Libby Asbestos Superfund Site
Operations and Maintenance Manual

Libby and Troy Residential and Commercial Properties, Parks, and Schools
Operable Units 4 and 7
Lincoln County, Montana

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Table of Contents

List of Tables

List of Appendices

List of Acronyms

AHERA	Asbestos Hazard Emergency Response Act
ARAR	applicable or relevant and appropriate requirements
ARP	Lincoln County Asbestos Resource Program
BMP	Best Management Practice
BNSF	Burlington Northern Santa Fe
BOH	City-County Board of Health for Lincoln County
CA	Cooperative Agreement
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
COC	Chain-of-Custody
DEQ	Department of Environmental Quality
EC	Engineered Control
EPA	U.S. Environmental Protection Agency
EQC	Environmental Quality Council
ESD	Explanation of Significant Difference
GIS	Geographic Information System
Grace	W.R. Grace Company
HEPA	High Efficiency Particulate Air
HI	Hazard Indices
ICIAP	Institutional Control Implementation and Assurance Plan
IC	Institutional Control
LA	Libby amphibole asbestos
LASOC	Libby Asbestos Superfund Oversight Committee
LUA	Limited Use Area
MCA	Montana Code Annotated
MDT	Montana Department of Transportation
MOA	Memorandum of Agreement
NCP	National Contingency Plan
NESHAP	National Emissions Standards for Hazardous Air Pollutants
NIST	National Institute of Standards and Technology
NOEC	Notice of Environmental Conditions
NOPEC	Notice of Potential Environmental Conditions
NPL	National Priority List
NUA	Non-use Area
NVLAP	National Voluntary Laboratory Accreditation Program

O&M	Operations and Maintenance
OSHA	Occupational Safety and Health Administration
OU	Operable Unit
PEN	Property Evaluation Notification
PLM	Polarized Light Microscopy
PLM-VE	PLM visual area estimation method
PM	Project Manager
QA	Quality Assurance
QAM	Quality Assurance Manager
QC	Quality Control
RA	remedial action
RACR	Remedial Action Completion Report
RAL	Remedial Action Level
RAO	remedial action objectives
RM	Response Manager
ROD	Record of Decision
RPM	Remedial Project Manager
s/cc	Structures per cubic centimeter
SARA	Superfund Amendments Reauthorization Act
SOP	Standard Operating Procedures
SOW	Statement of Work
TEM	Transmission Electron Microscopy
TIN	Taxpayer Identification Number
VCM	vermiculite containing material

1 Introduction

This Operations and Maintenance (O&M) Manual presents detailed protocols and procedures for implementing routine O&M activities at Operable Units (OUs) 4 and 7 of the Libby Asbestos Superfund Site (Site). It has been developed to meet the O&M objectives described in the *Final Operations and Maintenance Plan* (CDM Smith 2020a) in accordance with the U.S. Environmental Protection Agency's (EPA) *Guidance for Management of Superfund Remedies in Post Construction* (EPA 2017). The O&M Manual includes detailed information used to operate and maintain the remedy, while the O&M Plan is designed to be more of a management document. The completed O&M Plan and O&M Manual are submitted as part of the remedial action (RA) completion when remedy transitions to O&M. Both documents should be updated during O&M as conditions change.

This manual is intended to be a dynamic or "living" document whereby the most recent guidance and operating information is included for use by Montana Department of Environmental Quality (DEQ) staff and delegated authorities (i.e. Lincoln County, contractors, etc.). It is intended to provide detailed user information for use by entities charged with implementing O&M activities at the Site. DEQ will evaluate this manual annually to determine if updates are needed. If deemed necessary, DEQ will work with the affected entities to incorporate updates and/or revisions into the manual. All revisions will be provided to EPA for their record.

It should be noted that clean-up and response activities at the Site are referenced in various ways throughout this document. Prior to O&M, clean-up activities at the Site are generally referred to as removal or remedial actions which are specific programs under the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA, commonly referred to as Superfund). For O&M, clean-up activities will be referred to as abatement to reflect that CERCLA cleanup is complete. Since contaminated source material remains at the Site, O&M abatement activities are expected to continue in the future to address risk that may result from changes in property and/or use.

1.1 Background Information

1.1.1 History of Site

Libby is a community in northwestern Montana that is located near a former vermiculite mine. The vermiculite mine near Libby began limited operations in the 1920s and was operated on a larger scale by the W.R. Grace Company (Grace) from approximately 1963 to 1990. Vermiculite from the mine contains varying concentrations of amphibole asbestos, referred to as "Libby amphibole asbestos" or LA. Epidemiological studies revealed that workers at the mine had an increased risk of developing asbestos-related lung disease. In October 2002, the Site was listed on the National Priorities List (NPL). The Site includes homes and businesses that may have become contaminated with LA as a result of the vermiculite mining and processing conducted in and around Libby, as well as other areas that may have been affected by mining-related releases of LA.

Previous investigations conducted at the Site have demonstrated that LA is present in a variety of media (e.g., soil, bulk materials) from source materials (e.g., vermiculite insulation, vermiculite-containing soils, mine wastes) at properties throughout the Site. 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.

For long-term management purposes, the Site has been divided into eight (8) OUs. OU1 and OU2 include the former Export Plant and Screening Plant, respectively; OU3 includes the former vermiculite mine and the surrounding area; OU4 encompasses the residential, commercial, and public properties in and around Libby; OU7 includes residential, commercial, and public property in and around Troy (about 20 miles west of Libby); OU5 is the 400-acre industrial park (former Stimson Lumber Mill); OU6 contains all Burlington Northern Santa Fe (BNSF) railroad property in and between OU4 and OU7, including rights-of-way and rail yards; and OU8 consists of the U.S., state, and county rights-of-way within and between OU4 and OU7.

Between 2000 and 2019, EPA conducted numerous removal and remedial actions to address LA-contaminated materials. A Sitewide Human Health Risk Assessment, which evaluated potential health risks from exposures to LA in each of the OUs, was completed in 2015.

1.1.2 ROD

The Record of Decision (ROD) is the final decision document at the end of a detailed investigation and evaluation of conditions at the Site. The purpose of the ROD is to document the contaminants of interest and media of concern, establish the remedial action objectives (RAOs) and remedial goals, summarize the evaluation of alternatives, and identify the selected remedy. The RODs for the former processing areas (OU1 and OU2) were completed in 2010 (EPA 2010a, 2010b). The ROD for OUs 4 through 8 was completed in 2016 (EPA 2016). The Feasibility Study for OU3 is currently in preparation. The focus of this O&M manual is on OUs 4 and 7.

1.1.2.1 Media of Concern

In OUs 4 through 8, LA-contaminated soil and building materials are the primary source media that contain LA, and when disturbed, could result in unacceptable human health risks. Although indoor air and outdoor air are the exposure media that result in human health risks, these exposure media become impacted via disturbance of the source media (e.g., contaminated soil and building materials). Thus, they cannot be directly remediated, but are addressed by remediation of the contributing source media. EPA determined that not all vermiculite has LA, so analytical testing of soils for LA was necessary to determine if remediation was required. Accessible vermiculite insulation in any quantity required remediation and was not tested for LA due to the impartibility of separating the minerals.

1.1.2.2 Remedial Action Objectives

The RAOs were developed to restrict or mitigate through management of the continued release and migration of LA from contaminated soil and building materials, thus protecting human receptors from unacceptable exposure to LA. The RAOs for OUs 4 through 8 include:

- Minimize the inhalation of LA during disturbances of soil contaminated with LA such that the resulting exposures result in cumulative cancer risks that are within or below EPA's acceptable risk range of 10^{-6} to 10^{-4} and cumulative non-cancer hazard indices (HIs) that are at or below 1.

- Minimize the inhalation of LA during disturbances of building materials contaminated with LA such that the resulting exposures result in cumulative cancer risks that are within or below EPA's acceptable risk range of 10^{-6} to 10^{-4} and cumulative non-cancer HIs that are at or below 1.

1.1.2.3 Land Use Categories

There are four (4) separate land use categories that were identified in the ROD for non OU3 areas: (1) residential/commercial, (2) industrial, (3) transportation corridors, and (4) parks/schools. It should be recognized that while these land use categories were primarily identified to categorize existing land uses for properties, they also form the framework for evaluation of land uses when a property owner elects to change the land use in the future.

Residential/Commercial

This land use category includes private residential/commercial properties as well as public properties within the City of Libby (OU4) and the City of Troy (OU7) that are currently used, or will be used in the future, for residential and commercial/governmental purposes (excluding Federal agency land or structures) that are not involved in large-scale manufacturing of products for sale and export outside of the Site. This land use category includes alleyways and city streets within OU4 and OU7, as well as churches that do not provide primary, secondary, or higher education in a school setting. This land use category also includes future public and private school properties within the OU4 and the OU7 that do not currently exist but are planned to provide primary, secondary, or higher education.

Industrial

This land use category includes industrial properties that are currently or will be used in the future for industrial purposes (e.g., large-scale manufacturing of products for sale and export outside of the Site). This category includes rail spurs and roadways within an industrial property but excludes rail and highway transportation corridors (OU6 and OU8, respectively) that border industrial properties. It should be noted that, to date, there are no existing or future-planned industrial properties in OU4 or OU7.

Transportation Corridors

This land use category includes rail and highway corridors that are currently used, or will be used in the future, for vehicular transportation. This land use category also includes buildings and facilities used by transportation entities (e.g., Montana Department of Transportation [MDT], BNSF Railway).

Although none currently exist, this category excludes rail or highway corridors that are converted and repurposed in the future to non-vehicular recreational trails (e.g., "rails to trails" projects), which would be part of the park/school properties category.

Parks/Schools

This land use category includes the park properties within the OU4 and the OU7 that are currently used, or will be used in the future, for public or commercial recreational purposes. It also includes roadways within public or commercial parks.

This land use category also includes the public and private school properties within the OU4 and the OU7 that are currently used to provide primary, secondary, or higher education. Churches that do not provide primary, secondary, or higher education and schools established in the future are part of the residential/commercial properties land use category.

1.1.2.4 Remedial Action Levels and Criteria

Remedial criteria are qualitative and quantitative thresholds used to determine if cleanups of source media are warranted to address exposures that pose unacceptable risks, and when those cleanups result in acceptable risk reduction.

Remedial actions at the Site are guided based on two (2) types of remedial criteria – remedial action levels (RALs) and remedial clearance criteria. The RAL defines the condition when remedial action is and is not needed due to LA contamination in soil and building materials. For example, if surface soil conditions are below the RAL, no action is needed; if surface soil conditions are at or above the RAL, then action is warranted. The remedial clearance criteria define the conditions that must be met for the physical components or approaches of the remedial action to be deemed complete.

RALs for contaminated soil were developed separately for each of the following land use categories – residential/commercial, industrial, transportation corridors, and parks/schools. Within a residential/commercial property, outdoor exposures may differ as a function of location within a property (e.g., the amount of time spent in yards is expected to be different than time spent in a garden). For this reason, for residential/commercial properties, the RALs were further stratified based on the frequency of outdoor area use. The term “frequently used areas” applies to those areas of residential/commercial properties that are likely to be used on a regular basis by residents and outdoor workers such as yards, gardens, flowerbeds, play areas, unpaved walkways and driveways, lawns, and landscaped areas. The term “infrequently used areas” applies to those areas of residential/commercial properties that are likely to be used on a less regular basis such as pastures and fields, wooded lots, and areas beneath structures (e.g., soils beneath low clearance decks and raised sheds). The four (4) exposure areas as categorized in the ROD are as follows:

- Yards (frequent use area)
- Gardens/flowerbeds (frequent use area)
- Driveways (frequent use area)
- Limited-use areas (infrequent use area)

Likewise, the indoor clearance criteria for residential/commercial properties also considers the frequency of indoor area use. The term “living spaces” applies to areas with higher use, such as living rooms, bedrooms, kitchens, dining rooms, bathrooms, finished attics, finished basements, and offices. The term “non-living spaces” applies to areas with less frequent use, such as unfinished attics, unfinished basements, attached garages, and utility closets. The term “understructure” refers to the area at or below grade that is below the main structure of primary buildings. Understructures include crawlspaces, basements, and cellars. The clearance criteria for the understructure depends on the frequency of access. An understructure is considered frequently accessed if it is entered more than 12 times per year or if activities during access include significant soil disturbance such as digging. An

understructure is considered infrequently accessed if it is entered 12 times per year or less and activities during access involve minimal soil disturbance. Soils located within the living areas, not including understructures, of primary buildings are considered indoor soils. These may include, but are not limited to, indoor planters or soil floors.

Table 1 summarizes the RALs and remedial clearance criteria for contaminated soils and building materials established in the ROD.

Table 1 – RALs and Remedial Clearance Criteria for Contaminated Soils and Building Materials

Libby Asbestos Superfund Site Land Use Categories	Soil Remedial Action Level for Frequently Used Areas and Infrequently Used Areas	Remedial Clearance Criteria for Surface Soil	Remedial Clearance Criteria for Sub- Surface Soil	Remedial Action Level for Contaminated Building Materials	Remedial Clearance Criteria for Contaminated Building Materials in an Indoor Space
Residential/Commercial	*Frequently Used Areas:* LA soil concentrations of Bin B2 (0.2% to <1%) or Bin C (≥1%) by polarized light microscopy (PLM)-visual area estimation (VE) *Or* LA soil concentrations of Bin B1 by PLM-VE if the spatial extent of the Bin B1 area is more than 25 percent of the total soil exposure area at a property *Infrequently Used Areas*: LA soil concentrations of Bin B2 (0.2% to <1%) or Bin C (≥1%) by PLM-VE	*For Frequently Used Areas:* No LA soil concentrations of Bin B2 (0.2% to <1%) or Bin C (≥1%) by PLM-VE can be present *and* No more than 25% of the total soil exposure area can be Bin B1 by PLM-VE and the remainder of the total soil exposure area is Bin A *Infrequently Used Areas:* No LA soil of Bin B2 (0.2% to <1%) or Bin C (>1%) by PLM-VE can be present.	Confirmation soil samples collected at the depth of excavation are Bin A or Bin B1 by PLM-VE (i.e., LA is not present or is present at levels less than 0.2 percent) unless boundary conditions (e.g., depth of excavation reaches 36-inch depth) are reached.	Presence of accessible LA-containing vermiculite insulation in any quantity in living spaces, non-living spaces, and/or secondary structures *Or* Presence of accessible friable and/or deteriorated building materials containing greater than or equal to 0.25 percent LA by PLM using point counting (400 points examined) (PLM-PC400) (e.g., chinking, plaster, mortar, and other materials on boilers, pipes, or other appurtenances).	*Indoor Living Space:* No accessible vermiculite remaining *And* All five (5) clearance air samples, after remedial actions are complete have total LA air concentrations that are non-detect when analyzed by transmission electron microscopy (TEM) using Asbestos Hazard Emergency Response Act (AHERA) counting rules achieving analytical sensitivity of 0.005 cc^{-1} . *Indoor Non-Living Space*: No accessible vermiculite remaining *And* All five (5) clearance air samples, after remedial actions are complete have an average total LA air concentration <0.005 s/cc when analyzed by TEM using AHERA counting rules

Libby Asbestos Superfund Site Land Use Categories	Soil Remedial Action Level for Frequently Used Areas and Infrequently Used Areas	Remedial Clearance Criteria for Surface Soil	Remedial Clearance Criteria for Sub- Surface Soil	Remedial Action Level for Contaminated Building Materials	Remedial Clearance Criteria for Contaminated Building Materials in an Indoor Space
					achieving analytical sensitivity of 0.005 cc^{-1}.
Industrial	LA soil concentrations of Bin C (≥1%) by PLM-VE	No LA soil concentrations of Bin C (≥1%) by PLM-VE can be present.	Confirmation soil samples collected at the depth of excavation are Bin A, Bin B1 or Bin B2 by PLM-VE (i.e., LA is not present or is present at levels less than 1 percent) unless boundary conditions (e.g., depth of excavation reaches 36-inch depth) are reached.	Same as Residential/Commercial	Same as Residential/Commercial
Parks/Schools	LA soil of Bin B2 (0.2% to <1%) or Bin C (≥1%) by PLM-VE	No LA soil of Bin B2 (0.2% to <1%) or Bin C (≥1%) by PLM-VE can be present.	Confirmation soil samples collected at the depth of excavation are Bin A or Bin B1 by PLM-VE (i.e., LA is not present or is present at levels less than 0.2 percent) unless boundary	Same as Residential/Commercial	Same as Residential/Commercial

Libby Asbestos Superfund Site Land Use Categories	Soil Remedial Action Level for Frequently Used Areas and Infrequently Used Areas	Remedial Clearance Criteria for Surface Soil	Remedial Clearance Criteria for Sub- Surface Soil	Remedial Action Level for Contaminated Building Materials	Remedial Clearance Criteria for Contaminated Building Materials in an Indoor Space
			conditions (e.g., depth of excavation reaches 36-inch depth) are reached.		

AHERA = Asbestos Hazard Emergency Response Act
PLM= Polarized Light Microscopy
PLM-VE = PLM visual area estimation method
s/cc = structures per centimeter
TEM = Transmission Electron Microscopy

1.1.2.5 Boundary Conditions

Boundary conditions at OU4 and OU7 discussed in **Table 1** are features or conditions that limit the ability to further remediate LA contamination due to physical or technical constraints and the related lack of accessibility. Boundary conditions include the following:

- the presence of building foundations compromised by the response action;
- the presence of relatively permanent pavement (e.g., roadways and sidewalks);
- the presence of large tree root systems;
- the presence of bedrock;
- the presence of groundwater that was not seasonal or perched and thus could not be readily avoided;
- a pre-set maximum vertical extent of 3 feet below ground surface, due to limited future accessibility to subsurface soils under typical residential, commercial, and park and school activities; and,
- a maximum horizontal extent to the adjacent property boundary where cleanup occurred or where other boundary conditions (e.g., pavement, bedrock, and tree roots, as identified above) existed.

1.1.3 Historical and Relevant Documents

1.1.3.1 Remedial Action Completion Report

The purpose of the Remedial Action Completion Report (RACR) is to document that cleanup levels were achieved and that OU4 and OU7 have been remediated in accordance with the ROD. Information contained in the RACR (CDM Smith 2020b) includes site background and current site information, historical investigations and response action activities, ROD requirements, remedial design, completed construction activities, performance standards and quality control, final inspections and certifications, and O&M activities.

1.1.3.2 Operations and Maintenance Plan

The O&M Plan presents the administrative, financial, and technical aspects and requirements for inspecting, operating, and maintaining the remedial action for OU4 and OU7. Information contained in the O&M Plan (CDM Smith 2020a) includes site background and current site information, objectives for site inspections, physical remedy O&M activities, institutional control monitoring requirements, reporting requirements, summary of five-year review activities, and an O&M cost estimate.

1.1.3.3 Institutional Control Implementation and Assurance Plan

The Institutional Control Implementation and Assurance Plan (ICIAP) presents the institutional controls (IC) necessary to maintain the remedies or minimize encounters of LA in OU4 and OU7. Information contained in the ICIAP (CDM Smith 2020c) includes roles and responsibilities, site background and history, key elements for planned and implemented ICs, maintenance, enforcement, modification and termination of ICs.

1.1.3.4 Explanation of Significant Differences

Following public review and comment on the final ICIAP, a modification to the ROD will be prepared, known as an Explanation of Significant Differences (ESD). This document is intended to describe changes

to the remedy selected in the ROD that occur during the remedial design/remedial action process. The ESD will reference the ICIAP and will identify the specific IC requirements and IC tools to be used to implement the ICs selected in the ROD.

1.1.3.5 Repository of Relevant Historical Documents

The administrative record is the official repository for documents associated with the remedial actions completed at the site. Relevant historical documents that may require quick reference by personnel during O&M activities have been included in this manual for ease of access. These historical reference documents are included in **Appendix A.**

1.2 O&M Funding

DEQ is responsible for O&M funding of OUs 1, 2, 4, 5, 7, and 8. O&M funding for OUs 3 and 6 are the responsibility of the identified responsible parties. Three (3) different funding sources are currently available to DEQ for O&M activities at the site. These funding sources are subject to various funding restrictions and can only be used within the applicable OU boundaries. DEQ is responsible for managing all O&M funds in accordance with the applicable requirements. The various funding sources are described below. See **Appendix B** for more information on the use of O&M settlement funds.

1.2.1 Federal Funds

A settlement fund was set up for the Site. From the settlement fund, $11 million was placed into a separate interest-bearing account that will be used to help pay for future site-wide O&M (e.g., all OUs except OU3 and OU6). Currently, the funds in that account are nearly $12 million. These funds are administered by EPA to DEQ through a cooperative agreement (CA) and are subject to EPA eligibility requirements. Further guidance regarding funding for remedy maintenance activities during the O&M period are discussed in the *Guidance for Management of Superfund Remedies in Post Construction* (EPA 2017).

1.2.2 State Funds

In addition to Federal funds, under Montana's 2015 Senate Bill 20 legislation, DEQ received an appropriation of $600,000 annually from an orphan share transfer starting on July 1, 2018. The subsequent 2017 Senate Bill 315 provided a framework on how this money could be used and established a permanent trust fund to pay exclusively for costs of cleanup and long-term O&M for Libby. Annually, $48,000 is allocated from this account for oversight and support of the advisory team (i.e. Libby Asbestos Superfund Oversight Committee [LASOC]). As of September 2019, the trust fund balance was $852,536. In addition, DEQ also received approximately $5 million as part of the bankruptcy settlement with Grace. As determined by DEQ, after consideration of LASOC recommendations and state CERCLA policy and precedent, these funds can also be used to support O&M activities in OU4 and OU7.

2 O&M Roles and Responsibilities

This section describes the roles and responsibilities for each of the organizations responsible for specific O&M activities and the financial agreements to carry out the O&M activities.

2.1 EPA

EPA is the oversight agency responsible for determining whether the remedy at the Site is protective of human health and the environment. In making this determination, EPA is responsible for conducting five-year reviews. Additionally, EPA is responsible for developing and executing a CA with DEQ and determining O&M costs that are eligible under federal funding.

2.2 DEQ

DEQ is responsible for implementing O&M activities for the Site. Federal funds are provided to perform these activities through a CA with EPA. State funds are also available. DEQ is responsible for the execution, administration and management of state funds, and the CA with EPA, and for the performance of the activities as described in the CA.

DEQ will contract, in compliance with applicable federal and state procurement regulations, the performance of the activities as necessary to accomplish the objectives of the O&M Plan and ICIAP. DEQ must comply with and/or require contractors and subcontractors to comply with the applicable EPA general terms and conditions reflected in the official CA award document and any prescribed additional assurances and certifications made as part of the award and terms, conditions or restrictions.

O&M activities will include, but are not limited to, inspections, sampling, analyses, routine maintenance, and reporting at various intervals. These activities are governed by CERCLA and the regulatory framework of its Trust Fund program, and the Superfund Amendment and Reauthorization Act (SARA) of 1986. Many of these activities will be delegated to Lincoln County through a state and county developed Memorandum of Agreement (MOA). Those activities are described in **Section 2.3**.

The O&M tasks to be performed by the DEQ (or its designee) includes, but is not limited to the following:

- Project Oversight
    - Manage and coordinate all aspects of O&M;
    - Prepare CA;
    - Review and comment on project deliverables;
    - Prepare property status letters;
    - Keep management, project team members, and EPA informed of project progress and other relevant issues;
    - Develop and maintain cooperative working relationships with counterparts from other regulatory agencies (federal, State, and local), in-house staff members, and the public;
    - Ensure that the public participation requirements of CERCLA and the National Contingency Plan (NCP) are met so that the community is kept informed of Site activities and appropriately involved in Site decision-making;

- Annual Inspections
  - Provide and update the O&M Manual that defines the administrative and technical details and requirements for inspecting, operating, and maintaining the remedy;
  - Observe site conditions such as landscape, drainage, erosion, and integrity of the remedy on properties where access has been granted;
  - Inspect to identify failures or inefficiencies of ICs, and restrict access where needed when the integrity of physical remedies and engineered controls (ECs) are compromised;
- Routine Maintenance
  - Maintain the function and integrity of the remedy;
  - Maintain ICs;
- Reporting
  - Quarterly Progress Reports to EPA of site-specific expenditures and on operation, maintenance and adjustments of the remedy;
  - Summaries of annual sampling and monitoring results;
- Contracted Services
  - Procure asbestos laboratory and abatement services (when necessary);
  - Maintain a list of pre-qualified contractors to provide professional environmental engineering and other technical services for addressing investigation and abatement services at the Site;
  - Ensure contractors comply with the statement of work (SOW) to be completed under their agreement (**described further in Section 5.0)**;
  - Authorize and issue payments for cost reimbursement claims;
- DEQ Memorandum of Agreement with Lincoln County
  - Develop and implement MOA with local agencies and stakeholders;
  - Administer contracts, as necessary to implement ICs and to protect the physical remedy; and
  - Ensure past and future information regarding property investigations, response actions, and presence of known remaining LA and LA source materials is appropriately managed.

2.3 ARP

The City-County Board of Health for Lincoln County (BOH) - Lincoln County Asbestos Resource Program (ARP) is the current program staffed in Lincoln County, Montana, that was initially funded by EPA through the remedial action. The ARP works under the direction of the BOH. Through a DEQ and BOH developed MOA, ARP is the local presence responsible for implementing protective measures and selected ICs during O&M. ARP is a program that educates the public regarding the remaining risks of LA exposure, provides resources to manage the associated risks, and implements initiatives to reduce or prevent the risk of LA exposure. ARP will continue to provide information, as needed, to assist property owners and their contractors in understanding the appropriate best management practices (BMP) and ICs that apply to their properties.

The O&M tasks anticipated to be performed by ARP includes, but is not limited to, the following:

- Data Entry into DEQ's Libby Instance of Response Manager (RM)
    - Document communications with property owners and/or contractors, address updates, Montana811, ARP Hotline calls, and Property Evaluation Notification (PEN) requests, site assessments, analytical data, SOWs, reimbursement calculations and approvals, oversight activities, as-builts, etc.;
- PEN/Montana811 Requests or ARP Hotline calls
    - Respond to PEN/Montana811 requests and/or ARP Hotline calls;
    - Track each disturbance request;
    - Evaluate the existing data and information related to LA at and adjacent to the property;
    - Conduct a site assessment if deemed necessary to determine the potential for LA to be encountered and provide relevant information to the homeowner and/or contractor;
- Property Status Requests
    - Conduct database queries for property status requests;
- Site Assessments
    - Perform site assessments to determine if investigation and/or abatement work is necessary;
- SOW Development
    - Prepare investigation and abatement SOWs for third-parties;
    - Assist DEQ and third-parties with the reimbursement process;
- Project Oversight
    - Provide oversight of third-party[1] sampling investigation and/or abatement work;
    - Review analytical results;
    - Conduct final inspection and document results, complete as-built drawings, and photo log;
    - Conduct confirmation sampling, as approved by DEQ;
    - Provide Lincoln County Solid Waste Class IV Asbestos Cell and LA-waste disposal information, as necessary;
- Education and Outreach
    - Provide information and updates through the website, social media, newspapers, promotional materials and mailings;
    - Provide brochures that contain BMP and information about reducing exposures;
    - Educate the public through training and outreach events;
    - Provide general contractor and sampling training;
- Supplies and Equipment
    - Procure, maintain, and provide equipment and supplies (e.g., Tyvek suits, polyurethane sheeting, asbestos disposal bags) for use by local residents for minor property disturbances to be addressed by the property owner;
    - Track and maintain the EPA-provided backfill materials;

[1] Third-party individuals can be the property owner or a contractor hired by the property owner.

