developers and data users are cautioned to understand how data are generated from this SOP.

APPROVALS:

USEPA Region 8

Dania Zinner
 Signature

9-25-12
 Date

Dania Zinner
 Print Name

RPM
 Title

ESAT Region 8

[Signature]
 Signature

9-21-12
 Date

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ESAT Team Lead
 Title

Revision	Date	Principal Changes and Author
0	11/12/2002	Initial Author: Sally M. L. Gibson (Syracuse Research Corporation)
1	5/20/2003	Provided clarification on dealing with very small particles
2	4/21/2004	Included statements on limitations of intended use
3	9/19/2012	Entire SOP review and update provided by ESAT Region 8

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LIST OF ATTACHMENTS

Attachment 1: Libby Asbestos Superfund Site Analysis Bench Sheet (PLM-Grav)

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1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide a standard approach for quantitative analysis of asbestos in samples of coarse soil or other soil-like materials using stereomicroscopy with confirmation of asbestos identification by Polarized Light Microscopy (PLM). This SOP is specifically intended for application at the Libby Asbestos Superfund Site (referred to as the Libby Site from this point forward).

2.0 SCOPE AND APPLICATION

This method is intended for analysis of asbestos in coarse soil or other similar soil-like materials in which the soil has been taken through the preparation process described in Section 4.0. This method is appropriate for the analysis of all types of asbestos fibers (chrysotile and amphiboles), including Libby Amphibole (LA). For the purposes of this SOP, the term 'asbestos' refers to the six regulated asbestos minerals (chrysotile, amosite, crocidolite, anthophyllite, tremolite, and actinolite), as well as LA.

3.0 RESPONSIBILITIES

- 3.1 It is the responsibility of the laboratory supervisor to ensure that all analyses and quality control (QC) procedures are performed in accordance with this SOP and to identify and take appropriate corrective action to address any deviations that may occur during sample preparation or analysis.
- 3.2 The Laboratory Manager, Quality Assurance Coordinator (or equivalent), and/or Analytical Lead will communicate with the client, any situations where a modification to or deviation from the SOP may be useful and/or required. The laboratory supervisor must receive approval from the client for any modification to or deviation from the SOP before incorporating any such modification or deviation into the sample preparation and analysis process (refer also to Section 8.2).
- 3.3 It is the responsibility of the laboratory to maintain a PLM SOP for Bulk Asbestos Materials, Quality Assurance Manual (QAM), or an equivalent document(s) that meets all the requirements of the National Voluntary Laboratory Accreditation Program (NVLAP) Handbook 150 and Handbook 150-3. It is also the responsibility of the laboratory to ensure its testing activities stay in compliance with the requirements of NVLAP Handbooks 150 and 150-3 and the regulatory and accrediting agencies that provide oversight of the laboratory's operations and all Libby Site project-specific requirements.

4.0 METHOD DESCRIPTION

- 4.1 This test method describes a quantitative analysis of asbestos in samples of coarse soil or other soil-like materials using stereomicroscopy, with identification of any suspicious components by PLM. It is based on the National Institute of Occupational Safety and Health (NIOSH) Method 9002 and United States

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Environmental Protection Agency (EPA) Method 600/R-93/116, with project-specific modifications provided in this SOP. Although acid-washing, solvent dissolution, and ashing described as part of the gravimetry technique in EPA Method 600/R-93/116 are not part of this test method, the techniques described in this SOP still aim to isolate any asbestos from the sample, allowing its weight to be determined (EPA, 1993). Therefore, for the purpose of this SOP, this method is referred to as PLM-Grav.

- 4.2 Soil samples from the Libby Site are processed according to the current version of SOP *ISSI-LIBBY-01, Soil Sample Preparation*, before submittal to the laboratory for analysis. This process separates the coarse fraction of the soil from the fine fraction. The fine fraction constitutes all material passing through a ¼-inch sieve, while the coarse fraction is all material retained in the ¼-inch sieve. The fine fraction is homogenized and ground to a maximum particle size of approximately 250 microns (µm). This fine fraction is further sub-divided into four fractions using a riffle splitter. This SOP is specific to the analysis of the coarse fraction soil samples. Fine fraction soil samples are analyzed according to the current version of Libby-specific SOP SRC-LIBBY-03, *Analysis of Asbestos Fibers in Fine Soil By Polarized Light Microscopy*.
- 4.3 The coarse fraction soil sample to be evaluated for asbestos content is first weighed on an analytical balance and then examined using a low magnification stereomicroscope. Microscope slide mounts of fibers suspected of being asbestos are then prepared by immersing the fibers in a liquid medium of known refractive index (RI). These slide mounts are then analyzed visually by PLM for fiber identification. Asbestos and non-asbestos phases are identified on the basis of their morphology and optical properties. Quantification of asbestos concentrations is calculated by separating the asbestos fibers from the remaining sample and weighing them. This fiber weight is divided by the total sample weight to produce the mass percent of the asbestos fibers relative to the sample.
- 4.4 All samples from the Libby Site are identified by either one or two-characters followed by a hyphen and a five digit number (referred to as the Client Sample Number). The first characters identify the type of sample as indicated by the site-specific Summary Analytical Procedure (SAP). The five digit number is assigned by the field sampling teams. All samples from the Libby Site also have an associated tag to further identify the sample (e.g., a tag of C is the coarse soil retained by the ¼-sieve for a given parent sample). At all stages of documentation, this sample number and tag must be used to properly identify the sample (as many samples have multiple tags associated with them, especially PLM samples).

5.0 ACRONYMS

ACM	Asbestos Containing Material
CHP	Chemical Hygiene Plan
COC	Chain of Custody

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EDD	Electronic Data Deliverable
EPA	United States Environmental Protection Agency
HASP	Health and Safety Plan
HEPA	High Efficiency Particulate Air
LA	Libby Amphibole asbestos
LADT	Libby Asbestos Data Tool
LDC	Laboratory Duplicate – Cross-check
LDS	Laboratory Duplicate – Self-check
LIMS	Laboratory Information Management System
MSDS	Material Safety Data Sheet
NIOSH	National Institute for Occupational Safety and Health
NIST	National Institute of Standards and Technology
NVLAP	National Voluntary Laboratory Accreditation Program
PLM	Polarized Light Microscopy
PPE	Personal Protective Equipment
QA	Quality Assurance
QAM	Quality Assurance Manual
QC	Quality Control
RI	Refractive Index
SAP	Summary Analytical Procedure
SOP	Standard Operating Procedure
SRM	Standard Reference Material
USGS	United States Geological Survey

6.0 HEALTH AND SAFETY

- 6.1 Follow general laboratory health and safety policies and regulations in the laboratory's Health and Safety Plan (HASP), Chemical Hygiene Plan (CHP), or equivalent.
- 6.2 All sample handling and preparation activities must be performed in a ventilated hood with an operating High Efficiency Particulate Air (HEPA) filtration system, a class 1 biohazard hood, or glove box with continuous airflow (negative pressure). Never have a sample container open except when the sample is inside of the sample preparation hood. Appropriate personal protective equipment (PPE) should be worn at all times.
- 6.3 Avoid repeated or prolonged contact with the RI liquids and inhalation of fumes from the RI liquids. Refer to the Material Safety Data Sheet (MSDS) forms for RI liquids for additional information and cautions.

7.0 CAUTIONS

- 7.1 The toxicity or carcinogenicity of the RI liquids used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be avoided.

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- 7.2 After processing each sample, use water and paper towels to thoroughly decontaminate all work surfaces and utensils that came into contact with a sample and/or RI liquid. Never have more than one sample container open at any one time.

8.0 GENERAL LABORATORY PRACTICES

8.1 Quality Assurance Program

- 8.1.1 Each laboratory operates under a quality assurance (QA) program appropriate to the type, range, and volume of work it performs.
- 8.1.2 It is the responsibility of the laboratory to maintain a QAM, or equivalent, in which the laboratory's QA program is detailed. Additional QA/QC requirements specific to the PLM laboratory and the Libby Site are described in Section 17.0.
- 8.1.3 All work is performed at a permanent laboratory location. Even if a laboratory is part of a larger organization, it is able to carry out all testing, calibration, and daily QA/QC activities independently, and at one location. There are no remote or sub-facilities where testing work is performed.

8.2 Documenting SOP Modifications

- 8.2.1 Any deviation from the SOP shall be documented in a laboratory modification form and then addressed in the technical Case Narrative prepared as part of the test report.
- 8.2.2 Additionally, when there is reason to suspect a departure from the SOP has affected the result or validity of data provided to the client, the client must be notified of the nature of the departure from the SOP and informed about the possible effect on the result or validity of the analysis. The course of action taken to keep the departure from recurring must also be discussed with the client.

9.0 PERSONNEL QUALIFICATIONS

- 9.1 The use of this SOP is limited to microscopists knowledgeable in the production and evaluation of asbestos data.
- 9.1.1 All personnel analyzing samples from the Libby Site are expected to be familiar with routine chemical laboratory procedures, principles of optical mineralogy, and proficient in EPA Method 600/R-93/116 and NIOSH Method 9002.
- 9.1.2 Personnel at laboratories with less than one year of experience specific to the Libby Site are required to participate in the laboratory mentoring program to obtain additional guidance and instruction. This training is provided by personnel familiar with the particular problems and types of asbestos encountered at the Libby Site.

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- 9.2 Before performing any analyses, each analyst must demonstrate the ability to generate acceptable accuracy and precision with this method. This includes successfully completing NVLAP proficiency testing.

10.0 EQUIPMENT

- 10.1 Each laboratory must be equipped with all instrumentation, hardware, software, and reference materials required for the correct performance of calibrations and tests.
- 10.2 All equipment must be properly maintained and calibrated (as appropriate) prior to use. Refer to the Libby-specific SOP SCR-LIBBY-03 (current version), Section 12.0, for further details regarding microscope calibration.
- 10.3 The following is a general list of equipment available at the PLM laboratory to perform this SOP:
- 10.3.1 Polarized Light Microscope, with:
 - 10.3.1.1 Light source and replacement bulbs
 - 10.3.1.2 Binocular observation tube
 - 10.3.1.3 Blue daylight filter
 - 10.3.1.4 Oculars (10X)
 - 10.3.1.5 Objectives: 10X, 20X, and 40X (or similar magnification)
 - 10.3.1.6 10X Dispersion Staining Objective
 - 10.3.1.7 360 degree rotatable and centerable stage
 - 10.3.1.8 Polarizer and analyzer aligned at 90 degrees to one another
 - 10.3.1.9 Bertrand lens (optional)
 - 10.3.1.10 Substage condenser with iris diaphragm
 - 10.3.1.11 Accessory slot for compensator plate
 - 10.3.1.12 First order red (550 nanometer) compensator plate
 - 10.3.1.13 Crosshair reticle
 - 10.3.1.14 Adjustment tools
 - 10.3.2 HEPA-filtered hood, class 1 biohazard hood, or glove box with continuous airflow (negative pressure)
 - 10.3.3 Binocular stereomicroscope, 10-50X magnification (approximate)
 - 10.3.4 Light source for stereomicroscope
 - 10.3.5 Muffle furnace
 - 10.3.6 Analytical balance, accurate to 1mg (0.001g)
 - 10.3.7 Libby Asbestos Data Tool (LADT) or other computer software capable of generating a project-specific Electronic Data Deliverable (EDD) that meets the current client data reporting requirements
 - 10.3.8 Mortar and Pestle (agate or porcelain)
 - 10.3.9 Vaneometer
 - 10.3.10 Wet/dry vacuum with HEPA filtration
 - 10.3.11 Decontamination equipment (disposable lint-free wipes, wet mop with bucket, etc.)

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11.0 STANDARDS, REAGENTS AND SUPPLIES

- 11.1 High Dispersion RI Liquid(s) from 1.620 to 1.640
- 11.2 1.550 High Dispersion RI Liquid
- 11.3 1.680 to 1.700 RI Liquid(s)
- 11.4 Solid RI Standards (precision optical glass, RI from 1.48 to 1.72, in gradations of 0.01, 25 standards)
- 11.5 National Institute of Standards and Technology (NIST) Standard Reference Material (SRM) 1866b - Common Commercial Asbestos consisting of chrysotile, amosite, and crocidolite
- 11.6 NIST SRM 1867a - Uncommon Commercial Asbestos consisting of tremolite, actinolite, and anthophyllite
- 11.7 Controlled Libby Amphibole Asbestos (prepared for EPA by the United States Geological Survey [USGS]), a finely-milled composite of a selected subset of 30 samples taken from the mine at the Libby Site
- 11.8 NIST Bulk Asbestos Proficiency Testing Round M12001, Sample 4, a sample of un-milled rock-form winchite/richterite taken from the mine at the Libby Site
- 11.9 Non-asbestos reference materials (gypsum, calcite, fiberglass, etc.)
- 11.10 Instrument maintenance/calibration logbooks, document controlled
- 11.11 RI liquid calibration logbooks, document controlled
- 11.12 Analytical bench sheet (example provided in Attachment 1)
- 11.13 RI liquid calibration conversion tables (Refer to the Libby-specific SOP SCR-LIBBY-03 (current version), Attachment 2)
- 11.14 Thermometer, NIST traceable
- 11.15 Permanently mounted test slides of anthophyllite (or other orthorhombic mineral), or the synthetic fiber polypropylene, for alignment of microscope's polars and crosshairs
- 11.16 Thin section of biotite for alignment of microscope's lower polar (recommended but not required)
- 11.17 Glass microscope slides and cover slips

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- 11.18 Slide trays
- 11.19 Sampling utensils (tweezers, dissecting needles, scalpels, probes, etc.) for sample manipulation
- 11.20 Clean, asbestos-free sample containers (ceramic evaporating dishes, foil weighing dishes, watchglasses, etc.)
- 11.21 Aluminum ashing tins
- 11.22 Water in spray bottles
- 11.23 Plastic re-sealable sample bags (4 mil poly bags)
- 11.24 Asbestos Containing Material (ACM) disposal bags
- 11.25 Crucible tongs
- 11.26 Autoclave gloves
- 11.27 Disposable examination gloves (latex or nitrile)
- 11.28 Lens paper and lens cleaning solution
- 11.29 Safety glasses (Z-87 rated)
- 11.30 Paper towels
- 11.31 Disposable lint-free wipes
- 11.32 Additional PPE required by the laboratory-specific HASP, CHP, or equivalent

12.0 CALIBRATION OF THE ANALYTICAL BALANCE

- 12.1 The analytical balance must be calibrated and certified by a third-party vendor on an annual basis.
- 12.2 Weights used for daily verification checks by laboratory personnel must be certified and traceable to national standards for weights and measures. These weights must be certified by a third-party vendor on a regular basis, at a minimum of once every five years.
 - 12.2.1 Labels should be placed on both the analytical balance and weight sets with the following information: date of the certification, initials of the individual performing the calibration and certification, and the date the next service is to be performed.

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- 12.3 The analytical balance must have a lower range accurate to 1mg (0.001g). The upper range is not specified; however, it is recommended that laboratories have a balance with an upper range of at least 100g or access to a second balance with a greater upper range.
- 12.3.1 If a sample exceeds the weight limit of the laboratory's analytical balance, the analyst will need to split the sample, weigh each split section separately, and then add the weights together (all weights must be recorded on the analytical bench sheet for QC purposes). If the weight of a single particle in a sample exceeds the weight limit of the balance, the laboratory must have access to a second balance with a greater upper range.
- 12.3.2 Although the coarse fraction is prepared by sieving with a ¼-inch screen, particles smaller than ¼-inch may be present in the fraction due to adherence between coarse and fine particles, or fine particles that adhere together during the drying process. This may include very fine asbestos fibers.
- 12.3.3 Because of the technical difficulty of isolating and weighing very small particles, the analyst should not attempt to physically segregate and weigh particles smaller than about 1mm.
- 12.4 Each day samples are analyzed by PLM-Grav, a verification check of the analytical balance must be performed, and the results of the check must be recorded in a document-controlled logbook.
- 12.4.1 Allow the analytical balance to warm-up for approximately 30 minutes before the check is performed.
- 12.4.2 Weights used for verification checks should be acclimated to the temperature in which the analytical balance resides. For example, if the balance is kept in a hood with air flow, the temperature inside the hood will be different than outside the hood, and if the weights are not kept in the hood with the balance they will not be the same temperature.
- 12.4.2.1 When objects are a different temperature than the surrounding air, air currents are created as the two temperatures come to equilibrium. These currents, however subtle, affect the pressure applied to the balance weigh pan, which in turn create drift in the reading of the object's weight.
- 12.4.2.2 When the temperatures between the object being weighed and the surrounding air are the same, the weight value displayed by the balance will be stable and not fluctuate.
- 12.4.2.3 This difference in temperatures applies to all objects being weighed, so the samples and weigh containers must also acclimate to the air temperature surrounding the analytical balance.
- 12.4.3 The analytical balance must be free of debris, especially on or underneath the balance weigh pan.
- 12.4.3.1 Always remove the balance pan when it needs to be cleaned,

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- since pressing down on the pan while it is on the scale can damage the sensitivity of the weighing mechanism.
- 12.4.4 Ensure that the balance is level by checking the bulls-eye bubble level. When the balance is not leveled, the scale may not check correctly and may produce inaccurate weight readings for samples.
- 12.4.5 A minimum of three weights must be checked, but it is recommended that four be used when weighing Libby samples; the actual value of the weights will vary depending on the upper weight limit of the analytical balance.
- 12.4.5.1 For a balance with an upper range of 60g, the four recommended weights are 1mg, 1g, 10g, and 50g (this covers both the lower range for LA fibers, the upper range of the balance, and the range of weights observed for coarse soil fractions).
- 12.4.5.2 Close all doors on the analytical balance, then tare (or zero) it. Once the balance displays '0.000' or '0.0000', place the lowest weight onto the balance pan and close the door.
- 12.4.5.3 When the display indicates the weight is stable, record the weight in the logbook.
- 12.4.5.4 A 1.0% tolerance range is the permitted deviation between the assigned value of a calibration weight and the value displayed by the balance. If the weight falls outside this range, it should still be recorded in the logbook, along with a comment describing the action taken to rectify the improper weight.
- 12.4.5.4.1 If the weight is outside tolerance limits, refer to the analytical balance user manual troubleshooting section for further information.
- 12.4.5.5 Once all weights read within tolerance ranges, the analytical balance is ready for use.
- 12.4.6 It is recommended that the analytical balance verification check logbook contain the following information:
- 12.4.6.1 Analytical balance manufacturer and model number
- 12.4.6.2 Type and class of calibration weights
- 12.4.6.3 Date of verification check
- 12.4.6.4 Initials of person performing the verification check
- 12.4.6.5 Certified weight value (e.g., 1.0000g)
- 12.4.6.6 Observed weight value from the balance (e.g., 0.9998g)
- 12.4.6.7 Pass/Fail information
- 12.4.6.8 Comments

13.0 CALIBRATION AND OPTIMIZATION OF THE PLM

Refer to the current version of Libby-specific SOP SRC-LIBBY-03, Section 12.0, *Calibration and Optimization of the PLM*, for information regarding equipment, standards, and the general maintenance and calibration of the microscopes.

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14.0 DETAILED METHOD FOR ASBESTOS TESTING OF COARSE SOIL AND SOIL-LIKE MATERIALS

14.1 Weighing the Sample

- 14.1.1 Once the verification check of the analytical balance is complete for the day, analysis of coarse Libby soils may begin.
- 14.1.2 Ensure that the weigh containers and samples have acclimated to the air temperature surrounding the analytical balance by leaving both the containers and samples near the balance for a minimum of 30 minutes.
- 14.1.3 Place an empty container onto the scale, close the doors, and wait for the weight to stabilize. Record the empty container weight onto the analytical bench sheet.
- 14.1.4 Remove the empty container from the analytical balance and close the doors. Slowly pour the soil sample into the container, ensuring that as much of the sample as possible is collected in the container.
 - 14.1.4.1 Never pour the soil into the container while it rests on the balance pan to avoid contaminating the analytical balance and to keep it as clean as possible.
 - 14.1.4.2 Some of the sample material may stick to the inside of the sample bag due to static electricity. Using tweezers, try to remove the larger pieces and place them into the weigh container.
- 14.1.5 Place the weigh container with the soil sample onto the balance, close the doors, and wait for the weight to stabilize. Record the container plus sample weight onto the analytical bench sheet.
- 14.1.6 If the analytical balance is not kept in the same hood as the stereomicroscope, the samples need to be safely transferred from one hood to the other, either by tightly covering the weigh container with the sample or by pouring the sample back into the original sample bag.
 - 14.1.6.1 Do not place the weigh container into the original inner or outer sample bags in order to avoid contaminating the outside of the weigh container, which will in turn unnecessarily contaminate the analysts' gloves, the stereomicroscope, and/or the prep hood.
 - 14.1.6.2 A paper towel (or a lint-free wipe) may be used to cover the weigh container. Secure it down with a rubber band or tape so it does not come off during transfer. Any method of transfer may be used which prevents contamination of the air and cross-contamination between samples.
 - 14.1.6.2.1 Materials used to transfer samples must either be cleaned with water and lint-free wipes between uses, or disposed of as asbestos-containing material (ACM).

14.2 Stereomicroscopic Examination

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- 14.2.1 The entire sample must be examined using the stereomicroscope.
 - 14.2.1.1 Look for stray bundles or fibers of asbestos, but also closely examine the coarse material for fibers that have electromagnetically stuck to their surfaces.
 - 14.2.1.2 Manipulate the sample to look at all sides of the coarse material and to look underneath or within mats of cellulose (if present).
 - 14.2.1.3 When clumps of fine soil are present, gently break them up in order to see inside the clump and look for fine asbestos fibers.
- 14.2.2 Observe the homogeneity, texture, and color of the sample. Record this information on the analytical bench sheet.
- 14.2.3 If no asbestos fibers are observed in the sample, record 'ND' (no asbestos observed) in the qualifier field on the analytical bench sheet.
- 14.2.4 Fibers suspected of being asbestos are observed must be confirmed as asbestos by PLM.
 - 14.2.4.1 Mount the suspected fiber in the appropriate RI liquid. For further information on PLM techniques and how to properly identify asbestos fibers, refer to the current version of Libby-specific SOP SRC-LIBBY-03, Sections 13.3, 13.5 and 13.6.
 - 14.2.4.2 For confirmed asbestos fibers, record the optical properties on the analytical bench sheet.
- 14.2.5 Once the fibers are confirmed as asbestos, the remaining fibers need to be separated from the rest of the sample and weighed.
 - 14.2.5.1 Fibers and fiber bundles $\leq 1\text{mm}$ may be too fine to separate from the sample material and/or too light to weigh and quantify. When this is the case, record the qualifier for that particular asbestos type as 'Tr' (trace amount of asbestos observed but not quantified), indicating that trace levels of asbestos were observed but not quantified.
 - 14.2.5.2 If fibers and fiber bundles are present at lengths $> 1\text{mm}$, separate them from the sample material.
 - 14.2.5.3 Place an empty weigh container on the balance pan, and once the weight is stabilized, record the weight on the analytical bench sheet.
 - 14.2.5.4 Remove the container from the balance pan. In the sample preparation hood, place all asbestos fibers (of the same type) into the container. If multiple types of asbestos are observed, they must be weighed separately and in separate containers.
 - 14.2.5.5 Cover the container with the asbestos fibers to ensure the air does not become contaminated (refer to Section 14.1.6).
 - 14.2.5.6 Place the container with the asbestos fibers onto the balance pan, and once the weight is stabilized, record the weight on the analytical bench sheet.
 - 14.2.5.7 To calculate the mass percent of asbestos for the sample,

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divide the weight of the asbestos fibers (weight of the container with fibers minus the weight of the empty container) by the total sample weight (weight of the container with the entire sample minus the weight of the empty container), then multiply by 100. Record this percent in the appropriate field on the analytical bench sheet.

- 14.2.5.8 If asbestos fibers are weighed and their mass percent is less than 0.1%, record the qualifier as 'Tr'.
- 14.2.6 Once fibers are identified as asbestos or non-asbestos, record the type and visual percent of non-asbestos fibers present within the sample.
- 14.2.7 Return the sample to its original sample bag for storage, and if the weigh container is disposable, treat it as ACM. If the container is not disposable, clean it with water and paper towels.
- 14.2.8 Clean any equipment and/or utensils that came in contact with the sample, including the analytical balance if necessary.

15.0 RECORDING DATA AND RESULTS

15.1 Analytical Bench Sheets

- 15.1.1 Analysts record, by hand, on analytical bench sheets, analytical results at the time the observations are made. Refer to Attachment 1 for an example of a PLM-Grav analytical bench sheet.
 - 15.1.1.1 Additional bench sheets may be created by the laboratory as long as all of the required fields are included.
- 15.1.2 Completed bench sheets are the original, hard-copy records on which test data on client samples is stored.

15.2 Stereomicroscopic Examination Reportables

- 15.2.1 Homogeneity (Yes or No)
- 15.2.2 Sample appearance, including color and texture
- 15.2.3 Type and estimated percent non-asbestos fibrous materials, such as fiberglass, cellulose, synthetic fibers, etc.
- 15.2.4 Non-fibrous matrix material(s), if known

15.3 Reporting Positive Asbestos Results

- 15.3.1 If asbestos is positively identified in the sample, record the following data for each asbestos type that is present in the sample.
- 15.3.2 Habit
- 15.3.3 Fiber color in plane light
- 15.3.4 Pleochroism (Yes or No)
- 15.3.5 Indices of refraction (α and γ)
- 15.3.6 Birefringence

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- 15.3.6.1 Low if birefringence is ≤ 0.010 ; medium if birefringence is 0.011 to 0.050; high if birefringence is > 0.050
- 15.3.6.2 Extinction characteristics (parallel or inclined)
- 15.3.6.3 Sign of elongation (positive or negative)
- 15.3.6.4 After PLM confirmation, weigh the asbestos and record the appropriate weights on the bench sheet.

15.4 Other Reportables

- 15.4.1 Record if there was any deviation from the SOP or the analytical method.
- 15.4.2 Record the QC type as Not QC, Laboratory Duplicate – Self-check (LDS), or Laboratory Duplicate – Cross-check (LDC).
- 15.4.3 Record any pertinent comments.
- 15.4.4 Sign or initial the bench sheet, and record the date of analysis.

16.0 DATA REPORTING

16.1 EDD Report Generation

- 16.1.1 Results of PLM analyses are provided to the client in an EDD in the form of an Excel spreadsheet.
 - 16.1.1.1 The LADT is a Laboratory Information Management System (LIMS) specifically designed to generate EDDs that meet all of the current client data reporting requirements, as well as minimize data entry errors. The EDD generated by the LADT is intended to replace the Libby EDDs used in previous years.
 - 16.1.1.3 It is the responsibility of the laboratory to check with the client that they are using the most recent version of the LADT.
 - 16.1.1.3 Laboratories can elect to generate their own EDDs rather than use the LADT; however, their EDDs must meet all of the current client data reporting requirements.
 - 16.1.1.2 Laboratories that do elect to use the LADT will receive the LADT User's Manual, which includes installation and data entry instructions.
- 16.1.2 After generating an EDD, save the file electronically.
 - 16.1.2.1 The EDD file name is generated automatically by the LADT.
 - 16.1.2.2 If a laboratory does not use the LADT to generate the EDD, they must use the following naming convention to name their EDD files:

Laboratory ID_Work Order Number_Analytical Method_Correction Number

Example: ESATR8_0920120002_PLM-Grav_C0

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- 16.1.3 The EDD serves as an electronic version of the test report submitted to the client.
 - 16.1.3.1 Only one EDD is produced for each chain of custody (COC) received by the laboratory.
 - 16.1.3.2 A hardcopy of the test report is also delivered to the client (see Section 16.2 for further details about hardcopy test reports).
 - 16.1.3.3 The laboratory retains all original records until otherwise instructed by the client.

16.2 Test Report Generation

- 16.2.1 Hardcopy test reports of the raw analytical data are submitted to the client for archival.
- 16.2.2 A completed test report consists of a cover sheet signed and dated by an approved signatory, as well as the following information and documentation:
 - 16.2.2.1 The laboratory work order number, COC number, number of samples received, and copies of the signed COCs.
 - 16.2.2.1.1 A work order number is a unique number assigned by the laboratory to a set of samples from a single COC. Work order numbers are never duplicated.
 - 16.2.2.2 The date of sample receipt and condition of samples.
 - 16.2.2.3 A Case Narrative, including any opinions and interpretations; deviations, modifications, additions to, or exclusions from the test method; descriptions of any problems encountered in the analysis; or any specific conditions that could affect the results. Also include the following disclaimer: "This test report relates only to items tested."
 - 16.2.2.4 PLM-Grav Analysis Results, as presented in the EDD and containing the analytical data (including all LDC and LDS analyses performed on any samples in the work order).
 - 16.2.2.9 Copies of the handwritten bench sheets containing the analyst's original data and observations.
- 16.2.3 Refer to the current version of Libby-specific SOP SRC-LIBBY-03, Attachment 3, for a complete list of items required for each test report.
- 16.2.4 When opinions and interpretations are provided in a test report, the laboratory will:
 - 16.2.4.1 Document the basis on which the opinions and interpretations were made.
 - 16.2.4.2 Clearly indicate on the test report which items are opinions and interpretations.
- 16.2.5 Once the test report is complete, all pages must be paginated prior to delivery to the client.

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16.3 Delivery of Results to Client

- 16.3.1 The following items will be submitted electronically (via e-mail) to the client:
 - 16.3.1.1 The completed EDD containing the analytical data. This spreadsheet is presented in a format that can be imported into the client's data management software.
 - 16.3.1.2 A scanned .pdf of the completed test report as described above. All signatures must be originals, or if electronic signatures are used, the e-signature must be controlled by a password-protected login that allows its application only by the signer.
 - 16.3.1.3 The two above files are e-mailed to the client, including all parties on the distribution list submitted by the client to the laboratory.
- 16.3.2 Once the results of a work order number have been delivered to the client, the hardcopy test report is retained until further instruction by the client.

17.0 QUALITY ASSURANCE AND QUALITY CONTROL

17.1 General

- 17.1.1 The laboratory must operate under a quality system appropriate to the type, range, and volume of testing work that it performs.
- 17.1.2 Results of QC analyses are used to track the precision and accuracy of the laboratory's analyses and to identify areas that require or could benefit from improvement.
- 17.1.3 The following types of QC analyses are performed on a scheduled basis at the laboratory:
 - 17.1.3.1 Re-analysis of client samples by the same analyst (LDS) or by a different analyst (LDC)
 - 17.1.3.2 Routine analyses on calibration standards of known asbestos concentration
 - 17.1.3.3 NIST proficiency testing
 - 17.1.3.4 Inter-laboratory analyses (also referred to as Round Robin analyses)
- 17.1.4 Records must be kept of all QA documentation.
- 17.1.5 All QC analyses must be performed in real-time.

17.2 LDS and LDC QC Analyses (Duplicates and Replicates)

- 17.2.1 For all Libby samples received by the laboratory, a minimum of 10% must be re-analyzed within the laboratory.
- 17.2.2 A QC analysis (LDS or LDC) can be performed on any sample.
 - 17.2.2.1 QC analyses need to be performed on samples over the entire range of asbestos concentrations that are

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- encountered in site samples.
- 17.2.2.2 Any sample that is considered especially unusual or difficult should be re-analyzed for QC purposes.
- 17.2.3 The frequency of LDS analyses on client samples will be 2 per 100 samples analyzed (2%). LDS analyses should be performed as a re-analysis of the original sample by re-weighing and re-examining the entire sample. All sample weights (empty container, container with sample, or asbestos fibers) must be recalculated and recorded on the analytical bench sheet by the original analyst.
- 17.2.4 The frequency of LDC analyses on client samples will be 8 per 100 samples analyzed (8%). The original sample will be re-weighed and re-examined by an analyst other than the original. All sample weights (empty container, container with sample, or asbestos fibers) must be recalculated and recorded on the analytical bench sheet by the LDC analyst.
- 17.2.4.1 All analysts performing QC analyses must be experienced with PLM analysis of soil samples from the Libby Site and the specific requirements of this SOP.
- 17.2.4.2 If there is only one primary analyst at the laboratory performing PLM analysis on these samples, the laboratory must send all LDC samples to another Libby laboratory with the proper experience and qualifications.
- 17.2.5 For samples containing asbestos, LDS and LDC analyses are considered acceptable if results for both the original and QC analyses are $\leq 1\%$. For samples containing $>1\%$ LA, laboratories should defer to their own internal QA/QC system (such as control charting or similar tool) to determine QC acceptance criteria.
- 17.2.6 Corrective action(s) must be taken immediately if any QC analyses do not meet acceptance criteria. Examples of corrective actions that may be taken are re-analysis of the sample, analyst re-training, and/or notification of the client.
- 17.2.7 When performing a QC analysis, it is necessary to mark LDS or LDC in the "QC Type" section of the bench sheet.
- 17.3 Inter-Laboratory Analyses
- 17.3.1 The laboratory is involved in an ongoing sample exchange program with other PLM laboratories that analyze soil samples from the Libby Site. The purpose of this program is to help detect and minimize laboratory biases and unnecessary variance in results, as well as to characterize precision across laboratories performing PLM-Grav testing.
- 17.3.2 The frequency of the inter-laboratory sample exchange ranges from 1 in 100 samples exchanged amongst laboratories on a quarterly basis. However, higher frequencies of inter-laboratory sample analysis are required when a laboratory is new to the program, when systematic errors or biases are observed, or when a new version of the SOP is distributed. Whether or not the frequency to be performed is the

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- minimum or higher is determined by the client.
- 17.3.3 Results of the inter-laboratory analyses are reviewed by the client.
 - 17.3.4 The inter-laboratory analysis is acceptable if results for both the original and inter-laboratory analyses are $\leq 1\%$. If both the original and inter-laboratory result is $>1\%$ LA, acceptance of the inter-laboratory analysis will be determined by the client.
 - 17.3.5 Corrective action(s) must be taken immediately if analyses do not meet acceptance criteria. The specific course of action based on these results will be determined by the client. Common actions include re-analysis of the samples, collaboration between and amongst laboratories performing the test to root out biases and/or variance, and analyst re-training.