- Annual Inspections
  - Support DEQ-led annual Sitewide O&M inspections;
  - Provide property information for a selected number of properties;
  - Participate in field reviews and conduct property owner interviews;
  - Assist with evaluation of ICs;
- Reimbursement Program
  - Work with property owners, developers, and contactors to develop SOWs and provide associated reimbursement eligibility determinations;
  - Provide recommendations to DEQ for approval and disbursement of funds for eligible activities based on verified work.

2.4 Libby Asbestos Superfund Oversight Committee

In 2017, the 65th Montana Legislature passed Senate Bill 315, which was signed into law by Governor Bullock and established a liaison position and a Libby Asbestos Superfund Advisory Team attached to DEQ for administrative purposes. In the 66th Montana Legislature, House Bill 30 was passed to eliminate the Liaison position, to rename the Advisory Team to the LASOC (Oversight Committee) and to add duties. Per Montana Code Annotated (MCA) 75-10-1601, the Oversight Committee consists of five (5) members as identified in the O&M contacts list in **Appendix C**.

Money has been deposited in a state special revenue account to fund cleanup and long-term operation and maintenance costs at the Site, as well as the administrative costs of the Committee. The Committee was established to monitor activities within the Site and is required to meet at least quarterly to:

- Assist in the implementation of final cleanup and long-term operation and maintenance plans for the Site;
- Review documents and provide comments and recommendations to the DEQ and to local governments and appropriate federal agencies regarding the Site;
- Provide recommendations to DEQ regarding the administration of the Libby asbestos cleanup trust fund (MCA 75-10-1603) and the Libby asbestos cleanup operation and maintenance account (MCA 75-10-1604);
- Initiate and strive to maintain negotiations with DEQ, EPA, and any other entity with a goal of reducing the state and federal roles in the long-term operation and maintenance work at the Site while increasing the role of Lincoln County in expending funds to manage and implement operation and maintenance activities; and
- Submit a report to the Environmental Quality Council (EQC) by July 1st of each year.

2.5 Montana Asbestos Control Program

As delegated by the EPA and the Asbestos Control Act of Montana, DEQ administers regulatory requirements from sections of the National Emissions Standards for Hazardous Air Pollutants (NESHAP) and Montana's Administrative Rules, governing building renovations/demolitions, asbestos disposal, and other asbestos-related activities. Montana's asbestos control program is not specific to LA. Rather, it applies to asbestos that is a group of naturally occurring fibrous minerals including chrysotile, amosite, crocidolite, anthophyllite, actinolite and tremolite that presents a potential exposure and health hazard

found in building materials. It is important to note that, although there is some overlap, the regulations governing asbestos under the Asbestos Control Act of Montana and the federal NESHAP are not the same as for LA.

The Asbestos Control Program regulates asbestos projects at facilities involving the encapsulation, enclosure, removal, repair, renovation, placement in new construction, demolition of asbestos in a building or other structure, or the transportation or disposal of asbestos-containing waste. Facilities are defined as institutional, commercial, public, industrial, or large residential complexes. Asbestos demolition or renovation projects at facilities require a project permit from the Asbestos Control Program and must be completed by persons with a Montana Contractor/Supervisor or Worker accreditation. However, the following exemptions to these requirements apply:

- Projects involving less than 10 square feet, 3 linear feet, or 3 cubic feet of material containing more than 1% asbestos;
- Single residential buildings having four (4) or fewer units, provided:
    - It is the only structure impacted and under the control of the same owner within 660 feet (roughly a city block);
    - The structure in question is free from being included in any previous or future projects;
    - The structure is free of any previous use for commercial, industrial, or other non-residential use; and
    - The structure is free from any historical scenario that would change the findings above.

Additionally, Occupational Safety Health Administration (OSHA) enforces its asbestos standards in the construction industry. EPA maintains jurisdiction over asbestos in schools in Montana. However, schools are still subject to Montana asbestos statutes and rules.

2.6 Property Owners

General property maintenance and management will be the responsibility of the property owner to ensure activities on their property do not disturb the physical protective remedy in place. Property owners are expected to:

- Understand the status of their property information with respect to investigations and/or remedial actions completed on the property; and,
- Contact ARP with questions regarding the potential for encountering LA or LA source materials and how to reduce exposure.

Table 2 includes typical activities of a property owner and the measures available to manage the potential for encountering LA or LA source materials.

Table 2 – Measures for Managing LA or LA Source Materials for Property Owners

Type of Activity	Activity	Measures to Take for Managing LA or LA Source Materials
Soil	Digging/Excavation	Call Montana 811 program
		Obtain an MDT permit if property is in right-of-way
		Obtain PEN authorization
		Call ARP
		Review educational programs for managing LA
		Learn what contractors are approved to manage LA
	Landscaping/ Gardening	Submit PEN Request
		Call ARP
		Review educational programs for managing exposure
	Waste Disposal	Call ARP and Lincoln County Solid Waste
Building Materials	Renovation/ Remodeling	Submit PEN Request
		Obtain PEN authorization
		Call ARP
		Review educational programs for managing exposure
		Learn what contractors are approved to manage LA
	Demolition	Submit PEN Request
		Obtain PEN authorization
		Call ARP
		Review educational programs for managing exposure
		Learn what contractors are approved to manage LA
	Repair & Maintenance	Call ARP
		Review educational programs for managing exposure
		Learn what contractors are approved to manage LA
	Waste Disposal	Call ARP and Lincoln County Solid Waste
Other Activities	Wood Harvesting	Obtain a Forest Service permit if gathering firewood in a National Forest
		Call ARP
		Review educational programs for managing exposure
	Stove/Fireplace Ash Disposal	Call ARP or and Lincoln County Solid Waste
		Review educational programs for managing exposure
	Snowplowing / Gravel Lots	Call ARP
	Education	Review educational programs for managing exposure

3 Public Education and Outreach

ARP, through a MOA with DEQ, will be the entity primarily responsible for implementing education and outreach activities in Lincoln County to educate the public regarding the risks of LA exposure and provide resources to manage associated risks. These activities include providing past clean-up information, resource materials and BMPs, and training to stakeholders and the public, as well as maintaining a list of contractors educated in LA-specific abatement practices.

3.1 Status Letters

When requested, ARP will research requests by homeowners, real estate agents, title companies, and lenders for historical property information. This information and the proposed status letter will be provided to DEQ for approval. Status letter templates are included in **Appendix D** and will be used to describe the current status of the property; however, the template can be revised if warranted. The purpose of these letters is to inform a property owner of the assessments and remediation work that has been completed in the past. BMP maintenance recommendations may also be included with the letter, if deemed relevant. ARP will upload copies of all status letters to RM and track the number of requests received each year. The need for issuing a status letters will be evaluated annually.

3.2 Educational Materials and Outreach

Educational and resource programs have been developed and are provided by ARP to the public so that they can be knowledgeable about recognizing LA and LA source materials and employ BMPs to ensure that potential for LA exposure is limited. ARP strives to make sure the public is aware of what to look for and how to deal with LA and LA source materials prior to or when they may encounter it on their property.

Outreach to the community to help the public manage LA contamination is provided through social media, the ARP website, site assessments, and outreach events. ARP makes site visits to schools, construction sites, and homes to help the public manage LA contamination as part of their outreach role. In addition, ARP organizes outreach events to further connect the community with information (e.g., annual health fair in Libby). During outreach events, they show examples of LA and LA source materials, explain what to look for within soil and buildings to identify LA and LA source materials, and provide ARP contact information.

A large part of ARP's educational program focuses on BMP awareness. ARP develops brochures with BMP information and user guides on what to look for and what to do when encountering vermiculite in yards or within houses to reduce exposure. The BMP education and training materials provided by ARP are listed below:

- *Best Management Practices Manual* (EPA 2019)
- LA Exposure Brochure
- Contractors & Tradesmen Working Indoors Brochure
- Contractors & Tradesmen Working Outdoors Brochure
- Demolition Activities Brochure
- Libby and Troy Residents Brochure

- Lincoln County Do-It-Yourselfer Brochure
- Yard Work and Gardening Activities Brochure
- Reducing Asbestos Exposure Fact Sheet
- Maintaining Engineered Controls Fact Sheet (to be developed)
- Libby and Troy PEN Regulation Fact Sheet (to be developed)
- LA Reimbursement Program Fact Sheet (to be developed)
- LA Sampling Guidance for Soils Fact Sheet (to be developed)
- LA Sampling Guidance for Building Materials Fact Sheet (to be developed)
- Transporting LA Waste to the Landfill Fact Sheet (to be developed)

These education and training materials will be reviewed annually and updated as appropriate by DEQ with assistance from ARP.

3.3 Training Events

In coordination with DEQ, ARP will establish and provide periodic training on LA-specific hazards, including contractor awareness training and sample training. Contractor awareness training targets new workers who have not previously worked within the NPL site and are not familiar with the potential hazards. Examples include general contractors, city and county maintenance crews, and school district maintenance workers. ARP also provides site primers for contractors and state workers when work plans are in known areas of contamination.

Sample training is for third-party individuals or contractors involved in the collection, packaging, and shipment of samples. Sample training topics include sampling procedures, sample handling and chain-of-custody (COC) protocols. No accreditation will be given from ARP for this training. However, ARP will maintain a list of trained samplers.

ARP also has the following training materials available for outreach events.

- LA/vermiculite samples box
- Libby Legacy Project Poster

4 Physical Remedy Protection

This section details activities pertaining to maintenance of physical remedies and/or ECs at the Site. Breaches to physical remedies or ECs could result from planned disturbances due to renovation, new construction, and/or demolition, as well as from unplanned disturbances such as vandalism, accidents, floods, fires, and/or neglect. Further, damage could be caused by erosion, vehicles, digging, normal wear and tear, and/or deteriorating encapsulated building material. Because disturbances may result in exposure to LA-containing material that would result in unacceptable human health risks, controls have been put in place to address these disturbances. Additionally, changes in use can result in new exposure risks that will need to be evaluated and remedied, as necessary.

When planned disturbances, unplanned disturbances, and changes in use are expected to occur at a property, ARP should be notified so that a site assessment can be completed. In general, the site assessment process involves review of a proposed disturbance and/or change of use, property file information, sample results, and findings from an on-site assessment, if conducted. The site assessment process is conducted in order to determine if further abatement activities are necessary at the property.

4.1 Planned Disturbance

For anticipated indoor and outdoor property disturbance activities, ARP utilizes the PEN regulation, the Montana811 system, ARP Hotline Calls, and information obtained through the Lincoln County Planning Department to further protect the remedy and public health, and to reduce the public's potential exposure to LA from disturbance activities performed on real properties at the Site.

4.1.1 Property Evaluation Notification Regulation

The PEN regulation (**Appendix E**), established by the BOH and administered by ARP, is intended to reduce the possibility of the public's exposure to LA. To initiate the PEN process, a property owner or property owner's contractor submits a completed *Libby/Troy Amphibole Asbestos Property Evaluation Notification* form (also included in **Appendix E)** for the anticipated activities on their property and/or submits a Montana811 request (see **Section 4.1.2,** below). Each notification is assigned a unique identification with details recorded in RM, including the request date, address, current status, and information that was provided by and to the property owner or contractor.

ARP reviews existing property data and information related to LA and the proposed disturbance activity, as well as the jurisdiction (i.e. location relative to NPL boundaries) of the property. The ARP confirms the location using the geographic information system (GIS) information (i.e. address, latitude/longitude, Geocode, etc.) contained in the Libby Site Viewer (under development) and compares to Montana Cadastral and/or Google Earth.

Once the property address and the associated Geocode are identified and verified, ARP reviews the property information contained in RM, as well as archived in the property files stored on a separate external hard drive (**See Section 11.1.4**). ARP also reviews the as-builts to identify whether sampling and/or clean-up activities have occurred. ARP then contacts the property owner/contractor to provide information relative to the potential for LA to be encountered, and if necessary, provides recommendations to protect the remedy and/or limit exposure to LA or LA source materials. Site

assessments are sometimes required to determine the potential for exposure and/or provide recommendations.

ARP's response to the property owner/contractor is dependent on the type of disturbance that is proposed. For example, excavation, demolition, changes or additions to structures and buildings are expected to include recommendations for BMPs and guidance for disposal of contaminated materials. ARP may also provide a SOW and eligibility determination for reimbursement of abatement activities by DEQ. ARP records all recommendations in RM.

For proposed changes in land use or frequency of use, additional evaluations may be performed by ARP to determine if previous sampling events on or adjacent to the property adequately represent the proposed work area, whether the proposal increases the frequency of use on the land, and whether additional visual inspections and/or sampling events are warranted. Results from this evaluation, in combination with the RALs associated with the future land use, may suggest sampling is needed for the new use. If the proposal suggests changing the land use to a less restrictive use and/or a decrease of frequency of use, the evaluation is not needed because the current RALs of the property are already protective. If additional sampling is recommended, ARP will develop an investigation SOW for DEQ review and approval prior to sending recommendations to the property owner.

For proposed changes to interior structures, ARP would need to determine if the proposed changes impact vermiculite containing material (VCM) and/or vermiculite insulation. The ARP evaluation includes reviewing available records on the property and making a site assessment to visually confirm if material resides within the planned work area. An access agreement would need to be signed by the property owner allowing ARP and/or DEQ access onto their property. In the case where VCM is visually confirmed by ARP, an Investigation SOW would be developed to determine whether LA is present. Presence of vermiculite insulation does not require an investigation and should will be abated as necessary. ARP will submit this investigation SOW to DEQ for review and approval prior to sending recommendations to the property owner.

In conducting property evaluations, the File Review Checklist found in **Appendix F**, will be utilized. Research includes reviewing the surrounding properties near the proposed work area, collecting current status information, documenting any detection of visual vermiculite, evaluating previous sampling locations and sample results. Upon receiving consent to access the property, ARP will work with the property owner to schedule date(s) for site assessments. The presence of vermiculite, as well as the type(s) of material exposed, will be assessed and documented in the Site Assessment Form (**Appendix F**). Penetrating wall sections (e.g., drill, saw) may be necessary to look for suspect material. Completed File Review Checklists and Site Assessment Forms are uploaded into RM by ARP.

4.1.2 Montana811

Montana811 is a free service for a homeowner or contractor to request that existing subsurface utilities be located prior to conducting digging activities. If the project is located within the Site's NPL boundary, Montana811 will send a ticket to the ARP with the project location information so that ARP can evaluate how best to protect the remedy and limit exposure. Each Montana811 is assigned a unique identification (starting with LUD for Libby properties and TUD for Troy properties), with details recorded

in RM including the request date, address, current status, and information that was provided by and to the homeowner or contractor.

ARP follows the same evaluation process described above for the PEN regulation except that information is provided to the Montana811 requestor. Once the Montana811 request is received by ARP, the address is verified, and investigation details and sample data are gathered for the property. ARP evaluates if remnant material remains in the proposed work area and/or if visual vermiculite was observed during previous investigations. Most responses to Montana811 are informational. ARP will call the Montana811 requestor and make an introduction to explain why ARP received the Montana811 ticket and to provide information about previous investigations and the potential to encounter LA or vermiculite at work site areas. Occasionally, a site assessment is needed to understand the location of the work area. ARP will provide information about previous investigations, including the year the investigation was conducted and the current status of the property. A summary of details about the investigation and sample results, if any, is provided along with education materials and BMPs.

ARP may offer supplies to complete abatement work, such as poly, asbestos removal bags, duct tape, etc. and if needed, ARP can provide oversight on removal of contaminated material. ARP records all information in RM.

4.1.3 New Development/Subdivisions

In coordination with the Lincoln County Planning Department, ARP reviews new subdivision applications submitted for any property located within the NPL boundary. When applying for a subdivision approval, the property owner prepares their application in accordance with the Lincoln County Subdivision Regulations and submits the application to the Lincoln County Planning Department. As part of the County's application review process, ARP applies the same evaluation process as described above for the PEN regulation. ARP then provides information documenting the current status of the property, along with ARP recommendations for BMPs, guidance for disposal, and any additional steps that need to be taken. The Lincoln County Planning Department incorporates ARP recommendations into the subsequent subdivision written decision. ARP records all recommendations in RM.

4.2 Unplanned Disturbances

Abatement actions may be required for unplanned disturbances or damages to physical remedies or ECs due to natural causes, lack of maintenance, or unforeseen circumstances. If a disturbance results in LA or LA-contaminated material being encountered or suspected, work should be suspended immediately and DEQ and/or ARP contacted. An abatement to the physical remedy or EC is generally warranted if there is substantial likelihood of exposure from contaminated soil or building materials. DEQ, with assistance from ARP, is responsible for making the determination of remedy response severity.

Based on the degree of exposure anticipated, ARP may elect to provide the property owner with BMP advice and/or conduct a site assessment to determine if abatement work is necessary at the property. Should DEQ and ARP determine that abatement work is necessary, the following activities would typically be performed for the abatement action and identified in the SOW:

1. ARP obtains information about the disturbance and contact information from the property owner.

2. ARP educates the property owner on BMPs, including the necessary measures to secure and/or isolate the disturbed areas and to limit contaminant migration so that protection of human health is maintained.
3. ARP completes an initial site assessment to confirm and document the disturbance.
    a. For properties that have been previously characterized, ARP will review the property files in RM and document in the File Review Checklist.
    b. For properties that have not been previously characterized, ARP will review neighboring property information to determine the extent of sampling data available. ARP will utilize available sampling data, combined with onsite visual observations and data on historic land use, to determine if additional investigation sampling is warranted.
        i. If investigation sampling is recommended, ARP will provide justification and develop a SOW for review and approval by DEQ.
        ii. Once approved, ARP will provide the third-party with the approved SOW and sampling guidance and observe sample collection.
        iii. Samples will be submitted by the third-party to a laboratory from DEQ-approved list, with results emailed to DEQ and ARP.
4. ARP will compare the sample results to the RALs to determine if an abatement action is needed.
5. If warranted, ARP will develop an SOW for proposed abatement work utilizing the DEQ-approved format included in **Appendix F**.
6. ARP will determine reimbursement eligibility for proposed abatement work in accordance with **Section 8** of this manual.
7. ARP will verify the contractor, if utilized, has completed LA contractor awareness training.
8. ARP will provide the property owner the SOW which includes suggested BMPs, disposal protocol, and eligible abatement work to be conducted.
9. ARP will provide oversight of the abatement work, as necessary, and perform a final inspection to ensure SOW elements have been met.
10. ARP will confirm proper landfill disposal of LA-contaminated soils and building materials.
11. Once ARP has verified SOW elements are met, the property owner will submit eligible expenses to DEQ for reimbursement.

If it is determined that a “quick response” is needed to address contamination in an expeditious manner, ARP will first contact DEQ to discuss.

4.3 First Responder Emergency Response Action

During first responder emergency responses, ARP is contacted if emergency personnel find actual or suspected LA contaminants. ARP collects details of the incident and the potential for exposure and contacts DEQ to discuss. Depending on the potential exposure severity, DEQ will determine if ARP should follow the steps outlined above or whether a state-certified abatement contractor should be procured. If procurement is required, DEQ has the power to procure any contractor required for an emergency action without solicitation. ARP will assist DEQ in developing a SOW for the third-party contractor.

5 Statement of Work

ARP will develop a property-specific SOW to address investigation sampling and/or abatement actions at a given property. The SOW is intended to capture background information, property-specific information, BMPs, proposed sampling and/or abatement activities, disposal protocols, and reimbursement information. **Appendix F** includes a SOW template that shall be utilized during O&M.

Prior to conducting abatement work, the SOW will be reviewed and approved by DEQ. Once approved, ARP will provide the SOW to a third-party to perform the work. ARP will be required to verify the work is performed in accordance with the approved SOW.

Note, however, that additional regulations, such as the OSHA standards for the construction industry, Asbestos Control Program permits, etc. may apply. It is the responsibility of the person(s) conducting the work to adhere to all required regulations, permits, authorizations, and work practices.

5.1.1 Investigation SOW

As detailed in **Section 6**, investigation sampling will require justification detailing historical sample data and the rationale for collecting additional samples. The SOW shall detail the locations, scope, and total number of samples proposed. Site maps will be developed to illustrate the proposed investigation work.

Each property-specific sampling effort shall be detailed in the SOW, which serves as the blueprint for implementing the data collection to ensure that the technical and quality goals are met for the specific sampling effort. ARP will also consider the complexity of the property to meet the decision needs.

Prior to sample collection, the SOW will be reviewed and approved by DEQ. Once approved, ARP will provide the SOW, along with sampling guidance, to a third-party who will collect, process, and ship samples to the designated analytical laboratory in accordance with the SOW. ARP will be required to conduct oversight of those sampling efforts.

5.1.2 Abatement SOW

For proposed abatement actions, ARP will develop a SOW that describes the activities to take place at the property pertaining to the removal of LA and/or vermiculite insulation, including set-up, removal, clean-up, and confirmation sampling. The work description should include current and future use areas (land use and/or frequency of use), planned renovations and/or disturbances, proposed material sources and disposal sites, as well as the proposed timeframe for work and any applicable contractor and/or insurance information. Site maps will be attached depicting the proposed work.

5.1.3 Abatement Action – Contaminated Building Materials

For abatement to physical remedies and application of ECs for contaminated building materials, the following SOW will typically be required:

1. Install appropriate resources to isolate the work area.
2. Remove contaminated building materials (e.g., vermiculite insulation and/or LA-containing materials) using High Efficiency Particulate Air (HEPA) vacuum extraction.
3. Encapsulate any contaminated building materials to remain in place using in-place sealing and covering.

4. Clean interior, when required, using HEPA-filtered vacuum.
5. Clearance samples may be required to confirm that abatement actions were completed in accordance with the remedial clearance criteria established in the ROD and summarized in **Table 1** (in **Section 1.1.2.4**).
6. Follow proper waste transportation methods and dispose of waste appropriately at the landfill.

5.1.4 Abatement Action – Contaminated Soils

For abatement to physical remedies and application of ECs for soils, the following SOW will typically be required:

1. Provide appropriate resources to control dust at the site.
2. Excavate to required depths and place excavated soils in truck and/or container.
    a. Trace soils should be maintained separately for use as cover at the Lincoln County Landfill asbestos cell (Libby Landfill).
    b. Dispose of contaminated soils to the Libby Landfill.
3. Clearance samples may be required to confirm that abatement actions were completed in accordance with the remedial clearance criteria established in the ROD and summarized in **Table 1** (in **Section 1.1.2.4**).
4. Obtain fill material from an approved off-site borrow source that is analyzed in accordance with the *Fill Material Quality Assurance Project Plan* (CDM Smith 2018a). If available, EPA-provided backfill material may be utilized, with quantities tracked by ARP.
5. Transport, place, and compact the fill material.

6 Sampling

Although the majority of sampling activities were performed by EPA during removal and remediation actions, it is possible additional investigation sampling may be necessary under O&M to characterize property conditions at locations where properties were not previously characterized due to misses, limits of inspection, unforeseen conditions, property refusals, and/or where additional characterization may be necessary due to land use and/or frequency of use changes. In addition, confirmation sampling may be conducted at individual properties within the Site following abatement of LA-contaminated soil and/or LA-containing vermiculite or vermiculite insulation from a structure or building. The primary goal of confirmation sampling is to confirm that O&M abatement actions have met clearance criteria. This is accomplished using both visual inspections and collection of soil or air confirmation samples. Confirmation sampling requirements will be detailed by ARP in the abatement SOW. Borrow sources that will be used for fill material from the removal of contaminated materials on the Site will be analyzed in accordance with the *Fill Material Quality Assurance Project Plan* (CDM Smith 2018a).

This section describes the basic procedures for conducting O&M sampling. Detailed sampling procedures are provided in **Appendix G**.

6.1 Sitewide Quality Assurance

Quality Assurance (QA) activities include all processes and procedures that have been designed to ensure that samples are collected, analyzed, and documented properly, resulting data are of high quality, and any issues or deficiencies associated with field data collection, sample processing and preparation, analysis, and data reporting are quickly identified and rectified. The following sections describe several of the overarching components of the O&M Sampling QA program. Additional details on the QA procedures are provided in the *O&M Sampling Guidance* (WESTON 2020) provided in **Appendix G**.

6.1.1 Quality Assurance Management

DEQ is responsible for the quality management system for data collected and for decision-making during O&M. A DEQ Quality Assurance Manager (QAM) is assigned as an independent technical staff that reports directly to the DEQ Project Manager (PM) on QA matters. The QAM has the authority to objectively review all activities covered under the sampling guidance, to identify problems, and to resolve any quality-related problems and/or authorize changes to the sampling guidance. ARP is responsible for providing oversight of field sampling activities.

6.1.2 Approved Sampling Personnel

Investigation sample collection activities will be performed by a third-party in accordance with the SOW, with ARP oversight. In most instances, ARP will perform confirmation sampling following abatement activities. Consistency will be achieved to the extent possible through proper training, use of this guidance, and ARP oversight. It is the responsibility of each field sampling team member to review and understand DEQ's *O&M Sampling Guidance* and the property-specific SOW. It is the responsibility of the third-party to maintain training documentation. This documentation is to be made available upon request by ARP and/or DEQ.

The ARP is available for training third parties on the LA sampling requirements, COC protocols, and Site-specific guidance and Standard Operating Procedures (SOPs). A list of entities trained by ARP will be maintained and reviewed as part of oversight activities. At a minimum, ARP-provided training regarding LA-specific SOPs must be obtained. In addition, the third-party individuals should read and understand DEQ's *O&M Sampling Guidance*.

6.1.3 Approved Laboratories

Samples are to be collected and sent to a DEQ-approved analytical laboratory in accordance with the *O&M Sampling Guidance* and SOW. All analytical laboratories participating in the analysis of samples for the Site are subject to national, local, and Site-specific certifications and requirements. To comply with those requirements, a laboratory may be accredited by the National Institute of Standards and Technology (NIST) and National Voluntary Laboratory Accreditation Program (NVLAP) for the analysis of asbestos by polarized light microscopy (PLM) and/or transmission electron microscopy (TEM). Other methods to demonstrate proficiency are verified and evaluated by the DEQ QAM, as appropriate. Copies of recent proficiency examinations from NVLAP or an equivalent program, as well as certifications from other state and local agencies, are maintained by each participating analytical laboratory. 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 Site. These certifications require that each laboratory has all applicable state licenses and employs only qualified personnel. Copies of all proficiency examinations and certifications are maintained by the DEQ QAM. DEQ will work with the approved laboratories so that all eligible analytical costs are paid directly by DEQ.