18.0 REFERENCES

- 18.1 Camp, Dresser and McKee (CDM). April 2002. Sampling and Analysis Plan, Remedial Investigation, Contaminant Screening Study, Libby Asbestos Site, Operable Unit 4. 3282-116-PP-SAMP-14187. Denver, Colorado.
- 18.2 National Institute of Occupational Safety and Health. 1994. Method 9002, *Asbestos (bulk) by PLM*, Issue 2.
- 18.3 United States Environmental Protection Agency. 1993. *Method for Determination of Asbestos in Bulk Building Materials*. Method 600/R-93/116.

LIBBY ASBESTOS SUPERFUND SITE STANDARD OPERATING PROCEDURE
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ATTACHMENT 1

Libby Asbestos Superfund Site Analysis Bench Sheet (PLM-Grav)

LIBBY ASBESTOS SUPERFUND SITE ANALYSIS BENCH SHEET (PLM-Grav)

Laboratory Name: _____
 Work Order Number: _____
 Date Received: _____
 SOP Name/Revision: _____

Client Sample Number					
Tag					
Lab Sample ID					
Date Analyzed					
Analyst Initials					

TOTAL SAMPLE WEIGHT (g):

Wt of Empty Container					
Wt of Sample + Container					

STEREOMICROSCOPIC EXAMINATION:

Homogeneity (Y/N)					
Sample Color					
Sample Type/Texture					
Type and % of Non-Asbestos Fibers					
Non-Fibrous Matrix Materials (if known)					

MASS OF ASBESTOS PARTICLES (g):

LA Qual (ND, Tr)					
Wt of Empty Container					
Wt of LA + Container					
OA Qual (ND, Tr)					
OA Type					
Wt of Empty Container					
Wt of OA + Container					
CH Qual (ND, Tr)					
Wt of Empty Container					
Wt of CH + Container					

ASBESTOS OPTICAL PROPERTIES BY PLM:

Habit					
Fiber Color					
Sign of Elongation (+/-)					
Pleochroism (Y/N)					
Extinction Angle					
Refractive Index (α)					
Refractive Index (γ)					
Birefringence					
Becke Line or CSDS					
Temperature (°C)					

OTHER:

QC Type					
Deviation (Y/N)					
Comments					

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ANALYSIS OF ASBESTOS FIBERS IN FINE SOIL BY POLARIZED LIGHT MICROSCOPY

Date: July 27, 2012

SOP No.: SRC-LIBBY-03 (Revision 3)

ANALYSIS OF ASBESTOS FIBERS IN FINE SOIL
 BY POLARIZED LIGHT MICROSCOPY

SYNOPSIS: This is a semi-quantitative method for identifying and quantifying asbestos fibers in soil using polarized light microscopy. This method is based on NIOSH Method 9002, EPA Method 600/R-93/116, and CARB Method 435, with project specific modifications intended for application at the Libby Asbestos Superfund Site. Sampling and plan developers and data users are cautioned to understand how data are generated from this SOP.

APPROVALS:

USEPA Region 8

Dania Zinner
 Signature

7/30/12
 Date

Dania Zinner
 Print Name

RPM
 Title

ESAT Region 8

Mark McDaniel
 Signature

7/27/12
 Date

Mark McDaniel
 Print Name

ESAT Manager
 Title

Revision	Date	Principal Changes and Author
0	03/03/2003	Initial Author: William Brattin (Syracuse Research Corporation)
1	12/11/2003	Clarified binning assignment of samples at 0.2%. Author: William Brattin (Syracuse Research Corporation)
2	10/10/2008	Complete re-design of the SOP. Provided specific requirements for analytical sample preparation and analytical process. Authors: Douglas Kent and Nikki MacDonald, ESAT Region 8
3	07/27/2012	Entire SOP review and update provided by ESAT Region 8 with additional comments provided by QATS.

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1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide a standard approach for semi-quantitative analysis of asbestos in samples of soil or other soil-like materials using the visual area estimation technique by Polarized Light Microscopy (PLM). This SOP is specifically intended for application at the Libby Asbestos Superfund Site (referred to as the Libby Site from this point forward) and has been refined to focus testing on Libby Amphibole (LA) asbestos at levels below 1%.

2.0 SCOPE AND APPLICATION

This method is intended for analysis of asbestos in soil or other similar soil-like media in which the soil has been taken through the preparation process described in Section 4.0. This method is appropriate for the analysis of all types of asbestos fibers (chrysotile and amphiboles), including LA. For the purposes of this SOP, the term 'asbestos' will refer to the six regulated asbestos minerals (chrysotile, amosite, crocidolite, anthophyllite, tremolite, and actinolite), as well as LA.

3.0 RESPONSIBILITIES

- 3.1 It is the responsibility of the laboratory supervisor to ensure that all analyses and quality control (QC) procedures are performed in accordance with this SOP and to identify and take appropriate corrective action to address any deviations that may occur during sample preparation or analysis.
- 3.2 The Laboratory Manager, Quality Assurance Coordinator (or equivalent), and/or Analytical Lead will communicate with the client, any situations where a modification to or deviation from the SOP may be useful and/or required. The laboratory supervisor must receive approval from the client for any modification to or deviation from the SOP before incorporating any such modification or deviation into the sample preparation and analysis process (refer also to Section 8.2).
- 3.3 It is the responsibility of the laboratory to maintain a PLM SOP for Bulk Asbestos Materials, Quality Assurance Manual (QAM), or an equivalent document(s) that meets all the requirements of the National Voluntary Laboratory Accreditation Program (NVLAP) Handbook 150 and Handbook 150-3. It is also the responsibility of the laboratory to ensure its testing activities stay in compliance with the requirements of NVLAP Handbooks 150 and 150-3 and the regulatory and accrediting agencies that provide oversight of the laboratory's operations and all Libby Site project-specific requirements.

4.0 METHOD DESCRIPTION

- 4.1 The test method describes a semi-quantitative analysis of asbestos in samples of soil or other soil-like materials using the visual area estimation technique by PLM, referred to as PLM-VE. The test method used for analyzing PLM asbestos samples specific to the Libby Site is based on the National Institute of Occupational Safety and Health (NIOSH) Method 9002, United States Environmental Protection Agency (EPA) Method 600/R-93/116, and the State of California Air Resources Board (CARB) Method 435, with project-specific modifications provided in this SOP.

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- 4.2 Soil samples for the Libby project are processed according to the current version of SOP ISSI-LIBBY-01, *Soil Sample Preparation*, before submittal to the laboratory for analysis. This process separates the coarse fraction of the soil from the fine fraction. The fine fraction constitutes all material passing through a ¼-inch sieve. The fine fraction is homogenized and ground to a maximum particle size of approximately 250 microns (µm). This fine fraction is further sub-divided into four fractions using a riffle splitter. One or more of these fractions is then submitted to an approved and accredited PLM laboratory for analysis. This SOP is specific to the analysis of the fine fraction soil samples. Coarse fraction soil samples are analyzed according to the current version of SOP SRC-LIBBY-01, *Qualitative Estimation of Asbestos in Coarse Soil by Visual Examination Using Stereomicroscopy and Polarized Light Microscopy*.
- 4.3 The fine fraction soil sample to be evaluated for asbestos content is first examined using a low magnification stereomicroscope. Microscope slide mounts of the sample are then prepared by immersing sample material in a liquid medium of known refractive index (RI). These slide mounts are then analyzed visually by PLM. Asbestos and non-asbestos phases are identified on the basis of their morphology and optical properties. Quantification of the amount of asbestos present is done using a visual estimation approach. The concentration of LA in the sample is a percent visual estimation based on the use of project-specific mass percent reference materials, as well as any laboratory-specific visual estimation reference materials.
- 4.4 All samples from the Libby Site are identified by either one or two-characters followed by a hyphen and a five digit number (referred to as the Client Sample Number). The first characters identify the type of sample as indicated by the site-specific Summary Analytical Procedure (SAP). The five digit number is assigned by the field sampling teams. All samples from the Libby Site also have an associated tag to further identify the sample (e.g., a tag of FG2 is the second fine ground soil split for a given parent sample). At all stages of documentation, both the sample number and tag must be used to properly identify the sample.

5.0 ACRONYMS

ACM	Asbestos Containing Material
CARB	State of California Air Resources Board
CHP	Chemical Hygiene Plan
COC	Chain of Custody
EDD	Electronic Data Deliverable
EPA	United States Environmental Protection Agency
E-W	East-West
HASP	Health and Safety Plan
HEPA	High Efficiency Particulate Air
LA	Libby Amphibole
LADT	Libby Asbestos Data Tool
LDC	Laboratory Duplicate – Cross-check
LDS	Laboratory Duplicate – Self-check
LIMS	Laboratory Information Management System
MSDS	Material Safety Data Sheet

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NIOSH	National Institute for Occupational Safety and Health
NIST	National Institute of Standards and Technology
N-S	North-South
NVLAP	National Voluntary Laboratory Accreditation Program
PE	Performance Evaluation
PLM	Polarized Light Microscopy
PLM-VE	Visual Area Estimation technique by Polarized Light Microscopy
PPE	Personal Protective Equipment
QA	Quality Assurance
QAM	Quality Assurance Manual
QC	Quality Control
RI	Refractive Index
SAP	Summary Analytical Procedure
SEM	Scanning Electron Microscopy
SOP	Standard Operating Procedure
SRM	Standard Reference Material
TEM	Transmission Electron Microscopy
USGS	United States Geological Survey

6.0 HEALTH AND SAFETY

- 6.1 Follow general laboratory health and safety policies and regulations in the laboratory's Health and Safety Plan (HASP), Chemical Hygiene Plan (CHP), or equivalent.
- 6.2 All sample handling and preparation activities must be performed in a ventilated hood with an operating High Efficiency Particulate Air (HEPA) filtration system, a class 1 biohazard hood, or glove box with continuous airflow (negative pressure). Never have a sample container open except when the sample is inside of the sample preparation hood. Appropriate personal protective equipment (PPE) should be worn at all times.
- 6.3 Avoid repeated or prolonged contact with the RI liquids and inhalation of fumes from the RI liquids. Refer to the Material Safety Data Sheet (MSDS) forms for RI liquids for additional information and cautions.

7.0 CAUTIONS

- 7.1 The toxicity or carcinogenicity of the RI liquids used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be avoided.
- 7.2 After processing each sample, use water and paper towels to thoroughly decontaminate all work surfaces and utensils that came into contact with a sample and/or RI liquid. Never have more than one sample container open at any one time.

8.0 GENERAL LABORATORY PRACTICES

- 8.1 Quality Assurance

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- 8.1.1 Each laboratory operates under a quality assurance (QA) program appropriate to the type, range, and volume of work it performs.
- 8.1.2 It is the responsibility of the laboratory to maintain a QAM, or equivalent, in which the laboratory's QA program is detailed. Additional QA/QC requirements specific to the PLM laboratory and the Libby Site are described in Section 16.0.
- 8.1.3 All work is performed at a permanent laboratory location. Even if a laboratory is part of a larger organization, it is able to carry out all testing, calibration, and daily QA/QC activities independently, and at one location. There are no remote or sub-facilities where testing work is performed.

8.2 Documenting SOP Modifications

- 8.2.1 Any deviation from the SOP must be documented in a laboratory modification form and then addressed in the technical Case Narrative prepared as part of the test report.
- 8.2.2 Additionally, when there is reason to suspect a departure from the SOP has affected the result or validity of data provided to the client, the client must be notified of the nature of the departure from the SOP and informed about the possible effect on the result or validity of the analysis. The course of action taken to keep the departure from recurring must also be discussed with the client.

9.0 PERSONNEL QUALIFICATIONS

- 9.1 The use of this SOP is limited to microscopists knowledgeable in the production and evaluation of asbestos data.
 - 9.1.1 All personnel analyzing samples from the Libby Site are expected to be familiar with routine chemical laboratory procedures, principles of optical mineralogy, and proficient in EPA Method 600/R-93/116, NIOSH Method 9002, and CARB Method 435.
 - 9.1.2 Personnel at laboratories with less than one year of experience specific to the Libby Site are required to participate in the laboratory mentoring program to obtain additional guidance and instruction. This training is provided by personnel familiar with the particular problems and types of asbestos encountered at the Libby Site.
- 9.2 Before performing any analyses, each analyst must demonstrate the ability to generate acceptable accuracy and precision with this method. This includes successfully completing NVLAP proficiency testing.

10.0 EQUIPMENT

- 10.1 Each laboratory must be equipped with all instrumentation, hardware, software, and reference materials required for the correct performance of calibrations and tests.
- 10.2 All equipment must be properly maintained and calibrated (as appropriate) prior to use. See Section 12.0 for further details regarding microscope calibration.

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- 10.3 The following is a general list of equipment available at the PLM laboratory to perform this SOP:
- 10.3.1 Polarized Light Microscope, with:
 - 10.3.1.1 Light source and replacement bulbs
 - 10.3.1.2 Binocular observation tube
 - 10.3.1.3 Blue daylight filter
 - 10.3.1.4 Oculars (10X)
 - 10.3.1.5 Objectives: 10X, 20X, and 40X (or similar magnification)
 - 10.3.1.6 10X Dispersion Staining Objective
 - 10.3.1.7 360 degree rotatable and centerable stage
 - 10.3.1.8 Polarizer and analyzer aligned at 90 degrees to one another
 - 10.3.1.9 Bertrand lens (optional)
 - 10.3.1.10 Substage condenser with iris diaphragm
 - 10.3.1.11 Accessory slot for compensator plate
 - 10.3.1.12 First order red (550 nanometer) compensator plate
 - 10.3.1.13 Crosshair reticle
 - 10.3.1.14 Adjustment tools
 - 10.3.2 HEPA-filtered hood, class 1 biohazard hood, or glove box with continuous airflow (negative pressure)
 - 10.3.3 Binocular stereomicroscope, 10-50X magnification (approximate)
 - 10.3.4 Light source for stereomicroscope
 - 10.3.5 Muffle furnace
 - 10.3.6 Analytical balance, accurate to 1mg (0.001g)
 - 10.3.7 Libby Asbestos Data Tool (LADT) or other computer software capable of generating a project-specific Electronic Data Deliverable (EDD) that meets the current client data reporting requirements
 - 10.3.8 Mortar and pestle (agate or porcelain)
 - 10.3.9 Vaneometer
 - 10.3.10 Wet/dry vacuum with HEPA filtration
 - 10.3.11 Decontamination equipment (e.g. disposable lint-free wipes, wet mop with bucket, etc.)

11.0 STANDARDS, REAGENTS AND SUPPLIES

- 11.1 High Dispersion RI Liquid(s) from 1.620 to 1.640
- 11.2 1.550 High Dispersion RI Liquid
- 11.3 1.680 to 1.700 RI Liquid(s)
- 11.4 Solid RI Standards (precision optical glass, RI from 1.48 to 1.72, in gradations of 0.01, 25 standards)
- 11.5 National Institute of Standards and Technology (NIST) Standard Reference Material (SRM) 1866b - Common Commercial Asbestos consisting of chrysotile, amosite, and Crocidolite

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- 11.6 NIST SRM 1867a - Uncommon Commercial Asbestos consisting of tremolite, actinolite, and anthophyllite
- 11.7 Controlled Performance Evaluation (PE) Reference Materials (prepared for EPA by United States Geological Survey [USGS])
 - 11.7.1 Soils containing LA in various known concentrations (provided by the client)
 - 11.7.2 Permanently mounted slides containing 0.2% LA by mass
 - 11.7.3 Permanently mounted slides containing 1.0% LA by mass
- 11.8 Controlled Libby Amphibole Asbestos (prepared for EPA by USGS), a finely-milled composite of a selected subset of 30 samples taken from the mine at the Libby Site
- 11.9 NIST Bulk Asbestos Proficiency Testing Round M12001, Sample 4, a sample of un-milled rock-form winchite/richterite taken from the mine at the Libby Site
- 11.10 Non-asbestos reference materials (gypsum, calcite, fiberglass, etc.)
- 11.11 Instrument maintenance/calibration logbooks, document controlled
- 11.12 RI liquid calibration logbook, document controlled
- 11.13 Analytical bench sheets (example provided in Attachment 1)
- 11.14 RI liquid calibration conversion tables (Attachment 2)
- 11.15 Thermometer, NIST traceable
- 11.16 Permanently mounted test slides of anthophyllite (or other orthorhombic mineral), or the synthetic fiber polypropylene, for alignment of microscope's polars and crosshairs
- 11.17 Thin section of biotite for alignment of microscope's lower polar (recommended but not required)
- 11.18 Calibration standards (see Sections 16.2 and 16.3)
- 11.19 Glass microscope slides and cover slips
- 11.20 Slide trays
- 11.21 Sampling utensils (tweezers, dissecting needles, scalpels, probes, etc.) for sample manipulation
- 11.22 Clean, asbestos-free sample containers (ceramic evaporating dishes, foil weighing dishes, watchglasses, etc.)
- 11.23 Aluminum ashing tins

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- 11.24 Water in spray bottles
- 11.25 Plastic re-sealable sample bags (4 mil poly bags)
- 11.26 Asbestos Containing Material (ACM) disposal bags
- 11.27 Crucible tongs
- 11.28 Autoclave gloves
- 11.29 Disposable examination gloves (latex or nitrile)
- 11.30 Lens paper and lens cleaning solution
- 11.31 Safety glasses (Z-87 rated)
- 11.32 Paper towels
- 11.33 Disposable lint-free wipes
- 11.34 Additional PPE required by the laboratory-specific HASP, CHP, or equivalent

12.0 CALIBRATION AND OPTIMIZATION OF THE PLM

12.1 Equipment and Standards

- 12.1.1 All measuring and testing equipment having an effect on the accuracy and/or validity of analytical testing must be calibrated at frequencies described for the individual components below.
- 12.1.2 “Standards” refers to any material used in calibration of a piece of equipment or analytical methodology.
 - 12.1.2.1 Standards used at the lab include slides used for alignment of a microscope’s polars, optical glass for calibration of RI liquids, NIST SRMs of the various asbestos minerals, Controlled PE Reference Materials of LA in soils, and samples from past NIST proficiency rounds.
 - 12.1.2.2 The laboratory uses NIST-traceable standards whenever possible, or other standards that have been calibrated by a respected organization. When internal standards are used, they are checked as extensively as technically and economically feasible.
 - 12.1.2.3 The laboratory stores its standards in such a way to avoid contamination of the standards and to protect their integrity.
 - 12.1.2.4 Any standard that is damaged, compromised, or judged to be unreliable must be recalled from service.
 - 12.1.2.5 Reference standards of measurement (e.g., optical glass for RI liquid calibration, slides for aligning the microscopes, and LA reference materials) are used for calibration purposes and for no other purpose.
- 12.1.3 Visual estimates of asbestos concentrations other than LA, as well as LA

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concentrations >1%, are calibrated using permanently mounted working slides of known asbestos concentration prepared by the laboratory. The use of these standards is described in Section 16.0.

- 12.1.4 Visual estimations of LA concentrations $\leq 1\%$ are calibrated using the Controlled PE Reference Materials.

12.2 General Maintenance and Calibration of the Polarized Light Microscope

- 12.2.1 Extinction angle is an optical property used to identify asbestos and non-asbestos minerals. In order to accurately determine a mineral's extinction angle, the microscope's upper and lower polars must be aligned north-south (N-S) and east-west (E-W), resulting in a 90 degree orientation to each other.
- 12.2.2 When aligned properly, the field of view in crossed polars will appear as dark as possible.
- 12.2.3 The microscope's optics must be kept clean and properly aligned so optimal image quality can be produced.
- 12.2.4 Check the microscope's alignment each working day prior to use. The microscope must be re-aligned any time it is found to be out of alignment.
- 12.2.5 An individual instrument maintenance logbook must be kept for each microscope in use at the laboratory.
- 12.2.5.1 Each day the microscope is used, the analyst must record an entry into this logbook. Record the date and analyst's initials confirming that all microscope alignment checks were made prior to analysis.
- 12.2.5.2 All maintenance activities performed on the microscope must be recorded into this logbook.

12.3 Checking Microscope Alignment

- 12.3.1 Place a permanently-mounted slide that contains large straight fibers of anthophyllite or polypropylene onto the microscope stage.
- 12.3.1.1 While looking at an empty portion of the slide under crossed polars, make sure the field of view in the microscope is as dark as possible (black, not dark gray).
- 12.3.1.2 When the field of view is black under crossed polars, the polars are oriented at 90 degrees to each other.
- 12.3.2 The fibers should be completely extinct in both the N-S and E-W directions under crossed polars, indicating proper polar alignment.
- 12.3.2.1 Once the fibers become completely extinct in either the N-S or E-W direction, pull out the analyzer to make sure they are still parallel to the crosshairs.
- 12.3.3 The stage and objectives must be centered so that a fiber centered in the field of view remains centered when the microscope stage is rotated.
- 12.3.4 The light path through the scope must be centered (see Section 12.5 for centering the optic axis).
- 12.3.5 The crosshairs must be properly oriented E-W and N-S.
- 12.3.6 If any of the above conditions are not met, it is necessary to re-calibrate the microscope.

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12.4 Centering the Stage and Objectives

- 12.4.1 Because centering of the highest magnification objective (40X or 50X) is the most critical, center the microscope stage to this objective.
 - 12.4.1.1 Adjust the centering screws on the stage so that a particle remains centered in the field of view when using the highest magnification objective as the stage is rotated.
 - 12.4.1.2 The remaining objective lenses must be centered so they coincide with the axis of rotation of the stage.
 - 12.4.1.3 Adjust the centering of the remaining objectives using the centering screws for each objective.

12.5 Centering the Optic Axis

- 12.5.1 Looking at the field of view in plane light under low magnification, insert the sub-stage condenser lens and then tighten the field iris diaphragm (not the condenser iris diaphragm) until it begins to eclipse the outer edge of the field of view.
- 12.5.2 Use the centering screws to center the image of the outer edge of the field diaphragm so it coincides with the edge of the field of view.
- 12.5.3 Tighten the field iris diaphragm until it is almost closed. With the 10X objective, only a small circle of light should be visible somewhere close to center of the field of view.
 - 12.5.3.1 Raise or lower the microscope substage until the edge of the image of the field diaphragm comes into as sharp a focus as possible.
- 12.5.4 Move the substage with the condenser and its iris diaphragm using its adjusting screws until the small circle of light is centered in the field of view.
- 12.5.5 Open the field iris diaphragm until it is just barely wide enough that the entire field of view is illuminated.
- 12.5.6 Remove the sub-stage condenser lens.

12.6 Using the Condenser Iris Diaphragm

- 12.6.1 When viewing a microscope slide under plane light, adjust the iris diaphragm on the sub-stage condenser (not the field iris diaphragm) to improve contrast and the viewing of subtle shades and textures.
 - 12.6.1.1 The iris diaphragm is not used for controlling brightness; the light source is used to control light and brightness.

12.7 Alignment of Lower Polar

- 12.7.1 Place the thin section containing large crystals of biotite on the microscope stage and examine it in plane light. This procedure allows for rapid and accurate alignment of the lower polar. Laboratories may use a different procedure to align the lower polar as long as it is documented in their internal SOPs.
- 12.7.2 Find a biotite crystal on the slide that exhibits a strong cleavage trace.
 - 12.7.2.1 The cleavage planes in the biotite crystal between the mica sheets should be as close to perpendicular with the plane of the slide as

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- possible.
 - 12.7.2.2 Crystals that show the strongest cleavage traces should have their cleavage plane at a high angle to the plane of the slide and will show the most distinctive pleochroism.
 - 12.7.2.3 After selecting a biotite crystal, orient the slide so that the cleavage traces of the biotite crystal are directly E-W.
 - 12.7.2.4 Observe the crystal's pleochroism as the stage is rotated.
 - 12.7.2.5 While viewing the crystal in plane light, slowly rotate the lower polar clockwise or counter-clockwise until the biotite crystal is as dark as it will become.
 - 12.7.2.6 When the cleavage traces of the biotite crystal are oriented directly E-W and the pleochroism of the crystal is as dark as possible, the lower polar is properly oriented E-W.
 - 12.7.3 Rotate the ocular that contains the crosshair reticle until the crosshairs are oriented directly N-S and E-W.
- 12.8 Alignment of Upper Polar
- 12.8.1 Once the lower polar has been properly aligned E-W, place a permanently-mounted test slide containing large straight fibers of anthophyllite or polypropylene on the stage.
 - 12.8.2 While looking at a portion of the slide relatively free of birefringent material, slowly rotate the upper polar until the field of view, under crossed polars, reaches maximum darkness. The field of view should be black, not dark gray.
 - 12.8.3 Rotate the stage and observe the extinction of the fibers.
 - 12.8.3.1 If the field of view is as dark as possible and the fibers become extinct in the N-S and E-W directions, the polars are properly aligned.
 - 12.8.3.2 Once the fibers become completely extinct in either the N-S or E-W direction, pull out the analyzer to make sure the fibers are still parallel to the crosshairs.
 - 12.8.3.3 If the polars are still not properly aligned, then repeat steps 12.7.1 through 12.8.3 until the microscope's polars are properly aligned.
- 12.9 Cleaning the Polarized Light Microscope
- 12.9.1 The oculars, objective lenses, and condenser should be cleaned whenever they become soiled with dust, oil, RI liquids, etc. At minimum, they should be cleaned monthly.
 - 12.9.2 Always use lens cleaning solution and lens paper to clean the lenses.
 - 12.9.2.1 Do not use a dry cloth because this can scratch the surface of the lens.
 - 12.9.2.2 Avoid applying excessive pressure to the lens surface when cleaning as this could also scratch the lens.
 - 12.9.2.3 Never use any solvents (such as alcohol, etc.) other than lens cleaning solution because this can dissolve the cement that holds the lenses together and/or etch the glass surface of the lens.
 - 12.9.3 If dust gets inside the microscope, it is necessary to completely disassemble and clean the microscope.

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- 12.9.3.1 The microscope must be re-calibrated after being re-assembled and this must be recorded in the microscope's maintenance logbook.
- 12.9.3.2 Disassembly of the microscope should only be performed by qualified personnel.

13.0 DETAILED METHOD FOR ASBESTOS TESTING OF SOIL AND SOIL-LIKE MATERIALS

13.1 Initial Stereomicroscopic Examination

- 13.1.1 All sample preparation activities, including stereomicroscopic examination, slide mounts, etc., must be performed in a HEPA-filtered hood, class 1 biohazard hood, or glove box with continuous airflow (negative pressure).
- 13.1.2 Due to the sample preparation requirements described in the current revision of SOP ISSI-LIBBY-01, *Soil Sample Preparation*, samples should never be wet. If the sample is wet, contact the client.
- 13.1.3 The stereomicroscope is a low magnification microscope (approximately 10X-50X) used for visual examination of specimens at a coarse scale. Stereomicroscopic examination is especially useful for soil samples where fibers may be unevenly or thinly distributed throughout the sample.
- 13.1.4 Begin the analysis by pouring the entire sample out of its sample bag onto a clean, asbestos-free substrate, such as an agate mortar, ceramic evaporating dish, watchglass, weighing dish, etc.
 - 13.1.4.1 For fine-ground soil samples, the mass of the sample will ideally be 20 to 50 grams; however, some samples submitted to the laboratory may be smaller or larger.
- 13.1.5 With the stereomicroscope, visually examine the entire sample for homogeneity, sample color and texture, and the presence of any suspect fibers.
- 13.1.6 If individual fibers suspected of being asbestos are observed during this initial examination, pick out one or more of these fibers with fine forceps (or other appropriate utensil) and mount them on a glass microscope slide in an appropriate RI liquid. These sample preparations are referred to as fiber-picks in this SOP.
 - 13.1.6.1 Each microscope slide must be wiped with disposable lint-free wipes prior to use to avoid contamination.
 - 13.1.6.2 Mount individual fibers in 1.550 RI liquid if chrysotile is suspected, 1.620 to 1.640 RI liquid if LA or anthophyllite is suspected, or 1.680 to 1.700 RI liquid if amosite or crocidolite is suspected.
 - 13.1.6.3 Only one drop of RI liquid is necessary to prepare a fiber-pick slide.
 - 13.1.6.4 Cover this preparation with a glass cover slip and identify the fibers using PLM analysis techniques (see Section 13.5).
- 13.1.7 Record all stereomicroscopic findings, including homogeneity, sample appearance (color and texture), an initial estimated percent LA, and an initial estimated percent other asbestos (chrysotile and other amphibole), in the appropriate fields on the analytical bench sheet.
 - 13.1.7.1 Stereomicroscopic examination does not provide positive identification of asbestos fibers. Later analysis by PLM will confirm, deny, or refine the preliminary estimated percent and type of asbestos.
 - 13.1.7.2 The procedure for performing a calibrated visual estimate using both

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stereomicroscopy and PLM is described in Section 13.7.4 and Attachment 8.

- 13.1.8 Regardless of whether or not a fiber-pick was performed during this initial stereomicroscopic examination, each sample must be prepared for PLM analysis following the procedures described in Sections 13.3 and 13.4, below.

13.2 Determination of Ashing the Sample

- 13.2.1 Soil samples containing a significant amount of twigs, leaves, tar, or other debris may need to be ashed prior to being prepared for random mounts for PLM.

13.2.1.1 Excessive cellulose fibers, tar or asphalt may obscure asbestos fibers, and ashing will assist in eliminating this interference.

- 13.2.2 Ashing consists of placing a representative portion of the whole sample into the muffle furnace to burn off organics that obscure asbestos fibers or keep the sample from breaking up on the slide during mounting. Approximately 480°C is hot enough to burn off organics without destroying the crystallinity of asbestos fibers. Do not ash the entire sample because a re-analysis of the sample may be required at a later date.

- 13.2.3 The ashed residue can then be examined under the stereomicroscope following the procedures in Section 13.1, above, and slide mounts can be prepared from the ashed residue for PLM analysis, according to the procedures in Section 13.3, below.

- 13.2.4 Following PLM analysis, calculate the percentage of asbestos in the pre-ash sample using the equation below:

$$\text{Pre-ash percent asbestos} = (\text{percent asbestos in ashed residue}) * (C-A)/(B-A)$$

Where:

A = weight of ashing tin in grams

B = weight of sample + ashing tin in grams (pre-ash)

C = weight of sample + ashing tin in grams (post-ash)

- 13.2.5 Record the required gravimetric measurements and calculations listed above in Section 13.2.4 on the analytical bench sheet in the comments field. Alternatively, attach a separate analytical bench sheet (specific to ashing samples) with the necessary measurements, and indicate the attachment in the comments section of the PLM-VE bench sheet.

13.3 Preparation of Samples for PLM-VE

- 13.3.1 Quantitative analysis preparation typically consists of preparing random mounts of a sample. The objective is to produce random sub-sample mounts representative of the original sample.

- 13.3.2 For each sample, a minimum of five slide mounts must be prepared for PLM analysis (not including any fiber-picks). These slide mounts are prepared from randomly selected sub-samples taken from the original sample, which are then immersed in a RI liquid in the range of 1.620 to 1.640 for easier measurement of

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LA optical properties.

13.3.2.1 After performing the initial stereomicroscopic examination (according to the procedures described in Section 13.1), use a spatula, the curved edge of a scalpel blade, or other similar utensil to collect randomly selected sub-samples (minimum of five) of the original sample. These sub-samples can be made into slide mounts immediately by following the procedures in Sections 13.3.2.3 through 13.3.2.9, or the analyst can place the sub-samples together into the mortar, set the original sample aside and proceed with the next Section.

13.3.2.1.1 Care should be taken to grab enough sub-sample material to prepare five slide mounts, but not enough to create excess material that will need to be disposed of as ACM.

13.3.2.2 Use the pestle to gently break up any coarse particles in the sub-sample material. Not all samples will require further grinding with the pestle. If this is the case, proceed to the procedures described in Sections 13.3.2.3 through 13.3.2.9.

13.3.2.2.1 Soil samples processed according to the current version of SOP ISSI-LIBBY-01, *Soil Sample Preparation*, should be ground to a maximum particle size of approximately 250 μ m. However, particles this size will still cause thick, uneven distribution of the sub-sample material under the cover slip and may lead to broken cover slips.

Note: If a sample seems particularly fine (like powder) or particularly coarse (particle sizes > 250 μ m), notify the client so that the Troy Sample Preparation Facility can be alerted to make sure that the grinder is properly calibrated.

13.3.2.2.2 While using the mortar and pestle to grind the sub-sample material, care should be taken to not pulverize the asbestos to a fiber size unidentifiable by PLM techniques. The material in the slide mounts must be coarse enough that asbestos fibers can still be identified by PLM and still be as representative as possible of the sample as a whole.

13.3.2.3 Place one to two drops of RI liquid onto a slide for each of the five slide mounts.

13.3.2.3.1 Each microscope slide must be wiped clean with an appropriate wipe prior to use in order to avoid contamination.

13.3.2.3.2 Note that the five slide mounts do not have to be on five separate slides. Analysts can choose how many slide mounts to put on each slides (for example, an analyst can use two slides – one with three slide mounts and the other with two slide mounts).

13.3.2.4 With the utensil, gently stir sub-sample material into the RI liquid to produce a homogeneous mixture.

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- 13.3.2.5 Cover each mixture of RI liquid and sub-sample material with a glass cover slip.
- 13.3.2.6 Gently agitate the mixture under the cover slip by pressing down and rubbing the top of the cover slip with something that will “grab” the cover slip and allow it to be translated from side to side, such as an etching scribe or the eraser end of a pencil.
 - 13.3.2.6.1 Use this action to spread the mixture of RI liquid and sub-sample material over the approximate area of the cover slip.
 - 13.3.2.6.2 The material should be spread out evenly under the cover slip with little to no overlapping particles.
- 13.3.2.7 Wipe any loose sample material or excess RI liquid from the slide with a disposable lint-free wipe.
- 13.3.2.8 The prepared slide can now be safely removed from the preparation hood for analysis by PLM-VE.
- 13.3.2.9 Additional slide mounts of sub-sample material can be prepared in an appropriate RI liquid at the analyst’s discretion.

13.4 Secondary Stereomicroscopic Evaluation

- 13.4.1 After the sub-samples have been taken from the original sample, aggressively agitate or tap the sample substrate containing the original sample to cause the particulate to settle and the asbestos fibers to sort to the surface.
 - 13.4.1.1 Re-examine the entire sample using the stereomicroscope, and repeat the fiber-pick procedures described in Section 13.1.6.

Note: If a fiber-pick was prepared during the initial stereomicroscopic examination, it is not required that another fiber-pick be prepared after agitating the substrate. However, regardless of whether or not a fiber-pick was performed during the initial stereomicroscopic examination, each sample substrate must be agitated and the sample re-examined using stereomicroscopy following the procedures in this section. Additional fiber-picks can be prepared at the analyst’s discretion.

- 13.4.1.2 Agitating the substrate should only be used as a qualitative technique following random slide mount preparation and not as a quantitative technique because it tends to make the sample inhomogeneous.

Note: Agitating the substrate and re-examining the sample using stereomicroscopy can be done prior to preparing the five slide mounts. However, do not agitate the substrate until all the sub-samples have been taken from the original sample and placed in the mortar in order to avoid collecting inhomogeneous sub-sample material.

- 13.4.2 Avoid contamination by maintaining a clean work space.
 - 13.4.2.1 After preparing each sample, clean all work surfaces, sample substrates, utensils, and any other items that came into contact with the sample, using water and paper towels.
 - 13.4.2.2 Dispose of gloves once they become excessively dirty.

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- 13.4.2.3 Only prepare one sample at a time. Never have more than one sample container open inside the preparation hood at any given time.
- 13.4.2.4 When placing drops of RI liquid on the slides, never touch the dropper directly to a different RI liquid or to liquid that already has sample material in it. Only touch the dropper to a clean slide.
- 13.4.2.5 Discard any RI liquids that become contaminated with sample debris.

13.5 Classification of Asbestos Mineral Type

- 13.5.1 Analysis of soil samples from the Libby Site consists of identification and quantification of any and all asbestos phases present within the sample, and when possible, the identification and semi-quantification of non-asbestos fibers and the identification of matrix materials within the sample.
- 13.5.2 Positive identification of asbestos, non-asbestos fibers and matrix material is conducted by examination of sample slide mounts by PLM.
- 13.5.3 Visually examine the entire area of all prepared slides using PLM (using both plane light and crossed polars) to find any fibrous constituents within the slide mounts.
- 13.5.4 Positive identification of asbestos requires the determination of the following six optical properties by PLM.
 - 13.5.4.1 Habit
 - 13.5.4.2 Color and pleochroism (if pleochroism is present)
 - 13.5.4.3 RIs, both alpha and gamma
 - 13.5.4.4 Birefringence
 - 13.5.4.5 Extinction angle
 - 13.5.4.6 Sign of elongation (positive if the fiber is length slow, negative if the fiber is length fast)
- 13.5.5 Asbestos cannot be reported in any quantity, including trace, until its optical properties are measured and recorded.
- 13.5.6 Based on the optical properties, asbestos in the sample is classified into one of three categories described in Table 13.1:

Table 13.1

Code	Description	Notes
LA	Libby Amphibole	The minerals winchite, richterite, tremolite, and actinolite, which are characteristic of the mine at the Libby Site. Also included are the minerals magnesio-arfvedsonite and magnesio-riebeckite, which are known to occur at the Libby Site in smaller quantities.
OA	Other amphibole asbestos	Regulated amphibole asbestos (amosite, crocidolite, and anthophyllite)
C	Chrysotile	Asbestiform serpentine

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- 13.5.7 Chrysotile $Mg_3Si_2O_5(OH)_4$
- 13.5.7.1 Serpentine is a phyllosilicate (sheet-silicate) mineral, and when it occurs in an asbestiform habit, it is referred to as chrysotile.
- 13.5.7.2 There are three varieties of the mineral serpentine: antigorite, lizardite, and chrysotile. All three have the same chemical composition but different habits.
- 13.5.7.3 Individual fibrils of chrysotile have been shown by transmission electron microscopy (TEM) to be in the form of scrolled tubes, or tightly rolled micaceous sheets, such that the fibril axis lies within the plane of the sheets (much as if a newspaper had been rolled up). In other types of serpentine, the sheets may be curved, but they are flat or platy, not rolled into tightly scrolled tubes.
- 13.5.7.4 If serpentine is observed and has a platy or massive (non-fibrous) habit, it is classified as non-asbestiform serpentine (antigorite if it is platy or lizardite if it occurs as a massive, fine-grained matrix).
- 13.5.7.5 If serpentine is observed and has a fibrous habit, it is classified as chrysotile asbestos.
- 13.5.7.6 Chrysotile sometimes appears silky or wavy. The fibers are flexible, and sometimes occur as tangled mats of many fibers.
- 13.5.7.7 Chrysotile can only be seen in PLM as bundles; the individual fibrils that make up a chrysotile bundle are beyond the resolution of all light microscopy. These bundles are often splayed. Kinked, chevron-style folds are sometimes seen within the bundles.
- 13.5.7.8 Chrysotile is usually colorless in PLM, although it sometimes shows a slight golden, yellow, or pale golden-green color. If exposed to very high temperatures, chrysotile is distinctly brown in plain light.
- 13.5.7.9 Chrysotile is never pleochroic.
- 13.5.7.10 Small particles of opaque magnetite can sometimes be seen in large, intact bundles of chrysotile.
- 13.5.7.11 The range for the lower RI (alpha, or α) for chrysotile is 1.545 to 1.553 as reported in the certificate for NIST SRM 1866b, although the range for chrysotile encountered in field samples may be somewhat wider.
- 13.5.7.12 The range for the higher RI (gamma, or γ) for chrysotile is 1.552 to 1.560 as reported in the certificate for NIST SRM 1866b, although the range for chrysotile encountered in field samples may be somewhat wider.
- 13.5.7.13 Exposure to high heat and dehydration of the crystal lattice will increase the RIs of chrysotile.
- 13.5.7.14 The birefringence (δ ; expressed numerically as the difference between α and γ) of chrysotile is low, usually around 0.008. In practice, this means that most chrysotile bundles of fine to medium size observed in samples will have low first-order gray to medium gray interference colors under crossed polars. Larger, thicker fibers can show first-order white to yellow interference colors; higher colors may be seen in the thickest bundles.
- 13.5.7.15 Chrysotile is most easily visible in plane light in the higher RI liquids, such as 1.620 or 1.680. However, measurement of the RIs of

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- 13.5.7.16 chrysotile should be done with the fibers mounted in the 1.550 liquid. Chrysotile is almost always length slow (positive sign of elongation), although length fast chrysotile has been observed on very rare occasions.
- 13.5.7.17 Chrysotile invariably has parallel extinction.
- 13.5.8 Amosite $\text{Fe}_7\text{Si}_8\text{O}_{22}(\text{OH})_2$
- 13.5.8.1 The name amosite is derived from an acronym for "Asbestos Mines of South Africa." It is a trade name and not a mineralogical name. Amosite is the fibrous variety of the mineral grunerite.
- 13.5.8.2 Amosite has an acicular (needle-like) habit. Bundles of amosite are composed of many fibrils, which are often straight and only somewhat flexible.
- 13.5.8.3 In plane light, amosite is usually colorless, green, brown, or greenish-brown. Heated amosite is brown to dark brown and can be nearly opaque. Amosite is sometimes weakly pleochroic.
- 13.5.8.4 The range for the lower RI (α) for amosite is 1.675 to 1.681 as reported in the certificate for NIST SRM 1866b, although the range for amosite encountered in field samples may be somewhat wider.
- 13.5.8.5 The range for the higher RI (γ) for amosite is 1.697 to 1.704 as reported in the certificate for NIST SRM 1866b, although the range for amosite encountered in field samples may be somewhat wider.
- 13.5.8.6 Exposure to high heat and dehydration of the crystal lattice will increase the RI's of amosite.
- 13.5.8.7 The birefringence of amosite is moderate, usually about 0.020. Most fibers observed will have first-order white to yellow interference colors under crossed polars; although, higher colors (first-order magenta to second-order or sometimes even higher) can be seen in the thicker bundles.
- 13.5.8.8 RI measurements should be done with the fibers mounted in 1.680 to 1.700 RI liquid.
- 13.5.8.9 Amosite is length slow (positive sign of elongation).
- 13.5.8.10 Even though grunerite is a monoclinic mineral, the extremely fine fibers that form bundles of amosite cause amosite to have parallel extinction.
- 13.5.9 Crocidolite $\text{Na}_2\text{Fe}_3^{2+}\text{Fe}_2^{3+}\text{Si}_8\text{O}_{22}(\text{OH})_2$
- 13.5.9.1 Crocidolite is a fairly uncommon type of asbestos. It is the fibrous variety of the mineral riebeckite.
- 13.5.9.2 Crocidolite has an acicular habit very similar to that of amosite. The fibers are only somewhat flexible.
- 13.5.9.3 Crocidolite is distinctly blue or blue-green in plane light and is pleochroic.
- 13.5.9.4 Normally, the range for the lower RI (α) for crocidolite is 1.680 to 1.698 (EPA, 1993).
- 13.5.9.5 Normally, the range for the higher RI (γ) for crocidolite is 1.685 to 1.706 (EPA, 1993).
- 13.5.9.6 The strong color of crocidolite makes measurement of the RIs very difficult. For this reason, select finer fibers of crocidolite, which have less color, when measuring RIs.

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- 13.5.9.7 The birefringence of crocidolite is low, usually about 0.006. Crocidolite often shows anomalous interference colors under crossed polars.
- 13.5.9.8 RI measurements on crocidolite should be done with the fibers mounted in 1.680 or 1.700 liquid.
- 13.5.9.9 Because crocidolite is length fast, the lower RI (α) should be measured with the fiber oriented in the E-W direction (parallel to the lower polar), and the higher RI (γ) should be measured with the fiber oriented in the perpendicular (N-S) direction.
- 13.5.9.10 Even though riebeckite is a monoclinic mineral, the extremely narrow fibers that form bundles of crocidolite cause crocidolite to have parallel extinction.
- 13.5.10 Anthophyllite $(\text{Mg,Fe})_7\text{Si}_8\text{O}_{22}(\text{OH})_2$
- 13.5.10.1 Anthophyllite is a rare type of asbestos used in construction materials.
- 13.5.10.2 Anthophyllite has a lamellar to acicular habit, and may occur as straight to slightly curved fibers or fiber bundles.
- 13.5.10.3 Anthophyllite is colorless to pale brown in plane light. It is sometimes weakly pleochroic.
- 13.5.10.4 The range for the lower RI (α) for anthophyllite is 1.593 to 1.694 (Deer et al., 1997). The commercial-grade anthophyllite in SRM 1867a has an α of 1.615.
- 13.5.10.5 The range for the higher RI (γ) for anthophyllite is 1.613 to 1.722 (Deer et al., 1997). The commercial-grade anthophyllite in SRM 1867a has a γ of 1.636.
- 13.5.10.6 The birefringence of anthophyllite is moderate, usually about 0.020.
- 13.5.10.7 Generally, RI measurements on anthophyllite should be done with the fibers mounted in 1.620 to 1.640 liquid.
- 13.5.10.8 Because anthophyllite is an orthorhombic mineral, all fibers of anthophyllite will invariably have parallel extinction. This helps to distinguish it from LA and the non-asbestos mineral wollastonite, which often show inclined extinction.
- 13.5.10.9 Anthophyllite is length slow (positive sign of elongation).
- 13.5.11 Libby Amphibole
- 13.5.11.1 LA consists of tremolite-actinolite, $\text{Ca}_2(\text{Mg,Fe})_5\text{Si}_8\text{O}_{22}(\text{OH})_2$; winchite, $\text{CaNaMg}_4(\text{Al,Fe}^{3+})\text{Si}_8\text{O}_{22}(\text{OH})_2$; richterite, $\text{NaCaNa}(\text{Mg,Fe})_5\text{Si}_8\text{O}_{22}(\text{OH})_2$; magnesio-arfvedsonite, $(\text{Na,K})\text{Na}_2\text{Mg}_4\text{Fe}^{3+}\text{Si}_8\text{O}_{22}(\text{OH})_2$; and magnesio-riebeckite, $\text{Na}_2\text{Mg}_3\text{Fe}^{3+}_2\text{Si}_8\text{O}_{22}(\text{OH})_2$. This group of minerals is generally described as sodic tremolite.
- 13.5.11.2 The optical properties for each individual mineral are provided below and in Attachment 4. There is a great deal of overlap in optical properties among the minerals that make up LA. As such, discreet mineral identification is not required by this SOP. If the sample exhibits the optical properties of a mineral listed in this section, the specific optical properties shall be noted on the analytical bench sheet and EDD, and the mineral identified as LA.

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- 13.5.11.3 The habit of LA ranges from prismatic to fibrous. The fibers that form a bundle of LA may be parallel to sub-parallel, or the fibers may sometimes cross one another at various angles giving the bundle a matted appearance. The aspect ratio of the fibers is highly variable, and all tremolite, actinolite, winchite, richterite, magnesio-arfvedsonite or magnesio-riebeckite encountered in a sample should be classified as LA regardless of the aspect ratio of the individual fibers. Refer to Attachment 5 for photomicrographs that show a wide range of LA habits that might be encountered during PLM analysis.
- 13.5.11.6 Laboratories should use the Controlled Libby Amphibole Asbestos (refer to Section 11.8) and NIST Bulk Asbestos Proficiency Testing Round M12001, Sample 4, as reference materials to familiarize themselves with the range of habits and optical properties of LA. Laboratories should contact the client, or their designee, if they do not have these reference materials.
- 13.5.11.7 The color of LA is highly varied in plane light. Tremolite is usually colorless. Actinolite is usually pale green to dark green. Darker colors and stronger pleochroism are associated with higher iron content for the tremolite-actinolite series (Deer et al., 1997). Winchite can be pale yellow, blue, blue-green, or blue-gray. Richterite can be brown, tan, pale green to dark green, pale yellow, or violet (Deer et al., 1997). Magnesio-arfvedsonite is yellowish-green, brownish-green, or gray-blue (Deer et al, 1997). Magnesio-riebeckite is blue, gray-blue, or pale blue to yellow (Deer et al, 1997). Winchite, richterite, magnesio-arfvedsonite, and magnesio-riebeckite can all be pleochroic.
- 13.5.11.8 LA generally has moderate birefringence, usually about 0.020.
- 13.5.11.9 RI measurements on LA should be done with the fibers mounted in 1.620 to 1.640 RI liquid.
- 13.5.11.10 LA usually shows inclined (or oblique) extinction, although fibers in certain crystallographic orientations will exhibit parallel extinction. The maximum extinction angle for tremolite-actinolite can be as high as 10 to 21 degrees. Winchite and richterite can show higher extinction angles, sometimes as high as 30 degrees or even higher for richterite.
- 13.5.11.11 Winchite, richterite, tremolite, and actinolite are all length slow (positive sign of elongation). Both magnesio-arfvedsonite and magnesio-riebeckite are length fast (negative sign of elongation).
- 13.5.11.12 On the analytical bench sheet (Attachment 1), record only one set of optical properties for LA for each sample that contains LA. Choose the fiber/and or bundle that shows the best Becke line and/or dispersion staining colors.
- 13.5.11.13 Refer to Attachment 4 for additional information on the optical properties used in LA identification.

13.6 Refractometry

13.6.1 Calibration of RI Liquids

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- 13.6.1.1 Accurate measurement of a mineral's RIs begins with proper calibration of the RI liquids. Each RI liquid used for routine sample preparation and analysis must be calibrated once each month.
- 13.6.1.2 Prepare a slide mount of the appropriate certified precision optical glass in the RI liquid to be calibrated.
- 13.6.1.3 Read the laboratory's thermometer to the nearest 2°C to determine the ambient temperature t , and record the temperature on the appropriate worksheet.
- 13.6.1.4 Next determine λ_0 . This is the wavelength at which the RI of the liquid is equal to the RI of the certified precision optical glass. Observe the dispersion staining color shown by the glass, and consult the appropriate dispersion staining color chart (McCrone, 1987). If the glass particles show a range of dispersion staining colors, use the most predominant color when determining λ_0 . Record the predominant dispersion staining color and corresponding λ_0 on the worksheet.
- 13.6.1.5 Consult the appropriate conversion table developed by Shu-Chun Su, Ph.D. (see Attachment 2). These tables are used to convert λ_0 and t into n_d^{25} , which is the calibrated RI of the liquid at a wavelength of 589 nm and a temperature of 25°C. Determine the value of n_d^{25} from the appropriate table for the known values λ_0 and t .
- 13.6.1.6 If conversion tables for liquids are used but not included in Attachment 2, laboratories can contact ESAT Region 8 to receive an Excel workbook developed by Shu-Chun Su, Ph.D. The workbook enables individuals to generate new conversion tables by entering the dispersion coefficients and values of n_d of the liquid and the glass, and the value of dn/dt (change of RI with temperature) of the liquid into the first sheet of the workbook. All of these values are provided by the manufacturer of the glass and liquid.
- 13.6.1.7 Record the value of n_d^{25} on the worksheet. This is the calibrated RI of the liquid at a standard temperature of 25°C.
- 13.6.1.8 Write this calibrated RI and the date of calibration on the bottle.
- 13.6.1.9 If the difference between the calibrated RI of the liquid and the manufactured RI of the liquid is greater than 0.004, then the liquid may not be used for analysis of samples.
- 13.6.1.10 Repeat the above steps for each liquid in routine use.
- 13.6.2 Measurement of RIs (refractometry) of minerals is performed using either the dispersion staining method or the Becke line method.
 - 13.6.2.1 All analysts must be proficient in both methods. The choice of which method to use is left to the analyst's discretion.
 - 13.6.2.2 The dispersion staining method requires a clean surface of the mineral to be in direct contact with the liquid and can only be performed if a conversion chart has been developed beforehand for a specific mineral in a specific RI liquid.
 - 13.6.2.3 The Becke line method will often work on relatively fine fibers, and also requires a clean surface of the mineral to be in contact with the liquid. However, this method does not require a specific mineral-

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liquid chart to be developed before it is used. For this reason the Becke line method can be used to measure the RIs of materials other than asbestos.

13.6.3 Measurement of RIs by the Dispersion Staining Method

- 13.6.3.1 Mount the fibers in the appropriate liquid. A clean surface of the mineral must be in direct contact with the RI liquid in order for the correct dispersion staining colors to be displayed.
- 13.6.3.2 It may be necessary to separate and spread out fibers bundles on the slide so a clean surface is exposed. Do this by agitating the bundles with an X-acto knife or other sample manipulation utensil, or rubbing the cover slip over the bundles to agitate and dis-aggregate them.
- 13.6.3.3 Examine the slide in plane light using the 10X dispersion staining objective. Ensure that the objective is centered.
- 13.6.3.4 Stop down the condenser iris diaphragm until dispersion colors are observed.
- 13.6.3.5 Read the thermometer to find ambient temperature of the laboratory's air to the nearest 2°C.
- 13.6.3.6 To measure α , orient the fiber E-W (parallel to the lower polar) if the fiber is suspected of being crocidolite, or N-S if the fiber is suspected of being chrysotile, amosite, or anthophyllite. LA shows biaxial optics and requires a more detailed treatment, described in Section 13.6.5.
- 13.6.3.7 Next, observe the dispersion staining color that is displayed.
- 13.6.3.8 For central stop dispersion staining, light of a wavelength equal, or approximately equal, to the matching wavelength (given the symbol λ_0 , where the RI of the liquid matches the RI of the mineral) is blocked from reaching the ocular. The color observed is a summation of the wavelengths of light that are higher or lower than the matching wavelength, which pass around the central stop.
- 13.6.3.9 For annular stop dispersion staining, the color observed is the light of a wavelength equal, or approximately equal, to the matching wavelength passing through the stop to the ocular. Wavelengths of light higher or lower than the matching wavelength are blocked by the annular stop.
- 13.6.3.10 Consult the dispersion staining color chart (McCrone, 1987), and find the matching wavelength (λ_0) that corresponds to the observed color.
- 13.6.3.11 When measuring α and a range of dispersion staining colors is displayed, choose the color that produces the lowest RI, i.e., the color that corresponds to the longest λ_0 .
- 13.6.3.12 Refer to the paper "Rapidly and Accurately Determining Refractive Indices of Asbestos Fibers by Using Dispersion Staining Method," by Shu-Chun Su, Ph.D. (1996).
- 13.6.3.13 For the appropriate RI liquid and mineral combination, find the column for the laboratory's temperature and row for λ_0 ; record the corresponding RI value.
- 13.6.3.14 To measure γ , rotate the stage 90 degrees.
- 13.6.3.15 The fiber should now be perpendicular to the lower polar (N-S) if the fiber is suspected of being crocidolite, or parallel to the lower polar (E-W) if the fiber is suspected of being chrysotile, amosite, or

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anthophyllite. Refer to Section 13.6.5 for orienting fibers of LA when measuring γ .

13.6.3.16 Observe the dispersion staining colors and find the corresponding λ_0 . When measuring γ , choose the color that produces the highest RI, i.e., the color that corresponds to the shortest λ_0 .

13.6.3.17 Consult the appropriate chart for the asbestos type and liquid being used; record the RI value for the temperature and λ_0 .

Note: There are two charts for each mineral and liquid combination - one for α and one for γ . Be sure to use the appropriate chart when measuring α or γ .

13.6.4 Measurement of RIs by the Becke Line Method

13.6.4.1 Becke line colors are observed in plane light when the RI of the mineral is close or equal to the RI of the liquid. Becke line colors are usually best observed using high magnification (200X to 500X).

13.6.4.2 To measure RIs using the Becke line method, mount the fibers in a liquid whose RI is close to that of the mineral.

13.6.4.3 To measure α , orient the fiber E-W (parallel to the lower polar) if the fiber is suspected of being crocidolite, or N-S if the fiber is suspected of being chrysotile, amosite, or anthophyllite. LA shows biaxial optics and requires a more detailed treatment, described in Section 13.6.5. Observe the Becke line colors produced.

13.6.4.4 As a rule, the Becke line moves into whichever medium (the grain or the liquid) has a higher RI when the microscope stage is lowered from the focused position.

13.6.4.5 Colored Becke lines are produced when the RI of the grain is higher than the liquid for some wavelengths of light in the visible spectrum and when the RI of the grain is less than the liquid for other wavelengths.

13.6.4.6 If a brownish or rust colored Becke line moves into the grain when the microscope stage is lowered, and a bluish-white Becke line moves into the liquid, the RI of the grain is less than that of the liquid.

13.6.4.7 If an orange-yellow, yellow, or lemon-yellow Becke line moves into the grain when the stage is lowered, and a violet or blue-violet Becke line moves into the liquid, the RI of the grain is higher than that of the liquid.

13.6.4.8 A match occurs when n_d (the RI for the wavelength of sodium light, 589 nm) is the same for both the grain and the liquid. When the n_d of mineral matches the n_d of the liquid, an orange Becke line with just a touch of red moves into the grain and a bluish line moves into the liquid when the stage is lowered.

13.6.4.9 If a match cannot be obtained, mount the mineral in two liquids that bracket the RI of the mineral, and interpolate where the RI of the mineral should be.

13.6.4.10 The Becke Line Chart by F. D. Bloss (Attachment 9) may be used to approximate the size of the difference between the RI of the liquid and the RI of the mineral.

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13.6.5 Biaxial Optics

- 13.6.5.1 Anthophyllite and LA often show biaxial optics. This is rarely a consideration for amosite or crocidolite. Even though chrysotile is a monoclinic mineral, it does not show biaxial optics due to the scrolled nature of the fibers.
- 13.6.5.2 When an asbestos fiber shows biaxial optics, it is easy to measure a RI called alpha prime (α') that is between true α and beta (β) when attempting to measure α .
- 13.6.5.3 True α can only be observed when a grain is oriented in exactly the correct position.
- 13.6.5.4 For the monoclinic minerals that display biaxial optics (LA), the crystals need to be oriented so the X and Z axes of the biaxial indicatrix corresponding to the directions of α and γ are parallel to the lower polar when measuring these indices (not necessarily oriented with the crystallographic axes). As a general rule, when these fibers show inclined extinction, select the fibers that show the highest extinction angle when measuring α and γ . RI measurements should be made on a fiber where the plane of X and Z in the biaxial indicatrix lies as close to parallel to the plane of the microscope stage as possible, such that the microscopist is looking directly down Y, which corresponds to the β RI (and also the b crystallographic axis for tremolite, actinolite, winchite, richterite, and magnesio-arfvedsonite). Fibers at or close to this orientation will tend to show the highest extinction angle.
- 13.6.5.5 Next, when measuring α for LA, orient the fiber approximately N-S, but at the inclined orientation where the fiber is extinct under crossed polars. The fiber should now be oriented away from N-S at an angle that is equal to its extinction angle, and the Z direction of the biaxial indicatrix is perpendicular to the lower polar.
- 13.6.5.6 Repeat this for a number of fibers. If the fibers show different Becke line or dispersion staining colors, measure α for those that display the lowest RI.
- 13.6.5.7 Similarly, it is easy to measure a RI called gamma prime (γ') that is between β and true γ when attempting to measure γ . True γ can only be observed when a fiber is oriented in exactly the correct position.
- 13.6.5.8 When measuring γ , orient a fiber of LA approximately E-W at the inclined angle where the fiber is extinct under crossed polars. The fiber should now be oriented away from E-W at an angle equal to its extinction angle, so that the Z direction of the biaxial indicatrix is parallel to the lower polar. Repeat this for a number of fibers. If the fibers show different Becke line or dispersion staining colors, measure γ for those that display the highest RI.
- 13.6.5.9 Biaxial Optics of Anthophyllite
- 13.6.5.9.1 When measuring α (the lower RI) for anthophyllite, the fiber should be oriented in the N-S direction. At this orientation, they can show either α or β , or anywhere in between. It is therefore necessary to examine a number

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of fibers oriented in the N-S position to find true α , which will be observed for fibers that display the lowest RI).

- 13.6.5.9.2 When measuring γ (the higher RI) for anthophyllite, the fiber should be oriented in the E-W direction. Fibers of anthophyllite lying flat on the slide will always show γ , not γ' , because the c-axis of the fiber will lie parallel to the plane of the slide.

13.7 Quantification of Asbestos Content

13.7.1 General

- 13.7.1.1 Asbestos is reported as either mass percent or area percent for LA, but only as area percent for chrysotile, amosite, crocidolite, and anthophyllite.
- 13.7.1.2 Asbestos must be positively identified, and its optical properties measured and recorded, before it can be reported in any quantity, including trace.
- 13.7.1.3 Quantification of asbestos concentration is performed by making a calibrated visual estimate by PLM on carefully prepared slide mounts of the sample material, in conjunction with stereomicroscopic examination of the bulk sample.

13.7.2 Calibrated Visual Estimate of Asbestos Concentration by PLM

- 13.7.2.1 To perform a calibrated visual estimate, first decide on the best optical set-up to maximize the contrast between asbestos and non-asbestos materials within the slide mounts.
- 13.7.2.2 Higher magnifications (200X or 400X) will improve the visibility of asbestos when it is very fine. Lower magnification (100X) should be used when the asbestos is coarse. Use of the compensator plate under crossed polars enhances the contrast between asbestos and non-asbestos on some samples.
- 13.7.2.3 Scan the entire area of the slides, paying attention to the relative proportion of asbestos to non-asbestos.
- 13.7.2.4 Draw on previous experience to make a precise and calibrated visual estimate. Making accurate calibrated visual estimates is an acquired and experience-based skill.

13.7.3 Use of Reference Materials for Visual Estimation of Asbestos Content

- 13.7.3.1 Visual area estimation is a semi-quantitative approach requiring the microscopist to estimate the area of asbestos as a percentage of the total material present over many fields of view. Visual area estimation may be difficult, especially at low concentration values.
- 13.7.3.2 Visual estimates of LA content less than or equal to 1% by weight will be performed using a set of site-specific reference materials as a frame of reference. These Controlled PE Reference Materials will contain either 0.2% or 1.0% LA by weight¹ and were prepared for

¹ The nominal mass fraction of the reference materials is based on the gravimetric fraction of the material that is soil and the amount that is spiking material, adjusted for the fraction of the spiking material that is LA. For example, if the spiking material were estimated to contain 85% LA by mass, then the 1.0% Controlled PE Reference Material would contain 1.18 grams of spiking material (1.00g of LA) per 100g of reference material. Because the estimated LA content of the spiking material is

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- analysis using the same approach as for field samples.
- 13.7.3.3 Visual estimates of LA content greater than 1% will be performed using calibration standards made in-house from NIST SRMs and archived NIST PE samples as reference (see Section 16.2).
- 13.7.3.4 Labs analyzing samples for LA should prepare five slide mounts each of the 0.2% and 1.0% Controlled PE Reference Materials in a permanent medium, such as epoxy or melt-mount. These permanently-mounted slides can then be readily referred to at the bench by analysts as needed. When using the 0.2% and 1.0% standards as calibration materials for visual estimates, always examine the entire area of all five slide preparations by PLM for each of these standards. This will guard against potential analytical bias that may be introduced by inhomogeneities in the calibration standards.
- 13.7.3.5 Photomicrographs of representative fields of view of the 0.2% and 1.0% LA reference materials are included as Attachment 7 of this SOP so that analysts may refer to them as needed.
- 13.7.3.6 Note that because these reference materials are based on LA, they are not appropriate for estimating the mass percent of other types of asbestos (chrysotile, amosite, crocidolite, or anthophyllite). Therefore, if any asbestos types besides LA are observed, the reported values for those asbestos types should be in units of area percent.
- 13.7.3.7 It is recommended that laboratories prepare their own permanently-mounted slides of other asbestos types (such as amosite and chrysotile) in low concentrations. This can be performed by weighing out small quantities of relatively pure asbestos (such as NIST SRM's 1866b and 1867a) and a non-asbestos matrix material (such as calcite or gypsum). The two fractions can then be mixed together, and the mixture can be mounted on a slide in a permanent medium, such as epoxy or melt-mount.
- 13.7.3.8 Visual comparison charts can be posted on the walls of the PLM laboratory within sight of the microscope(s) so that analysts may refer to them as necessary. A number of these charts are available, such as the Comparison Chart for Visual Percentage Estimation (after Terry and Chilingar, 1955) and the visual estimation charts developed by Dr. Shu-Chun Su (see References).
- 13.7.3.9 For LA, compare what is seen in the 0.2% and 1.0% Controlled PE Reference Materials and visual comparison charts as needed. The concentrations of LA in the 0.2% and 1.0% reference materials were placed at the bin concentration cut-offs, which place LA concentrations of each sample into one of four categories (see Section 13.7.5, below).
- 13.7.3.10 Other LA reference materials, such as the 0.5% and 2.0% reference materials, may also be used for comparison when performing visual estimates. However, analysts should rely primarily on the 0.2% and

approximate, the true concentration of a reference material may not be precisely equal to the nominal value.