DEQ reserves the right to conduct any laboratory audit or investigation deemed necessary to determine the ability of each laboratory to perform the work.

6.1.4 Chain of Custody

The COC record is employed as physical evidence of sample custody and condition from the sampling team to the receiving facility. A completed COC record is required to accompany each batch of samples shipped to the analytical facility. DEQ will ensure a pre-printed COC is sent to ARP with an approved SOW to provide to the third-party sampling personnel. The hard copy COC will accompany the sample shipment, and the field sample coordinator will provide a copy of the COC to ARP for the project file. A copy of the final COC will be included in the laboratory data package along with the final analytical results.

6.1.5 Laboratory Documentation and Reporting

Sample results will meet DEQ's data requirements and will be delivered electronically to DEQ and ARP in the appropriate format. Any deviations to the methods will be documented in a narrative summary, which is included in the report. The lab report will be uploaded into RM in the appropriate property file.

6.1.6 Data Verification and Validation

Data verification of field sample activities will be conducted by the third-party sampling team and verified by ARP. Review will typically include ensuring proper labeling and sample handling and cross-checking that sample IDs and sample dates have been reported correctly on the COC, noting any deviations from the SOW. Field notes with the deviations will be provided to ARP.

ARP will also provide data verification of sampling prior to uploading the analytical data into RM. This data verification includes cross-checking that sample IDs and sample dates have been reported correctly, and that analytical methods and required analytical sensitivities align with the sampling requirements identified in guidance, SOPs, and SOW. The goal of data verification is to identify and correct data reporting errors. Performing regular data verification reviews will ensure that any potential data reporting issues are quickly identified and rectified to limit any impact on overall data quality. If discrepancies are found, ARP will contact the DEQ PM or QAM who will then notify the appropriate entity (e.g. field or laboratory) in order to correct the issue.

Libby-specific spreadsheets will be used by the laboratory, which eliminates the hand-entering of data and includes automated quality control (QC) checks that perform initial data checking of the reported analytical results. In addition to these automated checks, more detailed manual data verification efforts will be performed on an as needed basis.

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 potential data quality issues. Data validation will be performed by DEQ (or their designee). As part of the data validation effort, DEQ will review field QC sample and laboratory QC analyses results as deemed necessary. DEQ may assess other information associated with sampling and analysis efforts to identify potential data issues and evaluate the data quality.

Data verification and validation reviews will be performed by technical support staff familiar with Libby-specific data reporting, analytical methods, and investigation requirements.

6.2 Investigation Sampling

As noted above, it is possible investigation sampling may be necessary under the O&M phase when no previous sample data is available and/or when conditions have changed such that previous sample data is not representative of the current site conditions. This is accomplished by sampling for LA in soil and building materials of exteriors (e.g., yards, flowerbeds, driveways, etc.) and/or interiors (buildings and structures) of a property. As such, there is no set schedule for collection of these samples, as they are opportunistic in nature, although may be set based on seasonal factors. The required samples and sampling locations are highly variable and based on the complexity of the property being investigated, and are defined in the property-specific SOW.

The primary goal of investigation sampling is to determine if LA is present in source materials at a level that would warrant abatement actions. The need for abatement action(s) will be based on a comparison of the investigation sample results to the RALs established in the ROD and described in **Section 1.1.2**. This determination will depend upon both visual inspections and the analysis of LA in collected soils and bulk materials. Detailed sampling and analysis procedures for property investigations are presented in the *O&M Sampling Guidance*.

6.3 Confirmation Sampling

Confirmation samples are collected following abatement of LA-contaminated soil and/or LA-contaminated vermiculite or vermiculite insulation from a structure or building. If soils are removed,

confirmation soil samples are collected at the depth of excavation during the removal phase of the abatement action. If building materials (i.e., LA-containing vermiculite or vermiculite insulation) are removed, clearance air samples are collected from an interior living space after the removal of building materials. There is no set schedule for collection of these samples, as they are in conjunction with abatement activities, which may be set based on seasonal factors for exterior removals or renovations/demolitions. The required samples and sampling locations are highly variable and based on the complexity of the abatement, and are defined in the property-specific SOW.

Following completion of the abatement activities, the primary goal of confirmation sampling is to confirm the abatement conditions meet the clearance criteria as defined in this O&M Manual and established in the ROD. Detailed sampling and analysis procedures for confirmation sampling are presented in the *O&M Sampling Guidance*.

7 NOEC and NOPEC

Some property owners chose not to participate in remedial activities. For properties where access was refused, EPA filed notices with the Lincoln County Clerk and Recorders Office to alert property owners, both current and future, that potential or actual environmental conditions exist at the property.

At the end of the EPA-led remedial work in 2019, there were approximately 220 residential and commercial properties located within OU4/OU7 that refused access to conduct and/or complete investigations. Additionally, there were six (6) residential and commercial properties within the Site boundaries that refused access to conduct or complete response actions for known LA contamination. For these refusal properties, EPA filed a Notice of Potential Environmental Conditions (NOPEC) where the presence of LA contamination is unknown and a Notice of Environmental Conditions (NOEC) for properties where known LA contamination was not remediated. Moving into O&M, there is potential to receive requests for these existing NOEC/NOPECs to be withdrawn by EPA.

7.1 NOEC/NOPEC Withdrawal

For EPA to issue a withdrawal notice for existing NOPEC or NOEC properties, it is expected that property evaluations, sampling, and abatement work will occur as described in this manual. If no further action is determined to be necessary following completion of investigation and/or abatement action, DEQ will forward property-specific investigation documentation, including sample results and as-builts to EPA. Once the “no further action” determination is confirmed, EPA will prepare a Withdrawal Notice of a NOEC/NOPEC (notarized document), which will be filed with Lincoln County. This Withdrawal Notice will reference the document number of the previous notice, as assigned by Lincoln County.

7.2 NOEC/NOPEC Issuance

Should future investigation and/or abatement work be necessary at a property and access is refused, DEQ may request that additional notices be filed with the Lincoln County Clerk and Recorders Office. ARP will be responsible for providing the applicable property information (e.g. sampling data, proposed disturbance type, etc.) to DEQ. DEQ will review the provided information and file a notice with Lincoln County when necessary.

8 Property Reimbursement Process

Future encounters with LA or LA-contaminated material is expected to occur at properties during the O&M period. These exposures to LA-containing material may result in unacceptable risk, necessitating abatement work in order to prevent further exposure and protect human health. An O&M reimbursement process has been established for property owners or contractors that perform future investigations and/or abatement activities on a property to recover all or a portion of these costs. It is expected that costs not associated with the sampling and removal of LA or LA source materials will be the responsibility of the property owner.

This section describes the reimbursement process for eligible expenses related to investigation and/or abatement work performed by the property owner and/or contractor to address LA issues that may be encountered during the O&M period.

Several tools have been developed to evaluate reimbursement eligibility. The *Property Reimbursement Eligibility Flowchart* and *Investigation Sampling Flowchart* included in **Appendix H** provide guidance on the potential funding sources (i.e. state and/or federal) that can be utilized for the proposed work. Once work has been deemed eligible for reimbursement through a screening process, element-specific eligibility costs can be evaluated.

8.1 Eligibility Screening

The first step in the eligibility determination process is to evaluate whether the property itself is eligible for reimbursement.

8.1.1 Basic Eligibility Requirements

For abatement costs to be eligible for reimbursement, the property must be located within the NPL boundary, have the potential for LA-contamination that requires an investigation or existing LA-contamination that exceeds or could lead to an exceedance of RALs defined in the ROD. Additionally, the property owner must be willing to provide consent for ARP and DEQ to access the property.

Properties that are outside of the NPL boundary, do not lead to the exceedance of the RALs, or refuse access for ARP/DEQ personnel will not be considered for reimbursement. If it is unknown whether RALs are or will be exceeded, ARP should proceed with developing a SOW to conduct investigation sampling as described in **Section 5.0**.

8.1.2 Insurance Considerations

In the event a property owner has insurance that covers all or a portion of the costs for the proposed abatement and/or repair, a portion of the cost may be eligible for reimbursement only if the property owner is willing to provide insurance contacts. Abatement costs will not be eligible for reimbursement if the insurance contact information is not provided or if the insurance company covers all costs for abatement and/or repair.

ARP will coordinate with the insurance company to define the SOW related to LA contamination and abatement and discuss financial responsibilities. In general, only costs not covered by the insurance company will be considered for reimbursement. Reimbursement of deductibles will be determined on a

case-by-case basis. The property owner will need to demonstrate that the deductible cost was incurred primarily due to LA abatement.

8.1.3 General Property Considerations

The following general property considerations should be considered during the screening process.

8.1.3.1 Federal Properties

Federal properties are not eligible for O&M reimbursement. Instead, other sources of federal funds are available for investigation and/or abatement actions during O&M. Disturbances that occur at federal properties remain subject to the reporting and documentation requirements described in this manual, however. Work plans, analytical results, and as-builts are expected to be provided to ensure up-to-date property records.

8.1.3.2 Schools

Public and non-profit private schools have distinct regulatory requirements to protect school children and school employees from asbestos exposure. These properties are subject to the AHERA regulations, which require public school districts and non-profit schools including charter schools and schools affiliated with religious institutions inspect their schools for asbestos-containing building material and to prepare management plans and take action to prevent or reduce asbestos hazards. These legal requirements are founded on the principle of "in-place" management of asbestos-containing material. Removal of these materials is not usually necessary unless the material is severely damaged or will be disturbed by a building demolition or renovation project. Because these properties are already required to manage asbestos containing materials, reimbursement for LA remedial work requires a case-by-case evaluation and will need to follow AHERA requirements.

8.1.3.3 Misses/Limits of inspections/Unforeseen Site Conditions

Misses are defined as LA and or LA source materials encountered in areas that have been previously inspected and not found during prior remedial investigation inspections and/or sampling. Misses and unforeseen site conditions require coordination between DEQ and EPA to identify the appropriate response and eligibility criteria.

It is important to note that during the remedial investigation, limits of inspection may have prevented all or portions of the property from being investigated or sampled. Limits of inspection areas may include, but are not limited to, inaccessible knee wall, LA and or LA source materials found at depths greater than sampling design depth, or LA and/or LA source materials found in non-use areas (NUA), limited use areas (LUA), under concrete slabs and non-permeable liners. Limits of inspection are not considered a miss and should be evaluated following the procedures identified herein.

8.1.3.4 Asbestos Types

Naturally occurring asbestos, including LA, is present in and around Libby and Troy. The goal of the remedy is to address mine-related releases. As such, abatement of naturally occurring LA is not eligible for reimbursement.

Additionally, only LA and vermiculate insulation abatement costs are eligible for O&M reimbursement. If other forms of asbestos are present, LA-specific abatement work will need to be identified and carefully

documented to ensure reimbursement eligibility. This situation may require additional tracking procedures be utilized in order to ensure abatement and disposal costs can be adequately segregated between asbestos types. These procedures should be described in the ARP-developed SOW.

8.1.3.5 NOPEC/NOEC Properties

During the O&M period, a property with a NOPEC or NOEC may require investigation and/or abatement. In accordance with EPA policy, federal O&M funds cannot be used for refusal properties. Instead, reimbursement for investigation and/or abatement costs, if approved by DEQ, may be considered on a case-by-case basis using state funds only.

If assessment and/or abatement work is conducted at a refusal property, the property owner will need to work with ARP and DEQ to verify the completed work meets the applicable RALs and clearance criteria specified in the ROD. Once confirmed, DEQ can request that EPA complete a withdrawal notice to file with the Lincoln County Clerk and Recorder's office to resolve the existing NOPEC and NOEC notice as described in **Section 7**.

8.1.4 Work-Specific Eligibility Determinations

As illustrated in the *Property Reimbursement Eligibility Flowchart* and *Investigation Sampling Flowchart* included in **Appendix H**, considerations for the type of property disturbance and/or change of use must be evaluated in order to complete the eligibility screening process. Considerations of past remedial actions, maintenance of controls, and potential developer windfall situations need to be assessed. If it is determined through this screening process that all or a portion of the work is eligible for reimbursement, ARP should identify the eligibility within the SOW.

8.2 Contractor Utilization

ARP will identify potential reimbursable costs within the SOW. If a contractor is used to complete all or a portion of the work, ARP will work with the property owner to ensure the following:

1. Owner solicits estimates from at least three (3) licensed abatement contactors in accordance with the SOW. Owner may select abatement contractor of their choice but will only be reimbursed for the unit cost(s) specified in the lowest, responsible estimate. Note that three (3) estimates do not need to be obtained, only solicited.
2. If a general contractor is already conducting work at the property, the owner will request that the general contractor provide proposed costs specifically associated with the abatement activities defined in the SOW, utilizing unit cost and quantity breakdowns. ARP will evaluate the estimate for reasonableness. In evaluating for reasonableness, ARP may compare the proposed cost to past estimates for similar type work or create an independent estimate. If ARP's cost estimate is within +/- 10% of contractor's costs, proposed unit costs may be accepted for use in reimbursement. Actual quantities will need to be provided, however, for use in calculating the total reimbursement.

The property owner can solicit estimates from any contractor capable of meeting the requirements described in the SOW. An *LA Abatement Estimate Sheet* (**Appendix F**) is available for property owner's use to solicit estimates from abatement contractors. For convenience, ARP can provide a list of contractors to solicit estimates. The contractor list is not all-inclusive, nor is it intended to provide any endorsement for the listed contractors.

Once estimates are received, ARP will coordinate with DEQ to evaluate the estimate for reasonableness and determine eligible reimbursement costs based on the unit cost(s) specified in the lowest, responsible contractor estimate and provide a final SOW with the approved unit costs(s) to the property owner. Reimbursement will be provided based on final quantities only at the approved unit cost(s). The property owner is not obligated to use the lowest, responsible bidder; however, reimbursement will be equivalent to the lowest bid. The property owner should retain all associated receipts and invoices for submittal of reimbursement claim.

8.3 Eligibility Calculations

While the *Property Reimbursement Eligibility Flowchart* and *Investigation Sampling Flowchart* included in **Appendix H** identify potential funding sources (i.e. state and/or federal), specific reimbursement costs are not expected to be finalized until the work is complete and verified by ARP. ARP will calculate preliminary eligible costs in the SOW based on estimated quantities and unit costs. Only material costs will be considered when a homeowner self-performs the LA abatement work. DEQ will require documentation from the property owner or contractor of final quantities and unit costs required to perform the work for actual reimbursement.

Depending on the specific circumstances and work performed, specific work elements may or may not be eligible for reimbursement under the various funding categories. The materials, equipment, labor, and other costs that are eligible for reimbursement will be identified in the SOW, which is included in **Appendix F**. The determination of eligible costs incorporates the federal O&M funding eligibility criteria described in EPA's August 19, 2019, letter to DEQ (**Appendix B**) which provides recommendations for use of settlement funds on O&M specific items.

8.4 Reimbursement Procedures

Upon completion of investigation and/or abatement work outlined in the approved SOW, the property owner may submit a claim to DEQ for expenses associated with the investigation and/or abatement activities conducted. In order to be reimbursable under O&M, the costs must be eligible, associated with the investigation or abatement activities defined in the ARP-approved SOW, be actually, reasonably, or necessarily incurred to clean up and dispose of the contamination, and be verified by ARP. As part of this verification, ARP will verify actual quantities and that the project has been completed in accordance with the SOW and will develop a closeout report with as-builts summarizing what occurred. ARP will update RM and provide the property owner with the closeout report and property as-builts (if applicable).

8.4.1 Evaluation of Claims

Claims for actual costs should be submitted along with receipts and/or invoices upon completion of the investigation or abatement activities using the *Reimbursement Claim Form* in **Appendix F.** In addition to the claim form, a State of MT Substitute Form W-9, *Request for Taxpayer Identification Number (TIN) Verification* is required to be completed and submitted in order to receive payment from DEQ. In order to evaluate claims received, DEQ shall verify the actual quantities of work performed and confirmed and/or the actual quantity of materials used and confirmed at the accepted unit cost under the approved SOW.

8.4.2 Processing of Claims

Payment will go directly to the property owner and the property owner will be responsible to make payments to the associated party(s) that performed the work. Reimbursement will only be paid once the ARP has completed an inspection and verified that work was done in accordance with the SOW. Once the request is approved, DEQ will issue a reimbursement check directly to the property owner and notify ARP of the payment. ARP will document all information in RM.

8.5 Reimbursement for Unusual Circumstances

Unusual circumstances (e.g., catastrophe) will be handled on a case-by-case basis and will require agency consultation to make appropriate determinations on how reimbursement will be handled. For unusual circumstances, ARP will contact DEQ to discuss specifics. Options will be identified, along with a preferred path forward. DEQ will then consult with EPA to confirm eligibility.

9 Annual Inspections

This section provides details of the annual site inspection process. DEQ is responsible for conducting annual inspections in order to assess the Site's status and to visually confirm and document the conditions of the remedy. DEQ will conduct a review of the Site with support from ARP and forward the findings to EPA on an annual basis. The annual inspection objectives are as follows:

- Observe the integrity of the ECs and physical remedies to ensure that the protection of human health is maintained.
- Evaluate the implementation of ICs to ensure protectiveness.

Activities to be performed during these inspections will include visual site inspections, property records review, homeowner interviews, and IC evaluations. The annual inspection should be documented in the Annual Inspection Report which is discussed further in **Section 12.1**.

9.1 Visual Site Inspections

Visual site inspections will observe site conditions of properties to ensure the integrity of engineered controls and physical remedies is being maintained. The visual site inspections will consist of non-intrusive (i.e. surficial) visual inspections of the immediate ground surface and remedies completed within OU4 and OU7.

9.1.1 Property Site Selection

DEQ will randomly select approximately 10 properties from RM with "Response Completed" status that have not been previously assessed during an annual inspection. Status will be indicated as: Indoor, Indoor/Outdoor, Outdoor, which will determine the extent of the visual inspection. This information can be identified through the RM database. Properties identified on the "No Call" list (**Appendix I**), or who have not provided access consent, will not be considered. ARP will continue to maintain these lists.

9.1.2 Property Records Review

An administrative review of all applicable records and the IC program instruments will be conducted. The records review is structured to allow evaluation of the current IC program instruments. A review of available property records will include, but is not limited to the following:

- Property records/filings
    - Comfort/Status letters
    - Property Notices
    - Refusals (e.g. NOECs / NOPECs)
    - Building permits
    - Zoning
- PEN records
- Montana811 records
- ARP Hotline Call records
- RM Property file review (i.e., work plans, as-builts, sample results)
- Property Hard Drives (e.g. EPA archived information)

Available information used for the property file reviews include analytical data, removal action work plans, as-builts, and removal and restoration completion forms. Property-specific information for the property file reviews can be obtained from the RM database, project hard drives, SCRIBE data, Cadastral, and the GIS Viewer (under development).

Property records review findings will be documented using the forms included **Appendix J:** Annual Inspection Information.

9.1.3 Property Owner Interviews

When necessary for a specific property, property owner interviews will be conducted by ARP. The goals of the interview are to verify that property owners are familiar with the cleanup activities that occurred on their property, to ensure the remedy remains protective and that the property owner understands their responsibilities in protecting the remedy, and to ensure the property owner is familiar with the resources available. These interviews will be conducted using the interview questions included as **Appendix J** and archived in RM. Interview outcomes should also be used to assess the effectiveness of the IC components (e.g. education, training, etc.).

9.2 IC Evaluation

ICs are non-engineering measures designed to prevent or limit exposure to LA left in place at OU4 and OU7. EPA has developed an ICIAP to ensure ICs applicable to OU4 and OU7 are properly documented, implemented, and operating effectively during their entire lifespan (CDM Smith 2020c). The annual inspection process should include evaluation of specific IC instruments on a representative subset of properties in OU4 and OU7 to assess whether:

- The selected IC instruments are effective based on each IC's use or other applicable metrics;
- The selected IC instruments remain in place; and
- The ICs are enforced such that they meet the stated objectives, performance goals and provide protection required by the response.

The following ICs as identified in the ICIAP that should be evaluated annually, include:

- Governmental Control
    - PEN Regulation
- Informational Devices
    - MDT encroachment permit application and addendum
    - NOECs/NOPECs
    - Montana utility locate service (Montana811)
    - *Working Draft Libby Asbestos Superfund Site – Operable Unit 4 and 7 Best Management Practices Manual. Libby Asbestos Superfund Site BMP Manual* (EPA 2019)
    - ARP Program Educational and Resource Pillars that include the following educational components:
        - BMP awareness for public
        - LA contractor awareness
        - Educational outreach at schools and businesses

        - Property transaction awareness
        - Health fairs and public outreach campaign
        - Financial awareness – information on reimbursement assistance
        - Lincoln County departmental procedures (with review and LA information provided by ARP and BOH)
        - City of Libby/Troy and Lincoln County Coordination
            - City utility maintenance and repair
            - City building property maintenance and repair
    - Data and Administrative Record Sources (refer to **Section 11.0**)

Boundary conditions at OU4 and OU7 are features or conditions that limit the ability to further abate LA contamination due to physical or technical constraints and the related lack of accessibility. Boundary conditions, as described in the O&M Plan may not require O&M. However, the ICs that are used to address these areas and potential LA exposures, will be evaluated through visual inspections.

The IC evaluation should be documented in the appropriate section of the O&*M/Remedy Evaluation Checklist* (**Appendix J**).

9.3 Analysis of Inspection Results (Metrics)

DEQ will analyze the results of the annual inspection, as applicable, to determine if:

- PENs regulation correlates to observed construction, building permits, aerial photography, etc.;
- Contracting resources are sufficient to ensure remedial criteria continues to be met for property abatement completed during O&M;
- Property owner interviews reflect public education outreach efforts to ensure knowledge of risks and responsibilities under O&M;
- ARP records adequately document assessment results, SOW, contractor oversight, etc.; and
- Database services are sufficient to effectively track and monitor implementation of O&M activities.

The results of this evaluation will be used to determine if ICs are sufficient to protect the remedy and the metrics included in **Appendix J** will be used to assist with this determination.

9.4 IC Modifications, as needed

If it is determined that an identified control is not meeting the metric identified, DEQ, ARP, and EPA will discuss possible revisions to the program that may improve efficacy. Changes to the IC program will be documented in ICIAP/O&M Plan, and associated updates to the O&M Manual will be made.

10 Lincoln County Class IV Asbestos Landfill

EPA has turned over operation of its 10.5-acre Class IV landfill unit to Lincoln County. Lincoln County operates four (4) landfills in the vicinity; however, only the Libby Landfill is capable of accepting material containing asbestos. The cell is located at 2501 Pipe Creek Road in Libby, Montana.

In accordance with the Lincoln County Solid Waste regulations, "All friable and nonfriable asbestos containing materials including all vermiculite, being transported to or disposed of in the Lincoln County Solid Waste system shall be packaged in such a way as to prevent contamination of the surrounding environment, protect landfill workers, and protect the public health. Disposal must be coordinated with the landfill manager or gate attendant prior to transporting the material."

Lincoln County's Solid Waste Division is responsible for operation and maintenance of the Class IV landfill unit. The Solid Waste Division coordinates with the ARP to track and monitor LA activities, including volume of material received. Contacts for the operation of the Libby Landfill and ARP are identified in **Appendix C**.

10.1 LA Waste Disposal Process

The following describes general practices of disposing vermiculite insulation and LA contaminated waste at the Libby Landfill:

- Homeowner/Contractor shall follow the BMPs and proper disposal procedures as outlined in the property-specific SOW developed by ARP.
- Homeowner/Contractor completes the required waste manifest available on the Lincoln County Solid Waste Division's website: http://lincolncountymt.us/solid-waste/landfill and coordinates with the landfill operator to provide potential amount of material and approximate date of generation for pre-planning. The asbestos cell does not have routine hours of operation, so planning and coordination are necessary to ensure that cell is available.
- The volume of LA-containing material disposed is recorded, with eligible tipping fees directly billed to DEQ.

This waste disposal process does not supersede Montana Asbestos Control Program regulations for asbestos inspections and contractor/abatement requirements for non-LA asbestos. Refer to S**ection 2.5** for additional information on DEQ's Asbestos Control Program.

10.2 Homeowner Disposal of Potentially Containing Asbestos

ARP also provides drop box locations that are clearly marked for asbestos disposal for homeowners to dispose of trash-sized garbage bags of potentially containing LA waste. This allows homeowners the opportunity and resources to dispose of LA-containing waste properly. This is an IC resource program to aid homeowners who may have not followed the ARP process for notifications but may still dispose of potentially containing LA waste.

10.3 Waste Tracking and Reporting for O&M

Lincoln County Solid Waste Division will track and maintain records at the Libby Landfill asbestos cell, as follows:

- Waste Manifests from Contractors/Homeowners
- Waste Manifested material from drop boxes – unknown location of generation
- Tipping fees and volume of LA-containing material
- Volume of trace soils provided for use as cover

Lincoln County Solid Waste Division will submit these records to ARP and DEQ for review on an annual basis.

11 Records

All available project property information generated during the remedial action has been archived and is available for review. Information obtained during O&M will be managed in RM, as described below.

11.1 Data Records

11.1.1 Libby Instance of Response Manager

RM was originally developed as an EPA application with modules for collecting specific types of data typically involved in EPA remediation projects. DEQ modified the existing RM database for continued use in O&M. RM has the same functionality of the previous version, but only contains data for OU4 and OU7. RM is used to manage and view data associated with historical remedial investigations and future O&M actions competed at Site. The database is hosted by DEQ and can accommodate multiple users at DEQ and ARP.

11.1.2 GIS Viewer (Under development)

DEQ is in the process of developing a tool similar to EPA's Libby Asbestos Site Viewer that was previously used during the remedial action.

11.1.3 Montana Cadastral

Montana Cadastral is operated and maintained by the State of Montana. It is a database used to store information about public and private land ownership in Montana. For site assessments, sampling investigations, or abatement actions conducted, ARP should verify the address and contact information using the Montana Cadastral website (http://svc.mt.gov/msl/mtcadastral) to ensure that property information in RM matches Montana Cadastral.

11.1.4 Property Information Hard Drives

All electronic and hardcopy of historical information obtained during the remedial action was scanned and saved to a hard drive. The information stored on the hard drives includes, but is not limited to:

- Property information and photos for OUs 1-5 and 7;
- Libby 2 and POTS SQL data and applicable user guides;
- POTS2 MS Access and User guide; and
- RM and backup files.

ARP and DEQ each have a copy of the hard drive and will continue to maintain the information on site.

11.2 Administrative Record

A copy of EPA's administrative record was provided to ARP and DEQ. Additionally, copies of all documents are available online, at EPA, and the Libby and Troy Public Libraries.

11.2.1 Informational Device Records

Informational devices intended to educate the public regarding the risks of LA exposure are managed by ARP. Current informational devices include Montana811, PEN, BMP information, approved contractor list, and educational and training programs. ARP will be responsible for updating and maintaining the informational devices.

In addition, the ARP website is maintained by Lincoln County to provide LA education and outreach information for the public and is located at: http://www.lcarp.org/.