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- 1.0% Controlled PE Reference Materials for assignment of samples to bin categories; the other reference materials should be used only as supporting tools for determining LA content.
- 13.7.4 Combining Stereomicroscopic and PLM Visual Estimates
- 13.7.4.1 Analysts must not place over-reliance on either stereomicroscopy or PLM when performing visual estimates. The advantage of stereomicroscopy is that the entire sample can be examined. However, once fibers are smaller than a certain size (approximately 250 μm or less in length) it becomes difficult to impossible to find them with the stereomicroscope and mount them in a RI liquid for positive identification by PLM. Conversely, only a small sub-sample of the whole sample is examined in the random slide mounts prepared for PLM analysis. This means a PLM result can be biased high or low if the prepared slides are not representative of the sample as a whole. Therefore, it is necessary to base a calibrated visual estimate of asbestos content on both detailed stereomicroscopic observation of the entire sample and examination of the entire area of all five prepared slide mounts by PLM, as both microscopic tools are complementary to one another.
- 13.7.4.2 Examine every sample stereomicroscopically to produce an initial estimate of asbestos content. As described in Section 13.1, this preliminary stereomicroscopic visual estimate of asbestos content is recorded on the analytical bench sheet.
- 13.7.4.3 Carefully analyze the entire area of all five prepared slide mounts of the sample by PLM. The PLM result is then compared to the original stereomicroscopic estimate of asbestos concentration. The PLM result will confirm, refine, or deny the original stereomicroscopic estimate.
- 13.7.4.4 The PLM result may indicate the need to re-examine the sample stereomicroscopically, and possibly, the need to re-mount and re-analyze the sample by PLM.
- 13.7.4.5 Decide what asbestos concentration to report based on both the stereomicroscopic estimation of asbestos content and the PLM visual estimate of asbestos content. Refer to Attachment 8 for a flow chart describing this entire process.
- 13.7.4.6 If the asbestos is fine, more weight should be placed on the PLM mounts when estimating asbestos content. If the asbestos is coarse, more weight should be placed on the stereomicroscopic estimate. However, both stereomicroscopic examination and PLM are required for every Libby soil sample analyzed at the laboratory.
- 13.7.4.7 If different asbestos concentrations are observed in the different slide mounts, then the PLM estimate should be an average of all prepared slides.
- 13.7.5 LA Bin Categories
- 13.7.5.1 All winchite, richterite, tremolite, actinolite, magnesio-arfvedsonite, and magnesio-riebeckite observed in a sample is recorded as LA and contributes to the bin category (described in Table 13.2), whether the habit observed is fibrous, straight, or prismatic. Refer to Attachment

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- 5 for examples of a wide range of LA habits. Also refer to Attachment 6 for photomicrographs of representative examples of LA habits as imaged by the USGS by Scanning Electron Microscopy (SEM).
- 13.7.5.2 Using the Controlled PE Reference Materials (0.2% and 1.0%) as a visual guide, the microscopist will evaluate the sample and report LA results as follows:

Table 13.2

PLM Laboratory Report			Description
Qual	CONC (%)	Bin	
ND		A	LA was not observed in the sample
Tr		B1	LA was observed in the sample at a level that appeared to be lower than the 0.2% reference material
<	1	B2	LA was observed in the sample at a level that appeared to be approximately equal to or greater than the 0.2% reference material but less than the 1% reference material.
	1, 2, 3, etc	C	LA was observed in the sample at a level that appeared to equal or exceed the 1% reference material. In this case, the area percent is estimated quantitatively as a whole number percentage.

- 13.7.5.3 **"ND" (not detected) in the Qualifier column** is used for all samples in which LA is not observed using stereomicroscopy and is also not positively identified in any of a minimum of five different PLM slides prepared using representative sub-samples of the test material. These samples are assigned to **Bin A**.
- 13.7.5.4 **"Tr" (trace) in the Qualifier column** is used for all samples in which LA is observed either using stereomicroscopy or in at least one of the five required PLM slide mounts prepared from representative sub-samples of the test material, and in which the amount of LA present appears to be less than the 0.2% reference material. These samples are assigned to **Bin B1**.
- 13.7.5.5 **"<" (less than) in the Qualifier column and "1" in the Concentration column** is used for all samples in which LA is observed either by stereomicroscopy or by PLM in the five required slide mounts prepared from representative sub-samples of the test material, and in which the average amount of LA present appears to be equal to or greater than the 0.2% reference material but less than the 1% reference material. These samples are assigned to **Bin B2**.
- 13.7.5.6 **A numeric value (1, 2, 3, etc.) in the Concentration column and no entry in the Qualifier column** is used for all samples in

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which LA is observed either by stereomicroscopy or by PLM in the five required slide mounts prepared from representative sub-samples of the test material, and in which the average amount of LA present appears to be equal to or greater than the 1% reference material. These samples are assigned to **Bin C**.

- 13.7.6 Visual Estimations for Chrysotile, Amosite, Crocidolite, and Anthophyllite
 - 13.7.6.1 Visual estimates for chrysotile, amosite, crocidolite, and anthophyllite are reported as area percent.
 - 13.7.6.2 Do not use the bins designed for LA content for concentrations of chrysotile, amosite, crocidolite, and anthophyllite. Rather, report area percent as ND if these asbestos types are not detected, "<1" if these asbestos types are detected but at a concentration of less than 1% by area, or to the nearest whole percentage (1%, 2%, 3%, etc.) if these asbestos types are detected at a concentration of 1% or higher.

13.8 Non-Asbestos Fibrous Constituents

- 13.8.1 When non-asbestos fibers are observed, measure and record on the bench sheet at least one optical property that distinguishes the fiber from asbestos.
- 13.8.2 There are several non-asbestos fibers that can be confused with asbestos, and the analyst must be aware of their properties and habits. Commonly encountered non-asbestos fibers are listed below.
- 13.8.3 Talc $Mg_3Si_4O_{10}(OH)_2$
 - 13.8.3.1 Talc is a magnesium silicate mineral that usually occurs in a platy or fibrous habit that looks similar to that of chrysotile.
 - 13.8.3.2 In plane light, talc is colorless.
 - 13.8.3.3 Talc has higher RIs than chrysotile ($\alpha = 1.538$ to 1.554 , $\gamma = 1.575$ to 1.602), but both are lower than those of other asbestos minerals.
 - 13.8.3.4 Talc has higher birefringence than chrysotile, in the range of 0.03 to 0.05, which gives relatively fine fibers of talc first order white to yellow interference colors under crossed polars. Chrysotile fibers of comparable size would have low first order gray interference colors.
 - 13.8.3.5 Grains of talc display parallel extinction.
- 13.8.4 Wollastonite $CaSiO_3$
 - 13.8.4.1 Wollastonite is one of the pyroxenoid minerals and has a characteristically bladed or prismatic habit.
 - 13.8.4.2 Wollastonite is colorless in plane light.
 - 13.8.4.3 The RIs of wollastonite ($\alpha = 1.616$ to 1.645 , $\gamma = 1.631$ to 1.656) are very close to that of tremolite; however, wollastonite has a lower birefringence (0.013 to 0.017).
 - 13.8.4.4 Wollastonite has an extinction angle of up to approximately five degrees, which makes it easy to confuse with tremolite.
 - 13.8.4.5 Grains of wollastonite can be spun about their long axis until they change from length slow to length fast or vice versa; whereas, grains of tremolite will always remain length slow regardless of their optical orientation.
 - 13.8.4.6 To spin a wollastonite grain about its long axis, agitate the mixture of RI liquid and sample material by repeatedly tapping the cover slip

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- with the point of a ball point pen. Unless the grains are lying flat on one crystal face, they should rotate as the RI liquid is agitated.
- 13.8.5 Kyanite Al_2SiO_5
- 13.8.5.1 Kyanite is an orthosilicate mineral that is commonly used in refractory materials and often has a bladed or columnar habit.
- 13.8.5.2 Kyanite is colorless to light blue in plane light and may display weak pleochroism. Both its color and pleochroism are much more subdued than that of crocidolite.
- 13.8.5.3 Kyanite's RIs are higher than those of both crocidolite and amosite ($\alpha = 1.710$ to 1.718 , $\gamma = 1.724$ to 1.734).
- 13.8.5.4 Birefringence for kyanite ranges from 0.012 to 0.016.
- 13.8.6 Hornblende $(\text{Ca,Na})_{2-3}(\text{Mg,Fe,Al})_5\text{Si}_6(\text{Si,Al})_2\text{O}_{22}(\text{OH})_2$
- 13.8.6.1 Hornblende is one of the most common minerals in the amphibole group and is often found in soils from the Libby Site.
- 13.8.6.2 Hornblende generally has a slender prismatic to bladed habit. Traces of cleavage planes are usually visible within the mineral grains.
- 13.8.6.3 In plane light, hornblende is distinctly colored and pleochroic displaying green, yellow-green, brown, green-brown, or blue-green colors.
- 13.8.6.4 Hornblende's RIs vary greatly with composition ($\alpha = 1.60$ to 1.70 , $\gamma = 1.62$ to 1.73), but most hornblende has $\alpha = 1.645$ to 1.665 and $\gamma = 1.660$ to 1.690 .
- 13.8.6.5 The birefringence of hornblende is moderate, ranging from 0.014 to 0.034, but most falls within 0.018 to 0.028.
- 13.8.6.6 Hornblende can have parallel or inclined extinction depending on optical orientation. When extinction is inclined, the extinction angle is usually 14 to 25 degrees.
- 13.8.7 Calcic Clinopyroxene
- 13.8.7.1 The calcic clinopyroxene group includes Augite, $(\text{Ca,Na})(\text{Mg,Fe,Al})(\text{Si,Al})_2\text{O}_6$, and the end members Diopside, $\text{CaMgSi}_2\text{O}_6$, and Hedenbergite, $\text{CaFeSi}_2\text{O}_6$. These are among the most common pyroxenes, and are often found in soils from the Libby Site.
- 13.8.7.2 The habit of calcic clinopyroxene is usually prismatic to columnar. As a group, the pyroxenes tend to form less slender, elongated grains than the amphiboles. Traces of cleavage planes are usually visible within pyroxene grains.
- 13.8.7.3 In plane light, augite is colorless, pale green, greenish-brown, pale brown, or gray. Diopside is colorless, but as iron content increases through the diopside-hedenbergite, the mineral develops a green color. These minerals may display weak pleochroism.
- 13.8.7.4 As a group, the pyroxenes tend to have high RIs, with calcic clinopyroxene in the range of $\alpha = 1.66$ to 1.75 and $\gamma = 1.69$ to 1.77 .
- 13.8.7.5 The birefringence of calcic clinopyroxene is moderate, as with the majority of other pyroxenes, ranging from 0.018 to 0.034.
- 13.8.7.6 Calcic clinopyroxene can have a very high extinction angle, up to 48 degrees. In grains with high extinction angles, the sign of elongation

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- becomes ambiguous, but is generally length slow for smaller extinction angles.
- 13.8.8 Fiberglass (Amorphous Silica, SiO₂)
- 13.8.8.1 Fiberglass usually occurs as straight, solid, cylindrical tubes with the diameter of the tube varying little along the length of the fibers.
- 13.8.8.2 Most fiberglass is colorless under plane light. However, the addition of impurities can impart various colors to fiberglass, such as yellow, dark brown or dark green.
- 13.8.8.5 The RI of fiberglass varies considerably depending on the composition of the glass (i.e. the addition of impurities, such as aluminum or iron). However, the RI of most fiberglass is close to 1.60.
- 13.8.8.4 Fiberglass is almost always isotropic (appears black at all orientations under crossed polars). Slight interference colors may appear under crossed polars when fiberglass is coated with other minerals or is has been devitrified (partial recrystallization of amorphous silica) due to prolonged exposure to very high temperatures.
- 13.8.9 Cellulose
- 13.8.9.1 The habit of cellulose is often like that of ribbons in which fibers are wider than they are thick. These fibers may be straight, curved, kinked, or crooked. The interiors of cellulose fibers often show a cellular or structured network.
- 13.8.9.2 Cellulose is usually colorless under plane light, although it can be yellow, tan, or brown. Sometimes it has been dyed to various colors, such as red, blue, green, etc.
- 13.8.9.3 Although sometimes similar in appearance to chrysotile, cellulose usually has a higher birefringence.
- 13.8.9.4 Cellulose displays undulatory (incomplete) extinction.
- 13.8.10 Diatoms
- 13.8.10.1 Diatoms are minute organisms that live in both salt and freshwater and secrete shells of amorphous silica. When they die, their shells accumulate to form what is called diatomaceous earth, which is mined and used in a variety of construction materials.
- 13.8.10.2 Fibrous diatoms generally have a cylindrical tube habit, sometimes with tapered ends. Not all diatoms are fibrous, but many are.
- 13.8.10.3 When fibrous diatoms are found in a sample, other diatoms having circular or other various (elliptical, lenticular, etc.) shapes are often found in the same sample.
- 13.8.10.4 Many diatom shells have complex internal structure.
- 13.8.10.5 Because they are made of amorphous silica, diatoms as a rule are isotropic. However, extreme heating or diagenetic processes can lead to de-vitrification, causing some diatoms to become weakly birefringent as a result.
- 13.8.11 Hair
- 13.8.11.1 Hair is usually cylindrical in shape; many fibers of hair are tapered.
- 13.8.11.2 Hair is usually colorless, tan, brown, or red-brown in plane light.
- 13.8.11.3 A central canal is often visible in hair fibers.

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13.8.12 Synthetic Fibers

13.8.12.2 Many synthetic fibers display a cylindrical habit, and typically lack the splayed ends that chrysotile bundles commonly exhibit.

13.8.12.1 Synthetic fibers can be any color, including white, pink, red, purple, blue, green, yellow, etc.

13.8.12.3 Synthetic fibers almost always have high to very high birefringence (0.1 or higher).

13.8.12.4 Many synthetic fibers show parallel extinction.

13.8.12.5 Polyethylene is a synthetic fiber that may be confused with chrysotile due to its wispy habit, which can be displayed by either mineral. However, polyethylene has a higher birefringence than chrysotile, and when placed on a hot plate, polyethylene will melt.

13.8.13 Rutile (TiO₂)

13.8.13.1 Rutile occurs as an accessory mineral in certain types of igneous rocks, and because of its durability and resistance to weathering, it can sometimes be found as very small loose needles in soils. Rutile can sometimes be seen as needles that are inclusions in quartz grains (referred to as rutilated quartz).

13.8.13.2 Rutile generally occurs as small prisms or fine acicular needles.

13.8.13.3 In plane light, rutile can be gray, brown, reddish-brown, or nearly opaque.

13.8.13.4 RIs for rutile are extremely high ($\alpha = 2.6$ to 2.7 , $\gamma = 2.8$ to 2.9).

13.8.13.5 Needles of rutile have high birefringence, are length slow, and show parallel extinction.

14.0 RECORDING DATA AND RESULTS

14.1 Analytical Bench Sheets

14.1.1 Analysts record, by hand, on analytical bench sheets, analytical results at the time the observations are made. Refer to Attachment 1 for one example of a PLM-VE bench sheet.

14.1.1.1 Additional bench sheets may be created by the laboratory as long as all of the required fields are included.

14.1.2 Completed bench sheets are the original, hard-copy records on which test data on client samples is stored.

14.2 Stereomicroscopic Examination Reportables

14.2.1 Homogeneity (Yes or No)

14.2.2 Sample appearance, including color and texture

14.2.3 Estimated percent LA

14.2.4 Estimated percent other asbestos (other amphibole and chrysotile)

14.3 Reporting Positive Asbestos Results

14.3.1 If asbestos is positively identified in the sample during PLM analysis, record the following data for each asbestos type that is present in the sample on the bench

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- sheet.
- 14.3.2 Habit
- 14.3.3 Fiber color in plane light
- 14.3.4 Pleochroism (Yes or No)
- 14.3.5 Indices of refraction (α and γ)
- 14.3.6 Birefringence
 - 14.3.6.1 Low if birefringence is ≤ 0.010 ; medium if birefringence is 0.011 to 0.050; high if birefringence is > 0.050
- 14.3.7 Extinction characteristics (parallel or inclined)
- 14.3.8 Sign of elongation (positive or negative)
- 14.3.9 Qualifier and percentages of the following materials in the sample
 - 14.3.9.1 LA
 - 14.3.9.2 Other amphibole (amosite, anthophyllite, or crocidolite)
 - 14.3.9.3 Chrysotile
- 14.4 Other Reportables
 - 14.4.1 Record the percent non-asbestos fibrous materials, such as fibrous glass, cellulose, synthetic fibers, etc.
 - 14.4.1.1 Record at least one optical property that identifies the material as a non-asbestos fiber (see Section 13.8).
 - 14.4.2 Record the identity of the matrix material(s), if known.
 - 14.4.3 Record if there was any deviation from the SOP or the analytical method.
 - 14.4.4 Record the QC type as Not QC, Laboratory Duplicate – Self-check (LDS), or Laboratory Duplicate – Cross-check (LDC).
 - 14.4.5 Record any pertinent comments.
 - 14.4.6 Sign or initial the bench sheet, and record the date of analysis.

15.0 DATA REPORTING

15.1 EDD Report Generation

- 15.1.1 Results of PLM analyses are provided to the client in an EDD in the form of an Excel spreadsheet.
 - 15.1.1.1 The LADT is a Laboratory Information Management System (LIMS) specifically designed to generate EDDs that meet all of the current client data reporting requirements, as well as minimize data entry errors. The EDD generated by the LADT is intended to replace the Libby EDDs used in previous years.
 - 15.1.1.3 It is the responsibility of the laboratory to check with the client that they are using the most recent version of the LADT.
 - 15.1.1.3 Laboratories can elect to generate their own EDDs rather than use the LADT; however, their EDDs must meet all of the current client data reporting requirements.
 - 15.1.1.2 Laboratories that do elect to use the LADT will receive the LADT User's Manual, which includes installation and data entry instructions.
- 15.1.2 After generating an EDD, save the file electronically.
 - 15.1.2.1 The EDD file name is generated automatically by the LADT.

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- 15.1.2.2 If a laboratory does not use the LADT to generate the EDD, they must use the following naming convention to name their EDD files:

Laboratory ID_Work Order Number_Analytical Method_Correction Number
Example: ESATR8_0920120002_PLM-VE_C0
- 15.1.3 The EDD serves as an electronic version of the test report submitted to the client.
 - 15.1.3.1 Only one EDD is produced for each chain of custody (COC) received by the laboratory.
 - 15.1.3.2 A hardcopy of the test report is also delivered to the client (see Section 15.2 for further details about hardcopy test reports).
 - 15.1.3.3 The laboratory retains all original records until otherwise instructed by the client.
- 15.2 Test Report Generation
 - 15.2.1 Hardcopy test reports of the raw analytical data are submitted to the client for archival.
 - 15.2.2 A completed test report consists of a cover sheet signed and dated by an approved signatory, as well as the following information and documentation:
 - 15.2.2.1 The laboratory work order number, COC number, number of samples received, and copies of the signed COCs.
 - 15.2.2.1.1 A work order number is a unique number assigned by the laboratory to a set of samples from a single COC. Work order numbers are never duplicated.
 - 15.2.2.2 The date of sample receipt and condition of samples.
 - 15.2.2.3 A Case Narrative, including any opinions and interpretations; deviations, modifications, additions to, or exclusions from the test method; descriptions of any problems encountered in the analysis; or any specific conditions that could affect the results. Also include the following disclaimer: "This test report relates only to items tested."
 - 15.2.2.4 PLM-VE Analysis Results, as presented in the EDD and containing the analytical data (including all LDC and LDS analyses performed on any samples in the work order).
 - 15.2.2.5 Copies of the handwritten bench sheets containing the analyst's original data and observations.
 - 15.2.3 Refer to Attachment 3, the Analytical Test Report Standard Laboratory Data Package Checklist, for a complete list of items required for each test report.
 - 15.2.4 When opinions and interpretations are provided in a test report, the laboratory will:
 - 15.2.4.1 Document the basis on which the opinions and interpretations were made.
 - 15.2.4.2 Clearly indicate on the test report which items are opinions and interpretations.
 - 15.2.5 Once the test report is complete, all pages must be paginated prior to delivery to the client.

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15.3 Delivery of Results to Client

- 15.3.1 The following items will be submitted electronically (via e-mail) to the client:
 - 15.3.1.1 The completed EDD containing the analytical data. This spreadsheet is presented in a format that can be imported into the client's data management software.
 - 15.3.1.2 A scanned .pdf of the completed test report as described above. All signatures must be originals, or if electronic signatures are used, the e-signature must be controlled by a password-protected login that allows its application only by the signer.
 - 15.3.1.3 The two above files are e-mailed to the client, including all parties on the distribution list submitted by the client to the laboratory.
- 15.3.2 Once the results of a work order number have been delivered to the client, the hardcopy test report is retained until further instruction by the client.

16.0 QUALITY ASSURANCE AND QUALITY CONTROL

16.1 General

- 16.1.1 The laboratory must operate under a quality system appropriate to the type, range, and volume of testing work that it performs.
- 16.1.2 Results of QC analyses are used to track the precision and accuracy of the laboratory's analyses and to identify areas that require or could benefit from improvement.
- 16.1.3 The following types of QC analyses are performed on a scheduled basis at the laboratory:
 - 16.1.3.1 Re-analysis of client samples by the same analyst (LDS) or by a different analyst (LDC)
 - 16.1.3.2 Routine analyses on calibration standards of known asbestos concentration
 - 16.1.3.3 NIST proficiency testing
 - 16.1.3.4 Inter-laboratory analyses (also referred to as Round Robin analyses)
- 16.1.4 Records must be kept of all QA documentation.
- 16.1.5 All QC analyses must be performed in real-time.

16.2 Calibration Standards

- 16.2.1 Visual estimates of asbestos concentrations are calibrated with the use of the calibration standards.
- 16.2.2 The calibration standards are a set of permanently mounted slides of known asbestos concentrations. They should cover a wide range of asbestos concentrations.
- 16.2.3 Reference materials used to prepare calibration standards are NIST SRM's 1866b and 1867a, Controlled PE Reference Materials, and samples from past NIST proficiency testing rounds.
 - 16.2.3.1 Controlled PE Reference Materials at concentrations of 0.2% and 1.0% LA in soils are required to delineate between the bin assignments; however, those concentrations, as well as

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concentrations of 0.5% and 2.0%, are useful for the calibration of visual area estimates for low end samples.

- 16.2.3.2 "Working standard" refers to any calibration standard that was prepared internally at the laboratory. Laboratories are encouraged to prepare these standards over a range of asbestos concentrations. These slides should not just be prepared of LA but for other asbestos types as well.

16.3 Use of Calibration Standards for Precision and Accuracy Testing

- 16.3.1 The best way to track analyst precision and accuracy is by the analysis of standards of known asbestos concentration.
- 16.3.1.1 All analysts need to analyze calibration standards on a regular basis.
- 16.3.1.2 Calibration standards should be read at a minimum frequency of one per 100 client samples.
- 16.3.2 Vary the calibration standards read each month so that analysts are constantly presented with standards of different asbestos concentrations, various asbestos types, and various matrix material types.
- 16.3.3 The analysts must be blind to the known values of the calibration standards so as to prevent biased results.
- 16.3.4 The laboratory should designate someone other than the analyst performing the test to review the results for acceptability.
- 16.3.5 After completion of analyses of calibration standards, analysts are advised of the reference values of the standards so they can see how they performed and calibrate their readings on client samples accordingly.
- 16.3.6 Repeated analysis of the calibration standards provides a benchmark upon which analysts can base their visual estimations of percentage levels of asbestos in client samples. Use of control charts for concentrations 1% or greater is recommended.
- 16.3.7 Corrective action(s) must be taken immediately if the results of reading calibration standards do not meet acceptance criteria. Examples of corrective actions that may be taken are re-analysis of calibration standards, re-preparation of calibration standards, and analyst re-training.
- 16.3.8 Analyses of the calibration standards are not reported as part of an EDD or test report. Rather, laboratories are responsible for maintaining an internal system for tracking analyses of calibration standards.

16.4 LDS and LDC QC Analyses (Duplicates and Replicates)

- 16.4.1 For all Libby samples received by the laboratory, a minimum of 10% must be re-analyzed within the laboratory.
- 16.4.2 A QC analysis (LDS or LDC) can be performed on any sample.
- 16.4.2.1 QC analyses need to be performed on samples over the entire range of asbestos concentrations that are encountered in site samples.
- 16.4.2.2 Any sample that is considered especially unusual or difficult should be re-analyzed for QC purposes.
- 16.4.3 The frequency of LDS analyses on client samples will be 2 per 100 samples analyzed (2%). LDS analyses are performed as a remount of the sample (see

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- Section 13.3 for slide preparation procedures). All five slide mounts of the remounted sample should be analyzed by the original analyst and the results recorded on the analytical bench sheet as an LDS QC sample.
- 16.4.4 The frequency of LDC analyses on client samples will be 8 per 100 samples analyzed (8%). LDC analyses are performed on the five original slide preparations by an analyst other than the original and the results recorded on the analytical bench sheet as an LDC QC sample.
- 16.4.4.1 All analysts performing QC analyses must be experienced with PLM analysis of soil samples from the Libby Site and the specific requirements of this SOP.
- 16.4.4.2 If there is only one primary analyst at the laboratory performing PLM analysis on these samples, the laboratory must send all LDC QC samples to another Libby laboratory with the proper experience and qualifications.
- 16.4.5 For samples containing LA, LDS and LDC analyses are considered acceptable if results are within one bin category (i.e., ± 1 bin) of the original analysis and the %LA for both the original and QC analyses is $\leq 1\%$. For samples containing $>1\%$ LA, laboratories should defer to their own internal QA/QC system (such as control charting or similar tool) to determine QC acceptance criteria.
- 16.4.6 For samples containing all other asbestos types, LDS and LDC analyses are considered acceptable if both the original and QC analyses are $<1\%$ asbestos. If the original and QC analysis result is $\geq 1\%$ asbestos, laboratories should defer to their own internal QA/QC system (such as control charting or similar tool) to determine QC acceptance criteria.
- 16.4.6 Corrective action(s) must be taken immediately if LDS and LDC analyses do not meet acceptance criteria. Examples of corrective actions that may be taken are re-analysis and/or re-preparation and re-analysis of original and duplicate or replicate samples, analyst re-training, and notification of the client.
- 16.4.7 When performing a QC analysis, it is necessary to mark LDS or LDC in the "QC Type" section of the bench sheet.

16.5 Inter-Laboratory Analyses

- 16.5.1 The laboratory is involved in an ongoing sample exchange program with other PLM laboratories that analyze soil samples from the Libby Site. The purpose of this program is to help detect and minimize laboratory biases and unnecessary variance in results, as well as to characterize precision across laboratories performing PLM-VE testing.
- 16.5.2 The frequency of the inter-laboratory sample exchange ranges from 1 in 100 samples analyzed exchanged amongst laboratories on a quarterly basis. However, higher frequencies of inter-laboratory sample analysis are required when a laboratory is new to the program, when systematic errors or biases are observed, or when a new version of the SOP is distributed. Whether or not the frequency to be performed is the minimum or higher is determined by the client.
- 16.5.3 Results of the inter-laboratory analyses are reviewed by the client.
- 16.5.4 The inter-laboratory analysis result is considered acceptable if it is within one bin category (i.e., ± 1 bin) of the original analysis for reported concentrations of $\leq 1\%$ LA. If both the original and inter-laboratory result is $>1\%$ LA, acceptance of the

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- inter-laboratory analysis will be determined by the client.
- 16.5.5 Corrective action(s) must be taken immediately if analyses do not meet acceptance criteria. The specific course of action based on these results will be determined by the client. Common actions include re-analysis and/or re-preparation and re-analysis of original and duplicate or replicate samples, collaboration between and amongst laboratories performing the test to root out biases and/or variances, and analyst re-training.

17.0 REFERENCES

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LIBBY ASBESTOS SUPERFUND SITE STANDARD OPERATING PROCEDURE
APPROVED FOR USE AT THE LIBBY ASBESTOS SUPERFUND SITE ONLY

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ATTACHMENT 1

Libby Asbestos Superfund Site Analysis Bench Sheet (PLM-VE)

Laboratory Name:
 Work Order No.:
 SOP: SRC-LIBBY-03 (REV 3)

**LIBBY ASBESTOS SUPERFUND SITE
 ANALYSIS BENCH SHEET (PLM-VE)**

STEREOMICROSCOPIC
 EXAMINATION

ASBESTOS MINERALS OBSERVED

ASBESTOS OPTICAL PROPERTIES

OTHER

Client Sample No.	Tag	Lab Sample ID	Date Analyzed	Analyst Initials	Homogeneity	Sample Color	Sample Type/Texture ¹	Est. % LA	Est. % Other Asbestos	LA-Qual	LA-%	OA-Qual	OA-AF %	OA Type	CH-Qual	CH-AF %	Habit	Fiber Color	Sign of Elongation	Pleochroism	Extinction Angle	Ref. Index (α)	Ref. Index (γ)	Birefringence	RI Determined By	Temperature (C°)	Type and % of Non-Asbestos Fibers (w/ optical properties ²)	Non-Fibrous Matrix Materials (if known) ³	QC Type	Deviation	Comments ⁴
					Yes No	Tan Brown Gray	SF SC			ND Tr < DET		ND < DET		AMOS CROC ANTH	ND < DET		FB P ST		POS NEG	Yes No	I P			L M H	BL DS		___ CELL ___ ___ FBGL ___ ___ SYN ___ ___ OTHR ___	S C O Q F M	Not QC LDC LDS	Yes No	1 2 3 4
					Yes No	Tan Brown Gray	SF SC			ND Tr < DET		ND < DET		AMOS CROC ANTH	ND < DET		FB P ST		POS NEG	Yes No	I P			L M H	BL DS		___ CELL ___ ___ FBGL ___ ___ SYN ___ ___ OTHR ___	S C O Q F M	Not QC LDC LDS	Yes No	1 2 3 4
					Yes No	Tan Brown Gray	SF SC			ND Tr < DET		ND < DET		AMOS CROC ANTH	ND < DET		FB P ST		POS NEG	Yes No	I P			L M H	BL DS		___ CELL ___ ___ FBGL ___ ___ SYN ___ ___ OTHR ___	S C O Q F M	Not QC LDC LDS	Yes No	1 2 3 4
					Yes No	Tan Brown Gray	SF SC			ND Tr < DET		ND < DET		AMOS CROC ANTH	ND < DET		FB P ST		POS NEG	Yes No	I P			L M H	BL DS		___ CELL ___ ___ FBGL ___ ___ SYN ___ ___ OTHR ___	S C O Q F M	Not QC LDC LDS	Yes No	1 2 3 4
					Yes No	Tan Brown Gray	SF SC			ND Tr < DET		ND < DET		AMOS CROC ANTH	ND < DET		FB P ST		POS NEG	Yes No	I P			L M H	BL DS		___ CELL ___ ___ FBGL ___ ___ SYN ___ ___ OTHR ___	S C O Q F M	Not QC LDC LDS	Yes No	1 2 3 4
					Yes No	Tan Brown Gray	SF SC			ND Tr < DET		ND < DET		AMOS CROC ANTH	ND < DET		FB P ST		POS NEG	Yes No	I P			L M H	BL DS		___ CELL ___ ___ FBGL ___ ___ SYN ___ ___ OTHR ___	S C O Q F M	Not QC LDC LDS	Yes No	1 2 3 4
					Yes No	Tan Brown Gray	SF SC			ND Tr < DET		ND < DET		AMOS CROC ANTH	ND < DET		FB P ST		POS NEG	Yes No	I P			L M H	BL DS		___ CELL ___ ___ FBGL ___ ___ SYN ___ ___ OTHR ___	S C O Q F M	Not QC LDC LDS	Yes No	1 2 3 4
					Yes No	Tan Brown Gray	SF SC			ND Tr < DET		ND < DET		AMOS CROC ANTH	ND < DET		FB P ST		POS NEG	Yes No	I P			L M H	BL DS		___ CELL ___ ___ FBGL ___ ___ SYN ___ ___ OTHR ___	S C O Q F M	Not QC LDC LDS	Yes No	1 2 3 4
					Yes No	Tan Brown Gray	SF SC			ND Tr < DET		ND < DET		AMOS CROC ANTH	ND < DET		FB P ST		POS NEG	Yes No	I P			L M H	BL DS		___ CELL ___ ___ FBGL ___ ___ SYN ___ ___ OTHR ___	S C O Q F M	Not QC LDC LDS	Yes No	1 2 3 4

¹ SF = Soil/Fine, SC = Soil/Coarse; ² I = Isotropic, H = Habit, B = High Birefringence, U = Undulatory Extinction, RI = Refractive Index, O = Opaque, S = Sign of Elongation, P = Parallel Extinction; ³ S = Sand, C = Clay, O = Opaques, Q = Quartz, F = Feldspar, M = Mica; ⁴ 1 = Only prismatic LA observed, 2 = LA grain has striations, 3 = Some fibers length fast, 4 = Not analyzed per client request

ATTACHMENT 2

RI Liquid Calibration Conversion Tables

λ_m (nm)	1.550 (Cargille Series E)					1.615 (Cargille Series E)				
	Cargille Glass 1.55 (Lot B or C)					Cargille Glass 1.62 (Lot C)				
	<i>21°C</i>	<i>23°C</i>	<i>25°C</i>	<i>27°C</i>	<i>29°C</i>	<i>21°C</i>	<i>23°C</i>	<i>25°C</i>	<i>27°C</i>	<i>29°C</i>
400	1.520	1.521	1.522	1.523	1.524	1.602	1.603	1.604	1.605	1.606
420	1.526	1.527	1.528	1.529	1.529	1.605	1.606	1.607	1.608	1.609
440	1.530	1.531	1.532	1.533	1.534	1.607	1.608	1.609	1.610	1.611
460	1.534	1.535	1.536	1.537	1.538	1.610	1.611	1.611	1.612	1.613
480	1.537	1.538	1.539	1.540	1.541	1.611	1.612	1.613	1.614	1.615
500	1.540	1.541	1.542	1.543	1.544	1.613	1.614	1.615	1.616	1.617
520	1.543	1.544	1.545	1.546	1.546	1.614	1.615	1.616	1.617	1.618
540	1.545	1.546	1.547	1.548	1.549	1.616	1.617	1.617	1.618	1.619
560	1.547	1.548	1.549	1.550	1.551	1.617	1.618	1.619	1.619	1.620
580	1.548	1.549	1.550	1.551	1.552	1.618	1.619	1.620	1.620	1.621
589	1.549	1.550	1.551	1.552	1.553	1.618	1.619	1.620	1.621	1.622
600	1.550	1.551	1.552	1.553	1.554	1.619	1.620	1.620	1.621	1.622
620	1.551	1.552	1.553	1.554	1.555	1.619	1.620	1.621	1.622	1.623
640	1.553	1.554	1.555	1.556	1.557	1.620	1.621	1.622	1.623	1.624
660	1.554	1.555	1.556	1.557	1.558	1.621	1.622	1.623	1.624	1.624
680	1.555	1.556	1.557	1.558	1.559	1.621	1.622	1.623	1.624	1.625
700	1.556	1.557	1.558	1.559	1.560	1.622	1.623	1.624	1.625	1.626
750	1.558	1.559	1.560	1.561	1.562	1.623	1.624	1.625	1.626	1.627
800	1.560	1.561	1.562	1.563	1.564	1.624	1.625	1.626	1.627	1.628

λ_m (nm)	1.605 (Cargille Series E)									
	Cargille Glass 1.60 (Lot B)					Cargille Glass 1.61 (Lot D)				
	<i>21°C</i>	<i>23°C</i>	<i>25°C</i>	<i>27°C</i>	<i>29°C</i>	<i>21°C</i>	<i>23°C</i>	<i>25°C</i>	<i>27°C</i>	<i>29°C</i>
400	1.583	1.584	1.585	1.586	1.587	1.584	1.585	1.586	1.586	1.587
420	1.586	1.587	1.588	1.589	1.590	1.589	1.589	1.590	1.591	1.592
440	1.589	1.590	1.591	1.592	1.592	1.592	1.593	1.594	1.595	1.596
460	1.591	1.592	1.593	1.594	1.594	1.596	1.597	1.597	1.598	1.599
480	1.593	1.594	1.595	1.595	1.596	1.599	1.599	1.600	1.601	1.602
500	1.594	1.595	1.596	1.597	1.598	1.601	1.602	1.603	1.604	1.605
520	1.596	1.597	1.597	1.598	1.599	1.603	1.604	1.605	1.606	1.607
540	1.597	1.598	1.599	1.600	1.600	1.605	1.606	1.607	1.608	1.609
560	1.598	1.599	1.600	1.601	1.601	1.607	1.608	1.608	1.609	1.610
580	1.599	1.600	1.601	1.602	1.602	1.608	1.609	1.610	1.611	1.612
589	1.599	1.600	1.601	1.602	1.603	1.609	1.610	1.611	1.612	1.612
600	1.600	1.601	1.602	1.602	1.603	1.610	1.610	1.611	1.612	1.613
620	1.601	1.601	1.602	1.603	1.604	1.611	1.612	1.613	1.613	1.614
640	1.601	1.602	1.603	1.604	1.605	1.612	1.613	1.614	1.615	1.615
660	1.602	1.603	1.604	1.605	1.605	1.613	1.614	1.615	1.616	1.616
680	1.602	1.603	1.604	1.605	1.606	1.614	1.615	1.616	1.617	1.617
700	1.603	1.604	1.605	1.606	1.607	1.615	1.616	1.616	1.617	1.618
750	1.604	1.605	1.606	1.607	1.608	1.617	1.617	1.618	1.619	1.620
800	1.605	1.606	1.607	1.608	1.609	1.618	1.619	1.620	1.621	1.622

1.635 (Cargille Series E)						1.640 (Cargille Series E)				
λ_m (nm)	Cargille Glass 1.64 (Lot B)					Cargille Glass 1.64 (Lot B)				
	$21^\circ C$	$23^\circ C$	$25^\circ C$	$27^\circ C$	$29^\circ C$	$21^\circ C$	$23^\circ C$	$25^\circ C$	$27^\circ C$	$29^\circ C$
	400	1.612	1.613	1.614	1.615	1.616	1.611	1.611	1.612	1.613
420	1.617	1.618	1.619	1.620	1.621	1.616	1.617	1.618	1.619	1.620
440	1.622	1.623	1.624	1.625	1.626	1.621	1.622	1.623	1.624	1.625
460	1.626	1.627	1.627	1.628	1.629	1.625	1.626	1.627	1.628	1.629
480	1.629	1.630	1.631	1.632	1.633	1.628	1.629	1.630	1.631	1.632
500	1.632	1.633	1.634	1.634	1.635	1.631	1.632	1.633	1.634	1.635
520	1.634	1.635	1.636	1.637	1.638	1.634	1.635	1.636	1.637	1.637
540	1.636	1.637	1.638	1.639	1.640	1.636	1.637	1.638	1.639	1.640
560	1.638	1.639	1.640	1.641	1.642	1.638	1.639	1.640	1.641	1.642
580	1.640	1.641	1.642	1.643	1.644	1.640	1.641	1.642	1.643	1.644
589	1.641	1.642	1.643	1.643	1.644	1.641	1.642	1.643	1.643	1.644
600	1.642	1.642	1.643	1.644	1.645	1.642	1.642	1.643	1.644	1.645
620	1.643	1.644	1.645	1.646	1.647	1.643	1.644	1.645	1.646	1.647
640	1.644	1.645	1.646	1.647	1.648	1.644	1.645	1.646	1.647	1.648
660	1.645	1.646	1.647	1.648	1.649	1.646	1.647	1.647	1.648	1.649
680	1.646	1.647	1.648	1.649	1.650	1.647	1.648	1.649	1.649	1.650
700	1.647	1.648	1.649	1.650	1.651	1.648	1.649	1.650	1.651	1.651
750	1.650	1.650	1.651	1.652	1.653	1.650	1.651	1.652	1.653	1.654
800	1.651	1.652	1.653	1.654	1.655	1.652	1.653	1.654	1.655	1.656

1.680 (Cargille Series B)										
λ_m (nm)	Cargille Glass 1.68 (Lot A)					Cargille Glass 1.68 (Lot B or C)				
	$21^\circ C$	$23^\circ C$	$25^\circ C$	$27^\circ C$	$29^\circ C$	$21^\circ C$	$23^\circ C$	$25^\circ C$	$27^\circ C$	$29^\circ C$
	400	1.633	1.634	1.635	1.636	1.637	1.634	1.635	1.636	1.637
420	1.641	1.642	1.643	1.644	1.645	1.642	1.643	1.644	1.645	1.646
440	1.648	1.649	1.650	1.651	1.652	1.649	1.650	1.650	1.651	1.652
460	1.653	1.654	1.655	1.656	1.657	1.654	1.655	1.656	1.657	1.658
480	1.658	1.659	1.660	1.661	1.662	1.659	1.660	1.661	1.662	1.663
500	1.662	1.663	1.664	1.665	1.666	1.663	1.664	1.665	1.666	1.667
520	1.666	1.667	1.668	1.669	1.670	1.667	1.668	1.668	1.669	1.670
540	1.669	1.670	1.671	1.672	1.673	1.670	1.671	1.672	1.673	1.674
560	1.672	1.673	1.674	1.675	1.676	1.673	1.673	1.674	1.675	1.676
580	1.674	1.675	1.676	1.677	1.678	1.675	1.676	1.677	1.678	1.679
589	1.675	1.676	1.677	1.678	1.679	1.676	1.677	1.678	1.679	1.680
600	1.677	1.677	1.678	1.679	1.680	1.677	1.678	1.679	1.680	1.681
620	1.679	1.680	1.680	1.681	1.682	1.679	1.680	1.681	1.682	1.683
640	1.680	1.681	1.682	1.683	1.684	1.681	1.682	1.683	1.684	1.685
660	1.682	1.683	1.684	1.685	1.686	1.683	1.684	1.685	1.686	1.687
680	1.684	1.685	1.686	1.687	1.688	1.685	1.685	1.686	1.687	1.688
700	1.685	1.686	1.687	1.688	1.689	1.686	1.687	1.688	1.689	1.690
750	1.688	1.689	1.690	1.691	1.692	1.689	1.690	1.691	1.692	1.693
800	1.691	1.692	1.693	1.694	1.695	1.692	1.693	1.694	1.695	1.696

1.700 (Cargille Series B)					
λ_m (nm)	Cargille Glass 1.70 (Lot B or D)				
	<i>21°C</i>	<i>23°C</i>	<i>25°C</i>	<i>27°C</i>	<i>29°C</i>
400 #	1.664	1.665	1.666	1.667	
420 #	1.671	1.672	1.673	1.674	
440 #	1.677	1.678	1.679	1.680	
460 #	1.682	1.682	1.683	1.684	
480 #	1.686	1.687	1.688	1.689	
500 #	1.689	1.690	1.691	1.692	
520 #	1.693	1.694	1.694	1.695	
540 #	1.695	1.696	1.697	1.698	
560 #	1.698	1.699	1.700	1.701	
580 #	1.700	1.701	1.702	1.703	
589 #	1.701	1.702	1.703	1.704	
600 #	1.702	1.703	1.704	1.705	
620 #	1.704	1.705	1.706	1.707	
640 #	1.706	1.706	1.707	1.708	
660 #	1.707	1.708	1.709	1.710	
680 #	1.708	1.709	1.710	1.711	
700 #	1.710	1.711	1.712	1.713	
750 #	1.712	1.713	1.714	1.715	
800 #	1.715	1.716	1.717	1.718	

LIBBY ASBESTOS SUPERFUND SITE STANDARD OPERATING PROCEDURE
APPROVED FOR USE AT THE LIBBY ASBESTOS SUPERFUND SITE ONLY

ANALYSIS OF ASBESTOS FIBERS IN FINE SOIL BY POLARIZED LIGHT MICROSCOPY

Date: July 27, 2012

SOP No.: SRC-LIBBY-03 (Revision 3)

ATTACHMENT 3

Analytical Test Report Standard Laboratory Data Package Checklist

ANALYTICAL TEST REPORT

Bulk Asbestos Analysis by PLM-VE

Prepared For:

Address: _____

Laboratory Name:

Address: _____

Report Reviewed by: _____

_____ Date

Standard Laboratory Data Package Checklist

Instructions: For Analytical Test Reports, complete the following checklist and attach supporting documentation as outlined below.

- 1 Laboratory Job No.:
- 2 Chain of Custody No.:
- 3 Date of sample receipt:
- 4 Number of samples received:
- 5 Analytical Method:
- 6 Method SOP:
- 7 SAP Analytical Summary No.:
- 8 Test Report Correction No.:
- 9 Condition of samples:
- 10 Technical Direction Form No.:
- 11 Attachments:
 - Chain of Custody form(s)*
 - Case Narrative and any modification forms*
 - Analysis Results*
 - Analytical Bench Sheet(s)*

Verification:

Laboratory and Validator Verification signifies that all laboratory QA/QC tasks were performed for the samples in this Laboratory Job Number and that this Analytical Test Report is accurate and complete. Laboratory Verification is done by the person who performed data entry of the test results and Validator Verification is done by the person who performed the QC check of the data entry.

Laboratory Verification (Initials and Date) _____

Validator Verification (Initials and Date) _____

ATTACHMENT 4

Optical Properties of Fibrous Amphiboles Associated with Libby Amphibole

OPTICAL PROPERTIES OF FIBROUS AMPHIBOLES ASSOCIATED WITH LIBBY AMPHIBOLE^A

Libby Amphibole (LA) is a term used to categorize a group of minerals generally described as sodic tremolite. The solid solution series of sodic tremolite is comprised of the following group of minerals: tremolite, actinolite, winchite, richterite, magnesio-riebeckite, and magnesio-arfvedsonite. The optical properties for each individual mineral are provided below. There is a great deal of overlap in optical properties among the minerals that make up LA. As such, discreet mineral identification is not required in SOP SRC-LIBBY-03 (Revision 3). Rather, if a grain in the sample exhibits the optical properties of a mineral listed below, the specific optical properties will be recorded on the analytical bench sheet and reported on the Electronic Data Deliverable (EDD) and test report file, and the grain identified as LA.

Mineral	Habit and Color	Refractive Indices		Birefringence	Extinction	Elongation Sign
		α	γ			
Tremolite ⁷	Straight to curved fibers and bundles. Colorless to pale green.	1.600-1.628	1.625-1.655	0.017-0.028	Oblique (up to 21 °);	+ (length slow)
Actinolite ⁷		1.604-1.612	1.627-1.635			
		1.599-1.612	1.625-1.637			
		1.6063	1.6343			
		1.600-1.628	1.625-1.655	0.017-0.028		+ (length slow)
		1.612-1.668	1.635-1.688			
		1.613-1.628	1.638-1.655			
		1.6126	1.6393			
Winchite	Straight to curved fibers or bundles. Colorless to pale blue Pleochroism weak to moderate: X=colorless, Y=light blue-violet, Z=light blue ³	1.618-1.626 ¹	1.634 ¹	0.008-0.019 ¹	Oblique, 22 ^{o1} 15.8 ^{o2} Oblique, 7- 29 ^{o8}	+ (length slow)
		1.618-1.621 ²	1.642 ¹	0.016 ²		
		1.629 ³	1.634 ¹	0.021 ³		
		1.636 ⁴	1.637 ²	0.022 ⁴		
			1.650 ³			
			1.658 ⁴			
Richterite	Straight to curved fibers or bundles. Colorless, pale yellow, brown, pale to dark green, or violet ⁸ Pleochroism weak to strong in pale yellow, orange, and red ⁵	1.622-1.623 ¹	1.638 ¹	0.012-0.017 ¹ 0.017-0.022 ⁵	Oblique, 21 - 22 ^{o1} Oblique, 5- 45 ^{o8}	+ (length slow)
		1.605-1.624 ⁵	1.639 ¹			
		1.615 ⁶	1.627-			
			1.641 ⁵			
			1.636 ⁶			
Magnesio-riebeckite	Prismatic to fibrous aggregates. Blue, grey-blue, pale blue to yellow. Can be pleochroic. ⁸	1.650-1.673 ⁸	1.662-	Up to 0.015 ⁸	Oblique, 8- 40 ^{o8}	- (length fast) ⁸
Magnesio-arfvedsonite	Prismatic to fibrous aggregates. Yellowish green, brownish green, or grey-blue. Can be pleochroic. ⁸	1.623-1.660 ⁸	1.635-	0.012-0.026 ⁸	Oblique, 18- 45 ^{o8}	- (length fast) ⁸

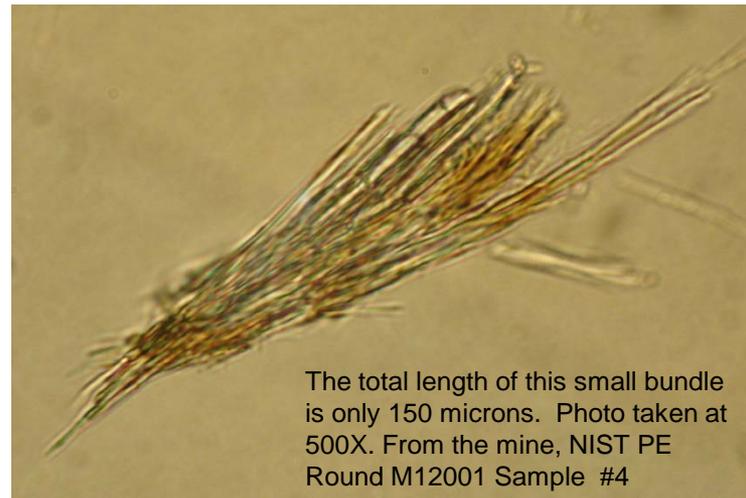
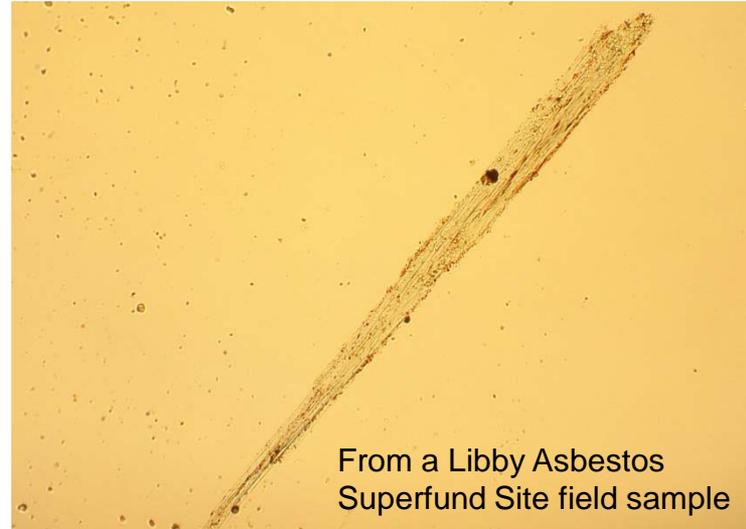
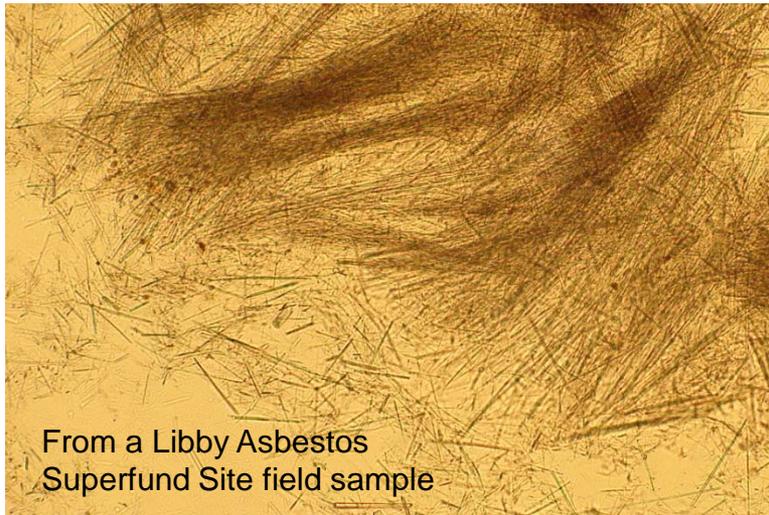
A. This table is adapted for use in SOP SRC-LIBBY-03 (Revision 3) from: Su, Shu-Chun, 2005. White paper: *Tables to Facilitate the Determination of Refractive Indices of Winchite and Richterite, (Libby, Montana) by Dispersion Staining*, August 8, 2005. Data on this table were compiled from data of amphiboles from Libby, Montana and other localities. The data in **bold** are samples from Libby, Montana. The data for tremolite/actinolite are adapted from Table 2-2 of EPA/600/R-93/116.

1. Bandli, B.R. et al. (2003) *Optical, compositional, morphological, and X-ray data on eleven particles of amphibole from Libby, Montana, U.S.A.* Canadian Mineralogist, 41, 1241-1253.
2. Wylie, A.G. and Verkouteren, J.R. (2000) *Amphibole asbestos from Libby, Montana: Aspects of nomenclature.* American Mineralogist, 85, 1540-1542.
3. www.minsocam.oeg/msa/Handbook/Winchite.PDF.
4. www.mindat.org/min-4296.html.
5. www.minsocam.oeg/msa/Handbook/Richterite.PDF.
6. www.webmineral.com/data/Richterite.shtml.
7. Adapted from: USEPA 1993. *Method for the Determination of Asbestos in Bulk Building Materials*. July 1993. (NTIS / PB93-218576).
8. W. A. Deer, R. A. Howie, and J. Zussman (1997). *Rock Forming Minerals Volume 2B: Double Chain Silicates, 2nd Edition*. The Geological Society, London. Optical properties for magnesio-riebeckite and magnesio-arfvedsonite inserted by Douglas Kent at ESAT Region 8, October 2008.

ATTACHMENT 5

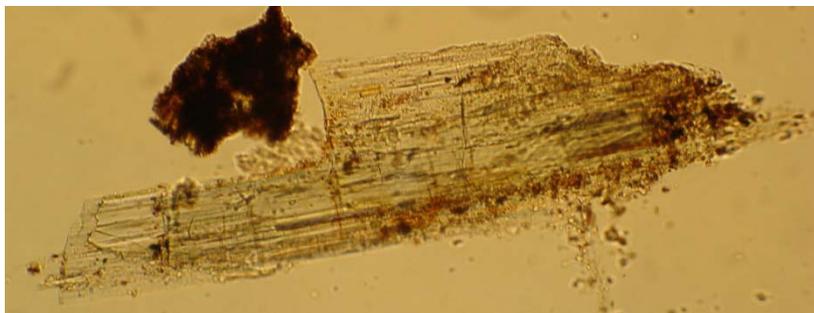
PLM Photomicrographs Demonstrating a Wide Range of LA Habits

PLM Photomicrographs Demonstrating a Wide Range of Libby Amphibole Habits



Prismatic Libby Amphibole

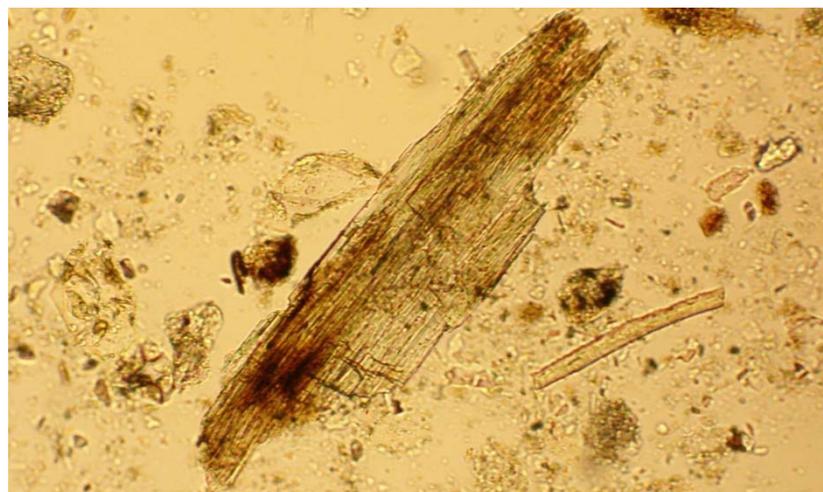
The optical properties are the same as they are for more fibrous forms of LA. Colors of winchite, richterite, tremolite, and actinolite are generally much paler than those of hornblende, which is usually dark green to dark blue-green to brownish green. Hornblende also has higher refractive indices (in the range of 1.65 to 1.68) than A.



From a Libby Asbestos Superfund Site field sample

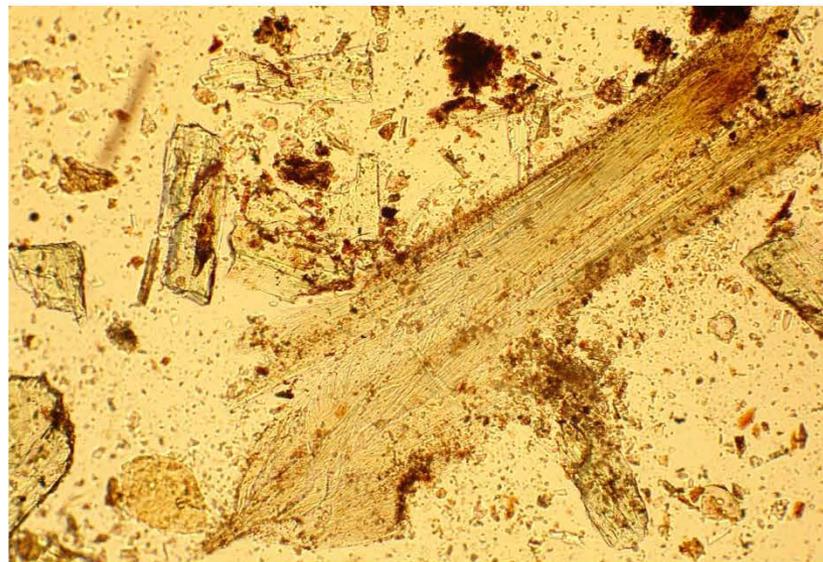


From the mine, NIST PE Round M12001 Sample #4

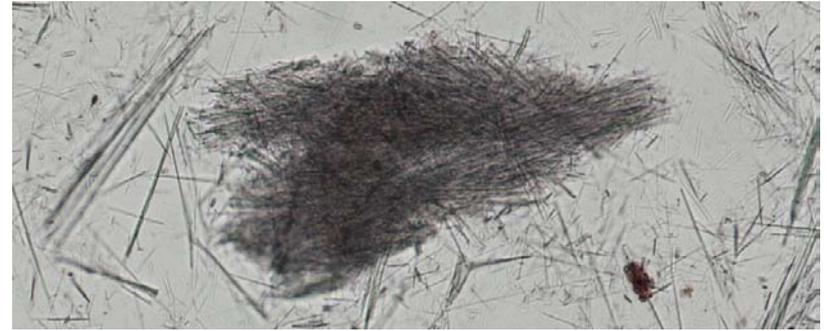


From a Libby Asbestos Superfund Site field sample

Some LA shows a “matted” or “felted” habit. The internal structure of these bundles is still fibrous. The green high-relief prismatic grains in the top right photo are hornblende. The bundles in the two top photos were found in Libby Asbestos Superfund Site field samples. The bundles in the lower two photos are from the NIST PE Round M12001 Sample #4, from the mine.

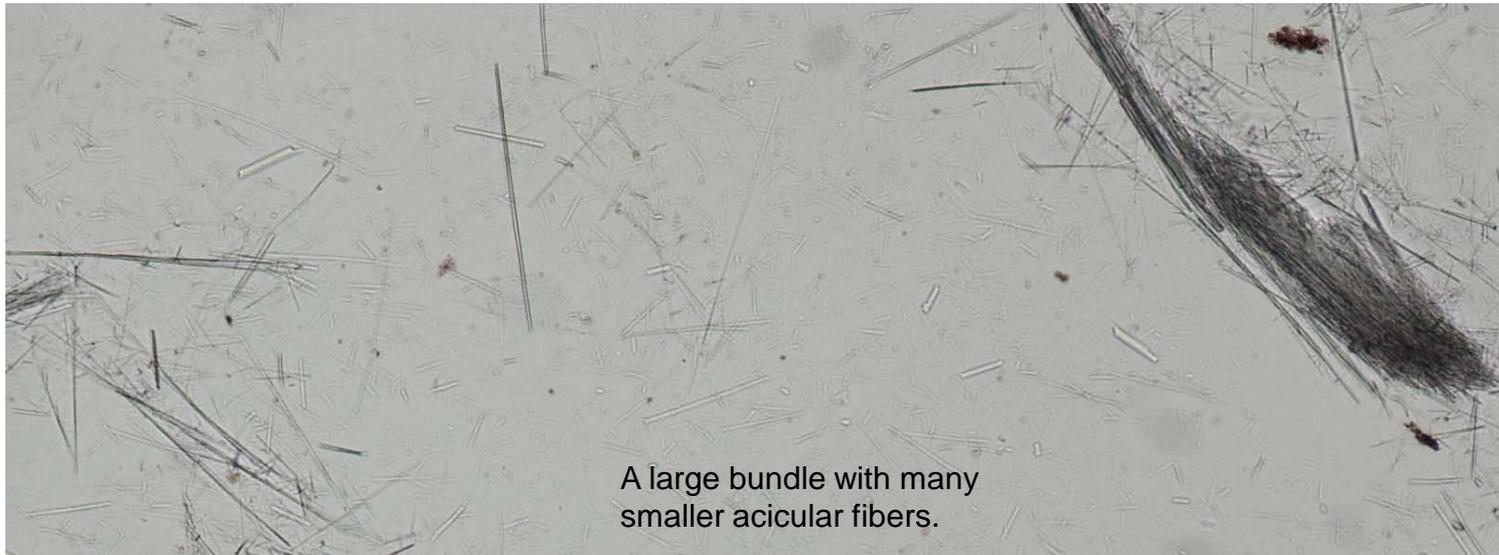


SOP SRC-LIBBY-03 (Revision 3)
For use at the Libby Asbestos Superfund Site only



A “felted” bundle plus some smaller acicular fibers. The photos on this page are all of bundles found in field samples collected from the Libby Asbestos Superfund Site.

The fibers on the right side of this bundle are completely matted.



A large bundle with many smaller acicular fibers.

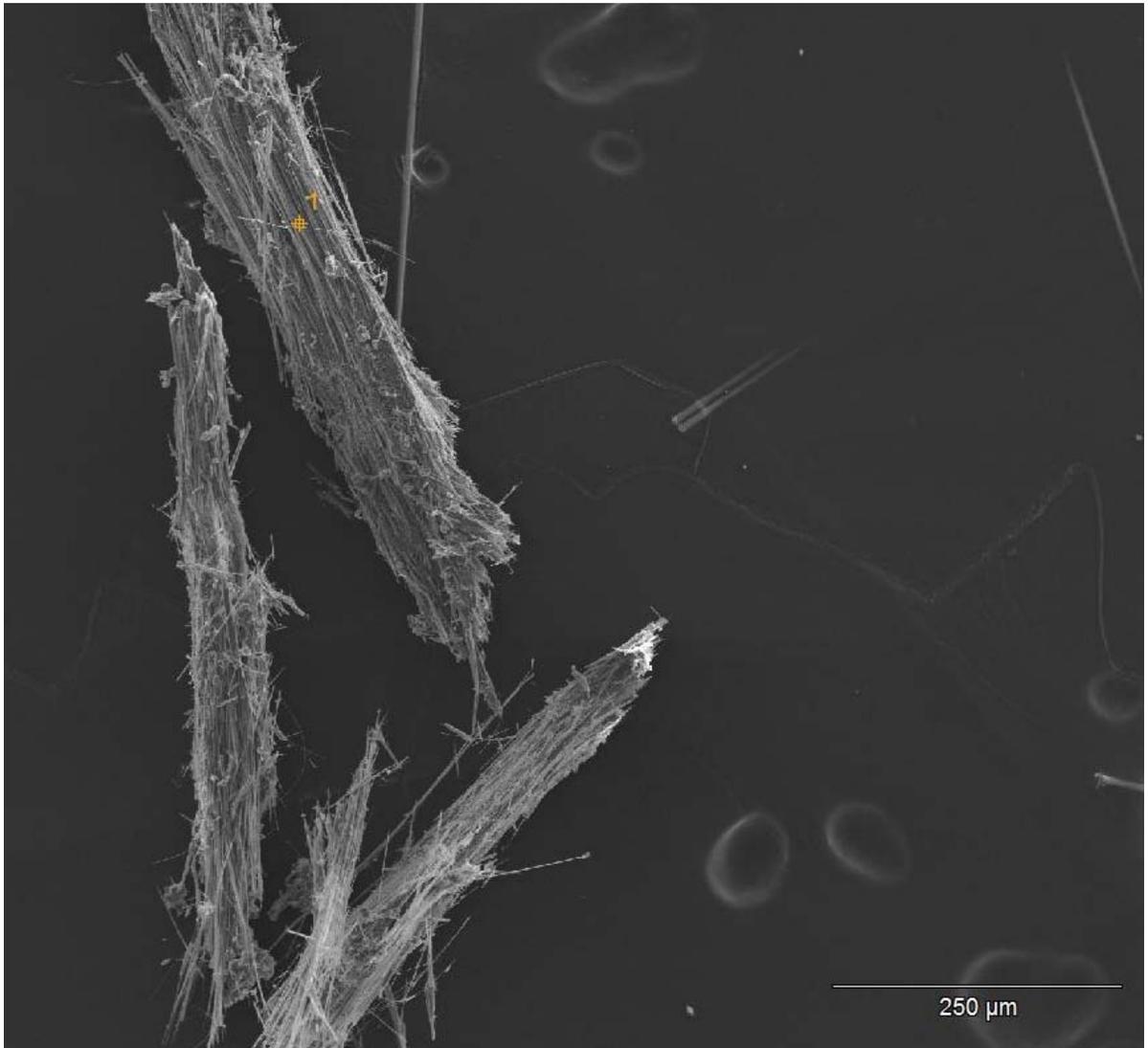
ATTACHMENT 6

SEM Photomicrographs of Representative Examples of LA Habits

SEM Photomicrographs of Representative Examples of Libby Amphibole Habits

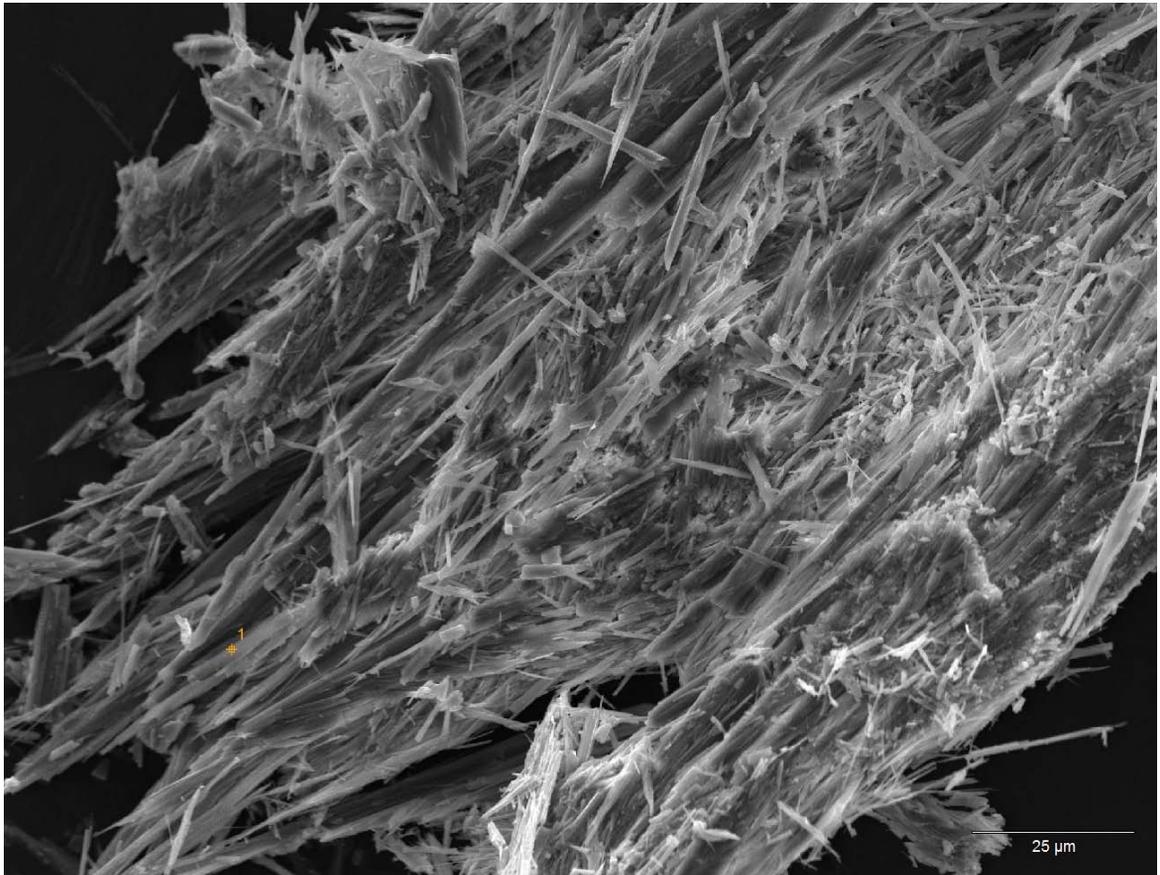
Individual bundles of Libby Amphibole (LA) were picked from soil samples at the ESAT Region 8 Laboratory and prepared for analysis by scanning electron microscopy (SEM). Slide mounts of these bundles were initially prepared in a refractive index (RI) liquid and the bundles were examined by PLM. Then the RI liquid was evaporated off the slides using a hot plate in a fume hood, and the bundles of LA were transferred to a SEM stub. Fibers were selected for SEM analysis that showed examples of the range of LA habits that may be encountered in field samples. During SEM analysis, energy dispersive spectrometry (EDS) was performed on these fiber bundles and their EDS spectra were found to be consistent with LA.