11.2.2 Montana811 Records

When Montana811 receives a request for a property located within the NPL boundary, they notify ARP of the potential disturbance via email. ARP tracks the ticket number, purpose of the disturbance, address and contact information and information obtained during the site assessment in RM.

11.2.3 PEN Records

PEN records will be retained at ARP and captured in RM.

11.2.4 Analytical Results

Prior to O&M, all investigation and remedial sampling results were entered into a spreadsheet (e.g. copy of information contained in Scribe). Scribe is a software tool developed by EPA to assist in the process of managing environmental data. Scribe captured sampling, observational, and monitoring field data. During O&M, ARP will save the analytical results to RM specific to each property identification.

11.2.5 Landfill Records

Lincoln County Solid Waste Division will track and maintain records at the Libby Landfill asbestos cell as described in **Section 10.3**.

11.2.6 EPA Stockpile Material Records

At the end of the remedial action (in 2019), EPA provided a stockpile of un-used fill material for use as LA soil related replacement during O&M. Records of the material types and volumes are to be maintained by ARP.

12 Reporting Requirements

This section describes the required reporting and the necessary documentation during O&M as described in EPA's *Guidance for Management of Superfund Remedies in Post Construction* (EPA 2017) and responsibilities for developing, reviewing, and approving the reports.

12.1 Annual Inspection Report

Comprehensive annual reports will be developed by DEQ that summarize the annual inspections described in **Section 9**.

The annual inspection report will identify the properties evaluated, a summary of the records reviewed, results of the visual inspection and information obtained during the phone interviews (**Appendix J**). The report will also describe the level of effort, available resources, potential data limitations, conclusions, and recommendations based on defined metrics for evaluating the ECs/ICs. Site photos from the inspection will be included with the report.

EPA has developed the *Annual O&M/Remedy Evaluation Checklist* (**Appendix J**) to capture data necessary to evaluate the efficiency and effectiveness of the remedy. DEQ will complete Sections I-IX of the checklist and the associated Appendix C - Containment Remedies to evaluate the technical data and remedy performance for inclusion with the annual report. Appendices A, B, D, and E of the checklist are not applicable to this Site.

In the event that any instrument of ICs are found to be inadequate, need to be modified, or additional ICs are necessary to ensure protectiveness of the remedy, this information will be included within the annual inspection report. These reports will assist the DEQ and EPA in evaluating the adequacy of O&M, the frequency of repairs, and how these factors relate to determining and ensuring protectiveness of the remedy. The report will be prepared by DEQ and submitted to the EPA remedial project manager (RPM).

12.2 Special Reports

DEQ will prepare special reports, as needed, to document abatement due to unforeseen events or conditions. One example of a special report is an incident report. Incident reports are used to document the details of accidents involving site personnel and other unusual events such as fires, floods, or weather damage. Special Reports could also be used to identify developments and changing applicable or relevant and appropriate requirements (ARARs).

These special reports will be based on the magnitude of the event as determined by DEQ and will be generated on a case-by-case basis. The report should be made available to EPA, ARP, the appropriate OU4 or OU7 property owner, and other interested parties in a timely manner.

12.3 Five-Year Review

A formal five-year review is performed and funded by EPA, "to evaluate the implementation and performance of a remedy in order to determine if the remedy is or will be protective of human health and the environment," for as long as they are required. The remedy will be re-evaluated in accordance with the review requirements of CERCLA Section 121(c). The five-year review process will follow EPA's

Five-Year Review Process in the Superfund Program and will consist of six components: 1) community involvement and notification, 2) document review, 3) data review and analysis, 4) site inspection, 5) interviews, and 6) protectiveness determination.

The five-year review will be summarized in a report prepared by EPA in accordance with EPA's *Comprehensive Five-Year Review Guidance* (EPA 2001).

13 References

CDM Smith 2018. Fill Material Quality Assurance Project Plan, Libby Asbestos Superfund Site, Libby, Montana, Revision 5. April 1, 2018.

CDM Smith 2020a. Final Operations and Maintenance Plan, Operable Units 4 and 7, Libby Asbestos Superfund Site. Libby Montana. April 2020.

CDM Smith 2020b. Final Remedial Action Completed Report, Operable Units 4 and 7, Libby Asbestos Superfund Site. Lincoln County, Montana. March 2020.

CDM Smith 2020c. Final Institutional Control Implementation and Assurance Plan, Operable Units 4 and 7, Libby Asbestos Superfund Site. Libby, Montana. March 2020.

EPA 2001. Comprehensive Five-Year Review Guidance, OSWER 9355.7-03B-P. Prepared by the EPA Office of Emergency and Remedial Response.

EPA 2010a. Record of Decision for Libby Asbestos Superfund Site, Former Export Plant, Operable Unit 1. Lincoln County, Montana. May 2010.

EPA 2010b. Record of Decision for Libby Asbestos Superfund Site, Former Screening Plant and Surrounding Properties, Operable Unit 2. Lincoln County, Montana. May 2010.

EPA 2016. Record of Decision for Libby Asbestos Superfund Site, Libby and Troy Residential and Commercial Properties, Parks and Schools, Transportation Corridors, and Industrial Park, Operable Units 4 through 8. Lincoln County, Montana. February 2016.

EPA 2017. Guidance for Management of Superfund Remedies in Post Construction. OSWER 9200.3-105. Prepared by the EPA Office of Superfund Remediation and Technology Innovation.

EPA 2019. Working Draft Libby Asbestos Superfund Site – Operable Unit 4 and 7 Best Management Practices Manual. Libby Asbestos Superfund Site. Lincoln County, Montana. June 2019.

WESTON 2020. Operations and Maintenance Sampling Guidance, Libby Asbestos Superfund Site. Lincoln County, Montana. March 2020.

APPENDIX A – RELEVANT O&M REFERENCE DOCUMENTS

Effective Date	Title	Revision Number	Author
2020	Final Remedial Action Completion Report, Operable Unit 4 and 7, Libby Asbestos Superfund Site, Lincoln County, Montana		CDM Smith
April 2020	Final Operations and Maintenance Plan, Operable Unit 4 and 7, Libby Asbestos Superfund Site, Lincoln County, Montana	0	CDM Smith
March 2020	Institutional Control Implementation and Assurance Plan, Operable Units 4 and 7, Libby Asbestos Superfund Site. Libby Montana	0	CDM Smith
March 2018	General Property Investigation Quality Assurance Project Plan, Libby Asbestos Site, Operable Unit 4	9	CDM Smith
April 2018	Response Action Quality Assurance Project Plan, Libby Asbestos Site, Libby, Montana	8	CDM Smith
April 2018	Response Action Work Plan, Libby Asbestos Site, Libby, Montana	10	ER
April 2018	Fill Material Quality Assurance Project Plan, Libby Asbestos Superfund Site, Libby, Montana	5	CDM Smith
April 2017	Class IV Asbestos Cell Landfill Quality Assurance Project Plan, Libby Asbestos Site	3	CDM Smith
2017	Guidance for Management of Superfund Remedies in Post Construction. OSWER 9200.3-105		EPA
April 2016	EPA Data Management Plan, Libby Asbestos Superfund Site	2	EPA
2016	Record of Decision for Libby Asbestos Superfund Site, Libby and Troy Residential and Commercial Properties, Parks and Schools, Transportation Corridors, Industrial Park. Operable Units 4–8, Lincoln County, Montana		CDM Smith
2014	Remedial Investigation Report, Residential and Commercial Properties, Operable Unit 4, Libby, Montana		CDM Smith

Property-Specific Information Summary - Libby Asbestos Project

Program Name	Program Duration	Applicable Operable Units	Data Collection Methods	Field Form Names	Data Stores/Reporting Tools
Phase 1 Investigation	1999-2009	all	paper field sample data sheets (FSDSs) and field forms transcribed into Libby2; logbooks; photos	-Access Agreement -Background Information Field Form (BIFF) -Property Status Evaluation Form (PSEF)	property folders; scanned documents; Libby2 database/standard reports; folder tags
2002 Contaminant Screening Study (CSS)	2002-2003	primarily 4 and 7	paper FSDSs transcribed into the electronic Libby Asbestos Sample Tracking Information Center (eLASTIC) field database with exports to Libby2; paper field forms transcribed into Libby2; logbooks; photos	-Access Agreement -Primary Structure Information Field Form (IFF) with sketch -Secondary Structure Information Field Form (SIFF) -PSEF	property folders; scanned documents; Libby2 database/CDM Smith standard reports; folder tags
2003 CSS and Remedial Investigation (RI)	2003-2009	primarily 4 and 7	paper FSDSs transcribed into eLASTIC with exports to Libby2; paper field forms transcribed into Libby2; logbooks; photos	-Access Agreement -IFF -SIFF -Additional Information Field Form (AIFF) -PSEF	property folders; scanned documents; Libby2 database/CDM Smith standard reports; folder tags
Pre-Design Inspection (PDI)	2003-2008	primarily 4 and 7	paper FSDSs transcribed into eLASTIC with exports to Libby2; paper (early PDIs) and electronic forms (later PDIs) captured in Mobile Surveyor database with exports to Libby2; logbooks; photos	-Exterior Inspection Checklist (EIC) with sketch -Supplemental Interior Inspection Checklist (SIIC) with sketch -PSEF	property folders; scanned documents; Libby2 database/CDM Smith standard reports; folder tags
Response Action (RA), including Building Demolition from 2005-2007	2000-2009	all	paper FSDSs transcribed into eLASTIC with exports to Libby2; paper (early RAs) and electronic form (later RAs) captured in Mobile Surveyor database with exports to Libby2; logbooks; photos	-Property Completion Checklist (PCC) -PSEF	property folders; scanned documents; Libby2 database/CDM Smith standard reports; folder tags
Troy Asbestos Property Evaluation (TAPE)	2007-2011	7	electronic FSDSs and field forms using tablets with exports to the TOAD database and then exports to Scribe; paper field forms, logbooks, and property sketches scanned/uploaded to Response Manager (RM); photos uploaded to RM	-Access Agreement -Field Modification Form -Reference Parcel Form -Property Report -PSEF	property folders; scanned documents; RM; Scribe/TetraTech Property Reports; CDM Smith standard reports; folder tags; Property Report
General Property Investigation (GPI)	2010-present	primarily 4 and 7	paper FSDSs transcribed into the Data Entry Tool (DE Tool) with exports to Scribe; paper field forms transcribed or direct-entered into RM; logbooks; milestone dates entered into POTS starting in 2012; logbooks; photos	-Removal Access Agreement -Property Access Deferral or Refusal Form -Exterior Property Inspection Form (EPIF) -Interior Property Inspection Form (IPIF) -PSEF	property folders; scanned documents; RM; POTS, Scribe/CDM Smith standard reports; folder tags; POTS contractor reports; RM reports
Response Action (RA)	2010-present	all	paper FSDSs transcribed into the DE Tool with exports to Scribe; paper field forms transcribed or direct-entered into Response Manager (RM); logbooks; milestone dates entered into the Property Operations Tracking System (POTS) database starting in 2012; logbooks; photos	-Removal Access Agreement -Property Access Deferral or Refusal Form -PCC -As-Built Drawing -Quality Assurance Report (details on why an area was removed or not) -Completion Form (useful for pre-As-Built properties) -PSEF	property folders; scanned documents; RM; POTS, Scribe/CDM Smith standard reports; folder tags; POTS contractor reports; RM reports

Notes:

1. This summary includes key information used to evaluate or confirm property-specific remediation statuses (e.g., Removal Not Required, Removal Required, Removal Completed) and access statuses. Information from studies that supported risk assessment (e.g., Phase 2, post-cleanup evaluation, ambient air, activity-based sampling [ABS]), versus property-specific characterization or removal confirmation, is not included. Additionally, items with no bearing on statuses (such as general correspondence/letters, removal volumes forms) are not included.
2. In addition to Scribe sample results, historical sample results from Libby2 are accessible through CDM Smith standard reports for each media - air, bulk materials, dust, soil, water.
3. Folder tags and PSEFs are optional, as-needed property status evaluation tools developed by CDM Smith so may not be needed/available for all properties. Folder tags are an output from CDM Smith Remediation Status Queries (RSQ) that consider property status, property investigation information, and sample results from Response Manager, Scribe, and Libby2.
4. Similar to folder tags, Tetra Tech Property Reports are an output from Tetra Tech queries that consider property investigation information and sample results from TOAD and Scribe.

Evaluating Property Information for Remediation Status
Operable Units 4 and 7
Libby Asbestos Superfund Site
May 2019

The purpose of this document is to provide a high-level summary to the following questions:

- What were the different types of investigation programs that have been implemented at the site?
- What process was taken to verify properties meet current cleanup criteria (e.g., review of property inspection files, analytical data)?
- What informational tools (e.g., Response Manager, POTS 2, Scribe, property file scans, photos) are available and how do these work together (e.g., what information are extracted from each)?

Investigations Summary

Phase 1 Study: late 1999 through early 2002 – designed as a rapid pilot-scale investigation to (1) gather information to help determine whether a time-critical intervention was needed to protect public health and (2) screen properties using visual inspection and sampling to obtain data on asbestos levels in source materials to determine the most appropriate analytical methods.

Contaminant Screening Study (CSS): 2002 through 2009 – initial property screening performed primarily from 2002 through 2004; employed visual inspections and soil sampling to identify sources of Libby amphibole asbestos (LA) in high-traffic areas.

Pre-Design Investigation (PDI): 2003 through 2009 – collected additional information (e.g., delineation sampling for LA in soil) for response action planning and property-specific work plan preparation.

General Property Investigation (GPI): 2010 to present – revised process addressing both the screening investigation (SI) (formerly CSS) and detailed investigation (DI) (formerly the PDI) processes to streamline investigation and consider the revised investigation and cleanup criteria described in Amendments A and B to the 2003 Tech Memo and the Libby Asbestos Site-wide Record of Decision (ROD).

Troy Asbestos Property Evaluation (TAPE): 2007 to 2010 – screening-level investigation program employed at Troy, which paralleled the SI.

Remedial Design Investigation (RDI): 2007 to 2010 – detailed-level investigation program employed in Troy, which paralleled the DI.

Property Status Evaluation History and Process

2002 to 2003 – Manual Review of Property Field Forms and Sample Results

- CSS and PDI field forms and sample results reports were manually reviewed to identify properties requiring response action
- Evaluated against the 2003 Tech Memo for response action status (e.g., required, not required)
- Property information and analytical data were maintained in an EPA Libby database (Libby2)

- Quality control (QC) included independent review of the manual review and review by EPA as needed

2004 to 2010 – Remediation Status Query (RSQ)

- RSQ included a series of database queries and supporting business logic to evaluate Libby2 data against the 2003 Tech Memo for response action status (e.g., required, not required, pending)
- Property information was manually reviewed to confirm the RSQ statuses
- TAPE/RDI data were maintained in a separate database by state contractor staff and manually reviewed for status by those staff
- QC included independent review of the manual review and review by EPA as needed

2010 to Present – Response Manager and Scribe

- Data were transitioned from Libby2 to Response Manager and Scribe for improved support using EPA-nationwide systems; essential Troy data was also migrated
- Historical property statuses were back-populated into Response Manager, which was then used to maintain statuses moving forward
- The 2004 RSQ process ended; property statuses were manually evaluated against the 2003 Tech Memo (and later, the ROD) using Response Manager and Scribe data
- Statuses reviews were documented on a form called the Property Status Evaluation Form (PSEF); updated or new forms prepared as properties moved through the investigation and cleanup process
- Notes to file were also prepared, as needed, to clarify data use and/or status decisions regarding a property
- QC included independent review of the manual review and review by EPA as needed
- An EPA webtool was implemented in 2011 to supplement large-scale investigation and cleanup planning and communicate sitewide progress; webtool information was cross-referenced with state cadastral information and EPA property survey data to ensure every property within OUs 4 and 7 was accounted for and boundaries accurately represented to the extent possible

2014 to 2018 – Revised RSQ

- Amendment B to the Tech Memo was finalized in 2014, memorializing the updated cleanup approach (i.e., response action required if trace LA in >25% area of frequently used areas) ultimately recorded in the ROD
- A new RSQ was developed to evaluate Response Manager and Scribe data against Amendment B (and later, the ROD)
- The new RSQ process included evaluating newly collected data and re-evaluating subset of properties (even those with only CSS-level investigation) having statuses assigned prior to Amendment B
- Similar to the 2004 RSQ process, property statuses were manually confirmed using Response Manager and Scribe data; status reviews were documented on a PSEF or folder tag (the folder tag captures essential status information from RSQ; thus avoiding manual completion of forms)

2018 to Present – Manual Evaluation of Property Information

- As EPA property investigation and cleanup work slowed toward project completion in 2018, the 2014 RSQ process ended and property statuses were again manually evaluated
- In 2019, POTS 2 replaced Response Manager for managing site property information

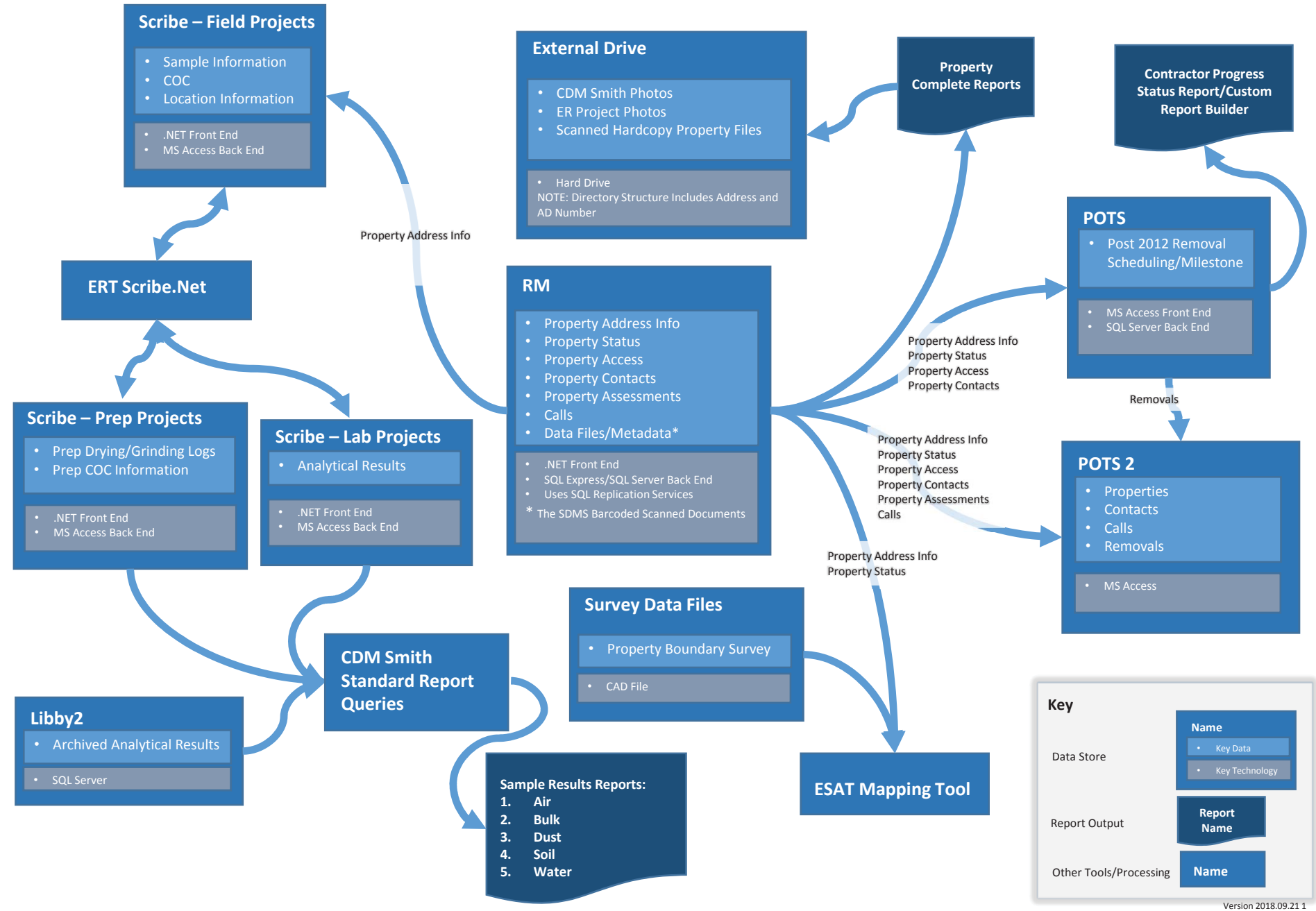
Information Tools for Property Evaluation

- **Property Ownership:** Check state cadastral information for current property ownership and boundary information. If there are owner changes, update contact information in POTS 2 or current system. If there is a split or merge of the property, edit POTS 2 or current system property information and any GIS system accordingly.
- **Property Status:** Look up the current property status in POTS 2 or current system.
- **Review Property Files:** Review investigation information, and cleanup documentation if applicable, using the property documents available in the current system or on the EPA archive hard drive.
- **Review Property Photos:** Review property photos and additional scanned information (available on the EPA archive hard drive) to answer questions about specific portions of a property, if necessary.
- **Review Sample Results:** Review sample results from Libby2 and Scribe (results are combined into one MS Excel report per sample media). CDM Smith currently provides these "standard reports" to users as needed/requested (via email, jump disk, etc.) as new data are collected. All Libby2 sample results were delivered to the state/county from CDM Smith on 5/7/19, which serves as the final deliverable from EPA for Libby2 data. State/county use of Scribe (via Scribe subscription) for managing sample information moving forward is being discussed as part of monthly project Scribe calls.

Notes

Tech Memo – *Libby Asbestos Site Residential/Commercial Cleanup Action Level and Clearance Criteria Technical Memorandum. 2003.*

Libby Data Flow Diagram



Version 2018.09.21 1

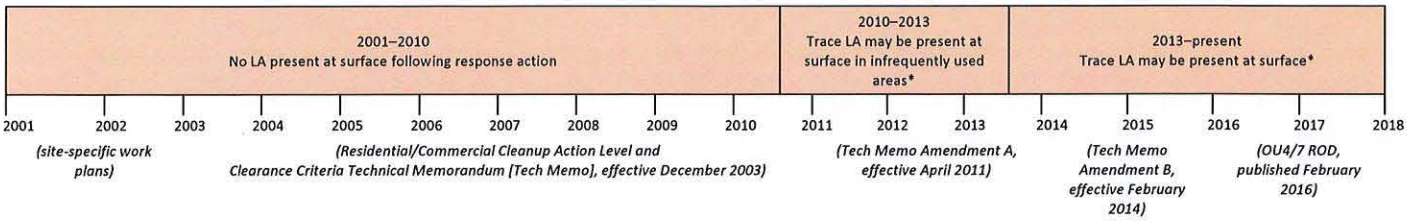
Response Action Timeline

Libby Asbestos Superfund Site Operable Units 4 and 7

Throughout development of the risk assessment, EPA learned more about Libby amphibole asbestos (LA) and the risk of exposure. All response actions that EPA conducted, either prior to the record of decision for operable units 4 and 7 (OU4/7 ROD) or after, have been protective. The timelines below illustrate property conditions where response action was completed or not required. The effective periods of the governing documents are included.

Response Action Completed

The timeline below indicates property conditions where a response action was completed. Current conditions meet OU4/7 ROD cleanup criteria based on the risk assessment.



Response Action Not Required

Response action is not required when there is no LA present at a property. Additionally, the risk assessment determined that small areas of trace LA could be present at the property and not require response action. The timeline below indicates property conditions where response action was not required.

*The risk assessment determined that small areas of trace LA may be left at a property without posing an unacceptable risk. The area of trace LA does not exceed 25% of a typical residential property. The area of trace LA may exceed 25% for infrequently used areas.

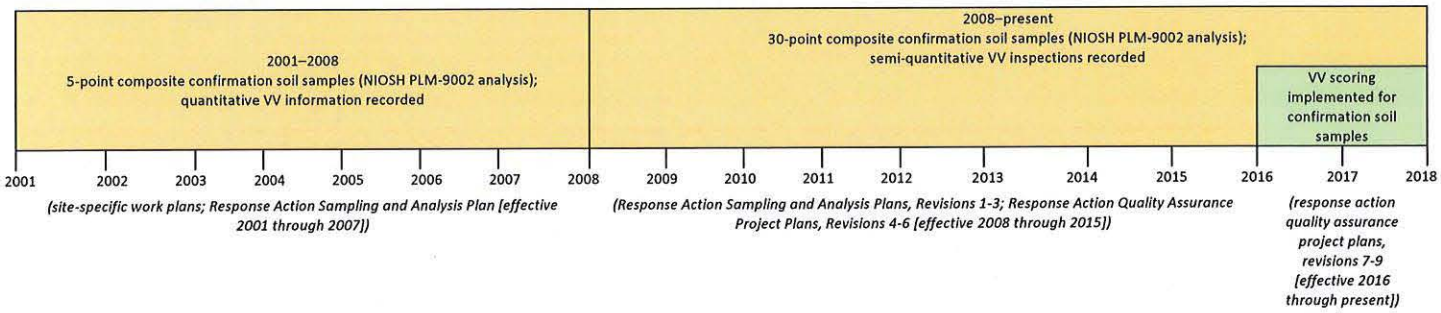
Point Composite and Visual Vermiculite Discussion

A 2002 EPA technical memorandum, *Technical Memo 1: Concordance Between Visible Vermiculite and the Occurrence of Asbestos by PLM in Soil and Soil-Like Media,* summarized data evaluating the assumption that when VV was present in a sample of soil-like media, asbestos would be observable in that sample when analyzed by National Institute for Occupational Safety and Health (NIOSH) method polarized-light microscopy 9002 (PLM-9002). Subsequently, EPA performed response action activities for soils at properties based, in part, on a conclusion of the memorandum that presence of VV in soil will usually be associated with elevated levels of LA.

From 2001 to 2007, confirmation soil samples were 5-point composites with qualitative visible vermiculite (VV) observations (i.e., present or not present) generally recorded. Beginning in 2008, confirmation soil sampling changed to 30-point composites combined with semi-quantitative VV inspections. A 2015 EPA technical memorandum, *Technical Memorandum: Use of Visible Vermiculite Status in Soil Removal Decisions*, re-evaluated the relationship between LA results by PLM by visual

estimation (PLM-VE) analysis and VV in soil, and re-assessed the role of VV in determining the need for soil response actions. This memorandum illustrated the correlation of VV with both PLM-VE and PLM-9002 analytical results by presenting frequency of detected LA stratified by a "VV score", and concluded: (1) the presence of VV is a good indicator of the probable presence of LA in soil by PLM-VE, particularly when the VV score is above 1.0, and (2) VV scores above 3.0 are a good indicator of LA in soil at or above 1 percent (%) by PLM-9002.