The SEM analysis was performed by the United States Geological Survey (USGS). Ten of the photomicrographs taken of the LA bundles by the USGS are provided here as a reference to help laboratories understand the range of habits of Libby Amphibole that they may encounter in field samples. All of the following pictures are of bundles that were found in field samples collected from the Libby Asbestos Superfund Site in Montana.



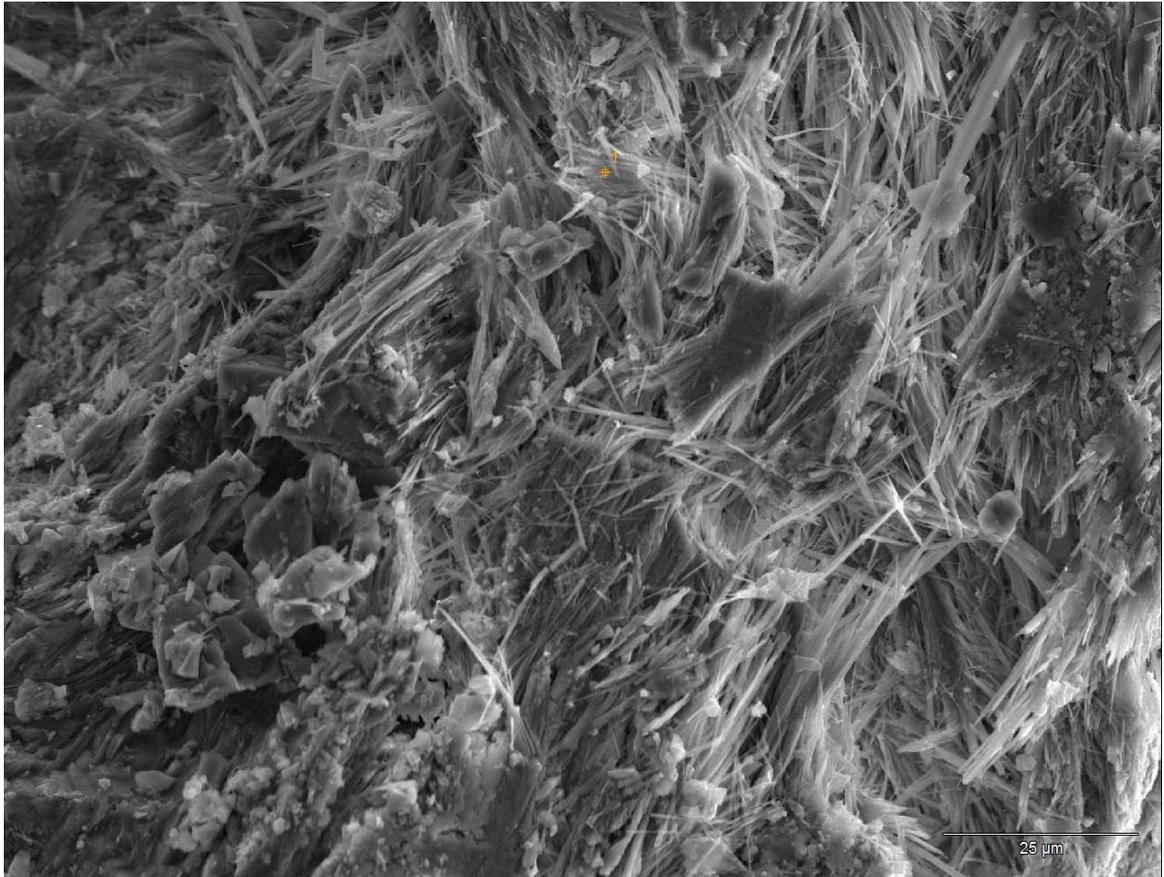
These are typical bundles of LA where the average aspect ratio of the fibers is high and most of the fibers are nearly parallel to one another. Note the scale in microns at the bottom of the photo. These three bundles are all of a size that can be seen with a stereomicroscope and picked out to be placed on a slide for analysis by PLM. The small number “1” at the top of the photo indicates where an EDS spectrum was taken and saved to a file.

Photograph provided by the USGS and used by permission. Photo for use by the Libby Lab Team only- do not cite or distribute.



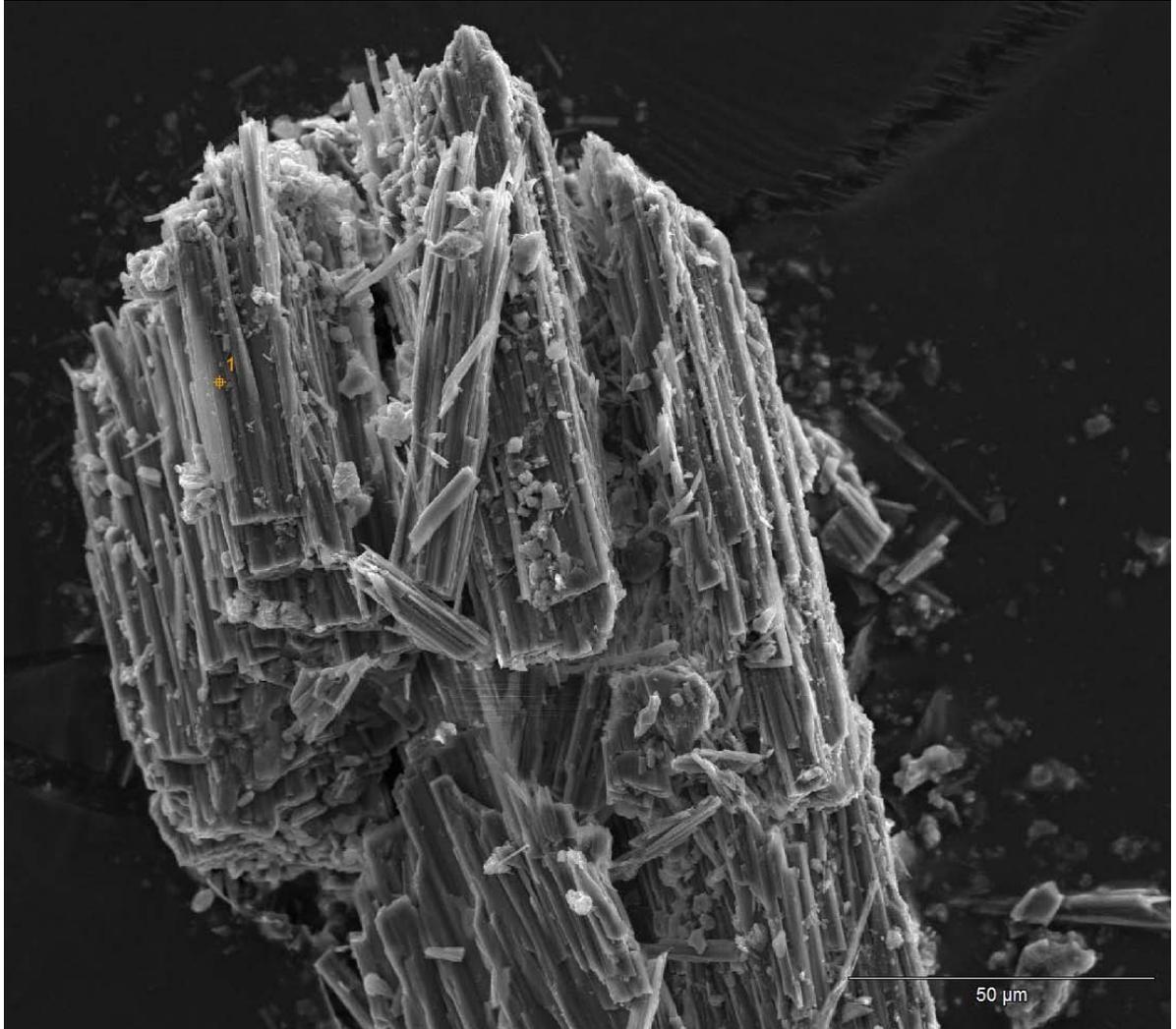
Varying degrees of parallelism can be seen in the fibers that compose bundles of LA. Note that the fibers in this bundle of LA are less parallel than the fibers in the bundles in the previous example.

Photograph provided by the USGS and used by permission.
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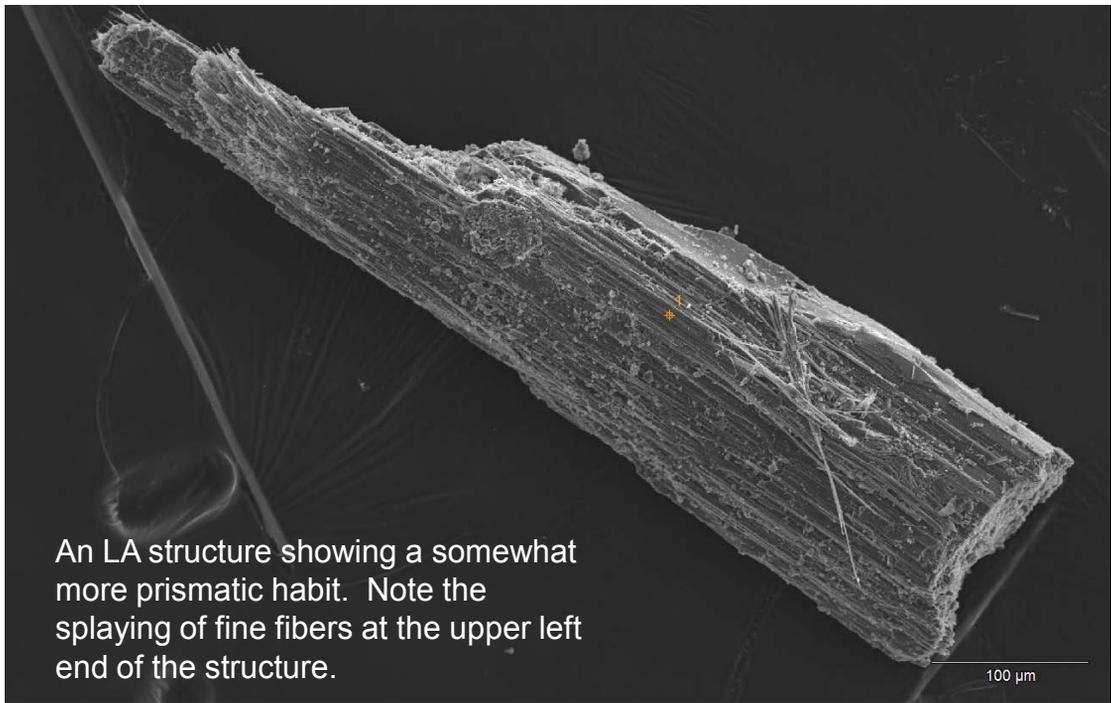
When this bundle of LA was viewed under PLM, its habit was described as “felted” or “matted” with the fibers crossing at high angles to one another. This is how the bundle appeared when it was subsequently viewed by SEM. The fibrous nature of the “felted” or “matted” habit is clear at this scale.

Photograph provided by the USGS and used by permission. Photo for use by the Libby Lab Team only- do not cite or distribute.

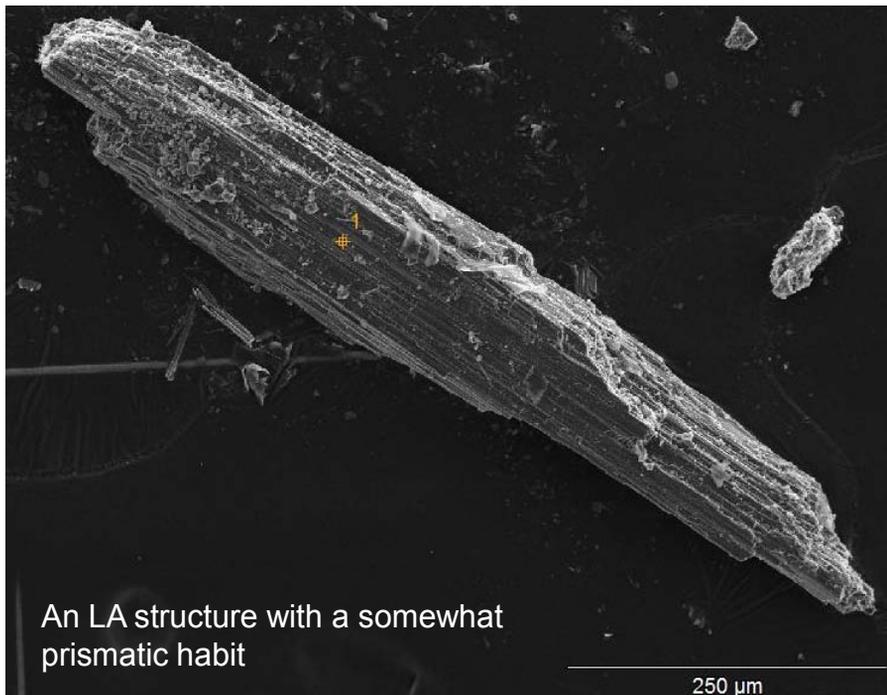


The average aspect ratio of the fibers in this bundle of LA is lower than those of the bundles in the previous examples. However, as seen by SEM, the bundle still splits readily into many small fibers.

Photograph provided by the USGS and used by permission. Photo for use by the Libby Lab Team only- do not cite or distribute.

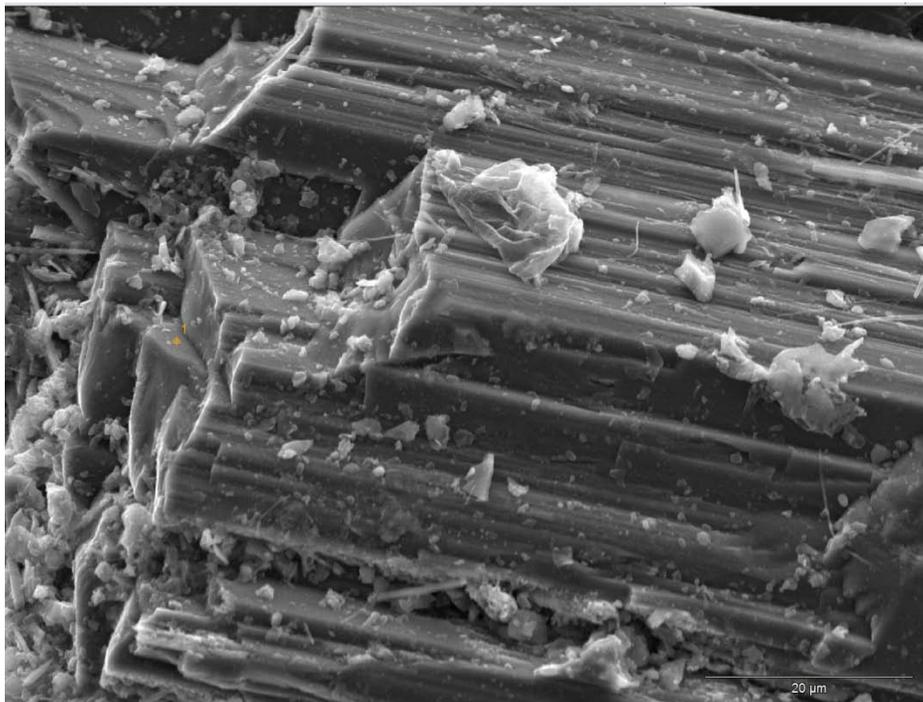
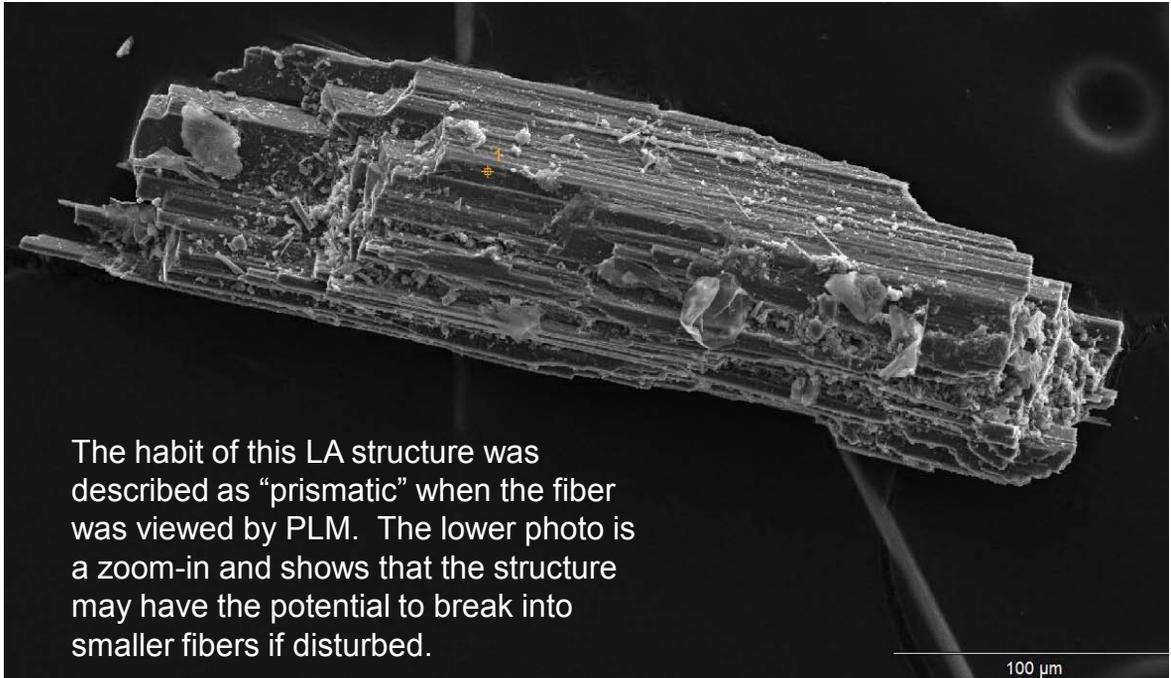


An LA structure showing a somewhat more prismatic habit. Note the splaying of fine fibers at the upper left end of the structure.



An LA structure with a somewhat prismatic habit

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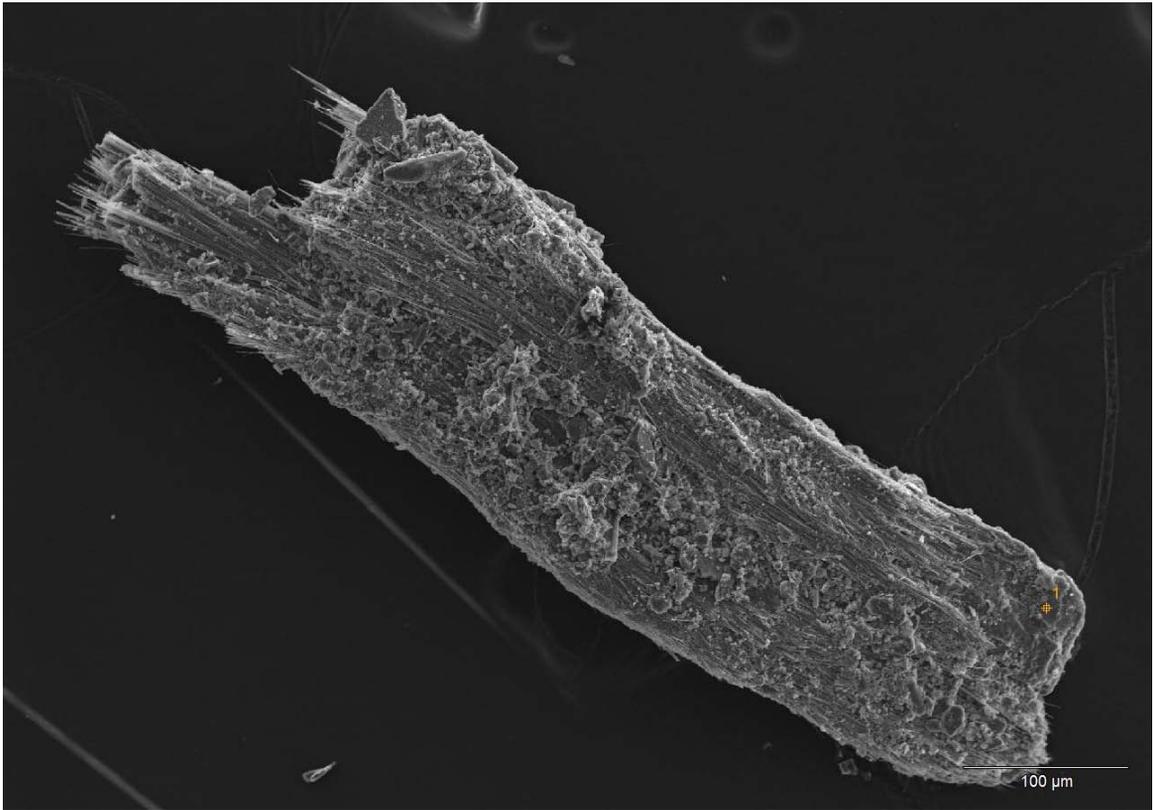


Photographs provided by the USGS and used by permission. Photos for use by the Libby Lab Team only- do not cite or distribute.



This bundle of LA was found either adhered to or grown on a piece of feldspar. EDS of the blocky material on the left half of the structure was found to be consistent with potassium feldspar. EDS of the fibrous material on the right, as with all other fiber bundles shown in these photos, was found to be consistent with LA.

Photograph provided by the USGS and used by permission. Photo for use by the Libby Lab Team only- do not cite or distribute.



This is a bundle of LA that was found in PLM as either adhered to or grown on a piece of mica. This is how the bundle appeared when it was subsequently viewed by SEM. The EDS spectrum of the platy, rounded material at the lower right end of the structure was found to be consistent with biotite. The EDS spectrum of the fibrous material on the upper left end of the structure was found to be consistent with LA.

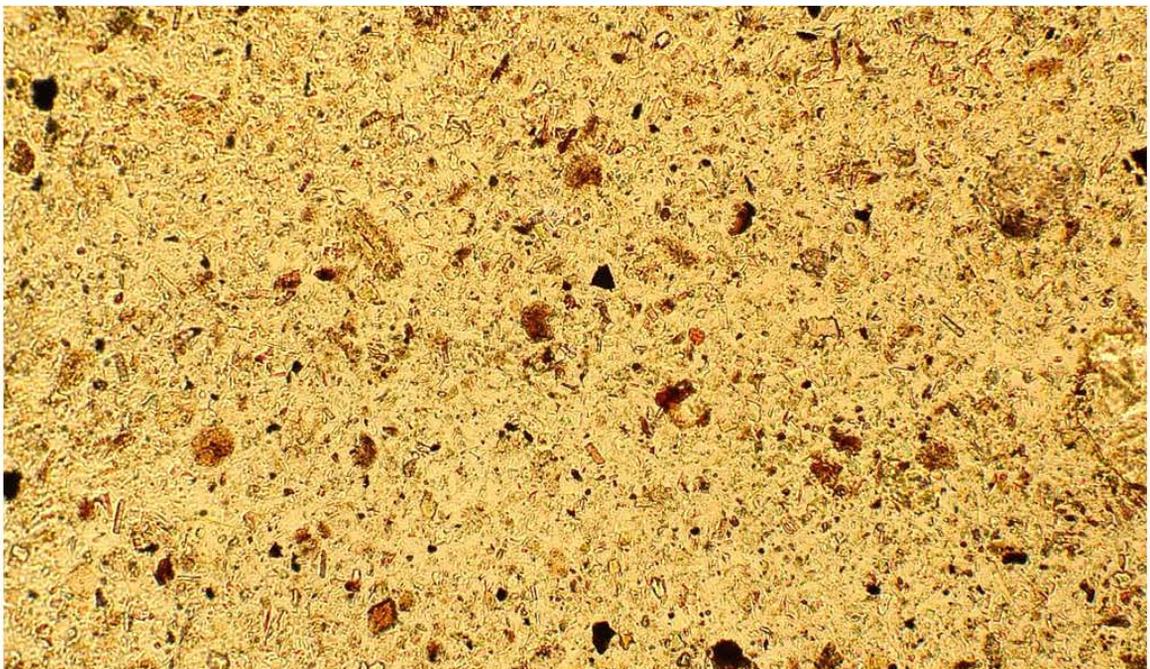
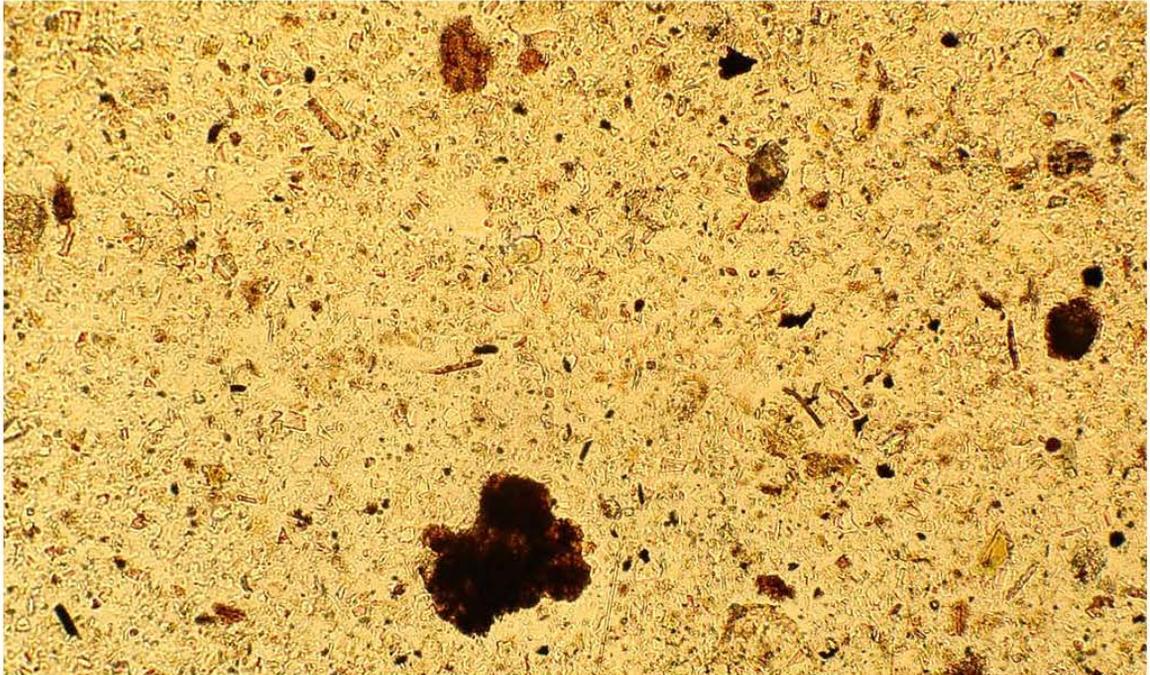
Photograph provided by the USGS and used by permission. Photo for use by the Libby Lab Team only- do not cite or distribute.

ATTACHMENT 7

Photomicrographs of Representative Fields of View of 0.2% and 1.0% LA Controlled PE Reference Materials

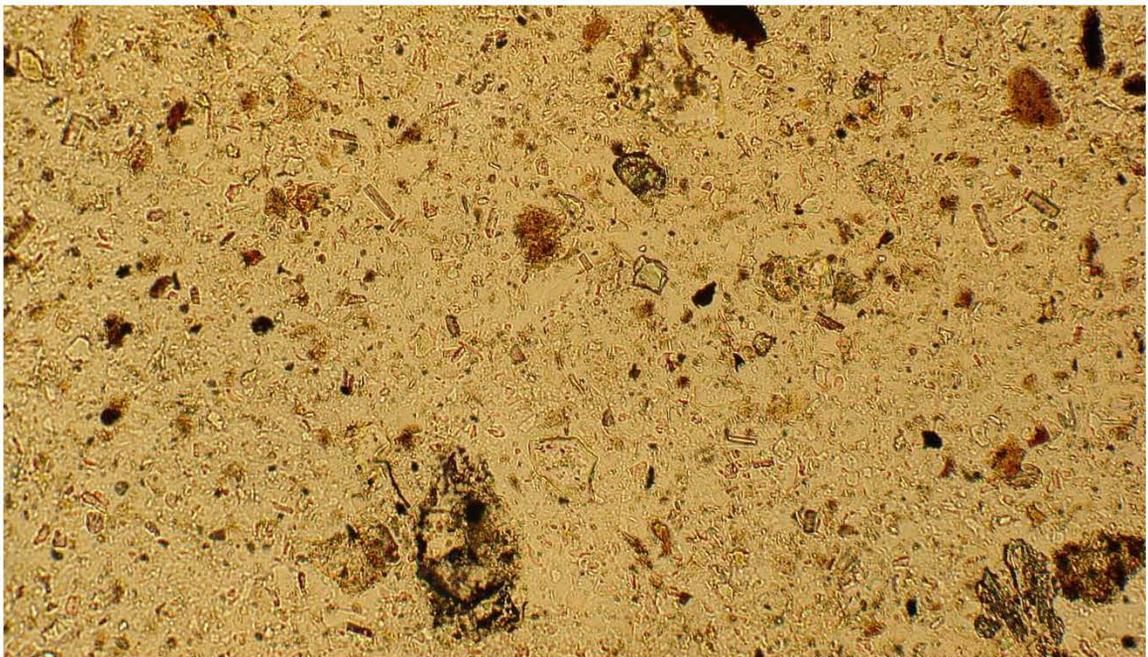
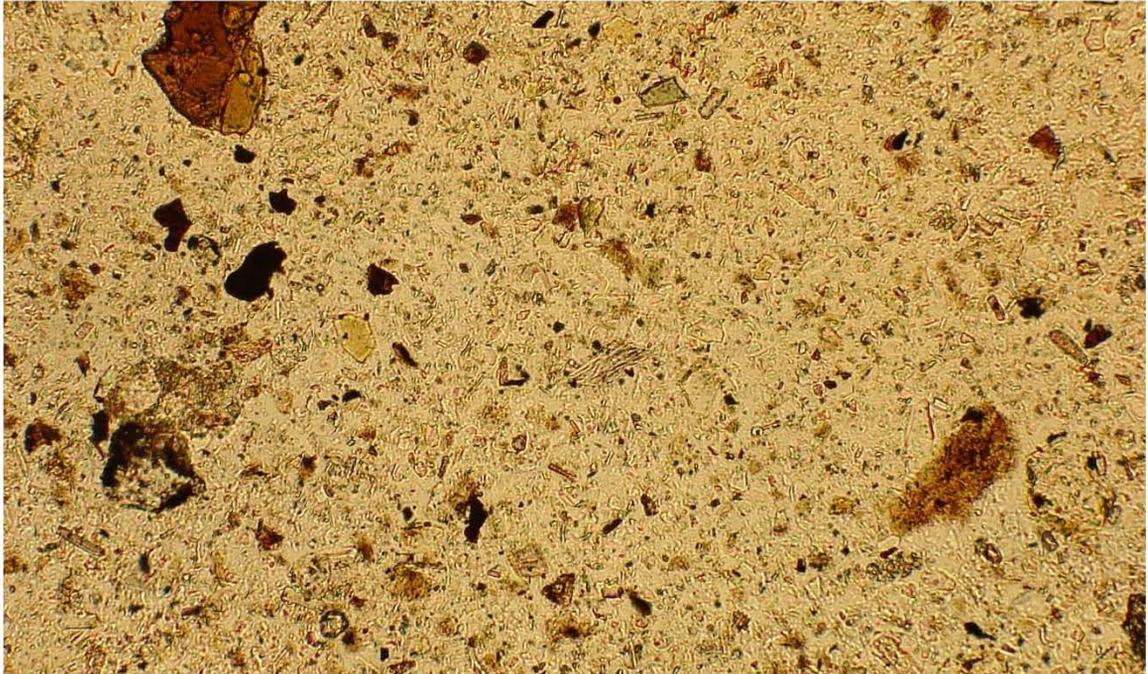
0.2% Libby Amphibole

Photomicrographs of representative fields of view of the 0.2% Libby Amphibole by weight Controlled PE Reference Material. All photos taken at 100x, plane light in 1.55 refractive index oil. Width of each picture is approximately 1,500 microns.