The OU4/7 ROD specifies clearance criteria for subsurface soil in terms of PLM-VE. However, because the PLM-VE method requires special sample preparation (sieving, grinding) prior to performing the PLM analysis, there is a significant lag time between sample collection and results receipt. Because of this, the PLM-VE method is not optimal for supporting real-time decisions during soil removal activities. The PLM-9002 method does not require the use of special sample preparation prior to analysis, which reduces the lag time between sample collection and results receipt, but the reported results do not allow for the discernment of LA concentrations below 1% to determine if they are less than (<) 0.2% (Bin B1 by PLM-VE) or greater than or equal to 0.2% (Bin B2 by PLM-VE). However, EPA's 2015 technical memorandum suggests that use of PLM-9002 coupled with field observations of VV can be used to approximate the likely PLM-VE bin associated with the PLM-9002 result. If the PLM-9002 result is reported as <1% and the associated VV score is <1.0, this is likely to be equivalent to Bin B1 by PLM-VE. Therefore, PLM-9002 is the selected analytical method for confirmation soil samples to determine if clearance criteria are achieved. The timeline below indicates the soil confirmation sampling protocols.



Response Action Timeline_050918

APPENDIX B – USE OF O&M SETTLEMENT FUNDS

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 8
1595 Wynkoop Street
Denver, Colorado 80202-1129
Phone 800-227-8917
www.epa.gov/region8

August 19, 2019

Ref: 8SEM-RBB

Carolina Balliew
Federal Superfund Section Supervisor
Montana Department of Environmental Quality
1225 Cedar Street
Helena, Montana 59601

Re: Use of Settlement Funds on O&M-Specific Items, Libby Asbestos Superfund Site

Dear Carolina:

As the U.S. Environmental Protection Agency (EPA) and Montana Department of Environmental Quality (MTDEQ) have previously discussed, multiple unresolved issues involving performance of Operations and Maintenance (O&M) and available O&M funding have arisen at the Libby Asbestos Superfund site (Site). The EPA provides recommendations for resolving these issues, as follows:

- At private residential properties, where previous sampling results identified the presence of Libby Amphibole (LA) but not at levels high enough to require a cleanup, and the owner now expresses an interest in changing a use area (i.e., home addition, adding a building, etc.), Region 8 recommends that O&M funding be available for sampling after the changes are complete to ensure that the area is still protective. If sampling determines that further cleanup is needed, the EPA recommends that O&M may be used for the necessary clean up. Availability of O&M funding should be contingent on the owner contacting the Asbestos Resource Program (ARP) prior to the work.

- For a commercial or for-profit developer, Region 8 does not recommend use of O&M funds for sampling; instead, the developer or business owner is to be responsible for costs of any needed sampling. However, the property owner should work with the ARP program to ensure sampling is conducted in an acceptable manner (i.e., proper QA and sampling plans, etc.) as outlined in the O&M plan. If, through adequate sampling, it is determined that cleanup is needed, O&M funding could be used for associated clean up.

- In cases of commercial improvement or development, where the owner/developer does not contact ARP prior to changes in use area, the EPA does not recommend use of O&M funding for sampling or for a cleanup, if it is determined later that a cleanup is needed.

- O&M funding should not be provided for per diem for properties under going remodeling, damage, repair due to fire, etc. However, if the EPA missed components of a cleanup (such as LA in the attic, etc.) due to error or oversight during that original inspections/clean up, the EPA recommends that cost be covered with O&M funds.

- The EPA recommends that O&M funding not go towards payment to replace material when a building is being remodeled, and O&M funding only address areas needing clean up due to presence of LA.

The EPA is providing the above recommendations to MTDEQ upon MTDEQ's request and following up on discussions between the EPA and MTDEQ. If you have additional comments or questions, please contact me at 303-312-6694.

Sincerely,

Stan Christensen
Superfund Section Chief

cc: Lisa Dewitt, MTDEQ
Jenny Chambers, MTDEQ
Tom Stoops, MTDEQ
Jessica Wilkerson, MTDEQ

Mike Cirian, EPA
Dania Zinner, EPA
Aaron Urdiales, EPA
Max Greenblum, EPA

DEQ
Montana Department of Environmental Quality

October 11, 2019

Stan Christensen
Region 8 EPA Superfund Section Chief
1595 Wynkoop St.
Denver, CO 80202

Re: DEQ Response to Use of Settlement Funds of O&M-Specific Items, Libby Asbestos Superfund Site

Dear Stan,

The Department of Environmental Quality (DEQ) has received and reviewed the Environmental Protection Agency's (EPA) recommendations within the August 19, 2019 letter regarding some unresolved issues involving the performance of Operations and Maintenance (O&M) and available EPA settlement O&M funding at the Libby Asbestos Superfund site (Site). DEQ is mindful of the EPA's recommendations and recognizes the constraints of EPA O&M funding. DEQ looks forward to continuing to work with all stakeholders to finalize the Institutional Control and Implementation Plan (ICIAP) and O&M Plan that will further guide discussions of the availability of EPA O&M funding.

Both EPA and DEQ recognize that the Asbestos Resource Program's (ARP) role in O&M is to protect the remedy in the Libby and Troy communities. As indicated in your letter, DEQ agrees that property owners should contact ARP prior to work and coordinate with ARP, as appropriate.

DEQ appreciates our collaborative discussions on these and other O&M issues. We value our ongoing teamwork with EPA and Lincoln County and these committed partnerships will help us finish developing and implement the O&M Program to maintain a protective remedy with consideration of long-term planning of all available funds.

Sincerely,

Carolina Balliew

Carolina Balliew
Federal Superfund Section Supervisor
1225 Cedar Street
Helena, MT 59601

Cc: Lisa Dewitt
Jenny Chambers
Tom Stoops
Jessica Wilkerson JW
Mike Cirian, EPA
Dania Zinner, EPA
Aaron Urdiales, EPA
Max Greenblum, EPA

APPENDIX C – O&M CONTACTS

O&M Contact List

Contact Name	Representing	Email	Phone
Montana Department of Environmental Quality			
Jason Rappe	Libby Superfund Project Manager	Jason.rappe@mt.gov	406-444-6802
Libby Asbestos Superfund Site Oversight Committee Members			
Shaun McGrath	DEQ, Director	shaun.mcgrath@mt.gov	406-444-2544
Mark Peck	Lincoln County Commissioner	mark.peck@libby.org	406-283-2317
George Jamison	Citizen	gjamison@libby.org	406-293-8567
Steve Gunderson	State House Representative	steve.gunderson@mtleg.gov	406-334-4370
Mike Cuffe	State Senator	mike.cuffe@mt.leg.gov	406-889-5777
City County Board of Health for Lincoln County (Health Department: 406-283-2442)			
Jan Ivers	Lincoln County Representative	jivers@libby.org	
George Jamison	Lincoln County Representative	gjamison@libby.org	406-293-8567
Sara Mertes	Lincoln County Representative	smertes@libby.org	
Mark Peck	Lincoln County Commissioner	mark.peck@libby.org	406-283-2317
Laura Crismore	City of Libby Representative	lcrismore@libby.org	
Maggie Anderson	City of Troy Representative	manderson@libby.org	
Debra Armstrong	Town of Eureka Representative	darmstron@libby.org	
City County Board of Health for Lincoln County – Lincoln County Asbestos Resource Program (ARP)			
Virginia Kocieda	Program Director	vkocieda@libby.org	406-283-2446
Amanda Harcourt	Field Operations Manager	aharcourt@libby.org	406-283-2445
Elzhon Anderson	Resource Coordinator	elanderson@libby.org	406-283-2462
Lincoln County Solid Waste Division - Landfill Operations			
Kathi Hooper	Health Department Director	khooper@libby.org	406-283-2440
Bryan Alkire	Solid Waste Manager	lclandfill@libby.org	406-293-7146
Virginia Kocieda	ARP Program Director	vkocieda@libby.org	406-283-2446
Elzhon Anderson	LA Waste Coordinator	elanderson@libby.org	406-283-2462
U.S. Environmental Protection Agency			
Mike Cirian	Project Manager	cirian.mike@epa.gov	406-293-6194
Dania Zinner	Project Manager	Zinner.dania@epa.gov	303-312-7122

APPENDIX D – STATUS LETTERS (UNDER DEVELOPMENT)

Month XX, 2020

RE: **Address**
Property ID: **AD######**
Legal Description: **[Copy from MT Cadastral]**

Dear Property Owner:

The Montana Department of Environmental Quality (DEQ) is confirming that cleanup is not required at the above-referenced property. This determination is based on EPA's final remedy as defined in the 2016 Record of Decision for the Libby Asbestos Superfund Site (Site) and the results from any previous investigation at this property (see enclosure).

To help manage possible future encounters with Libby Amphibole asbestos (LA), a long-term management plan was developed by DEQ, the United States Environmental Protection Agency (EPA), local government agencies, and the community. This long-term management plan outlines the responsibilities of DEQ, Lincoln County, and property owners to ensure long term protection of human health and the environment. It is important to contact the Lincoln County Asbestos Resource Program (ARP) at (406) 291-5335 before beginning any excavation, renovation, or demolition work at this property or if a new possible source of vermiculite is encountered.

EPA has completed cleanup at the Site. The agencies have implemented a final plan for institutional controls, along with the long-term management plan. Institutional controls are non-engineered instruments such as administrative and legal limits that help minimize the potential for human exposure to contamination and/or protect the integrity of the remedy. At that time, DEQ oversees operations and maintenance at the site. Both DEQ and Lincoln County are responsible for maintaining records describing the status of investigation and/or cleanup for each property within the Site and providing information to the community on how to address situations where contaminated vermiculite or LA may be discovered. We recommend that you retain this information.

If you have any questions about the Site or this property status, please contact ARP in Libby at (406) 291-5335.

Sincerely,

Jason Rappe
Federal Superfund Project Officer
Montana Department of Environmental Quality
1225 Cedar Street
Helena, MT 59601

Month XX, 2020

RE: **Address**
Property ID: **AD######**
Legal Description: **[Copy from MT Cadastral]**

Dear Property Owner:

The Montana Department of Environmental Quality (DEQ) is confirming that a removal has been performed at the above-referenced property based on EPA's final remedy as defined in the 2016 Record of Decision for the Libby Asbestos Superfund Site (Site). Based on the results from any additional previous investigation and clearance sampling at this property, it is determined that an additional removal is not required. The removal completion documentation has been provided (see enclosure).

To help manage possible future encounters with Libby Amphibole asbestos (LA), a long-term management plan was developed by DEQ, the United States Environmental Protection Agency (EPA), local government agencies, and the community. This long-term management plan outlines the responsibilities of DEQ, Lincoln County, and property owners to ensure long term protection of human health and the environment. It is important to contact the Lincoln County Asbestos Resource Program (ARP) at (406) 291-5335 before beginning any excavation, renovation, or demolition work at this property or if a new possible source of vermiculite is encountered.

EPA has completed cleanup at the Site. The agencies have implemented a final plan for institutional controls, along with the long-term management plan. Institutional controls are non-engineered instruments such as administrative and legal limits that help minimize the potential for human exposure to contamination and/or protect the integrity of the remedy. At that time, DEQ oversees operations and maintenance at the site. Both DEQ and Lincoln County are responsible for maintaining records describing the status of investigation and/or cleanup for each property within the Site and providing information to the community on how to address situations where contaminated vermiculite or LA may be discovered. We recommend that you retain this information.

If you have any questions about the Site or this property status, please contact ARP in Libby at (406) 291-5335.

Sincerely,

Jason Rappe
Federal Superfund Project Officer
Montana Department of Environmental Quality
1225 Cedar Street
Helena, MT 59601

APPENDIX E – CITY-COUNTY BOARD OF HEALTH FOR LINCOLN COUNTY PEN INFORMATION

HEALTH AND ENVIRONMENT REGULATIONS
CHAPTER 1: Control of Air Pollution
Subchapter 2: Libby Amphibole (LA) Property Evaluation Notification (PEN)
Date Adopted: March 11, 2020
Date Effective: *to be determined*

I. REGULATION, AUTHORITY AND PURPOSE

A. The City/County Board of Health for Lincoln County (Board of Health) was created as the Local Board of Health for Lincoln County by an Inter-local Agreement between the City of Libby and Lincoln County with authority under Mont Code Ann. § 50-2116(2)(c)(v)(A) to enact public health regulations to protect public health, safety, and welfare and to facilitate Institutional Controls selected by the United States Environmental Protection Agency (USEPA) and Montana Department of Environmental Quality (DEQ) for the Libby Asbestos Superfund Site.

B. The Board of Health finds there is a threat to public health, safety, and welfare posed by the environmental conditions that led the USEPA to designate the Libby Asbestos Superfund Site. That threat was largely mitigated by completion of remedial actions performed by the USEPA. The remedial action included leaving some contamination in place. As such, the final remedial action condition as well as ongoing and future changes on properties must be maintained to ensure protectiveness of the remedy.

C. The Board of Health collaborates with the DEQ and the USEPA to continue to protect public health, safety, and welfare by ensuring that the Libby Asbestos Superfund Site remedies remain protective and LA asbestos is properly managed to ensure protectiveness of the remedy.

D. The Lincoln County Asbestos Resource Program (ARP) is a Board of Health directed public health program that was established in 2012 with the mission of reducing potential exposure to LA asbestos that is found within the Libby Asbestos Superfund Site and the surrounding areas of Lincoln County. A key goal of the Board of Health directed ARP to minimize burden on the community members themselves. The program was developed by the USEPA as a pilot study as the Environmental Resource Specialist (ERS) program and through a cooperative agreement passed on to Lincoln County ARP program in January 2014 and modified under the guidance of the Board of Health to its current program under the guidance of the Board of Health and is currently funded through a cooperative agreement/grant from the USEPA.

E. DEQ is responsible for future Operation and Maintenance (O&M) of the Site, and funding from DEQ is anticipated for ARP to support O&M activities.

F. The Board of Health has chosen to implement this Property Evaluation Notification Regulation pursuant to its authority under Mont Code Ann. § 50-2-116(2)(c)(v)(A) to protect public health, safety, and welfare.

II. GENERAL PROVISIONS

A. Title: These regulations shall be known as the "LIBBY AMPHIBOLE (LA) ASBESTOS PROPERTY EVALUATION NOTIFICATION (PEN)".

B. Authority: Authorization for these regulations is through Montana Code Annotated (MCA) § 50-2-116(2)(c)(v)(A).

C. Purpose: The purpose of this regulation is to reduce the possibility of the public's exposure to LA asbestos as a result of Applicable Activities, as defined in Definitions in Section F.2 of this regulation. These activities shall be referred to as Applicable Activities. This PEN regulation is focused on providing LA asbestos property information, data, education, and evaluations to protect the public during Applicable Activities. This PEN regulation is an institutional control listed within the Operating Unit 4 and Operating Unit 7 Institutional Control Implementation and Assurance Plan (ICIAP). Note that this PEN regulation is separate from the Montana Asbestos Control Act, DEQ Asbestos Control Program requirements, or other due diligence processes, and does not replace or supersede the associated regulations on asbestos in Montana.

D. Contingent Applicability: Implementation and execution of this regulation is dependent upon the existence and continued functionality and funding of the ARP. Similarly, success of the ARP is highly dependent upon the existence of this regulation. If the ARP ceases to exist or is unable to effectively function from lack of funding or other reasons, then this regulation will be suspended until the ARP, or other BOH designated organization, is functional and able to again support implementation and execution. Such suspension shall not be effective until the Board of Health affirmatively votes to suspend this regulation.

E. Jurisdiction: This LA PEN regulation governs activities within the Libby Asbestos Superfund Site National Priorities List boundary which is composed of eight Operable Units, all of which are located in Lincoln County, Montana. Jurisdiction includes Operable Units 1, 2, 4, 5, and 7. Operable Unit 3 (the Former Libby Vermiculite Mine), Operable Unit 6 (Burlington Northern Santa Fe Railroad and Rail corridors) and Operable Unit 8 (Roadways) are excluded from the requirements of this LA PEN regulation. Descriptions of the jurisdictional areas included within each Operable Unit governed by this PEN regulation are detailed in each respective Record of Decision and summarized below:

1. Operable Unit 1 is the former Export Plant, and is situated on the south side of the Kootenai River, just north of the downtown area of the City of Libby, Montana. OU1 includes the embankments of Montana Highway 37, the former Export Plant, and the Riverside Park. The property is bounded by the Kootenai River on the north, Highway 37 on the east, the Burlington Northern Santa Fe railroad thoroughfare on the south, and the State of Montana property on the West (EPA, May 2010a). These areas and boundaries are shown the Operable Unit 1 Record of Decision Exhibit 2-2 (EPA, May 2010a). Currently in the final stages of Deletion from the NPL.
2. Operable Unit 2 includes area impacted by contamination released from the former Screening Plant. These areas include the former Screening Plant, the Flyway property, a privately-owned property, and the Rainy Creek Road Frontage and Highway 37 right-of-way adjacent to Rainy Creek Road (EPA, May 2010b). These areas and boundaries are shown in the Operable Unit 2 Record of Decision Exhibit 22 (EPA, May 2010b). Formally Deleted from the NPL on April 10, 2019.

3. Operable Unit 4 is called Libby Residential/Commercial areas. Operable Unit 4 is defined as the residential, commercial, industrial (not associated with Grace Mining Operations), and public properties, including schools and parks, in and around the City of Libby (EPA, February 2016). The boundaries for Operable Unit 4 are shown in Exhibit 1-2, Figure 1-2, and Figures 5-2 through 5-16 in the Operable Unit 4 through 8 Record of Decision (EPA, February 2016.

4. Operable Unit 5 is called the Former Stimson Lumber Company. Operable Unit 5 is defined geographically by the parcel of land that included the former Stimson Lumber Company. OU5 is bounded by the high bank of Libby Creek to the east, the Burlington Northern Santa Fe railroad to the north, and properties within Operable Unit 4 to the south and west (EPA, February 2016). The boundaries for Operable Unit 5 are shown in Exhibit 1-2, Figure 1-2, and Figures 5-17a through 5-17b in the Operable Unit 4 through 8 Record of Decision (EPA, February 2016).

5. Operable Unit 7 is called Town of Troy, and is defined as the residential, commercial, and public properties in and around the Town of Troy, Montana located 20 miles west of downtown Libby (EPA, February 2016). The boundaries for Operable Unit 7 are shown in Exhibit 1-2, Figure 1-2, and Figures 5-21 through 5-25 in the Operable Unit 4 through 8 Record of Decision (EPA, February 2016).

F. Definitions: The following definitions shall apply in the interpretation and enforcement of this regulation. The word "shall" as used in this regulation indicates a mandatory requirement.

1. LA asbestos is specific to the form of naturally occurring amphibole asbestos comprised of a range of mineral types and morphologies, and associated with the Libby vermiculite deposits in the region near the Libby Asbestos Superfund Site (EPA, February 2016). LA asbestos forms durable, long, thin structures that are generally respirable, can reasonably be expected to cause disease, and is considered to be the contaminant of concern at the Libby Asbestos Superfund Site (EPA, February 2016).

2. "Applicable Activities" means activities related to real property to include:
    a. Excavation, grading, and landscaping;
    b. Interior or exterior demolition, repair, modification, disturbance of material, or remodeling of permanent or temporary structures;
    c. Transfer of real property regardless of whether any comfort letter has been issued by USEPA or any other agency;
    d. Change in Land Use Category or Property Use Area as used in Sections 2.3 and 4.2 of the *Remedial Design Report, Revision 1, Libby Asbestos Site Operable Units 4 & 7* (April 5, 2017); and
    e. Any dividing of land, including through subdivision, family transfer, Court-ordered division, or other division of land.

3. "LA Asbestos Property Evaluation" means a required evaluation, performed by the ARP, to include evaluation of data and information related to LA asbestos based on the notification by a property owner or interested party who has submitted a PEN due to planned Applicable Activities within the jurisdiction

(Section E above). The LA Asbestos Property Evaluation will be performed by the ARP to provide information relative to the potential for LA Asbestos exposure related to the Applicable Activity as detailed. This regulation details the PEN notification requirements and the associated LA Asbestos Property Evaluation elements to be provided in an effort to protect the remedy and public health.

4. "Days" means business days (i.e., Monday, Tuesday, Wednesday, Thursday, and Friday), excluding holidays observed by Lincoln County and ARP.

5. "Person" is any individual, institution, partnership, business, corporation, association, or other private or government entity.

6. "Property" is real property that is fixed property, principally land and structures. This regulation applies to the Applicable Activities related to real property within the jurisdiction.

G. Severability: If any provision of this Regulation is declared invalid by any court or tribunal, the remaining provisions of this Regulation shall not be affected thereby.

III. LIBBY AMPHIBOLE ASBESTOS PROPERTY NOTIFICATION PROCESS

A. LA Asbestos Property Evaluation Notification (PEN) Process Requirements: Prior to performing Applicable Activities within the above defined jurisdiction, a person is required to notify the ARP of the proposed Applicable Activities through the PEN process.

B. Applicability Specifics:

1. The following Applicable Activities within the jurisdiction require a PEN:
    a. Excavation, grading, and landscaping;
    b. Interior or exterior demolition, repair, modification, disturbance of material, or remodeling of permanent or temporary structures;
    c. Transfer of real property regardless of whether any comfort letter has been issued by USEPA or any other agency;
    d. Change in Land Use Category or Property Use Area as used in Sections 2.3 and 4.2 of the *Remedial Design Report, Revision 1, Libby Asbestos Site Operable Units 4 & 7* (April 5, 2017); and
    e. Any dividing of land, including through subdivision, family transfer, Court-ordered division, or other division of land.

2. In addition to the defined Applicable Activities, the following activities within the jurisdiction also require a PEN:

    a. These requirements are applicable to modification or construction of wastewater systems requiring disturbance of surface or subsurface soils.
    b. These requirements are applicable to any division of property, including through subdivision, family transfer, Court-ordered division, or other division of land. Subdivision definitions, requirements, and permits are authorized by separate entities and regulations. The Lincoln County Subdivision regulations contain specific requirements related to

examination of potential LA related issues as a condition of approval of the subdivision. Division of property exempt from the Subdivision regulations is however an Applicable Activity requiring a PEN.

c. These requirements are applicable to government entities performing Applicable Activities within the jurisdiction.
d. Emergency response activities (such as floods, fires, natural disasters, building collapse, sinkholes, earthquakes, etc.) where the excavation, modification, or demolition activities are conducted in response to a property emergency. In this case, the ability to submit a PEN form beforehand is not feasible. Thus, the property owner shall notify ARP of the emergency response activity within three (3) business days to determine if a post-facto PEN notification or inspection is required.

3. Exclusions to PEN Process include the following:
    a. Remodeling activities that are cosmetic in nature (e.g. wallpaper installation or removal, carpet installation or removal, painting, installing built-in furniture, etc.) that will not disturb the existing interior flooring (excluding carpet), interior walls, ceilings, structural elements, exterior siding, roofing, foundations, utility penetrations or insulation;
    b. Exterior landscaping or remodeling that will not disturb surface or subsurface soil (e.g., concrete repair/staining, replace slats on decking, staining or painting fencing, etc.); or
    c. Emergency response activities (such as floods, fires, natural disasters, building collapse, sinkholes, earthquakes, etc.) where the excavation, modification, or demolition activities are conducted in response to a property emergency. In this case, the ARP shall be notified the next business day to determine if a post-facto PEN notification or inspection is required.

C. PEN Requirements: The notification of intent to perform Applicable Activities for a property shall be made to the ARP by the owner of the property, or the owner's authorized agent, on a form provided by the ARP (electronic or hard-copy) and/or through the Montana811 utility locate request process.

1. Notification for those Applicable Activities regulated by Montana811 through MCA Title 69, Chapter 4, Part 5 are automatically notified to the ARP when submitted through the Montana811 notification process and will serve as notification to ARP relative to the PEN process. If activities are limited to those regulated by Montana811 then no additional PEN-specific ARP form is required.

2. Applicable Activities not captured under Montana811 Notifications within the jurisdiction will require preparation and submittal of the ARP PEN form signed and dated by the applicant, and will include the following information, at a minimum:

    a. The name, address, email address, and telephone number of the person who owns the real property;
    b. The name, address, email address and telephone number of the person submitting the PEN.
    c. The physical address of the property or a legal description if a physical address is not assigned where the Applicable Activity will take place;

d. The name, address, email address, and phone number of the person who will be responsible for performing the Applicable Activity, if it is not the owner of the real property. If a contractor is to be used, provide their name, address, telephone number, and any asbestos related credentials or certifications;
e. Confirmation that Montana811 has been notified, if applicable; and
f. A description of the proposed activity, including:
    i. The general nature and extent of the project including the project objective, including a specific statement regarding whether division of property is an objective;
    ii. Estimated location, mass, area, and volume (as applicable) of the media or building materials that will be disturbed or removed;
    iii. If already proposed, any mitigating or best management practices that are planned to reduce or eliminate the exposure to LA asbestos and/or vermiculite, if anticipated, and measures to reduce the generation of dust;
    iv. Planned activities for transporting and disposing of building materials, soil, waste, disturbed materials, and potential LA asbestos and/or vermiculite; and
    v. If the Applicable Activity is the sale of real property or change in Land Use Category, the description should state "sale of property" or "Change in Land Use Category".

D. Fee: No fee will be associated with a PEN for the owner or person submitting the notification.

E. PEN and LA Asbestos Property Evaluation Process: PEN forms shall be submitted to ARP and a subsequent LA Asbestos Property Evaluation conducted. In addition to the "ARP Required Response" outlined in Section III.E. below, ARP is authorized to do none, any, or all of the following activities in response to a PEN submission:

1. Collection of prior information related to LA investigations, inspections, site records, evaluations, designs, remedies, communications, etc. as may be available from EPA documents and database, DEQ Libby Instance Response Manager database, or other accessible sources;

2. Site observations, including reference to available maps/figures and other available records, and an ARP site visit of the subject property (on or near the property depending on access permission granted by the owner);

3. Discussion with owner, PEN applicant, or contractor representatives related to property conditions and proposed activities;

4. An evaluation of prior information and site observations in relation to former and current land use, existing conditions, future land use, and proposed activities at the property;
5. Summarization of collected information, site observations, evaluations;

6. Recommendations as may be specific to the Subdivision approval process for follow up activities, such as sampling, evaluations, and cleanups;

7. Recommendations for Best Management Practices, available resources to support the activity, and informational/educational materials;

8. Follow up site visit, if applicable;

9. Dialog and communication summary;

10. Assistance in identifying a remediation contractor, if applicable;

11. Guidance related to possible mitigation of expenses for the incremental cost to the project attributable to the presence of LA;

12. Evaluations and/or recommendations specific to the Subdivision review and approval process;

13. Updates to property evaluation and pertinent applicable activities or inspections will be uploaded and tracked by ARP in the DEQ Libby Instance Response Manager database.

F. ARP Required Response:

1. Notifications shall be submitted at least three (3) full business days prior to the initiation of Applicable Activities. Once notified, the ARP has two full business days to discuss activities to be performed and to respond by giving the current property status. Day one begins the next operating business day after the PEN form submittal to the ARP. The timeline for ARP's discussion with the applicant is based on expected circumstances. If there are unforeseen circumstances, ARP will provide notice to the applicant of a modified timeline.