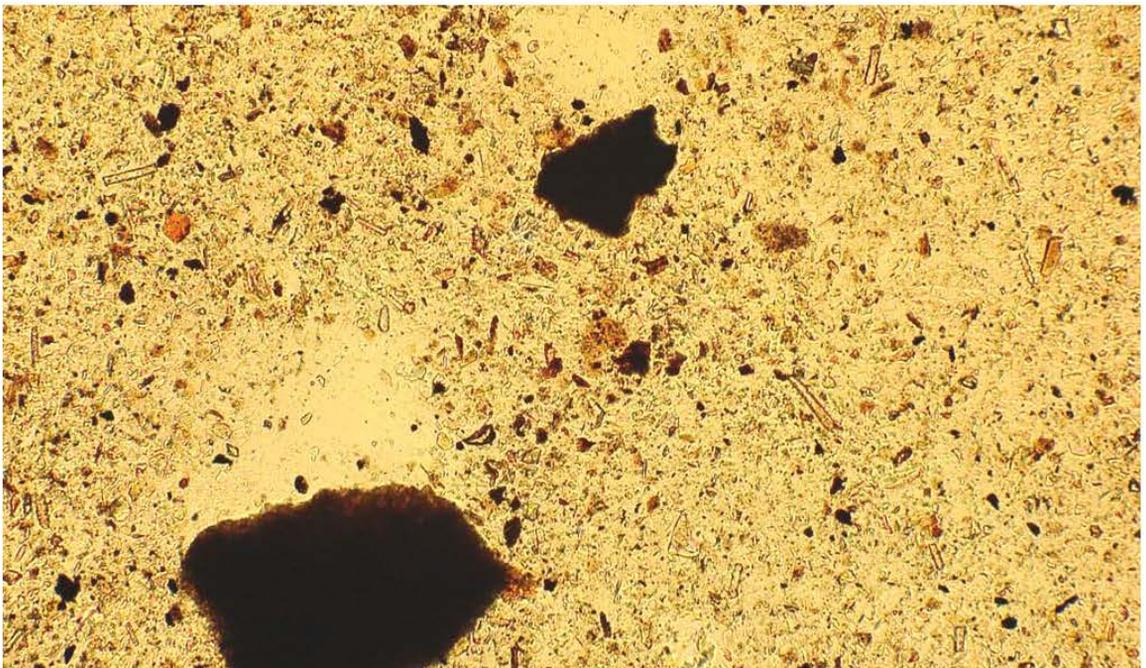
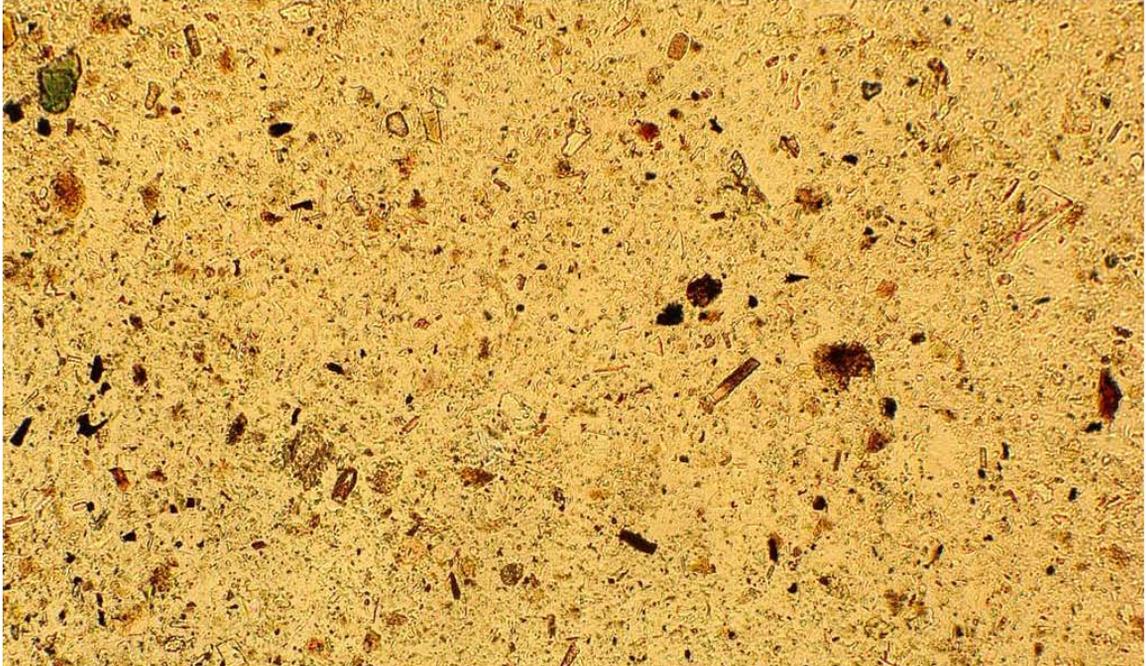
0.2% Libby Amphibole

Photomicrographs of representative fields of view. Width of each picture is approximately 1,500 microns.



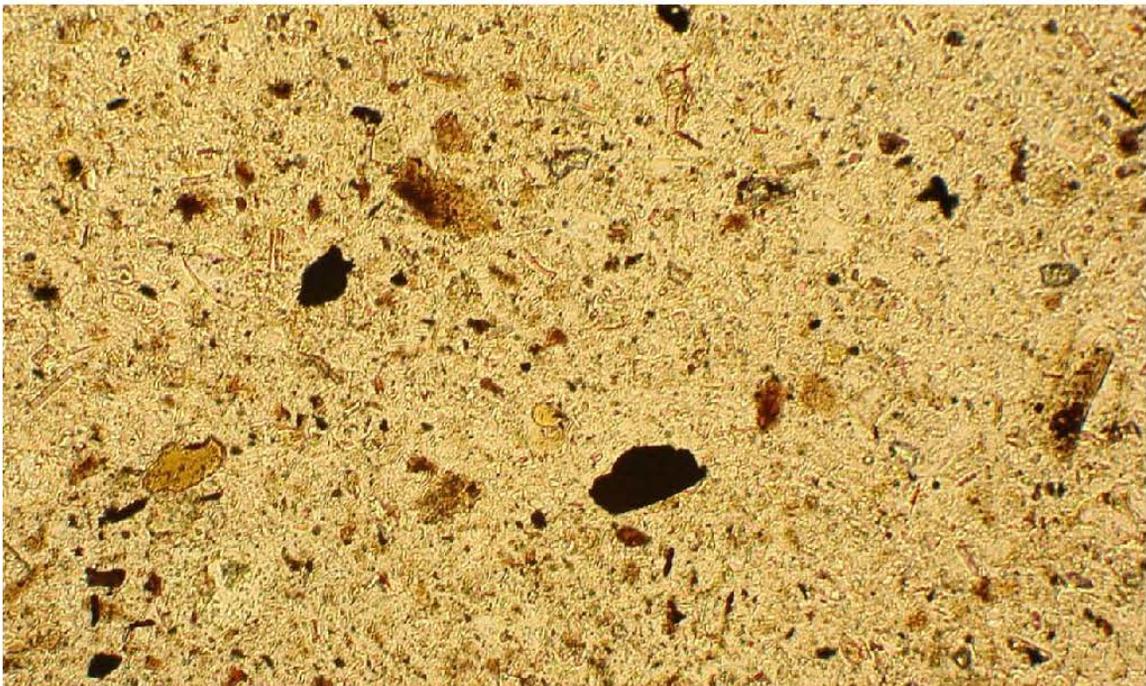
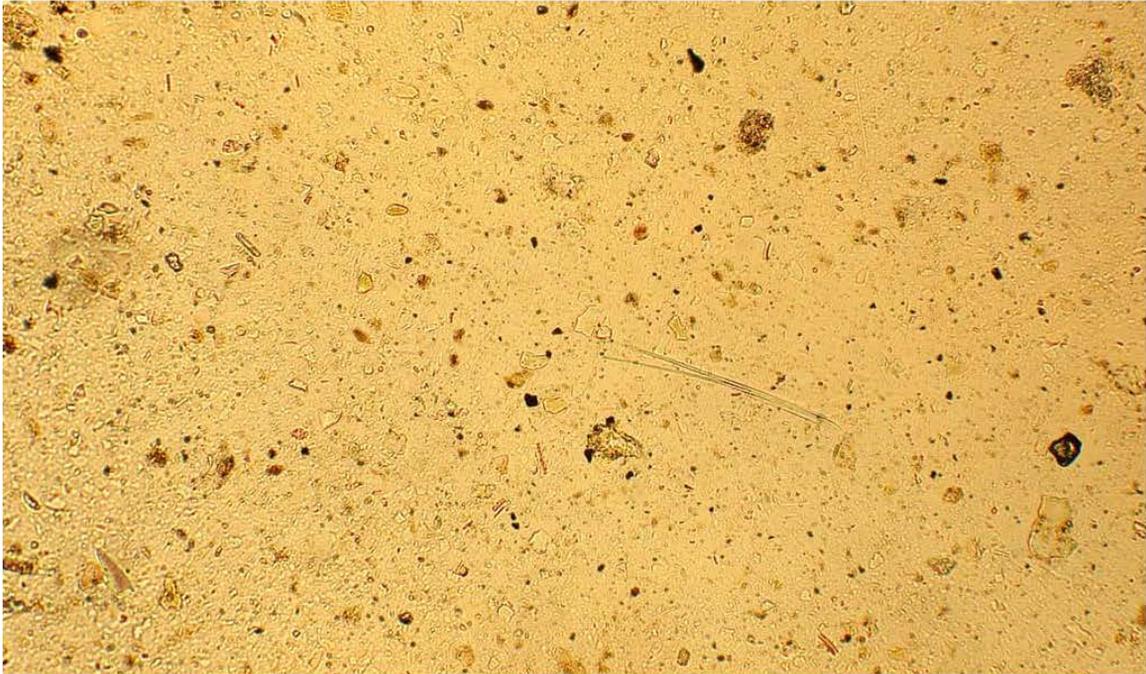
0.2% Libby Amphibole

Photomicrographs of representative fields of view. Width of each picture is approximately 1,500 microns.



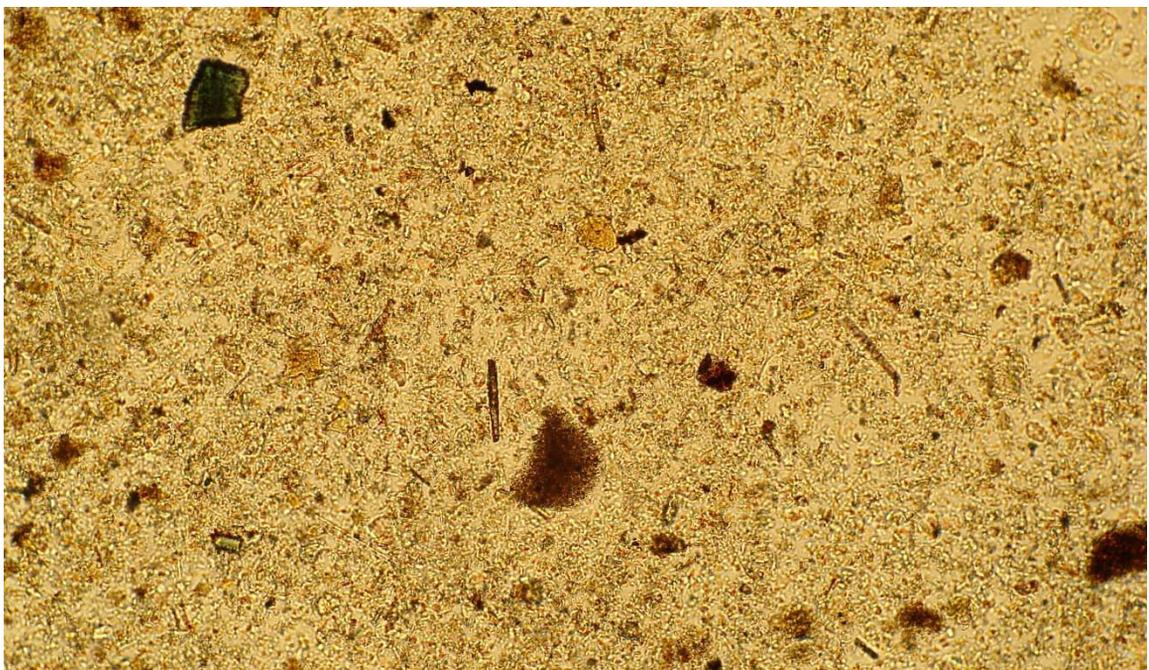
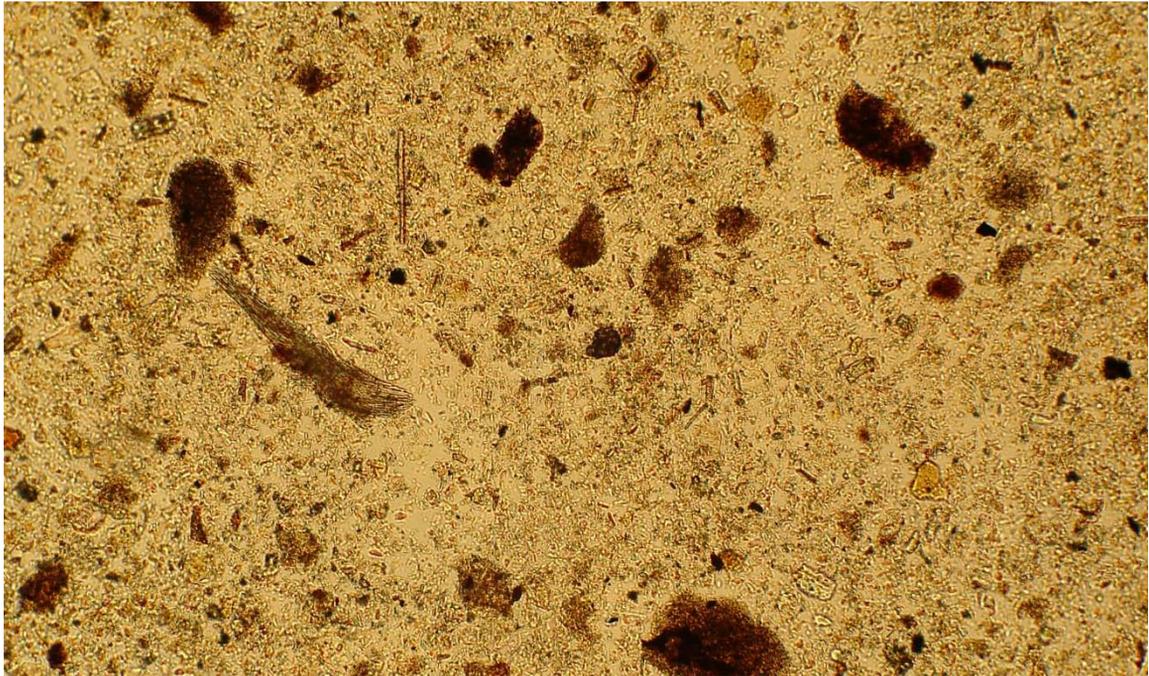
0.2% Libby Amphibole

Photomicrographs of representative fields of view. Width of each picture is approximately 1,500 microns.



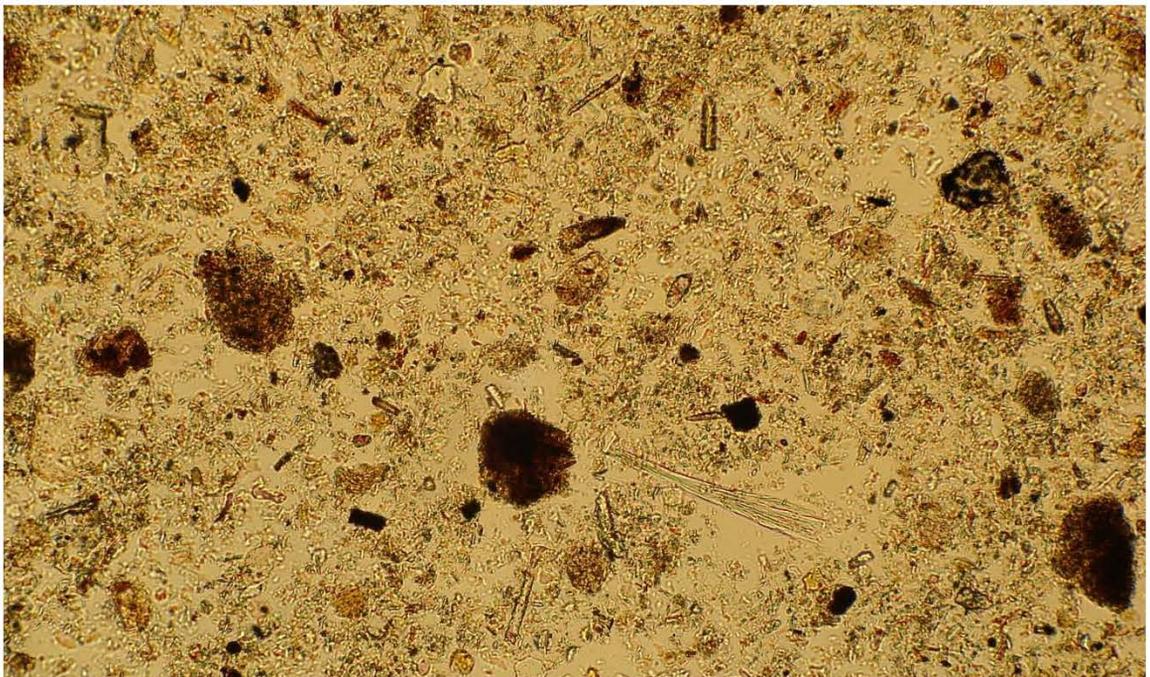
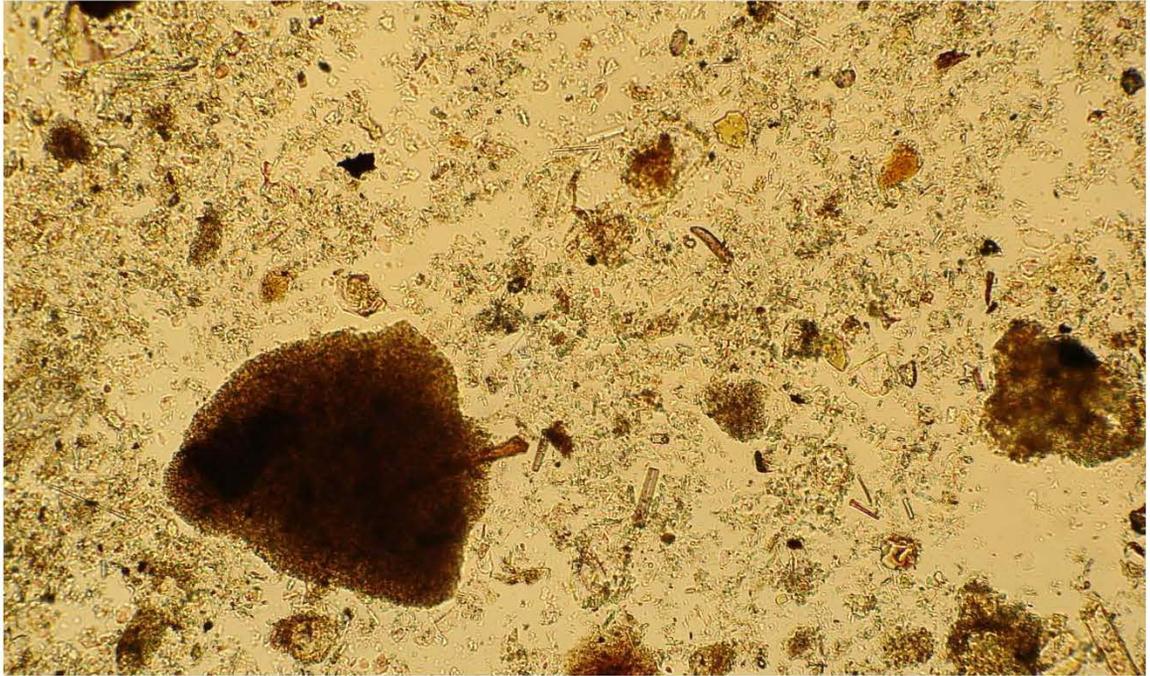
1.0% Libby Amphibole

Photomicrographs of representative fields of view of the 1.0% Libby Amphibole by weight Controlled PE Reference Material. All photos taken at 100x, plane light in 1.55 refractive index oil. Width of each picture is approximately 1,500 microns.



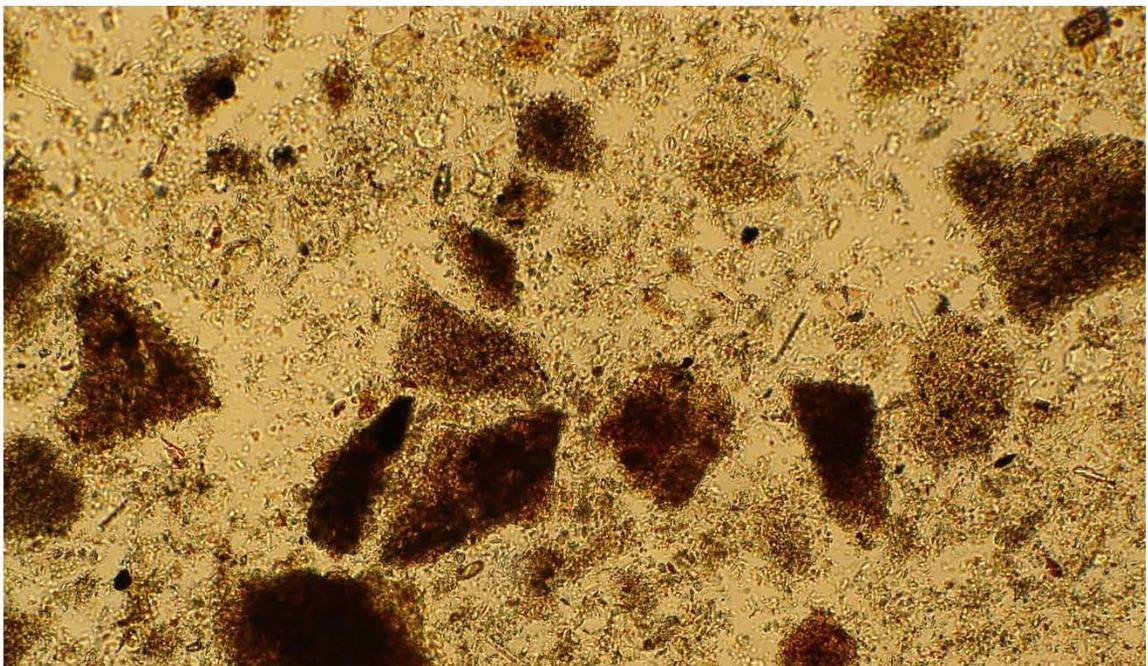
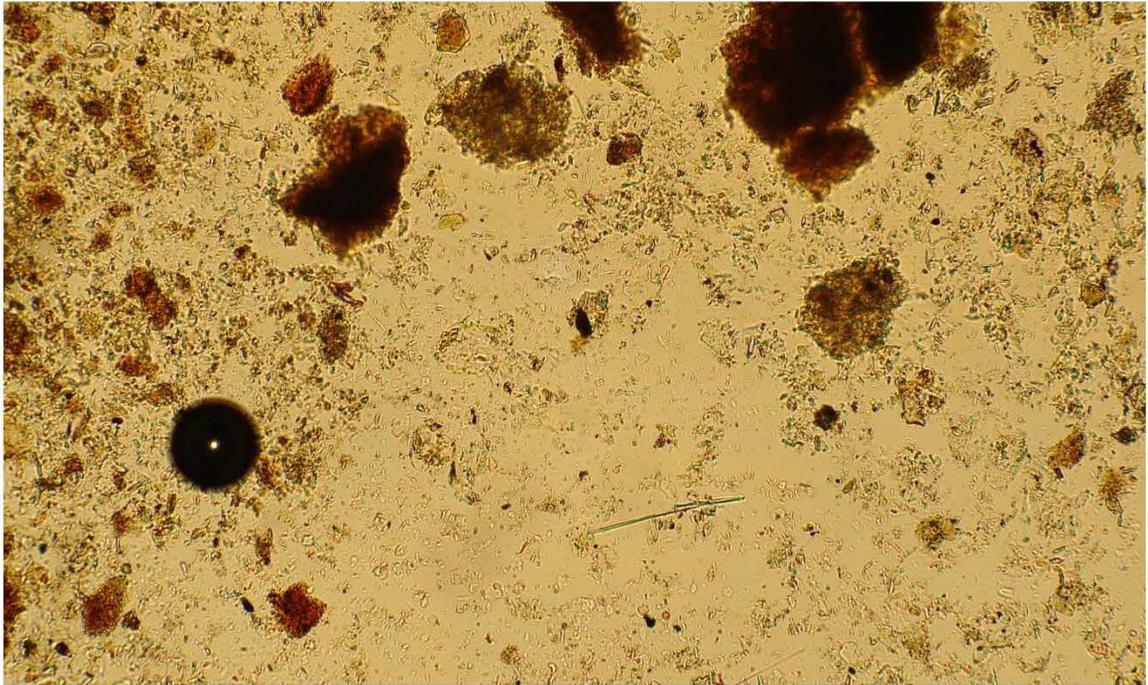
1.0% Libby Amphibole

Photomicrographs of representative fields of view. Width of each picture is approximately 1,500 microns.



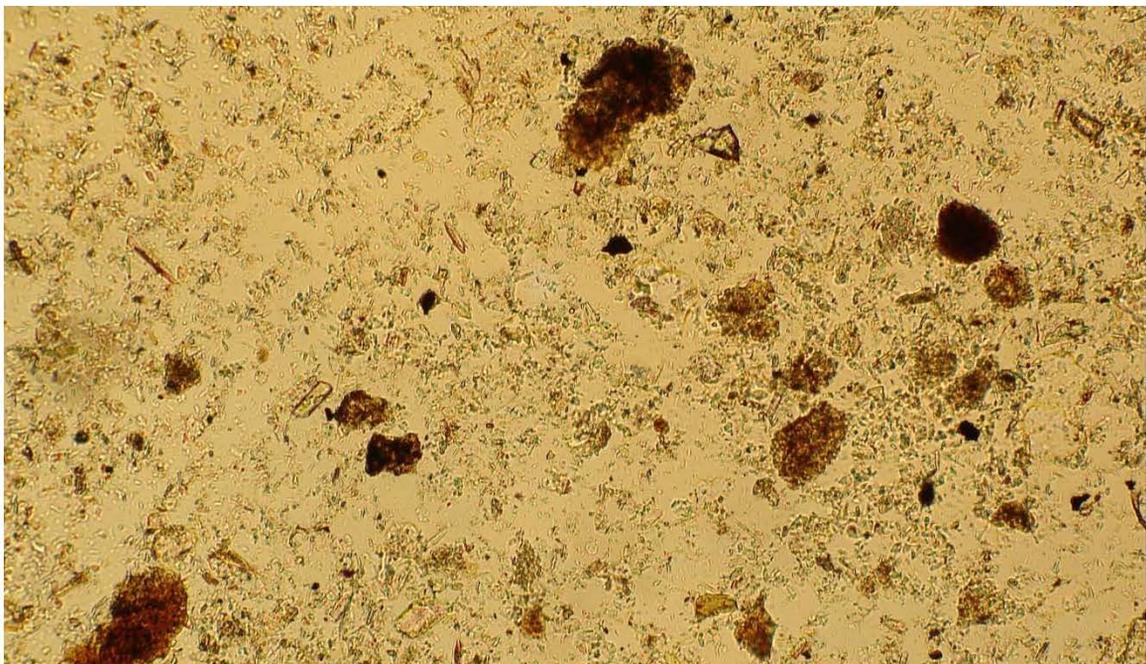
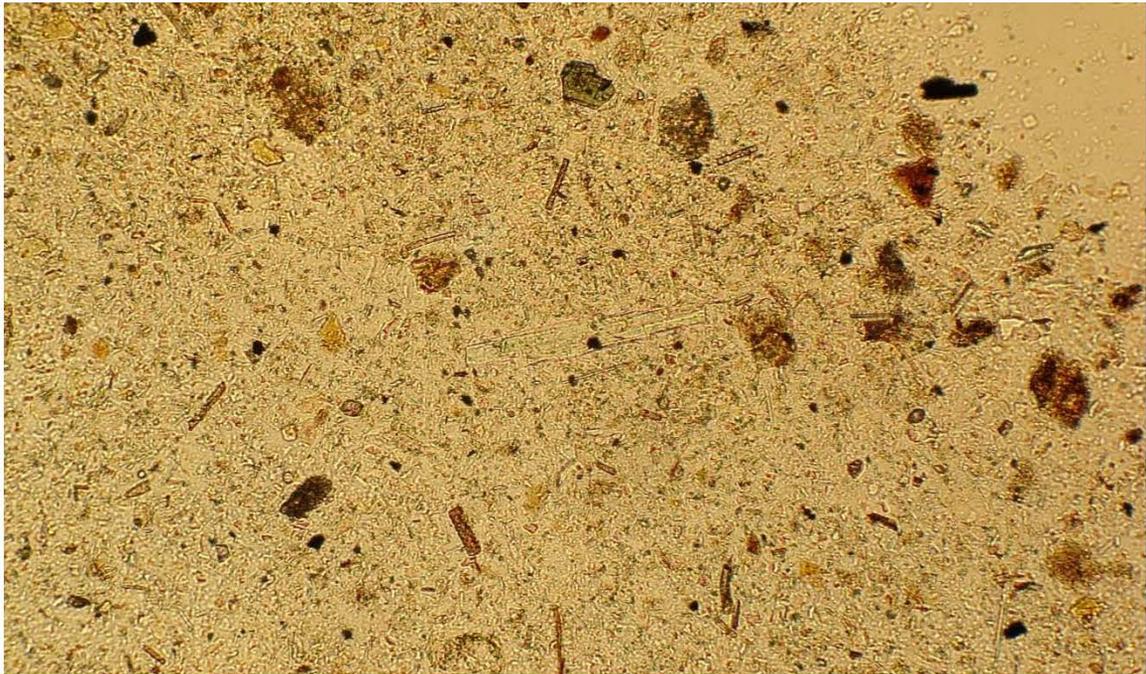
1.0% Libby Amphibole

Photomicrographs of representative fields of view. Width of each picture is approximately 1,500 microns.



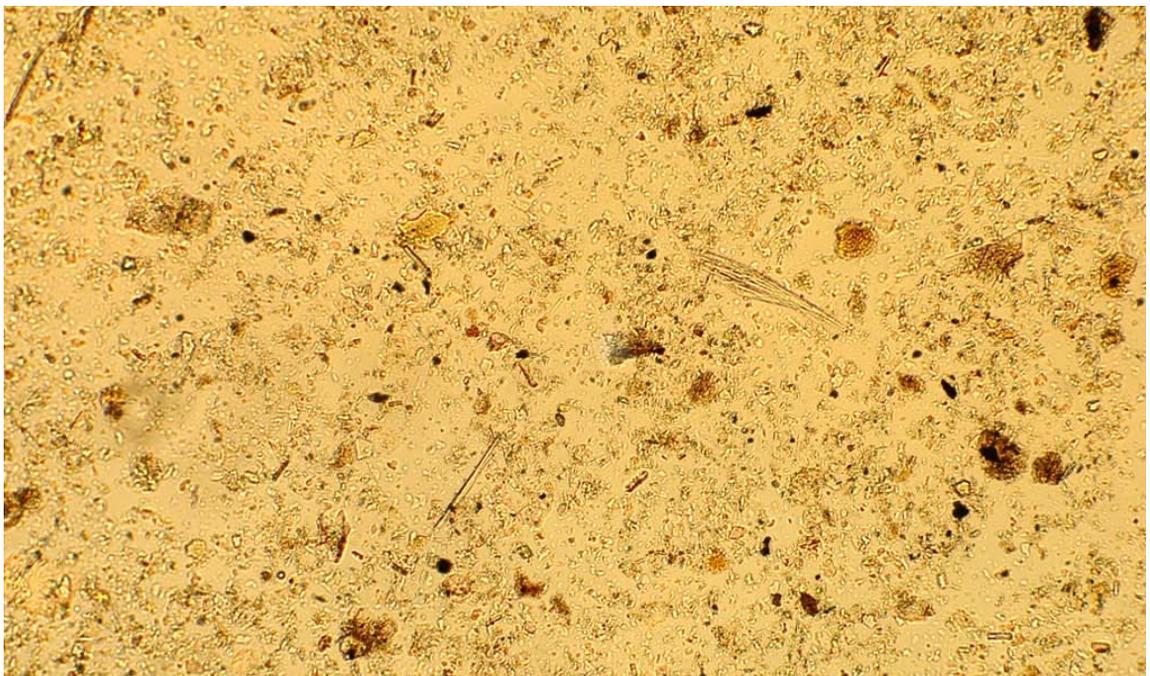
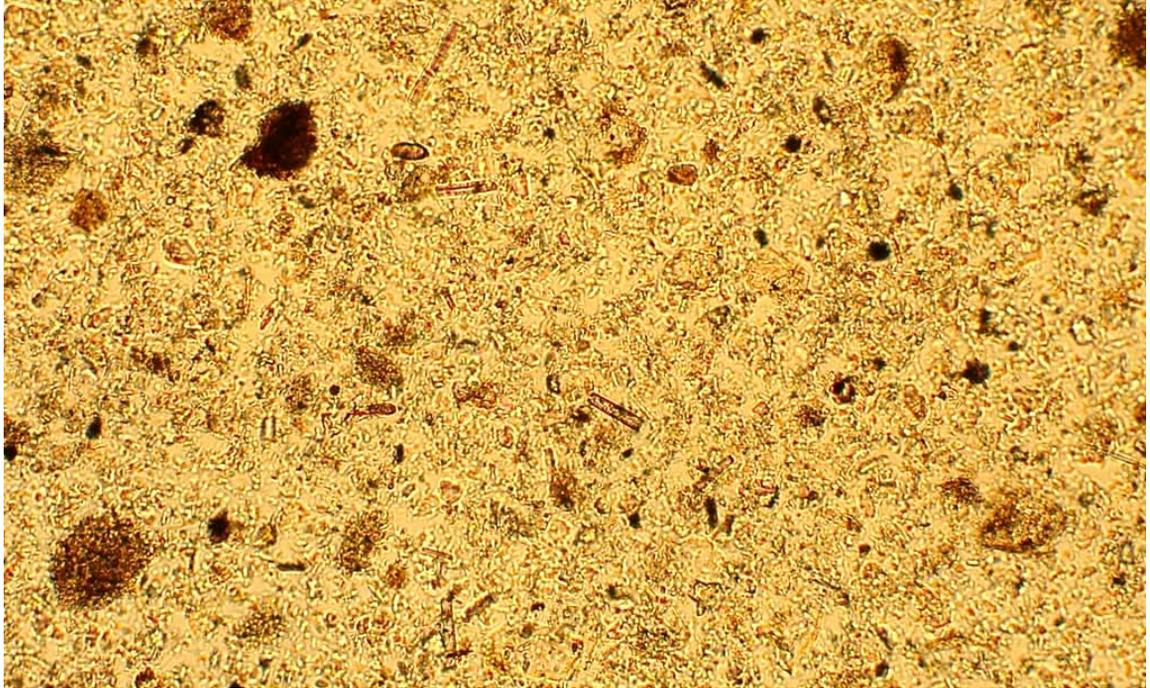
1.0% Libby Amphibole

Photomicrographs of representative fields of view. Width of each picture is approximately 1,500 microns.