2. Once a complete PEN form is submitted, the ARP shall review the notification and perform the ARP LA Asbestos Property Evaluation to assess the potential for LA asbestos exposure based on previous LA asbestos evaluations, remedies, and inspections. If the PEN notification is incomplete, the ARP may request additional information prior to performing or completing their Evaluation.

3. Notifications to ARP are separate from, and not limited to, other required notifications under local, county, state, or federal law.

G. Evaluation Reporting: Upon completion of the LA Asbestos Property Evaluation, the ARP will communicate the findings to the applicant and/or owner, and document the communication. Different PEN deliverables will be offered according to the applicable activity:

1. Response for excavation, grading, landscaping activities: After receiving a completed PEN form, a phone call and/or email to the PEN requestor explaining the current status of the property will suffice as a completed PEN response. Confirmation that Montana811 utility locate has been notified of planned digging activity will be requested. Please see Section III B (1) for details on Montana811 utility locates and the PEN notification. If follow-up is needed, an additional evaluation performed by ARP may be conducted. An additional phone call, email

and/or letter would summarize the findings of this additional evaluation and any additional steps that need to be taken. Best management practices and guidance for disposal, relevant to the applicable activity, will be shared with the PEN requestor. A summary of PEN activities, and associated records or documents, will be retained in DEQ and/or ARP databases or files.

2. Response for interior/exterior demolition, repair, modification, disturbance of material, or remodeling to permanent or temporary structures: After receiving a completed PEN form, a phone call and/or email to the PEN requestor explaining the current status of the property will suffice as a completed PEN response. If follow-up is needed, an additional evaluation performed by ARP may be conducted. An email and/or letter would summarize the findings of this additional evaluation and any additional steps that need to be taken. Best management practices and guidance for disposal, relevant to the applicable activity, will be shared with the PEN requestor. A summary of PEN activities, and associated records or documents, will be retained in DEQ and/or ARP databases or files.

3. Response for sale of real property: After receiving a completed PEN form, a phone call and/or email to the PEN requestor explaining the current status of the property will suffice as a completed PEN response. After communicating with the buyer and/or seller of real property, ARP will develop a letter detailing the current status of the property and activities performed on the property during cleanup. The letter can be delivered electronically or by mail. See Section E 3(G) on Disclosure of LA Asbestos Property Evaluation in Sale of Property. Maintenance requirements for installed engineering controls, relevant to the specific remedy on the property, will be shared with the PEN requestor. A summary of PEN activities, and associated records or documents, will be retained in DEQ and/or ARP databases or files.

4. Response for Change in Land Use Category or Property Use Area: After receiving a completed PEN form, ARP will make a phone call and/or send an email to the PEN requestor explain the current status of the property. An additional evaluation performed by ARP may be required which entails the analysis of previous sampling, if any, within the proposed work area, researching property files of surrounding properties near the proposed work area, and a visual soil inspection of the work areas. A detailed report summarizing the findings of this additional evaluation, along with an ARP recommendation for any additional steps that need to be taken will be given to the PEN requestor. Best management practices and guidance for disposal, relevant to the applicable activity, will be shared with the PEN requestor. A summary of PEN activities, and associated records or documents, will be retained in DEQ and/or ARP databases or files.

5. Response for any division of property, including through subdivision, family transfer, Court-ordered division, or other division of land: The Lincoln County Subdivision Regulations require an APR evaluation initiated through a PEN submission as part of the subdivision application review. After receiving a completed PEN form, ARP will make a phone call and/or email to the PEN requestor explaining the current status of the property. An additional evaluation performed by ARP is required which entails the analysis of previous sampling, if any, within the proposed work area, researching property files of surrounding

properties near the work area and a visual soil inspection of the proposed work areas. A detailed report summarizing the findings of this additional evaluation, along with an ARP recommendation and any additional steps that need to be taken will be given to the PEN requestor. This letter may be included in the new subdivision package for the County Planner to receive. Best management practices and guidance for disposal, relevant to the applicable activity, will be shared with the PEN requestor. A summary of PEN activities, and associated records or documents, will be retained in DEQ and/or ARP databases or files.

H. Disclosure of LA Asbestos Property Evaluation in Sale of Property: Sellers of real property shall submit a PEN application as outlined in Section III.B.2. above. Sellers shall provide a copy of the resulting LA Asbestos Property Evaluation to any buyer, or buyer's agent, prior to sale of seller's property. At buyer's request, seller shall also provide a copy of the resulting LA Asbestos Property Evaluation to any third parties (for example, lending institutions, insurers, etc.).

I. Individuals not performing Applicable Activities, but who wish to obtain a LA Asbestos Property Evaluation for a property, may contact ARP to submit a request for a LA Asbestos Property Evaluation. ARP, at its discretion, may initiate the PEN process on any property within the jurisdiction of this regulation. Those LA Asbestos Property Evaluation will be processed based on ARP availability.

J. Penalties: Violations of any provision of this regulation is counter to the USEPA Libby Asbestos Superfund Site remedy, operation and maintenance, and institutional control measures. Violations of this notification could result in exposure to or spreading of LA contamination and may be subject to enforcement provisions by the BOH under MCA § 50-2-124. Failure to comply may exclude consideration of any financial assistance that may be available.

K. Effective Date: Once the regulation is adopted by the City/County Board of Health for Lincoln County, the requirements of this regulation shall not become effective until the City/County Board of Health for Lincoln County passes a resolution stating the effective date of this regulation.

IV. REFERENCES

EPA, 2010a. *Record of Decision for Libby Asbestos Superfund Site, The Former Export Plant Operable Unit 1*. Libby Asbestos Site, Libby, Montana. Prepared for the EPA by CDM Federal Programs Corporation. EPA Document: 1154081.

EPA, 2010b. *Record of Decision for Libby Asbestos Superfund Site, The Former Screening Plant and Surrounding Properties Operable Unit* 2. Libby Asbestos Site, Libby, Montana. Prepared for the EPA by CDM Federal Programs Corporation. EPA Document: 1154082.

EPA, 2016. *Record of Decision for Libby Asbestos Superfund Site – Libby and Troy Residential and Commercial Properties, Parks and Schools, Transportation Corridors, and Industrial Park – Operable Units 4 through 8.* Libby Asbestos Site, Libby, Montana. Prepared for the EPA by CDM Federal Programs Corporation. EPA Document: 1563024.

EPA, 2020. *Operable Units 4 and 7, Institutional Control Implementation and Assurance Plan.* Libby Asbestos Superfund Site, Libby, Montana. Prepared for the EPA by CDM Smith. EPA Document: (to be determined). *In preparation.*

EPA, 2020. *Operable Units 4 and 7, Operations and Maintenance Plan.* Libby Asbestos Superfund Site, Libby and Troy Residential and Commercial Properties, Parks, and Schools. Prepared for the EPA by CDM Federal Programs Corporation. EPA Document: (to be determined). *In preparation.*

DEQ, 2020. *Operable Units 4 and 7, Operations and Maintenance Manual.* Libby Asbestos Superfund Site. Prepared for DEQ by Weston Inc. *In preparation.*

Lincoln County, 2019. *Lincoln County Subdivision Regulations.* Prepared to comply with the Montana Subdivision and Platting Act.

Libby/Troy Amphibole Asbestos Property Evaluation Notification

Your Name: _____

Telephone: _____

Email Address: _____

Address and/or Location Description: _____

Are you the property owner? Y/N _____

Work plan description (ie wall demolition in north bedroom, ceiling drop in kitchen)

_____
_____
_____
_____
_____

Planned Start Date:_____

Are you planning exterior excavations? If so Where? (Reminder that a UDIG still must be submitted before and excavation in order to locate utilities. UDIG can be reached by calling 811)

_____
_____
_____
_____
_____

***This form is used to evaluate your property for the presence of Libby Amphibole Asbestos (LA) that may be left sealed in place in buildings or at depth on the property. The Asbestos Resource Program (ARP) will evaluate the documentation of LA on your property and will respond within 3 working days of the status of your property. ARP may request to do drilling and scoping of walls before demolition.**

***If you have had an accidental spill of vermiculite insulation and need an emergency visit or if you have any questions please call 406-291-5335**

***This evaluation does NOT meet the requirement for asbestos inspection required by the state of Montana for demolition. The state regulation applies to contractors and facility owners. It generally does not apply to homeowners. More information on the requirements can be found here**

***If LA is found in the area where work is going to be conducted, ARP will provide resources on how to handle the material safely and may provide contracted support for removal/abatement.**

***This evaluation does NOT meet the requirement for asbestos inspection required by the state of Montana for demolition. The state regulation applies to contractors and facility owners. It generally does not apply to homeowners. More information on the requirements can be found here**

☐ **I understand that this evaluation form does not meet the requirements for asbestos inspection found in state code.**

APPENDIX F – SITE ASSESSMENT INFORMATION

(Under Development)

APPENDIX G – SAMPLING GUIDANCE

Operations and Maintenance Sampling Guidance

Operable Units 1, 2, 4, 5, 7 and 8
Lincoln County, Montana

Prepared for:

MONTANA DEPARTMENT OF ENVIRONMENTAL QUALITY
1225 Cedar St.
Helena, MT 59601

Prepared By:

WESTON SOLUTIONS, INC.
805 N. Last Chance Gulch
Helena, MT 59601
406-646-2401

May 2020

Table of Contents

List of Tables

List of Appendices

List of Acronyms

AHERA	Asbestos Hazard Emergency Response Act
ARM	Administrative Rules of Montana
ARP	Lincoln County Asbestos Resource Program
bgs	below ground surface
BMP	Best Management Practice
BOH	City-County Board of Health for Lincoln County
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
COC	Chain-of-Custody
CUA	Common Use Area
DEQ	Department of Environmental Quality
DI	Detailed Investigation
DQO	Data Quality Objectives
EDD	Electronic Data Deliverable
EPA	U.S. Environmental Protection Agency
ft^2	square feet
HEPA	High Efficiency Particulate Air
ID	Identifier
LA	Libby amphibole asbestos
L/min	Liter per minute
LUA	Limited Use Area
µm	Micron
MCE	Mixed Cellulose Ester

MT	Montana
N	Number
NCP	National Contingency Plan
NIOSH	National Institute of Occupational Safety and Health
NIST	National Institute of Standards and Technology
NPE	Negative-Pressure Enclosure
NUA	Non-use Area
NVLAP	National Voluntary Laboratory Accreditation Program
O&M	Operations and Maintenance
OU	Operable Unit
PB	Primary Building
PLM	Polarized Light Microscopy
PLM-VE	PLM Visual Area Estimation Method
PLM-GRAV	PLM Gravimetric Method
PM	Project Manager
PPE	Personal Protective Equipment
QA	Quality Assurance
QAM	Quality Assurance Manager
QC	Quality Control
RAL	Remedial Action Level
RM	Response Manager
ROD	Record of Decision
SB	Secondary Building
s/cc	Structures per centimeter
s/mm^2	Structures per square millimeter
SI	Screening Investigation
Site	Libby Asbestos Superfund Site
SOP	Standard Operating Procedure
SOW	Statement of Work
SS	Secondary Structure
SUA	Specific Use Area
TEM	Transmission Electron Microscopy
VCBM	Vermiculite-Containing Building Material

1 INTRODUCTION

This Operations and Maintenance (O&M) Sampling Guidance was developed by the Montana Department of Environmental Quality (DEQ) to outline the O&M sampling and analysis requirements for the Libby Asbestos Superfund Site (Site) for Operable Units (OUs) 1, 2, 4, 5, 7, and 8 to ensure that DEQ's environmental decisions during O&M are supported by data of known and documented quality. DEQ is the lead agency for Site activities during O&M, and their guidance is based on requirements for collecting environmental data established by the U.S. Environmental Protection Agency (EPA) for ensuring quality data. This guidance applies to activities performed by DEQ, the City-County Board of Health of Lincoln County (BOH) – Lincoln County Asbestos Resource Program (ARP), sampling personnel, as well as activities performed by others on behalf of DEQ.

This guidance only applies to Libby Amphibole asbestos (LA) investigation and confirmation sampling and analysis activities conducted during O&M for soils and building materials (e.g., log chinking, chimney mortar, plaster, or other building materials). There may be unique scenarios during O&M that may require a deviation from this guidance and a specific sampling and analysis plan may be developed.

This guidance details the roles and responsibilities of those who collect and use the analytical data for decision-making and provides a practical framework for managing the quality of activities, resulting in sound environmental determinations and controls.

1.1 Previous Sampling and Data Collection

Previous sampling conducted at the Site by EPA has demonstrated that a variety of media (e.g., soil, building materials) was contaminated with LA from source materials (e.g., vermiculite insulation, vermiculite-containing soils, mine wastes) at properties within OUs 1, 2, 4, 5, 7 and 8. EPA completed removal and remedial actions at a number of properties within the Site with known LA-contamination. However, not all source material was removed and some may still be present with varying levels of LA at individual properties. Materials left behind did not warrant removal during the original removal/remedial action as they did not pose an immediate health risk.

In developing this guidance, DEQ considered EPA documents regarding sampling and data collection since many of the processes are Site-specific. The following documents were considered:

- General Property Investigation Sampling and Analysis Plan/Quality Assurance Plan, Revision 9 (CDM Smith 2018a)
- Libby Standard Operating Procedures (various)
- Response Action Quality Assurance Project Plan, Revision 8 (CDM Smith 2018b)

1.2 Objectives

The primary goal of this sampling guidance is to provide data for the purposes of determining if additional actions are warranted during O&M at a given property. Although the majority of sampling activities were performed by EPA under the investigation, removal, and remedial programs, it is possible additional investigation sampling may be necessary under O&M to characterize property conditions at locations where properties were not characterized due to misses, limits of inspection, and/or property refusals. Here, additional characterization may be necessary due to land use or property use changes, or to evaluate soil borrow sources for use in the OUs identified above. In the event abatement activities

are deemed necessary under O&M, confirmation sampling may also be necessary to ensure the abatement goals are achieved.

2 PROJECT ORGANIZATION

The following sections summarize the entities and individuals that are responsible for providing project management, technical support, and quality assurance (QA) for this project.

2.1 Project Management

DEQ is the lead agency for Superfund activities during O&M. The DEQ Project Manager (PM) for the Libby Asbestos Project is Jason Rappe. DEQ will consult with EPA as provided for by Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), the National Contingency Plan (NCP), and applicable guidance in conducting Superfund O&M activities within the Site.

DEQ has entered into a Memorandum of Agreement (MOA) with the BOH. As described in the DEQ's and BOH's MOA, the ARP works under the direction of the BOH. The ARP Program Director is Virginia Kocieda.

2.2 Quality Assurance

DEQ will designate a Quality Assurance Manager (QAM) for this project who is independent of project technical staff and reports directly to the DEQ PM on QA matters. The QAM has the authority to objectively review all activities covered under this sampling guidance and identify problems, and to resolve any quality-related problems and/or authorize changes to the sampling guidance. The ARP will be responsible for providing oversight of sampling activities.

2.3 Field Sampling Activities

As described in the O&M Manual (WESTON 2020), the ARP is responsible for performing site assessments, developing a statement of work (SOW) that outlines the necessary samples required for a property in accordance with this guidance, and for providing oversight of sample data collection activities. Key sampling oversight personnel that are involved in this sampling program, include:

- Virginia Kocieda - Program Director
- Amanda Harcourt - Field Operations Manager
- Elzhon Anderson - Resource Coordinator

Prior to any sampling, the SOW is reviewed and approved by the DEQ PM or DEQ QAM.

The ARP will provide the DEQ-approved SOW to the third-party[1] who will be performing investigation sampling. All field sample collection activities will be conducted in accordance with the SOW as described in the O&M Manual (WESTON 2020). Consistency will be achieved to the extent possible through proper training, use of this guidance, and ARP oversight. It is the responsibility of the third-party to review and understand all applicable governing documents associated with this sampling program, including this guidance and the property-specific SOW. Samples are collected, processed, and sent to appropriate analytical laboratories as stated in this sampling guidance.

Additionally, for properties where abatement is conducted, clearance samples will be collected as outlined in the SOW.

[1] Third-party can be the property owner or a contractor hired by the property owner

2.4 Analytical Support

All samples collected as part of this sampling guidance are to be relinquished to a DEQ-approved laboratory for preparation and analysis for LA in accordance with the *Minimum Asbestos Laboratory Acceptance Criteria for Libby Asbestos Superfund Site* (**Appendix A**). DEQ will be responsible for coordinating the laboratory support for the Site.

2.5 Data Management

All data generated as part of this sampling effort is managed and maintained in DEQ's Libby Instance of Response Manager (RM). DEQ is responsible for the administration of all data management aspects of this project. The ARP is responsible for entering the data into RM.

3 SAMPLING RATIONALE

This sampling guidance provides a plan for collecting two (2) separate types of data during the O&M period: investigation sampling data and confirmation sampling data.

3.1 Investigation Sampling

Investigation samples are collected when no previous sample data is available and/or when conditions have changed such that previous sample data is not representative of the current site conditions. The primary goal of investigation sampling is to determine if LA is present in source materials at a level that would warrant O&M abatement actions. This is accomplished by sampling for LA in soil and building materials within interiors and/or exteriors of a property.

Investigation sampling is opportunistic sampling at individual properties within the Site where the degree of contamination is unknown because of the following scenarios:

- No previous sampling occurred (e.g., miss, limit of inspection, unforeseen condition, or refusal)
- Conditions have changed such that previous collected samples are not representative of current conditions at the property (i.e., change in frequency of use, land use change)
- Use of fill material that was not previously sampled

For misses, limits of inspection, or unforeseen condition scenarios, sampling was not performed by EPA prior to O&M to characterize the property. Misses are encounters with LA and/or LA-source materials in areas previously inspected by EPA that were not identified during their remedial action.

For refusal scenarios, the property owner did not allow access to complete characterization of the property during the Site remedial action performed by EPA.

The land use or frequency of use for an area may change during the O&M period. If the future use of an area changes such that it is used on a more frequent basis or is categorized as a more stringent land use category (e.g., industrial to residential), further investigations or sampling may be necessary to determine if contamination exists that could lead to exceedances of the Remedial Action Levels (RALs) associated with the new use. RALs are identified in the O&M Manual (WESTON 2020) and are based on the requirements in the Record of Decision (ROD) (EPA 2016). In instances where the frequency of use has or is anticipated to change, additional sampling will only occur in the specific area (i.e., the entire property will not be re-investigated).

Borrow sources that will be used for fill material from the removal of contaminated materials on the Site will be analyzed in accordance with the *Fill Material Quality Assurance Project Plan* (CDM Smith 2018c) and is not included in this guidance.

3.1.1 Sampling Locations and Variables

Investigation sampling may include exterior (e.g., yards, flowerbeds, driveways, etc.) and/or interior (i.e., buildings and structures) sampling. There is no set schedule for collection of these samples as they are opportunistic in nature, although may be based on seasonal factors. The required samples and sampling locations are highly variable and based on the complexity of the property being investigated, and will be defined in the property-specific SOW.

3.2 Confirmation Sampling

Confirmation sampling may be conducted at individual properties within the Site following abatement of LA-contaminated soil and/or LA-containing vermiculite or vermiculite insulation from a structure or building. The primary goal of confirmation sampling is to confirm that O&M abatement actions have met clearance criteria as identified in the O&M Manual (WESTON 2020) and the ROD (EPA 2016). This is accomplished using both visual inspections and collection of soil and/or air clearance samples.

3.2.1 Sampling Locations and Variables

Confirmation samples are collected in conjunction with abatement activities, which may be based on seasonal factors for exterior abatements or demolitions. The required number of samples and sampling locations are highly variable, based on the complexity of the abatement, and will be defined in the property-specific SOW.

3.2.2 Interior

If a property requires abatement of vermiculite or vermiculite insulation from an interior, clearance air samples may be collected following abatement activities to determine if contamination has been removed to meet clearance criteria as identified in the O&M Manual (WESTON 2020) and based on the requirements in the ROD (EPA 2016), as deemed necessary. Clearance air samples are collected from living spaces (e.g., living rooms, bedrooms, kitchens, dining rooms, bathrooms, finished attics, finished basements, etc.) and appropriate non-living spaces (e.g., unfinished attics, unfinished basements, attached garages, utility closets, etc.). Note that there may be instances where air clearance samples are unachievable (e.g., dirt floors, crawlspaces, etc.).

The location of clearance air samples is dependent upon the size, type, and dimensions of each building or structure requiring sample collection.

3.2.3 Exterior

If a property requires abatement of LA-contaminated soil, confirmation soil samples may be collected following abatement activities to determine if contaminated soils have been removed to Site-specific clearance criteria identified in the O&M Manual (WESTON 2020) and based on requirements in the ROD. If soils are removed, confirmation soil samples are collected from the floor of an excavation area during the soil removal phase of the abatement action.

4 SPECIAL TRAINING/CERTIFICATION

4.1 Field

LA is a hazardous substance that can increase the risk of cancer and result in serious non-cancer effects in people who are exposed by inhalation. Therefore, all individuals involved in the collection, packaging, and shipment of samples must ensure that sampling is conducted in accordance with their developed health and safety guidance document(s) and must maintain appropriate documentation of training by active field personnel.

Field investigation sample collection activities will be performed by a third-party in accordance with the SOW. Consistency will be achieved to the extent possible through proper training, use of this guidance, and ARP oversight. It is the responsibility of each third-party field sampling team member to review and understand all applicable governing documents associated with this sampling program.

The ARP is responsible for training all third parties on the sampling requirements, chain of custody (COC) protocols, and Site-specific guidance and Standard Operating Procedures (SOPs). A list of entities trained by ARP will be maintained and reviewed as part of oversight activities. At a minimum, ARP-provided training regarding LA-specific SOPs must be obtained. In addition, the third-party individuals should read and understand this guidance and follow all applicable worker safety programs (i.e. Occupational Safety and Health Administration (OSHA), etc.). It is the responsibility of the third-party to maintain training documentation. This documentation is to be made available upon request by ARP and/or DEQ.

For properties where abatement is conducted, ARP or the abatement contractor who performed the abatement work is responsible for conducting clearance samples as outlined in the abatement SOW. ARP personnel will be required, at a minimum, to complete OSHA 40-Hour Hazardous Waste Operations and Emergency Response (HAZWOPER) and relevant 8-hour refreshers, hold current HAZWOPER medical clearance certification (i.e. physician letter and respiratory protection training as required by 29 Code of Federal Regulations [CFR] 1910.134), and asbestos awareness training as required by 29 CFR 1910.1001.

4.2 Laboratory

All analytical laboratories participating in the analysis of LA samples during O&M are subject to national, local, and Site-specific certifications and requirements. To comply with those requirements, a laboratory may be accredited by the National Institute of Standards and Technology (NIST) and National Voluntary Laboratory Accreditation Program (NVLAP) for the analysis of asbestos by polarized light microscopy (PLM) and/or transmission electron microscopy (TEM). This includes the analysis of NIST/NVLAP standard reference materials, or other verified quantitative standards, and successful participation in two (2) proficiency rounds per year of airborne asbestos by TEM supplied by NIST/NVLAP. Other methods to demonstrate proficiency are verified and evaluated by the DEQ QAM, as appropriate.

Copies of recent proficiency examinations from NVLAP or an equivalent program, as well as certifications from other state and local agencies, are maintained by each participating analytical laboratory. 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 Site. These certifications require that each laboratory has all applicable state licenses and employs only qualified personnel. Copies of all proficiency examinations and certifications are maintained by the DEQ QAM.

DEQ reserves the right to conduct any laboratory audit or investigation deemed necessary to determine the ability of each laboratory to perform the work.

The *Minimum Asbestos Laboratory Acceptance Criteria for Libby Asbestos Superfund Site* is provided as **Appendix A**. Any laboratory utilized will adhere to these requirements.

It is the responsibility of the laboratory to follow their health and safety policies and regulations. All sample handling and preparation activities (drying, splitting, sieving, grinding, etc.) must be performed in an area fitted with a negative pressure, ventilated hood with an operating High Efficiency Particulate Air (HEPA) filtration system. Appropriate personal protective equipment (PPE) must always be worn during handling.

5 SAMPLING PROCESS

This section summarizes field activities that will be performed by a third-party in support of investigation and confirmation sampling.

5.1 Pre-Sampling Activities

Prior to beginning field sampling activities, the ARP will provide the DEQ-approved SOW that will define the following:

- Objectives and scope of the fieldwork
- Number, types, and location of samples to be collected
- Documentation requirements
- Supplies and equipment
- Best Management Practices (BMPs), as necessary
- Reimbursement determination

Third-party samplers will perform the following activities before field activities, as applicable:

- Ensure minimum training requirements defined in Section 4
- Review and understand applicable governing documents
- Obtain required sample containers and other supplies
- Obtain and maintain PPE in accordance with their health and safety guidance document(s)

The specific supplies and equipment required for sampling are dependent on the sample media. Third-party sampling staff will be responsible for ensuring supplies and equipment are readily available for field use. At a minimum, the following supplies are required for any sampling activities conducted under this guidance:

- Field logbook
- Indelible ink pens
- Digital camera
- COC and sample labels
- Plastic zipper-top bags
- Hand-held water sprayer
- Demineralized water (as sold in grocery stores)
- Paper towels
- Shipping materials (e.g., small cooler/box, packing tape, shipping labels, etc.)
- PPE, as required

5.2 Investigation Sampling Process

Standardized investigation sampling procedures for soils and building materials will be employed and are described below. For exterior investigation sampling, soil samples will be collected to fully characterize the property and results of the investigation sampling will be evaluated to determine whether abatement activities are required. In most cases, detailed investigation (DI) sampling will occur; however, in some cases, a screening investigation (SI) may be employed, dependent on the protocol defined in the SOW (**Table 1**). The level of investigation sampling will be determined on a case-by-case

basis. Regardless of the sampling protocol (i.e., DI or SI), all soil samples are 30-point composites and area(s) where vermiculite is observed will be segregated and sampled separately.

Table 1. Soil Sampling Protocol

Screening Investigation	
Area Type	**Maximum Area per Sample**
SUA[1] (flowerbed, garden, play area, etc.)	1 acre (43,560 ft^2)
SUA[1] (driveway)	1 acre (43,560 ft^2)
CUA[1] (yard, etc.)	1 acre (43,560 ft^2)
LUA[1] (field, pasture, etc.)	5 acres (217,800 ft^2)
PB (crawlspace, cellar, etc.)	Use area
SB[2] (shed, garage, barn, pumphouse, etc.)	Use area
SS[3] (carport, lean-to, etc.)	Use area
NUA[4] (wooded area, etc.)	No Sampling
Detailed Investigation	
Area Type	**Maximum Area per Sample**
SUA[1] (flowerbed, garden, play area, etc.)	1,000 ft^2
SUA[1] (driveway)	6,000 ft^2
CUA[1] (yard, etc.)	3,000 ft^2
LUA[1] (field, pasture, etc.)	5 acres (217,800 ft^2)
PB (crawlspace, cellar, etc.)	Use area
SB[2] (shed, garage, barn, pumphouse, etc.)	Use area
SS[3] (carport, lean-to, etc.)	Use area
NUA[4] (wooded area, etc.)	No Sampling

[1] Multiple SUAs, 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 can be combined (e.g., flowerbeds may only be combined with other flowerbeds).

[2] Secondary buildings (four walls and a roof) 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).

[3] Secondary structures (open on at least one side and/or mobile) may be combined with the surrounding area if the material is the same throughout (e.g., a carport and driveway can be sampled together if the same material is present in both areas).

[4] If inspection 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
ft^2 – square feet

If a screening investigation is performed and the results of the investigation indicate an exterior trigger is present (**Table 2**), the property will require more detailed sampling to determine the extent of contamination.

Table 2. Exterior Triggers for Screening Investigation

Maximum PLM Condition Present	SUAs, CUA, SBs, and SSs	LUAs
≥0.2% LA	Perform DI	If area ≤15,000 ft^2, perform DI If area >15,000 ft^2, delineate into areas <15,000 ft^2 and perform DI
<0.2% LA (trace)	If area ≥25% trace LA, perform DI	No further action

Investigation Soil Sampling Method (Exterior)

For exterior sampling, sample maps will be developed to show the general layout of the property, the locations of soil samples (if applicable), the locations of bulk samples (if applicable), and locations of observed vermiculite. Sample maps can be hand-drawn or based on an aerial image, however, should be of sufficient scale to be easily read and capture necessary details. An example sample map is provided in **Appendix B**.

Soil samples will be collected using one-gallon, zip-top plastic bags as the primary container. Sample bags will be marked with a unique identifier number (discussed in **Section 5.4**) that identifies the sample and corresponds to a marked-up area on the sample map. The unique soil sample identifier (ID) is specified in the SOW and listed on the COC provided by ARP/DEQ. Sample bags and the sample map should be labeled upon the collection of each sample to avoid labeling errors.

Each soil sample is a 30-point composite of soil subsamples or aliquots of approximately equal size for a final volume of approximately one third of a one-gallon, zipper-top plastic bag. Samples should be collected in "like" areas. Non-contiguous areas (such as separate flowerbeds) can be combined into one (1) soil sample, in consideration of vermiculite observed. For example, if there are six (6) flowerbeds sampled together as one (1) soil sample, the six (6) flowerbeds would have five (5) aliquots taken from each of the flowerbeds to combine for 30 aliquots in the soil sample. Sampling of non-contiguous areas should be "like with like" (i.e., flowerbeds can be combined, but a flowerbed should not be combined with a driveway or garden).

As each aliquot is collected, the soil will be inspected for vermiculite. Refer to the photographs of visible vermiculite in soil as provided in **Appendix C**. Areas with vermiculite should be sampled separately from areas that do not contain vermiculite. This will help avoid potentially biasing a soil sample result either high or low. For example, a flowerbed with vermiculite should not be sampled with a separate flowerbed that does not contain vermiculite. For larger contiguous areas, such as a yard, a section with vermiculite should not be sampled with a section that does not contain vermiculite.

To collect a soil sample, put on a pair of disposable gloves and collect the 30 aliquots that make up the soil sample. If sampling multiple samples (e.g., one (1) garden sample and one (1) flowerbed sample, each with 30 subsamples), put on a new pair of gloves for each sample collected to mitigate the potential for cross contamination. If conditions are dry and indicate sampling may generate dust, use a hand-held sprayer filled with demineralized water to wet each aliquot location prior to sampling and stand upwind of the sample area. For flowerbeds and gardens, use the trowel or shovel to dig to 6 inches below ground surface (bgs). For all other types of use areas, use the trowel or shovel to dig to 3

inches bgs. If vermiculite is observed, record the information on the sampling map. Vegetative material such as grass or weeds, rocks, and miscellaneous items such as nails or screws should be removed by hand from the aliquot before placing it into a one-gallon, zip-top plastic bag. Any excess soil from where the aliquot was taken should be placed back into the hole and tamped down lightly. Repeat this step for each subsequent aliquot until 30 aliquots have been collected. The soil sample from that area is then complete, and the unique sample ID must be affixed or written on the bag. The bag is then placed inside a second bag, with the same unique sample ID (listed in the SOW and COC provided by DEQ/ARP). This "double bagging" helps reduce potential spills of soil from the inner bag. Do not shake the sample to mix the aliquots—the laboratory will mix the sample as part of their processes.

Investigation Soil Sampling Method (Interior)

If vermiculite additives are observed in soils (e.g., soil floor of understructure), only one (1) soil sample will be collected per understructure, except for the following examples, which may require additional sampling:

- Multiple soil types are present in the understructure
- Significant elevation differences exist (e.g., understructure is part basement and part crawlspace)
- The understructure is physically separated into several smaller areas (e.g., the crawlspace for the original house and the addition are separated by a foundation wall)

To collect an interior soil sample, follow the same 30-point composite protocol as defined above for exterior soils samples.

Investigation Building Material Sampling Method (Exterior and Interior)

Building material samples from the interior or exterior may be collected for LA analysis from a variety of sources (e.g., log chinking, chimney mortar, plaster, or other building material) where vermiculite additives are visually identified. Vermiculite insulation will not be sampled, as it is assumed to contain LA and will be removed and/or managed accordingly. If vermiculite additives are observed in building materials (e.g., log chinking, chimney mortar, plaster, or other building material), those materials will be evaluated for friability (i.e., ability to be pulverized by hand). If the material is friable or deteriorated, or there are plans to demolish or remodel buildings or areas of buildings with vermiculite-containing building materials (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*. Friable vermiculite-containing building material will be sampled, from each homogenous material, as follows:

- Three (3) samples from each homogeneous material that is 1,000 square feet (ft^2) or less in area
- Five (5) samples from each homogeneous material that is greater than 1,000 ft^2 but less than or equal to 5,000 ft^2 in area
- Seven (7) samples from each homogeneous material that is greater than 5,000 ft^2 in area

Individual building material samples will be collected in plastic zipper-top bags, double-bagged, and labeled with a unique sample ID.

5.3 Confirmation Sampling Process

Following abatement activities, samples may be collected to confirm that abatement criteria was achieved. If building materials are removed air clearance samples will be collected. If soils are removed from the exterior, soil confirmation samples will be collected.

Interior Air Clearance

Based on the concept of physical accessibility, the evaluation of contaminated building materials within primary structures was divided into living and non-living spaces. Living spaces are areas that are generally occupied for long periods of time such as living rooms, bedrooms, kitchens, dining rooms, bathrooms, finished attics, finished basements, and offices. Non-living spaces are areas that are generally occupied infrequently or for short durations such as unfinished attics, unfinished basements, attached garages, and utility closets (CDM Smith 2017).

Prior to collecting air clearance samples, ARP will determine whether the area to be sampled is considered a "living space" or a "non-living" space in order to compare data collected to the action levels specified for the two (2) different areas. In cases where a "non-living space" shares air space with a "living space" area and is included within the same negative-pressure enclosure (NPE), the area must meet the Site-specific action level for a "living space" (CDM Smith 2018c).

Following abatement of vermiculite or vermiculite insulation from an interior, five (5) clearance samples will be collected in each containment area where abatement was performed. Each clearance air sample will be collected in accordance with TEM Asbestos Hazard Emergency Response Act (AHERA) sampling guidance (EPA, 1987) (Appendix A), with modifications, which is included in **Appendix D**. The general sampling steps are as follows:

1. Sampling for airborne LA following an abatement action must use commercially available cassettes. **Modification: 0.8-micron (µm) mixed cellulose ester (MCE) filter air sampling cassettes will be used in place of MCE cassettes having a pore size less than or equal to 0.45 µm.**
2. Prescreen the loaded air sampling cassette collection filters to assure that they do not contain concentrations of LA which may interfere with the analysis of the sample. A filter blank average of less than 18 structures per square millimeter (s/mm^2) in an area of 0.057 mm^2 (nominally ten 200-mesh grid openings) and a single preparation with a maximum of 53 s/mm^2 for that same area is acceptable for this method.
3. Use sample collection filters which are MCE having a pore size of 0.8 µm.
4. Place these filters in series with a 5.0 µm backup filter (to serve as a diffuser) and a support pad. See Figure 1 of **Appendix D**.
5. Reloading of used cassettes is not permitted.
6. Orient the cassette downward at approximately 45 degrees from the horizontal.
7. Maintain a log of all pertinent sampling information.
8. Calibrate sampling pumps and their flow indicators over the range of their intended use with a recognized standard. Assemble the sampling system with a representative filter (not the filter which will be used in sampling) before and after the sampling operation. Record all calibration information.

9. Ensure that the mechanical vibrations from the pump will be minimized to prevent transfer of vibration to the cassette.
10. Ensure that a continuous smooth flow of negative pressure is delivered by the pump by damping out any pump action fluctuations if necessary.
11. Ensure the final plastic barrier around the abatement area remains in place for the sampling period.
12. Use aggressive sampling conditions to dislodge any remaining dust.
13. Select an appropriate flow rate equal to or greater than 1 liter per minute (L/min) or less than 10 L/min for 25 mm cassettes. Larger filters may be operated at proportionally higher flow rates.
14. **Modification: A minimum of seven (7) samples are to be collected for each testing site consisting of the following:**
    a. Five (5) samples per abatement area.
    b. Two (2) field blanks taken by removing the cap for not more than 30 seconds and replacing it at the time of sampling before sampling is initiated in the abatement area.
15. Perform a leak check of the sampling system at each indoor sampling site by activating the pump with the closed sampling cassette in line. Any flow indicates a leak which must be eliminated before initiating the sampling operation.
16. Table I of **Appendix D** specifies the air volume sampling ranges to be used.
17. Ensure that the sampler is turned upright before interrupting the pump flow.

The individual cassettes will be labeled with a unique sample ID (listed in the SOW and COC provided by DEQ/ARP) and placed in a one-gallon plastic zip-top bag that is also labeled with the unique sample ID, affixed or written on the bag.

Exterior Soil Confirmation

Following the excavation of contaminated soils within the abatement area, a confirmation soil sample will be collected to determine if abatement goals have been achieved. At least one (1) composite sample will be collected per maximum of 2,500 ft^2 of excavation area. If an area is excavated to a maximum depth of 3 feet, the final confirmation soil sample will be collected and analyzed for informational purposes only since the excavation will not extend deeper than 3 feet. Sampling for areas that can't be excavated to design depth (e.g., adjacent to trees, curbs, foundations, sidewalks, etc.) are also for documentation purposes.

To collect a soil sample, put on a pair of disposable gloves and collect the 30 aliquots that make up the soil sample. If conditions are dry and indicate sampling may generate dust, use a hand-held sprayer filled with demineralized water to wet each aliquot location prior to sampling and stand upwind of the sample area. Individual confirmation soil samples may include composite points from different use areas (e.g., yard and flowerbed, yard and garden) if all areas have been excavated to design depth and pass visual inspection. Each sample will be collected from 0 to 2 inches bgs of the completed excavation, from both the bottom and sidewalls, and consist of nearly equal portions of soil from 30 locations within the delineated sample area. Soils will be collected with a trowel or shovel and should fill at least approximately one third of a one-gallon, zip-top plastic bag. Once the individual soil samples have been collected, the unique sample must be affixed or written on the bag. The bag is then placed inside a

second bag, with the same unique sample ID (listed in the SOW and COC provided by ARP). This "double bagging" helps reduce potential spills of soil from the inner bag.

5.4 Sample Label and Identification

A unique sample ID will identify each sample collected during investigation or confirmation sampling events. This provides a tracking record to allow retrieval of information about a particular sample and to ensure that each sample is uniquely identified. Sample IDs are defined in the SOW and documented on the COC. Preprinted adhesive sample ID labels will be provided to sampling personnel by ARP to control the samples collected, to prevent duplication in assigning sample IDs, and to prevent transcription errors in the documentation process. The labels will be affixed to both the sample cassette and sample bag for air samples, and both the inner and outer sample bags for soil samples.

The sample labeling scheme is as follows:

AD/BD Number - 0000

Where:

AD/BD represents numbers assigned to the property and/or building and **0000** represents a unique, 4-digit number assigned by ARP (e.g., BD005584-0001).

5.5 Sample Handling and Custody

Following collection, store samples in a safe location where they are protected from tampering, damage, contamination, or loss prior to shipping to the analytical laboratory. There is no hold time for samples for LA analysis, but it is prudent to ship samples as soon after collection as reasonable.

The COC form provided by ARP contains the appropriate analysis(es), sample information (e.g., sample ID, analytical methods, etc.), and billing information. If there are any deviations, please note those on the COC. When ready to ship, complete the sample collection date and time, then sign and date that you are relinquishing the samples and include the COC in a plastic zipper-top bag in the shipping container.

Place samples in a sturdy shipping container, such as a cardboard box or standard beverage cooler. Samples for LA analysis are not required to be maintained at a specific temperature. Containers should be just slightly larger than the volume of the samples to prevent jostling and should not be over-packed, which can compromise the container and seals. Bulk samples must be shipped in a separate container from soil samples. If a small amount of packing materials is needed to secure samples in the container, use bubble wrap. Seal the container closed with packing tape. A good practice is to add a custody seal to the outside and make several full passes around the container with the tape to ensure if dropped, the container and samples will remain intact. Samples will be shipped using the approved shipping provider.

5.6 Field Documentation

Documentation of field activities under the SOW will include sample maps, photos, and field logbooks as needed. Third-party samplers will provide field documentation to ARP electronically following investigation sampling. In addition, ARP collected field information during oversight activities will be included in RM.

5.6.1 Sample Location Maps

As described in **Section 5.2**, sample maps are used for exterior soil sampling and intended to show the general layout of the property, the locations and sample areas (in ft^2) of soil samples. Maps may also be used to show the locations and areas (in ft^2) of bulk samples (if applicable) and location and extent of vermiculite insulation in buildings. Sample maps can be hand-drawn, based on an aerial image, or created from property plans; however, maps should be of sufficient scale to be easily read and capture necessary details. An example sample map is provided in **Appendix B**.

5.6.2 Photos

Photos are taken to document site conditions at the time of the investigation or confirmation sampling. Exterior photos should be medium- and wide-angle shots of sampled site features such as yard, flowerbeds, etc., with close-ups of the sampled site features. Interior photos should be medium- and wide-angle shots of sampled site features.

5.6.3 Field Logbook

A field logbook should be used to document equipment and calibration information (as needed), field conditions, log of the photos taken, and any other relevant information about field sampling activities.

5.7 Field Decontamination

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 after use.

Potable water should be used to thoroughly rinse off soil sampling equipment (shovel, trowel) after each soil sample has been collected to minimize the potential for cross contamination during soil sample collection. Decontamination water will not be captured but will be discharged to the ground in the vicinity of the sample area.

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

1. Scrub the outside of the pump and other wetted parts with a brush.
2. The pump shall be set up in the same configuration as for sampling. Submerge the pump intake and all downhole-wetted parts (tubing, piping, foot valve) in a container of soapy water, collecting the discharge in a waste container. Pump soapy water through the pump assembly.
3. Immerse the pump intake in a container of potable water container while leaving the discharge outlet in the waste container. Pump potable water through the pump assembly until it runs clear, collecting the discharge in a waste container.
4. Allow the equipment to dry.
5. Decontamination water collected in the waste container will be discharged to the ground in the vicinity of the sample.

5.8 Field Equipment Maintenance

Air sampling pump calibrations will be conducted and documented in accordance with manufacturer maintenance and calibration procedures, which will be included with the pump. Record maintenance and calibration actions in the equipment log or field. If the pump cannot be calibrated or adjusted to

perform accurately, the user should discontinue use and contact ARP to determine appropriate resolution.

6 ANALYTICAL METHODS/LABORATORY OPERATIONS

The DEQ will coordinate with an approved lab(s) so that all eligible laboratory analytical costs are paid directly by DEQ. The prepared COC will contain pertinent sample and analysis information including sample IDs, analysis methods, and any special instructions. The third-party sampler is responsible for retaining a copy of all records and shipping all samples to the DEQ-approved laboratory. Once samples are received at the lab, DEQ is responsible for all eligible costs of sample analyses. Any issues identified by the laboratory will be communicated to DEQ's QAM.

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

The analytical requirements summary sheet (**Appendix D**) describing the preparation method, analysis method and laboratory modifications by medium will be provided to participating laboratories in this sampling program prior to any sample handling.

6.1 Investigation Sample Analysis

The COC will have the requested turn-around times for all relinquished samples. In general, it is expected that investigation soil analysis, including soil preparation will be completed within 20 (business) days from the time the laboratory receives them. Analysis of investigation building material samples by PLM-9002, and PLM-PC400 if needed, will be completed within 10 (business) days from the time the laboratory receives them. Adjustments to the standard turnaround times will be made with approval from DEQ. If the analytical laboratory determines the results will not be delivered within the requested turnaround time, the analytical laboratory is responsible for contacting DEQ.

For investigation samples, no hold time requirements apply.

6.1.1 Soil Sample Analysis

Soil Sample Preparation

Prior to analysis, all soil samples require processing by the analytical laboratory. Soil samples received at the laboratory are dried in a laboratory oven, and the 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 microscopic slides for PLM analysis. It is the responsibility of the laboratory preparing soil samples to specify the appropriate PLM method as it corresponds to the specific sample fraction being submitted (i.e., fine ground or coarse fraction) prior to analysis.

Analysis

All soil samples collected as part of this effort, including field duplicate samples, will be analyzed for LA by PLM-Visual Estimation (VE) and PLM-Gravimetric (Grav) in accordance with EPA SOPs *Analysis of Asbestos Fibers in Fine Soil by Polarized Light Microscopy* and *Qualitative Estimation of Asbestos in Coarse Soil by Visual Examination Using Stereomicroscopy and Polarized Light Microscopy* and *Analysis of Asbestos Fibers in Fine Soil by Polarized Light Microscopy*, respectively as well as in the most recent

versions of Libby laboratory modifications (LB-000073, LB-000088, LB-000097, LB-000098, and LB-000103), which are provided in **Appendix E**.

6.1.2 Building Material Analysis

Building material samples will be analyzed by PLM by National Institute of Occupational Safety and Health (NIOSH) 9002, Issue 2, Asbestos (bulk) by PLM, with subsequent analysis using point counting and examining 400 points in accordance with EPA/600/R-93/116 (PLM-PC400) if initial results are reported as less than (<) 1 percent LA.

6.2 Confirmation Sample Analysis

The standard turnaround time for confirmation sample results is one (1) day unless otherwise requested and noted on the COC form. When possible, the turnaround time may be extended in order to keep laboratory costs down.

6.2.1 Air Clearance Analysis

Clearance air samples will be analyzed by the TEM AHERA method in accordance with 40 CFR Chapter 1, Part 763, Subpart E, Appendix A, *Interim Transmission Electron Microscopy Analytical Methods – Mandatory and Non-mandatory – and Mandatory Section to Determine Completion of Response Actions*. All Site-specific laboratory modifications to the TEM AHERA method will be applied.

The laboratory will achieve the method analytical sensitivity of 0.005 structures per cubic centimeter (s/cc) using direct sample preparation techniques, with a minimum volume of 1200 L of air/sample.

6.2.2 Confirmation Soil Samples

Soil Sample Preparation

Prior to analysis, all soil samples require processing by the analytical laboratory. Soil samples received at the laboratory are dried in a laboratory oven, and the 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 microscopic slides for PLM analysis. It is the responsibility of the laboratory preparing soil samples to specify the appropriate PLM method as it corresponds to the specific sample fraction being submitted (i.e., fine ground or coarse fraction) prior to analysis.

Analysis

All soil samples collected as part of this effort, including field duplicate samples, will be analyzed for LA by PLM-Visual Estimation (VE) and PLM-Gravimetric (Grav) in accordance with EPA SOPs *Analysis of Asbestos Fibers in Fine Soil by Polarized Light Microscopy* and *Qualitative Estimation of Asbestos in Coarse Soil by Visual Examination Using Stereomicroscopy and Polarized Light Microscopy* and *Analysis of Asbestos Fibers in Fine Soil by Polarized Light Microscopy*, respectively as well as in the most recent versions of Libby laboratory modifications (LB-000073, LB-000088, LB-000097, LB-000098, and LB-000103), which are provided in **Appendix E**.

6.3 Laboratory Quality Assurance/Quality Control (QA/QC)

Each laboratory operates under a QA program. It is the responsibility of the laboratory to maintain a documented QA program manual, or equivalent, that details the laboratory's QA program. The overall

laboratory QA program consists of laboratory certifications, training, quality documents, laboratory audits, and external performance evaluation programs. Laboratories that analyze field samples must maintain required certifications and must satisfactorily complete internal training requirements to ensure that proper QA/QC practices are conducted during sample analysis.

Samples collected under this guidance will be analyzed in accordance with nationally recognized analytical procedures (i.e., Good Laboratory Practices) in order to provide analytical data of known quality and consistency. The lab must meet established criteria for accuracy, sensitivity, bias, and precision and that they comply with specified data quality needs or requirements according to the methods.

The laboratory will notify DEQ if there are non-conformances associated with the analysis methods that may impact the data quality. Other nonconformance issues, such as those found during performance evaluations or audits, will be addressed on a case-by-case basis by DEQ's QAM.

6.4 Laboratory Documentation and Reporting

Sample results will meet DEQ's data requirements and will be delivered electronically to DEQ and ARP in the appropriate electronic data deliverable (EDD) format. Any deviations to the methods will be documented in a narrative summary, which is included in the report. The lab report will be uploaded into RM in the appropriate property file.

6.5 Sample Disposal

All samples and grids (sample mounting structure for aiding in TEM examination) will be maintained in storage at the analytical laboratory unless otherwise directed by DEQ. The laboratory will be responsible for proper disposal of any remaining samples, sample containers, shipping containers, and packing materials in accordance with sound environmental practice, based on the sample analytical results. The laboratory will maintain proper records of waste disposal methods and will have disposal company contracts on file for inspection, as necessary.

7 DATA QUALITY OBJECTIVES AND CRITERIA

The primary goal of this sampling guidance is to provide data for the purposes of determining if additional actions are warranted during O&M at a given property. Data quality objectives (DQO) are established to identify the factors that affect the quality and usefulness of the data, thus impacting the decisions made based on those data. The DQO process was applied to define the type, quantity, purpose, and use of data to be collected to help ensure that data collected is adequate to support decision-making during O&M (**Appendix F**).

Not all decisions based on environmental data require the same degree of certainty; therefore, data is collected on a graded approach. This means that the level and rigor of data and quality required is based on the importance of decisions to be made.

7.1 Performance Criteria

In order to support the decision, the performance criteria for the data quality indicators (i.e., precision, bias, and sensitivity) has been defined and described below.

7.1.1 Precision

Soil and Bulk Material Sampling

For soil samples, a field duplicates for soil sampling will be collected at a rate of one (1) per 20 field samples collected. Individual composite points for the soil duplicate samples will be collected from different locations within the same use area as the original field sample. Analysis of these field duplicates will provide a measure of the precision of the sampling and analysis process. Bulk material duplicates will be collected from the same homogenous material and general location as the field sample (CDM Smith 2018a).

There are no acceptance criteria established for soil or bulk material field duplicates. Rather results are used to determine the magnitude of sampling variability to evaluate data usability. In general, if the discordance rate for field duplicate samples is greater than 20% for an investigation, the data usability assessment should alert users to this inherent variability (CDM Smith 2018a).

Laboratory QC analyses will provide information on analysis reproducibility and precision. Laboratory QC analyses consist of laboratory duplicates, and analysis of standard reference materials for PLM.

Air Clearance Sampling

For TEM analyses, the precision of asbestos measurements is determined mainly by the number (N) of asbestos structures counted in each sample. In general, when good precision is needed, it is desirable to count a minimum of 3 to 10 structures per sample, with counts of 20 to 25 structures per sample being optimal. Stopping rules have been established so that analyses may stop at 25 LA structures, otherwise the target sensitivity should be reached if the filter area stopping rule has not been met.

Laboratory QC analyses will provide information on analysis reproducibility and precision. Laboratory QC analyses consist of recount and re-preparation analyses for TEM analysis.

7.1.2 Bias/Accuracy and Representativeness

To the extent feasible, samples will be collected and analyzed in accordance with the procedures set forth in this sampling guidance, which are consistent with the previous sampling efforts conducted at

the Site by EPA. This will ensure that results are representative and appropriate for comparison to other data sets from historical and future data collection efforts.

7.1.3 Comparability

The data generated under this sampling guidance will be obtained using sample collection, preparation, and analysis methods used previously at the Site. The use of consistent methods and development of SOWs by ARP will yield data that are comparable to previous data, allowing for comparison to other historic and future data collected at the Site.

7.1.4 Method Sensitivity

For air clearance sampling by TEM, the laboratory will attempt to achieve the method analytical sensitivity of 0.005 s/cc using direct sample preparation techniques. The method sensitivity (analytical sensitivity) is not applicable for the analysis of LA by PLM because the method level of detection is estimated (at <1 percent LA).

8 ASSESSMENTS AND OVERSIGHT

To ensure consistency and adherence to the SOW, ARP will provide oversight to the third-party sampling activities.

The DEQ or designee may conduct a field surveillance for sampling activities detailed in this sampling guidance to assess the overall QA/QC program. During the surveillance, the assessor will examine activities and documentation to assess whether activities are conducted in conformance with the procedures and QA/QC requirements stated in this sampling guidance, and any other relevant governing documents. During the surveillance, the following field activities will be assessed: visual vermiculite inspections; sample collection, handling, shipping; and field documentation. A copy of the surveillance report will be made available to the EPA upon request.

Performance and system assessments/audits of the analytical laboratory may be conducted at each analytical laboratory as deemed appropriate by DEQ. Results of audits will be delivered to EPA upon request.

The result of the field assessment may warrant DEQ to implement improvements in the sampling process and address quality issues. Corrective response actions will be implemented on a case-by-case basis to address quality problems. Minor actions taken to immediately correct a quality problem will be documented via logbook and reported to the DEQ PM or QAM. Major corrective actions (i.e., those that impact or have the potential to impact O&M sampling objectives) will be approved by DEQ prior to implementation. The DEQ PM or QAM will be notified when quality problems arise that cannot be corrected quickly through routine procedures. Any changes to this guidance must be made by DEQ.

No regularly scheduled written reports are planned as part of this sampling guidance. However, QA reports may be provided for routine audits and whenever significant quality problems are encountered.

9 DATA REVIEW, VERIFICATION, AND VALIDATION

Key components to assessing the data collected as part of this guidance are data review, data verification, and data validation. This section outlines the general processes involved with each component.

9.1 Data Review

Data review of field sample activities will be conducted by the third-party sampling team and verified by ARP. Review will typically include ensuring proper labeling and sample handling and cross-checking that the sample IDs and sample dates have been reported correctly on the COC, noting any deviations from the SOW. Field notes with the deviations will be provided to ARP.

9.2 Data Verification

Libby-specific EDD spreadsheets will be used by the laboratory, which eliminates the hand-entering of data and includes automated QC checks that perform initial data checking of the reported analytical results. In addition to these automated checks, more detailed manual data verification efforts will be performed on an as needed basis.