1.0% Libby Amphibole

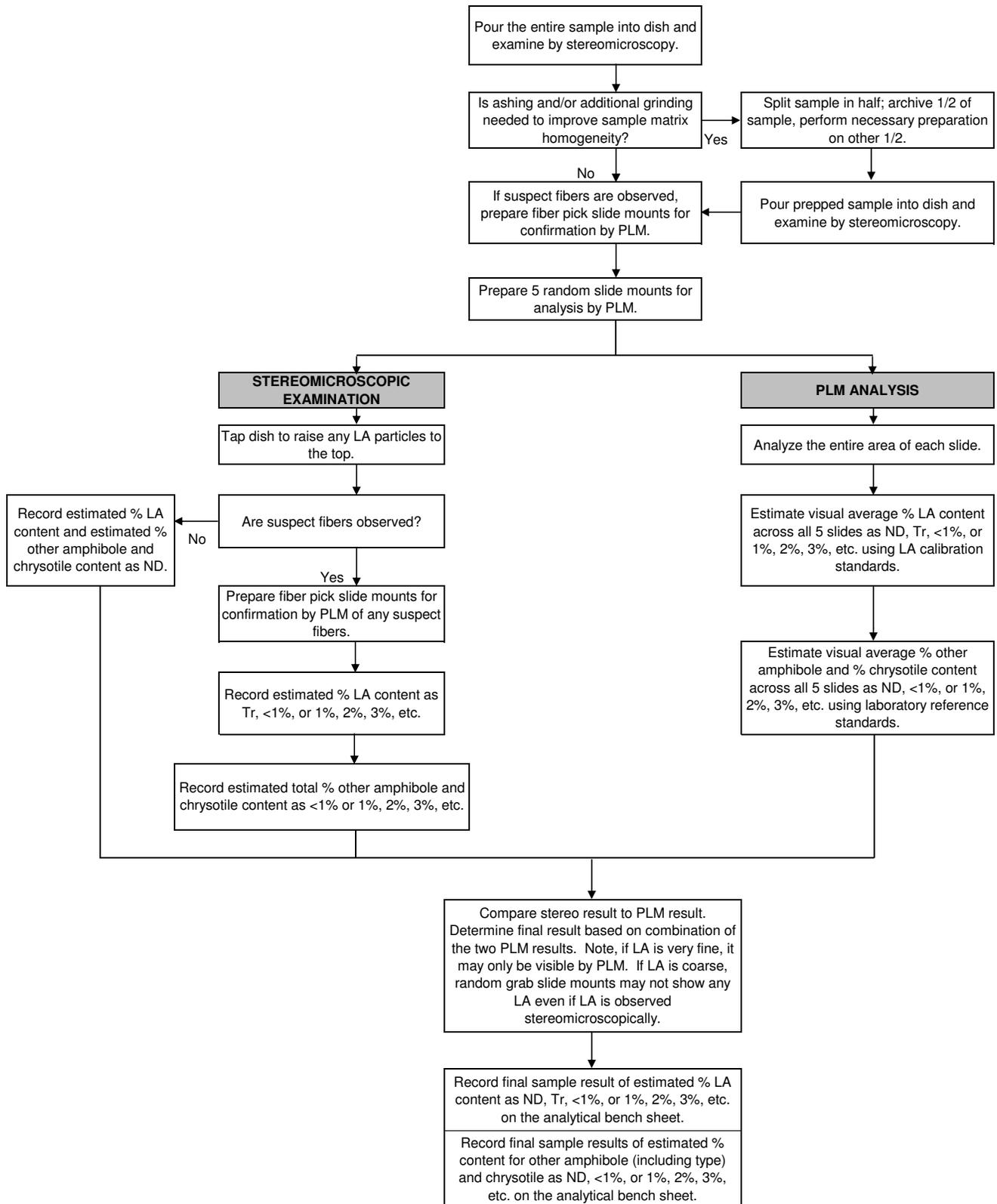
Photomicrographs of representative fields of view. Width of each picture is approximately 1,500 microns.



ATTACHMENT 8

Flow Chart for Determining Asbestos Content by Complementary Use of Stereomicroscopy and PLM Visual Estimation

Flow Chart for Determining Asbestos Content by Complementary Use of Stereomicroscopic Examination and PLM Visual Estimation



LIBBY ASBESTOS SUPERFUND SITE STANDARD OPERATING PROCEDURE
APPROVED FOR USE AT THE LIBBY ASBESTOS SUPERFUND SITE ONLY

ANALYSIS OF ASBESTOS FIBERS IN FINE SOIL BY POLARIZED LIGHT MICROSCOPY

Date: July 27, 2012

SOP No.: SRC-LIBBY-03 (Revision 3)

ATTACHMENT 9

Becke Line Chart by F. D. Bloss

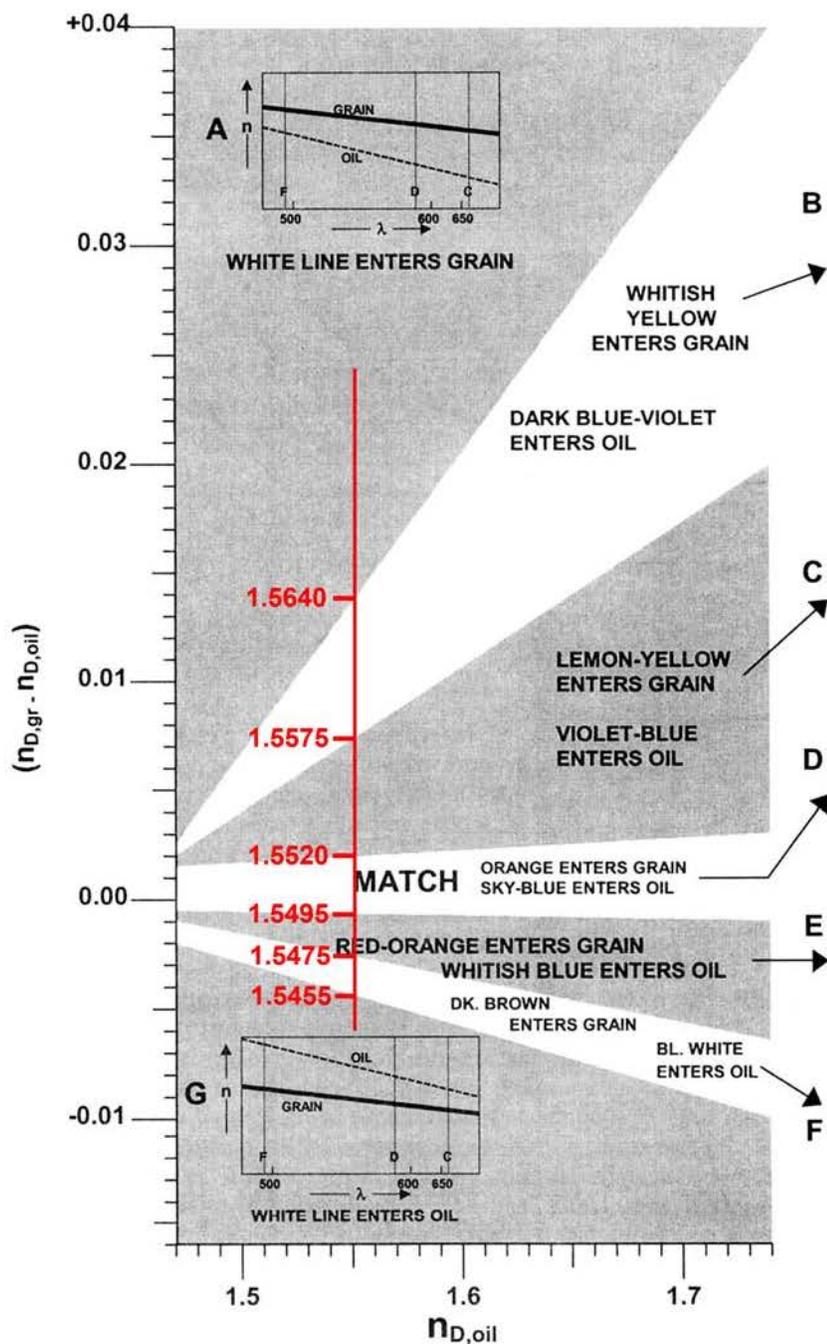


Figure 5-9. Use of the colors observed for the Becke lines or oblique illumination shadows to determine the amount by which n_D for the solid exceeds n_D for the liquid (if Cargille Series A or B liquids are being used). The Becke line movements are those observed when the objective is raised upward from sharp focus. The color observed depends upon the wavelength (λ_m) where—as shown to the right—the dispersion curves of liquid and solid intersect within the visible range. To correct the colors cited to your own perceptions of color, observe grains of salt ($n_D = 1.544$) mounted in liquids for which n_D equals 1.570, 1.560, 1.550, 1.544, 1.540, and 1.530. [Figure continued on facing page.]

OPTICAL CRYSTALLOGRAPHY. By F. Donald Bloss. Mineralogical Society of America (Monograph Series, Publication No. 5), 1999, Washington, D.C. 239 p

LIBBY SUPERFUND SITE STANDARD OPERATING PROCEDURE
COLLECTION AND ANALYSIS OF ASBESTOS IN INDOOR DUST

Date: August 12, 2003

SOP No. SRC-LIBBY-05 (Revision 0)

Title: **COLLECTION AND ANALYSIS OF ASBESTOS IN INDOOR DUST**

Author: William Brattin

SYNOPSIS: A standard method for collecting and analyzing indoor dust samples for asbestos is provided. This method is based on ASTM Method D5755-95, with project-specific modifications intended specifically for use at the Libby Superfund Site.

APPROVALS:

TEAM MEMBER	SIGNATURE/TITLE	DATE
USEPA Region 8	<u>John Goldade</u>	<u>8/12/03</u>
Syracuse Research Corp.	<u>WJ Brattin</u>	<u>8/12/03</u>

Revision	Date	Principal Changes
0	08/12/03	--

LIBBY SUPERFUND SITE STANDARD OPERATING PROCEDURE
COLLECTION AND ANALYSIS OF ASBESTOS IN INDOOR DUST

1.0 PURPOSE

The purpose of this standard operating procedure (SOP) is to provide a standard approach for collection of indoor dust samples and analysis of those samples for asbestos. This SOP is based on ASTM Method D5755-95, with project-specific modifications specifically intended for application at the Libby Superfund site.

2.0 SCOPE AND APPLICATION

This method is intended for preparation and analysis of samples collected for asbestos in indoor dust using ASTM Method D5755-95. This method is appropriate for the preparation and analysis of all types of asbestos fibers, including both chrysotile and amphiboles, including amphiboles that are characteristic of the Libby site

3.0 RESPONSIBILITIES

It is the responsibility of the laboratory supervisor to ensure that all analyses and quality assurance procedures are performed in accord with this SOP, and to identify and take appropriate corrective action to address any deviations that may occur during sample preparation or analysis. The laboratory supervisor should also communicate with project managers at EPA or their oversight contractors any situations where a change from the SOP may be useful, and must receive approval from the EPA Remedial Project Manager or Regional Chemist for any deviation or modification from the SOP before proceeding with sample preparation and analysis.

4.0 METHOD DESCRIPTION

Dust samples are collected in a filter cassette using a microvacuum device. Dust in the cassette is suspended in water and a portion of the suspension is applied to a filter and analyzed for asbestos using transmission electron microscopy (TEM).

5.0 DETAILED METHOD

Dust samples are collected and analyzed in accord with ASTM D5755-95, except for the project-specific modifications, clarifications, and requirements provided below.

LIBBY SUPERFUND SITE STANDARD OPERATING PROCEDURE
COLLECTION AND ANALYSIS OF ASBESTOS IN INDOOR DUST

1. Sample Collection

Samples will be collected using 25 mm MCE filters. The number of 100-cm² templates collected should be specified in the project-specific plan. In general, a composite of at least three template areas is desired in order to ensure that the sample is representative.

2. Classification of Asbestos Mineral Type

Based on fiber attributes (morphology, SAED, EDXA), asbestos in the sample is classified into one of three categories:

Mineral Class	Description
Libby Amphibole (LA)	Any amphibole asbestos similar to that observed in ores obtained from the mine in Libby. This solution series includes (but may not be limited to) actinolite, tremolite, richterite, and winchite, as well as magnesio-arfedsonite and ferro-edenite.
Other Amphibole (OA)	Other types of amphibole asbestos, including amosite, anthophyllite, and crocidolite. These forms of asbestos are not thought to be related to the mine in Libby.
Chrysotile (C)	Serpentine asbestos. This form of asbestos is the most common type in building materials, and is not thought to be related to the mine in Libby.

A discussion of the EDS spectrum associated with LA fibers is presented in USGS (2002).

3. Secondary Filter Loading

The volume of dust suspension applied to the secondary filter shall be sufficient to produce a total loading of $\leq 25\%$.

4. No Structures Detected

In a grid opening where no asbestos structures are detected, enter "ND" (rather than "NSD").

5. Analytical Sensitivity

Target sensitivity is 500 s/cm² or less, with a maximum of 1,000 s/cm². Whenever possible, sensitivity should be controlled by increasing the number of grid openings counted, up to a maximum of 20. If a sensitivity of 1,000 s/cm² cannot be achieved with 20 grid opening, the sample should be ashed in order to reduce debris loading (thereby allowing application of a

LIBBY SUPERFUND SITE STANDARD OPERATING PROCEDURE
COLLECTION AND ANALYSIS OF ASBESTOS IN INDOOR DUST

larger fraction of the original sample to the secondary filter). If the necessary sensitivity cannot be achieved even after ashing, then the laboratory should complete a laboratory modification form to summarize the issues associated with that sample.

6.0 APPARATUS AND MATERIALS

All equipment and materials are as described in ASTM D5755-95.

7.0 QUALITY ASSURANCE/QUALITY CONTROL

All QA/QC procedures are as described in ASTM D5755-95, except for the project-specific modifications, clarifications, and requirements provided below.

1. Field Blanks

Field blanks will be collected at a rate of one per sample team per day. The EPA regional chemist will specify the fraction of field blanks that must undergo analysis, as documented in a modification form LFO-000064. In the absence of any evidence of contamination, the rate will typically be one sample per team per week.

2. Re-Analysis

Each laboratory will prepare and analyze up to five different types of QC sample at the rates specified in Mod LB-000029.

8.0 RECORDS

8.1 Data Forms

Analysts will record analytical results using the electronic data sheets developed for the Libby project, as presented in the Dust Sampling and Analysis Plan (SAP). Once completed and checked, these spreadsheets are submitted to EPA for upload into the database. The laboratory should retain all original records for use in resolving any questions until otherwise instructed by EPA.

8.2 Instrument Maintenance Logbook

An individual instrument maintenance logbook should be kept for each piece of equipment in use at the laboratory. All maintenance activities must be recorded in the appropriate logbook.

8.3 Data Storage and Archival

Electronic Data. Each day of data acquisition, all electronic files will be saved onto two separate media. For example, the data may be saved onto a computer hard drive, but must also be backed up onto a type of portable media such as CD-ROM, floppy disc, or tape. Portable media will be maintained in a single location with limited access.

Hardcopy Data. All data sheets and micrographs must be stored in a secured location with limited access (e.g., locking file cabinet) when not in use.

Copies (hardcopy and electronic) of the raw analytical data will be submitted to USEPA for archival.

LIBBY SUPERFUND SITE STANDARD OPERATING PROCEDURE
COLLECTION AND ANALYSIS OF ASBESTOS IN INDOOR DUST

9.0 REFERENCES

ATSM. 1995. Standard Test method for Microvacuum Sampling and Indirect Analysis of Dust by Transmission Electron Microscopy for Asbestos Structure Number Concentrations. American Society of Testing and Materials. Method Designation D 5755-95. October, 1995.

APPENDIX C
Inspection Forms

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**LIBBY ASBESTOS SITE
Occupant Information Form (OIF)**

General Information	
Address:	
Property ID:	
Location ID:	
Location Type:	
Location Description:	
Survey Date (Investigation Date):	
Event ID (Investigation Name):	SI DI SI/DI ABS ERS
Field Logbook Number:	
Logbook Page Numbers:	
Surveyors (Investigation Team Members):	
Field Form Check (100% of forms):	

Occupant Information		
Is there any knowledge of former vermiculite miners, close relatives of vermiculite miners, or any highly exposed persons living at or visiting the property?	Yes	No
Is the resident, past or present, diagnosed with an asbestos-related disease?	Yes	No
	N/A	
Number of adult residents or employees?		
Number of child residents?		
Age range of child residents?	0 - 6	7 - 12
	13 - 18	N/A
Does the current resident have any outdoor pets?	Yes	No
	N/A	
If wood chips are present on the property, what is the source?		
Has the current resident purchased vermiculite or vermiculite containing soil from a commercial source (eg ACE, Home Depot). If yes, note known locations in comments field	Yes	No
Comments		

**LIBBY ASBESTOS SITE
Interior Property Inspection Form (IPIF)**

General Information	
Address:	
Property ID:	
Location ID:	
Location Type:	
Location Description:	
Survey Date (Investigation Date):	
Event ID (Investigation Name):	SI DI ABS ERS
Field Logbook Number:	
Logbook Page Numbers:	
Surveyors (Investigation Team Members):	
Field Form Check (100% of forms):	
Screening Field Check (2% of forms):	

*Circle all that apply.

Building Attributes	
Year of construction	
Heating source	Wood/Coal Propane/Oil Electric None
Heat distribution	Forced Air Radiant N/A
Cooling system	Air Conditioner Swamp Cooler None

**LIBBY ASBESTOS SITE
Interior Property Inspection Form (IPIF)**

Primary triggers for interior action checklist (Primary and Secondary buildings)		
Is open, non-contained, or migrating vermiculite insulation present in the attic?	Yes (note on sketch)	No N/A
Is open, non-contained, or migrating vermiculite insulation present in the living space (including frequently accessed understructures)?	Yes (note on sketch)	No N/A
Inspection Assessment		
Could all of building (other than understructure) be inspected?	Yes	No (note in comments)
Type of Understructure	Basement (note on sketch) Cellar (note on sketch)	Crawlspace (note on sketch) None
Are any areas of the understructure inaccessible?	Yes (note on sketch)	No N/A
Are any areas of the understructure frequently accessed? (frequently accessed defined as 12 times a year minimum)	Yes (note on sketch)	No N/A
Are any areas of the understructure infrequently accessed? (frequently accessed defined as 12 times a year minimum)	Yes (note on sketch)	No N/A
Bulk/Building Materials		
Evidence of vermiculite additives used in building materials?	Yes (note in comments)	No
Are vermiculite containing building materials friable/ deteriorated or are in an area planned for demo or re-model?	Yes (note on sketch and collect sample)	No N/A
Comments		

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APPENDIX D
Analytical Requirements Summary Sheet (OU4GI0410-Rev4)

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SAP ANALYTICAL SUMMARY # OU4GI0410
SUMMARY OF PREPARATION AND ANALYTICAL REQUIREMENTS FOR ASBESTOS

Title: Quality Assurance Project Plan for General Property Investigations, Revision 4, OU4 and OU7, Libby Asbestos Site

SAP Date/Revision: April 7, 2014 (Revision 4)

EPA Technical Advisor: Liz Fagen (303-312-6095, Fagen.Elizabeth@epamail.epa.gov)
 (contact to advise on DQOs of SAP related to preparation/analytical requirements)

Sampling Program Overview: The objectives of the GPI sampling program are two-fold: 1) perform screening investigations (SIs) at OU4 and OU7 properties to identify sources of Libby amphibole asbestos (LA); and 2) based on SI findings, perform detailed investigations (DIs) at the properties to determine the extent of LA removal required at properties. SI consists of a verbal interview with property owners, visually inspecting each property for potential sources of LA indoors and outdoors, and collecting 30-point composite surface soil samples. DIs consists of inspecting, in detail, indoor and outdoor areas and collecting 30-point composite surface soil samples. Routine health and safety monitoring of field personnel in accordance with OSHA guidelines.

Sample ID Prefix: CS- for archived dust samples
1D- for archived dust samples
2S- for SI samples and 2D- for DI samples (effective 04/23/10-04/15/12)
3G- (effective 04/16/12-04/14/13)
4G- (effective 04/15/13-present)

PLM Preparation and Analytical Requirements:

Medium Code	Sample Type	Preparation Method	Analysis Method	Applicable Laboratory Modifications (current version of)
A	Soil – all field samples and field duplicate samples	ISSI-LIBBY-01 Rev. 11	PLM-Grav: SRC-LIBBY-01 Rev. 3 PLM-VE: SRC-LIBBY-03 Rev. 3	LB-000073, LB-000088
D	Bulk Material	EPA/600/R-93/116	PLM-PC400	none
E	Bulk Material	PLM-9002	PLM-9002	LB-000087

Medium-Specific TEM/PCM Preparation and Analytical Requirements for Field Samples:

Medium Code	Medium, Sample Type	Preparation Details				Analysis Details			Applicable Laboratory Modifications (most current version)
		Investigative?	Indirect Prep?		Filter Archive ?	Method(s)	Recording Rules	Analytical Sensitivity/ Prioritized Stopping Rules	
			With Ashing	Without Ashing					
B	Health & Safety Personal Air	No	No	Yes ^[a] , if material is overloaded (>25%) or unevenly loaded on filter	Yes	PCM – NIOSH 7400, Issue 2 TEM–AHERA (upon request)	For PCM: NIOSH 7400, “A” rules If AHERA is requested: All asbestos ^[b] , L ≥ 0.5 μm AR ≥ 5:1	For PCM: Count until 100 fibers are detected. Count a minimum of 20 FOVs. Stop at 100 FOVs regardless of count. For AHERA: Count until one is achieved: i) Target S = 0.005 s/cc, or ii) Evaluate a minimum filter area of 0.1 mm ² , or iii) 25 LA structures are enumerated (finish GO where 50 th LA found)	For PCM: LB-000015 For AHERA: LB-000029, LB-000031, LB-000067, LB-000085 LB-000091
E	Dust	No	No	Yes [a]	Yes	SRC-LIBBY-05/ASTM D5755-09	All Asbestos; L:≥0.5μm AR:≥3:1	Target S = 1000 1/cm2	LB-000029, LB-000031, LB-000040A, LB-000067, LB-000085, LB-000091

^[a] See most current version of EPA-LIBBY-08 for preparation details

^[b] Recording of chrysotile can stop after 50 chrysotile structures have been recorded (finish GO where 50th chrysotile found).

TEM/PCM Preparation and Analytical Requirements for Field Quality Control Samples:

Medium Code	Medium, Sample Type	Preparation Details			Analysis Details			Applicable Laboratory Modifications (most current version)
		Indirect Prep?		Archive?	Method	Recording Rules	Stopping Rules	
		With Ashing	Without Ashing					
C	Air, Field Blank	No	No	Yes	PCM – NIOSH 7400, Issue 2	For PCM: NIOSH 7400, “A” rules	For PCM: Count until 100 fibers are detected. Count a minimum of 20 FOVs. Stop at 100 FOVs regardless of count.	For PCM: LB-000015
F	Dust, Field Blank	No	Yes[a]	Yes	SRC-LIBBY-05/ASTM D5755-09	All Asbestos; L:≥0.5μm AR:≥3:1	Evaluate a minimum filter area of 0.1 mm ²	LB-000029, LB-000031, LB-000040A, LB-000067, LB-000085, LB-000091

Laboratory Quality Control Sample Frequencies:**TEM [c]:** Lab Blank – 4%

Recount Same – 1%

Verified Analysis – 1%

Repreparation – 1%

Recount Different – 2.5%

Inter-laboratory – 0.5% [d]

PLM [e]:

Lab Duplicates – 10% (cross-check 8%; self-check 2%)

Inter-laboratory – 1% [d,f]

PCM [g]: Blind Recounts – 10%

[c] See LB-000029 for selection procedure and QC acceptance criteria

[d] *Post hoc* selection to be performed by the QATS contractor

[e] See SRC-LIBBY-03 for QC acceptance criteria

[f] See LB-000073 for inter-laboratory acceptance criteria

[g] See NIOSH 7400 for QC acceptance criteria

Requirements Revision:

Revision #:	Effective Date:	Revision Description
0	4/23/2010	N/A
1	4/16/2012	<ul style="list-style-type: none"> Sample ID prefix change from 2S- / 2D- to 3G- to correspond to GPI SAP revision Bulk samples of building materials containing vermiculite will no longer be collected or analyzed
2	10/31/2012	<ul style="list-style-type: none"> PCM and field blank preparation and analytical requirements added. Applicable lab modification for soil updated.
3	4/15/2013	<ul style="list-style-type: none"> Update title of guidance document (Revision 2) Sample ID prefix change from 3G- to 4G- to correspond to GPI SAP/QAPP revision Updated AHERA analysis details
4	03/21/2014	<ul style="list-style-type: none"> Added media codes D and E for bulk material sample analysis Revised stopping rules for medium code B to correspond to RA QAPP requirements

Analytical Laboratory Review Sign-off:

All laboratories signed the original version of this analytical summary sheet (Rev 0); this revision (Rev 4) did not require another signature process.

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APPENDIX E
Human Subjects Research Memorandum

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UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

REGION 8
999 18TH STREET- SUITE 300
DENVER, CO 80202-2466
Phone 800-227-8917
<http://www.epa.gov/region08>

May 1, 2013

Ref: 8RA

MEMORANDUM

SUBJECT: General Property Investigation for the Libby Asbestos Site, Operable Unit 4

FROM: Patti Lynne Tyler
Human Subjects Research Officer

TO: Dania Zinner
Remedial Project Manager

Mike Cirian
Onsite Remedial Project Manager

Elizabeth Fagen
Libby Asbestos Site, OU4, Remedial Project Manager

Rebecca Thomas
Libby Asbestos Project Team Leader

I have reviewed the below referenced Sampling Analysis Plan/Quality Assurance Project Plan and have determined that this project does not meet the regulatory definition of research involving human subjects, which is defined as a *systematic investigation, including research development, testing and evaluation, designed to contribute to generalizable knowledge* (40 CFR 26 (d) and (f)). The goals of the General Property Investigation are to *collect data to confirm the presence/absence of Libby Amphibole asbestos (LA) and/or LA source materials at residential, commercial, industrial and public properties within Operable Unit 4 and to collect data to determine the extent of removal activities at properties within OU4 where previous investigations indicate the presence of LA and/or LA source materials.* Since this project does not incorporate activities meeting the regulatory definition of human subjects research, the project is not subject to the Common Rule or to the additional requirements found in EPA Regulation 40 CFR 26 (Protection of Human Subjects), and there is no regulatory requirement for oversight by an Institutional Review Board.

Please be sure to contact me should the goals of the project change.

Project Title: General Property Investigation Sampling and Analysis Plan/Quality Assurance Project Plan for the Libby Asbestos Site, Operable Unit 4, Libby, Montana, Revision 2, April 2013

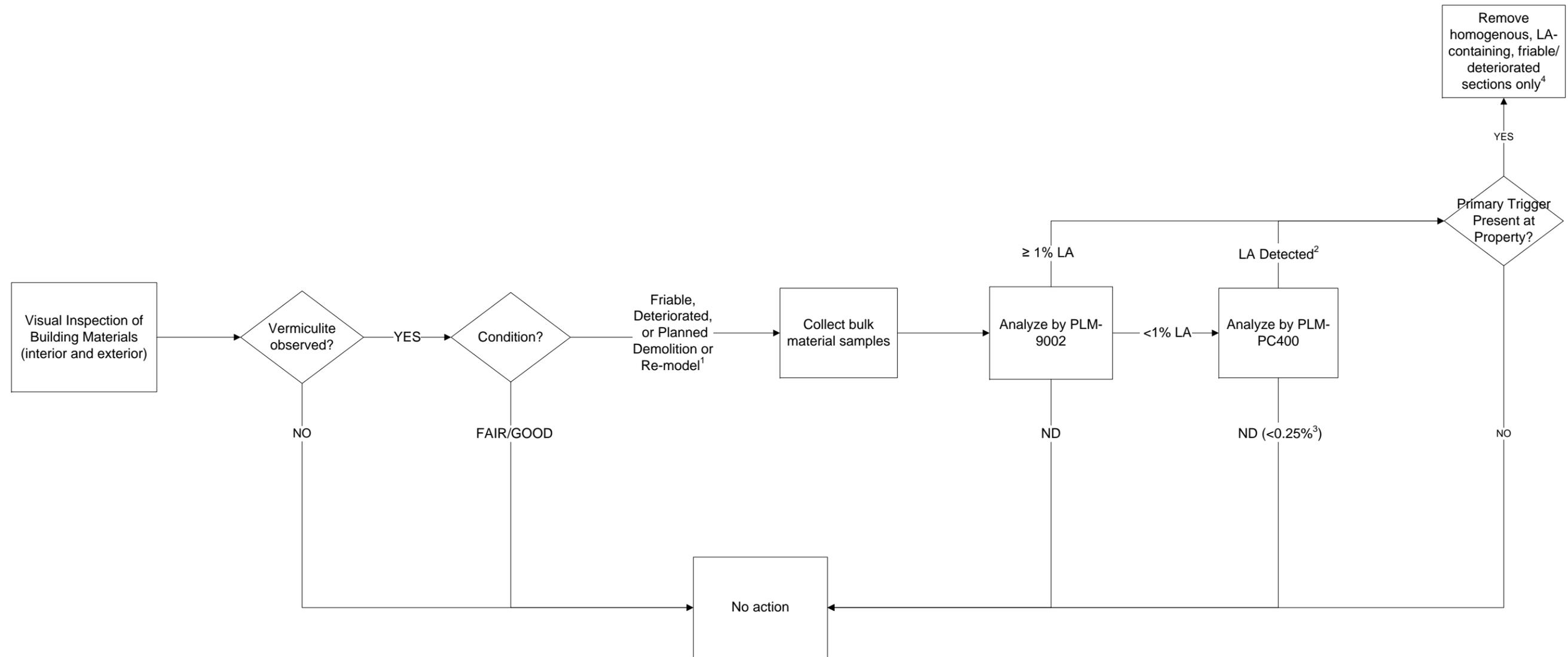
EPA Contract # and Project Managers: W912F-11-D-0023, Task Order No. 0003
U.S. Army Corps of Engineers, Omaha District
Mary Darling, USACE Project Manager
CDM Smith, CDM Federal Programs Corporation
Thomas Cook, CDM Smith, Project Manager

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APPENDIX F
Decision Tree for VCBM

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DECISION TREE FOR VERMICULITE-CONTAINING BUILDING MATERIALS (VCBM)



¹VCBM samples will be collected if the material is friable, deteriorated, or demolition or re-modeling is planned in buildings or areas of buildings with VCBM present.

²LA may be positively detected at a level of less than 0.25% if more than 400 points are evaluated during the PLM-PC400 analysis.

³The detection limit for PLM-PC400 is 0.25% if exactly 400 points are evaluated during analysis. A result of <0.25% by PLM-PC400 indicates LA was positively detected using PLM-9002, but not positively detected using PLM-PC400 analysis at the sample-specific detection limit.

⁴Owner request to remove homogenous, LA-containing, non-deteriorated material may be considered by the EPA

Definitions:

LA: Libby amphibole asbestos

ND: none-detected

PLM: polarized light microscopy

PLM-PC400: PLM by EPA/600/R-93/116 (400 points)

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