ARP provides data verification of sampling prior to uploading the EDD into RM. This data verification includes cross-checking that sample IDs and sample dates have been reported correctly, and that analytical methods and required analytical sensitivities align with the sampling requirements identified in guidance, SOPs, and the SOW. The goal of data verification is to identify and correct data reporting errors. Performing regular data verification reviews will ensure that any potential data reporting issues are quickly identified and rectified to limit any impact on overall data quality. If discrepancies are found, ARP will contact the DEQ PM or QAM who will then notify the appropriate entity (field or laboratory) in order to correct the issue.

9.3 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 potential data quality issues. Data validation is performed by DEQ or their designee. As part of the data validation effort, DEQ reviews laboratory QC analyses results, as deemed necessary. DEQ may assess other information associated with sampling and analysis efforts to identify potential data issues and evaluate the data quality.

9.4 Data Usability

It is the responsibility of DEQ to evaluate data to ensure that the data objectives and criteria have been met, and reported results are adequate and appropriate for their intended use based on the results of the data verification and data validation efforts. Data verification and validation reviews may also be performed by technical support staff familiar with Libby-specific data reporting, analytical methods, and investigation requirements.

The data usability assessment should evaluate results against data quality indicators, including precision, accuracy/bias, representativeness, comparability, and whether specified analytic requirements (e.g., sensitivity) were achieved. Non-attainment of project requirements may result in additional sample collection or field observations (if possible) or additional analysis in order to achieve project needs.

10 References

CDM Smith 2018a. General Property Investigation Sampling and Analysis Plan/Quality Assurance Project Plan, Libby Asbestos Site, Operable Unit 4, Libby, Montana, Revision 9. March 23, 2018.

CDM Smith 2018b. Response Action Quality Assurance Project Plan, Libby Asbestos Superfund Site, Operable Unit 4 and 7, Libby, Montana. Revision 8. April 1, 2018.

CDM Smith 2018c.Fill Material Quality Assurance Project Plan, Libby Asbestos Superfund Site, Libby, Montana, Revision 5. April 1, 2018.

EPA 1987. 40 CFR 763, Appendix A to Subpart E of Part 763, Interim Transmission Electron Microscopy Analytical Methods – Mandatory and Nonmandatory – and Mandatory Section to Determine Completion of Response Actions. EPA, 1987.

EPA 2016. Record of Decision for Libby Asbestos Superfund Site, Libby and Troy Residential and Commercial Properties, Parks and Schools, Transportation Corridors, Industrial Park. Operable Units 4–8, Lincoln County, Montana.

WESTON 2020. Operations and Maintenance Manual, Libby Asbestos Superfund Site. Lincoln County, Montana. May 2020.

APPENDIX A – MINIMUM ASBESTOS LABORATORY ACCEPTANCE CRITERIA FOR LIBBY ASBESTOS SUPERFUND SITE

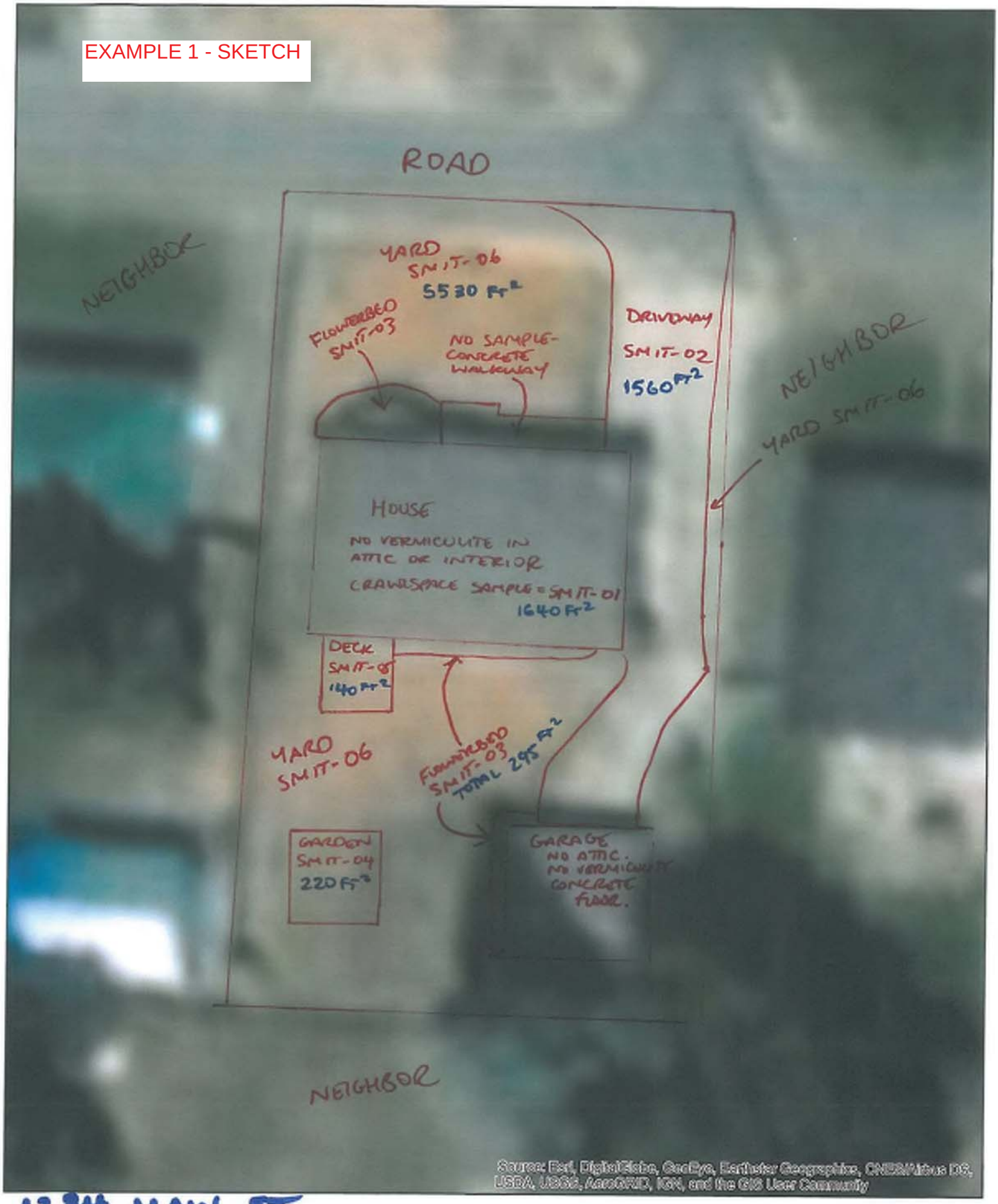
Minimum Asbestos Laboratory Acceptance Criteria for Libby Asbestos Superfund Site

1. Must be certified by the National Institute of Standards and Technology (NIST) National Voluntary Laboratory Accreditation Program (NVLAP) for the analysis of asbestos by PLM[1] and/or TEM[2] and provide performance evaluation samples to demonstrate proficiency.
2. Must have a laboratory-specific Quality Management Plan and all relevant SOPs in place for asbestos environmental sample processing and analysis.
3. Must have a minimum of 2 experienced analysts capable of running PLM Visual area estimation methods and/or TEM Methods, with documentation in place demonstrating all analysts work experience and training related to analyses performed.
4. Must be familiar with standard TEM and PLM preparation methods. TEM laboratories must have the ability to perform under indirect preparation for ashing. PLM must have the ability to dry samples and the ability to sieve and grind soil samples in accordance with the Libby-specific preparation method.
5. TEM laboratories must have Energy Dispersive Spectroscopy (EDS) and Selected Area Electron Diffraction (SAED) capability incorporated into their microscopes.
6. Must have the capacity to meet the required turnaround-times.

[1] NIST Handbook 150-3, NVLAP Bulk Asbestos Analysis (2006 Edition)
[2] NIST Handbook 150-13, NVLAP Airborne Asbestos Analysis (2006 Edition)

APPENDIX B – SAMPLING MAP EXAMPLES

EXAMPLE 1 - SKETCH



1234 MAIN ST
OWNER: SMITH
SAMPLING DATE: 06/20/19

0 5 10 20 30 40 Feet

1:240

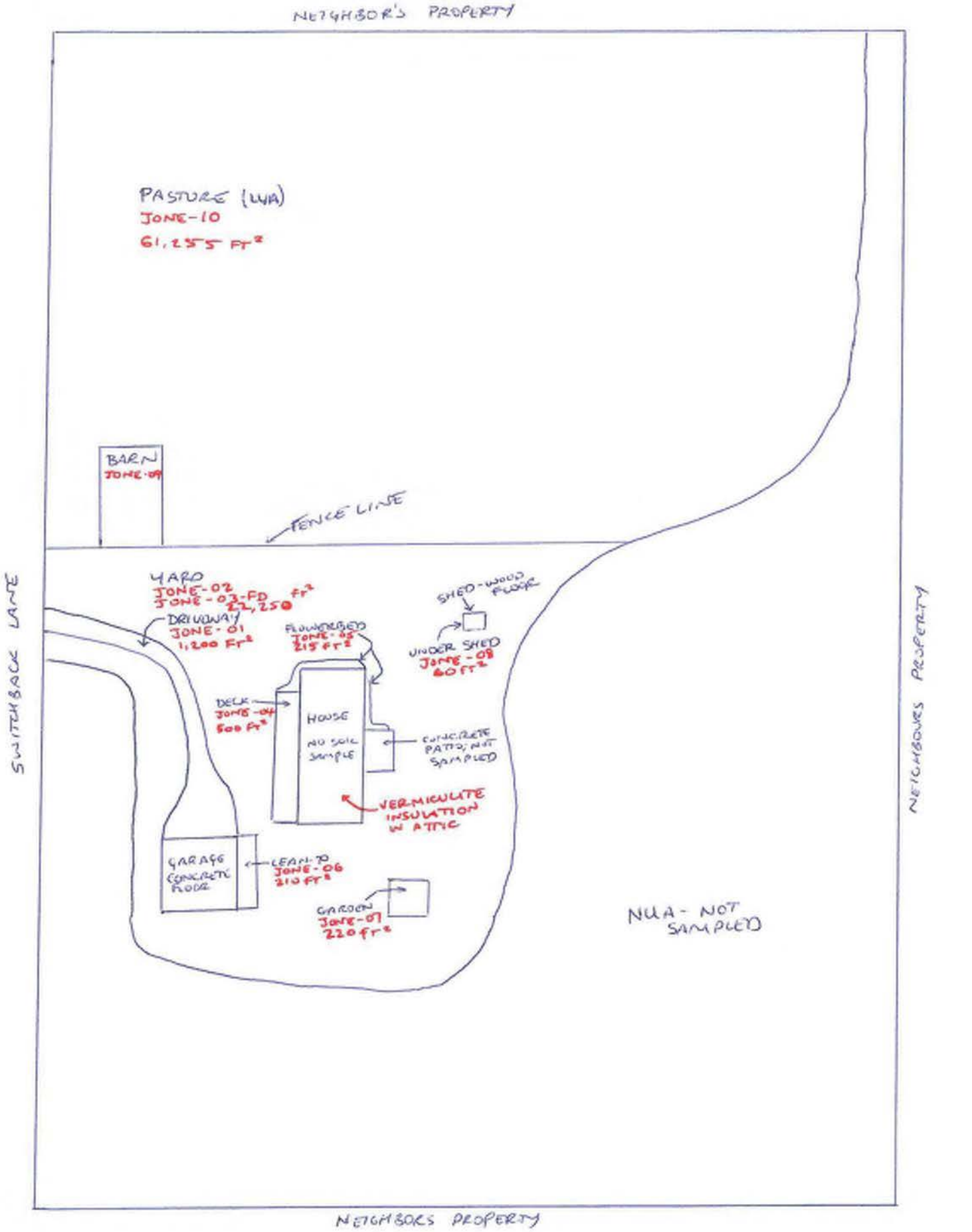
1 inch = 20 feet

N

840 SWITCHBACK LN
OWNER : JONES
SAMPLING DATE : 6/20/19

N

SCALE IS 1" = 50'



APPENDIX C – VERMICULITE PHOTO EXAMPLES

UNEXFOLIATED VERMICULITE IN SOIL

UNEXFOLIATED VERMICULITE

EXFOLIATED VERMICULITE INSULATION

EXFOLIATED VERMICULITE INSULATION IN AN ATTIC

VCBM - VERMICULITE USED IN PATCHING MORTAR

VCBM - VERMICULITE IN PLASTER (LATHE & PLASTER WALL)

APPENDIX D – TRANSMISSION ELECTRON MICROSCOPY ANALYTICAL METHOD

construction document for the building, or, to the best of his or her knowledge, no ACBM was used as a building material in the building. The local education agency shall submit a copy of the signed statement of the architect, project engineer, or accredited inspector to the EPA Regional Office and shall include the statement in the management plan for that school.

(b) The exclusion, under paragraphs (a) (1) through (4) of this section, from conducting the inspection under §763.85(a) shall apply only to homogeneous or sampling areas of a school building that were inspected and sampled before October 17, 1987. The local education agency shall conduct an inspection under §763.85(a) of all areas inspected before October 17, 1987, that were not sampled or were not assumed to be ACM.

(c) If ACBM is subsequently found in a homogeneous or sampling area of a local education agency that had been identified as receiving an exclusion by an accredited inspector under paragraphs (a) (3), (4), (5) of this section, or an architect, project engineer or accredited inspector under paragraph (a)(7) of this section, the local education agency shall have 180 days following the date of identification of ACBM to comply with this subpart E.

APPENDIX A TO SUBPART E OF PART 763—INTERIM TRANSMISSION ELECTRON MICROSCOPY ANALYTICAL METHODS—MANDATORY AND NON-MANDATORY—AND MANDATORY SECTION TO DETERMINE COMPLETION OF RESPONSE ACTIONS

I. Introduction

The following appendix contains three units. The first unit is the mandatory transmission electron microscopy (TEM) method which all laboratories must follow; it is the minimum requirement for analysis of air samples for asbestos by TEM. The mandatory method contains the essential elements of the TEM method. The second unit contains the complete non-mandatory method. The non-mandatory method supplements the mandatory method by including additional steps to improve the analysis. EPA recommends that the non-mandatory method be employed for analyzing air filters; however, the laboratory may choose to employ the mandatory method. The non-mandatory method contains the same minimum requirements as are outlined in the mandatory method. Hence, laboratories may choose either of the two methods for analyzing air samples by TEM.

The final unit of this Appendix A to subpart E defines the steps which must be taken to determine completion of response actions. This unit is mandatory.

II. Mandatory Transmission Electron Microscopy Method

A. Definitions of Terms

1. *Analytical sensitivity*—Airborne asbestos concentration represented by each fiber counted under the electron microscope. It is determined by the air volume collected and the proportion of the filter examined. This method requires that the analytical sensitivity be no greater than 0.005 structures/cm^3.

2. *Asbestiform*—A specific type of mineral fibrosity in which the fibers and fibrils possess high tensile strength and flexibility.

3. *Aspect ratio*—A ratio of the length to the width of a particle. Minimum aspect ratio as defined by this method is equal to or greater than 5:1.

4. *Bundle*—A structure composed of three or more fibers in a parallel arrangement with each fiber closer than one fiber diameter.

5. *Clean area*—A controlled environment which is maintained and monitored to assure a low probability of asbestos contamination to materials in that space. Clean areas used in this method have HEPA filtered air under positive pressure and are capable of sustained operation with an open laboratory blank which on subsequent analysis has an average of less than 18 structures/mm^2 in an area of 0.057 mm^2 (nominally 10 200-mesh grid openings) and a maximum of 53 structures/mm^2 for any single preparation for that same area.

6. *Cluster*—A structure with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group. Groupings must have more than two intersections.

7. *ED*—Electron diffraction.

8. *EDXA*—Energy dispersive X-ray analysis.

9. *Fiber*—A structure greater than or equal to 0.5 μm in length with an aspect ratio (length to width) of 5:1 or greater and having substantially parallel sides.

10. *Grid*—An open structure for mounting on the sample to aid in its examination in the TEM. The term is used here to denote a 200-mesh copper lattice approximately 3 mm in diameter.

11. *Intersection*—Nonparallel touching or crossing of fibers, with the projection having an aspect ratio of 5:1 or greater.

12. *Laboratory sample coordinator*—That person responsible for the conduct of sample

handling and the certification of the testing procedures.

13. *Filter background level*—The concentration of structures per square millimeter of filter that is considered indistinguishable from the concentration measured on a blank (filters through which no air has been drawn). For this method the filter background level is defined as 70 structures/mm^2.

14. *Matrix*—Fiber or fibers with one end free and the other end embedded in or hidden by a particulate. The exposed fiber must meet the fiber definition.

15. *NSD*—No structure detected.

16. *Operator*—A person responsible for the TEM instrumental analysis of the sample.

17. *PCM*—Phase contrast microscopy.

18. *SAED*—Selected area electron diffraction.

19. *SEM*—Scanning electron microscope.

20. *STEM*—Scanning transmission electron microscope.

21. *Structure*—a microscopic bundle, cluster, fiber, or matrix which may contain asbestos.

22. *S/cm^3*—Structures per cubic centimeter.

23. *S/mm^2*—Structures per square millimeter.

24. *TEM*—Transmission electron microscope.

B. Sampling

1. The sampling agency must have written quality control procedures and documents which verify compliance.

2. Sampling operations must be performed by qualified individuals completely independent of the abatement contractor to avoid possible conflict of interest (References 1, 2, 3, and 5 of Unit II.J.).

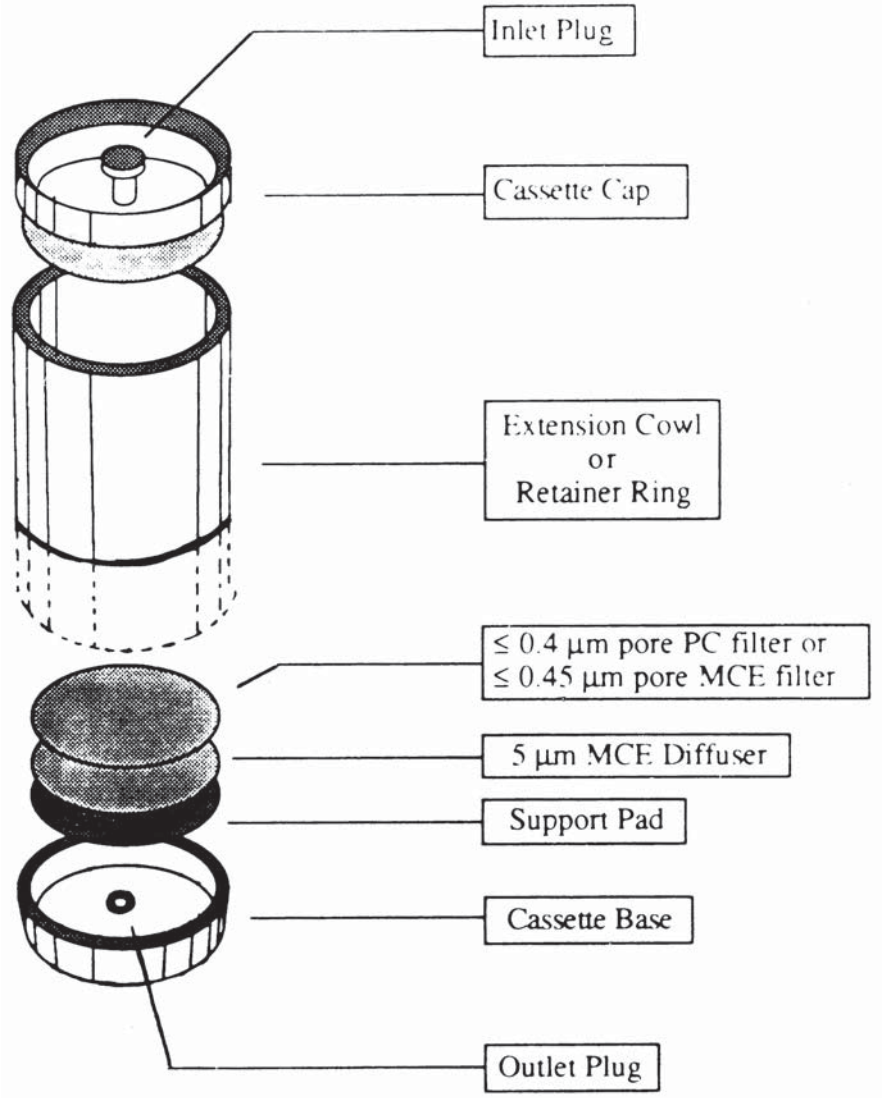
3. Sampling for airborne asbestos following an abatement action must use commercially available cassettes.

4. Prescreen the loaded cassette collection filters to assure that they do not contain concentrations of asbestos which may interfere with the analysis of the sample. A filter blank average of less than 18 s/mm^2 in an area of 0.057 mm^2 (nominally 10 200-mesh grid openings) and a single preparation with a maximum of 53 s/mm^2 for that same area is acceptable for this method.

5. Use sample collection filters which are either polycarbonate having a pore size less than or equal to 0.4 μm or mixed cellulose ester having a pore size less than or equal to 0.45 μm.

6. Place these filters in series with a 5.0 μm backup filter (to serve as a diffuser) and a support pad. See the following Figure 1:

FIGURE I--SAMPLING CASSETTE CONFIGURATION



7. Reloading of used cassettes is not permitted.

8. Orient the cassette downward at approximately 45 degrees from the horizontal.

9. Maintain a log of all pertinent sampling information.

10. Calibrate sampling pumps and their flow indicators over the range of their intended use with a recognized standard. Assemble the sampling system with a representative filter (not the filter which will be used in sampling) before and after the sampling operation.

11. Record all calibration information.

12. Ensure that the mechanical vibrations from the pump will be minimized to prevent transferral of vibration to the cassette.

13. Ensure that a continuous smooth flow of negative pressure is delivered by the pump by damping out any pump action fluctuations if necessary.

14. The final plastic barrier around the abatement area remains in place for the sampling period.

15. After the area has passed a thorough visual inspection, use aggressive sampling conditions to dislodge any remaining dust. (See suggested protocol in Unit III.B.7.d.)

16. Select an appropriate flow rate equal to or greater than 1 liter per minute (L/min) or less than 10 L/min for 25 mm cassettes. Larger filters may be operated at proportionally higher flow rates.

17. A minimum of 13 samples are to be collected for each testing site consisting of the following:

a. A minimum of five samples per abatement area.

b. A minimum of five samples per ambient area positioned at locations representative of the air entering the abatement site.

c. Two field blanks are to be taken by removing the cap for not more than 30 seconds and replacing it at the time of sampling before sampling is initiated at the following places:

i. Near the entrance to each abatement area.

ii. At one of the ambient sites. (DO NOT leave the field blanks open during the sampling period.)

d. A sealed blank is to be carried with each sample set. This representative cassette is not to be opened in the field.

18. Perform a leak check of the sampling system at each indoor and outdoor sampling site by activating the pump with the closed sampling cassette in line. Any flow indicates a leak which must be eliminated before initiating the sampling operation.

19. The following Table I specifies volume ranges to be used:

TABLE 1--NUMBER OF 200 MESH EM GRID OPENINGS ($0.0057\ MM^2$) THAT NEED TO BE ANALYZED TO MAINTAIN SENSITIVITY OF 0.005 STRUCTURES/CC BASED ON VOLUME AND EFFECTIVE FILTER AREA

	Effective Filter Area 385 sq mm	
	Volume (liters)	# of grid openings
	560	*24*
	600	23
	700	19
	800	17
	900	15
	1,000	14
	1,100	12
Recommended Volume Range	**1,200**	**11**
	1,300	**10**
	1,400	**10**
	1,500	**9**
	1,600	**8**
	1,700	**8**
	1,800	**8**
	1,900	7
	2,000	7
	2,100	6
	2,200	6
	2,300	6
	2,400	6
	2,500	5
	2,600	5
	2,700	5
	2,800	5
	2,900	5
	3,000	5
	3,100	4
	3,200	4
	3,300	4
	3,400	4
	3,500	4
	3,600	4
	3,700	4
	3,800	4

	Effective Filter Area 855 sq mm	
	Volume (liters)	# of grid openings
	1,250	*24*
	1,300	23
	1,400	21
	1,600	19
	1,800	17
	2,000	15
	2,200	14
	2,400	13
	2,600	12
	2,800	**11**
	3,000	**10**
	3,200	**9**
	3,400	**9**
	3,600	**8**
	3,800	**8**
Recommended Volume Range	**4,000**	**8**
	4,200	7
	4,400	7
	4,600	7
	4,800	6
	5,000	6
	5,200	6
	5,400	6
	5,600	5
	5,800	5
	6,000	5
	6,200	5
	6,400	5
	6,600	5
	6,800	4
	7,000	4
	7,200	4
	7,400	4
	7,600	4

Note minimum volumes required:
25 mm : 560 liters
37 mm : 1250 liters

Filter diameter of 25 mm = effective area of 385 sq mm
Filter diameter of 37 mm = effective area of 855 sq mm

20. Ensure that the sampler is turned upright before interrupting the pump flow.

21. Check that all samples are clearly labeled and that all pertinent information has been enclosed before transfer of the samples to the laboratory.

22. Ensure that the samples are stored in a secure and representative location.

23. Do not change containers if portions of these filters are taken for other purposes.

24. A summary of Sample Data Quality Objectives is shown in the following Table II:

TABLE II--SUMMARY OF SAMPLING AGENCY DATA QUALITY OBJECTIVES

This table summarizes the data quality objectives from the performance of this method in terms of precision, accuracy, completeness, representativeness, and comparability. These objectives are assured by the periodic control checks and reference checks listed here and described in the text of the method.

Unit Operation	QC Check	Frequency	Conformance Expectation
Sampling materials	Sealed blank	1 per I/O site	95%
Sample procedures	Field blanks	2 per I/O site	95%
	Pump calibration	Before and after each field series	90%
Sample custody	Review of chain-of-custody record	Each sample	95% complete
Sample shipment	Review of sending report	Each sample	95% complete

C. Sample Shipment

Ship bulk samples to the analytical laboratory in a separate container from air samples.

D. Sample Receiving

1. Designate one individual as sample coordinator at the laboratory. While that individual will normally be available to receive samples, the coordinator may train and supervise others in receiving procedures for those times when he/she is not available.

2. Bulk samples and air samples delivered to the analytical laboratory in the same container shall be rejected.

E. Sample Preparation

1. All sample preparation and analysis shall be performed by a laboratory independent of the abatement contractor.

2. Wet-wipe the exterior of the cassettes to minimize contamination possibilities before taking them into the clean room facility.

3. Perform sample preparation in a well-equipped clean facility.

NOTE: The clean area is required to have the following minimum characteristics. The area or hood must be capable of maintaining a positive pressure with make-up air being HEPA-filtered. The cumulative analytical blank concentration must average less than 18 s/mm^2 in an area of 0.057 mm^2 (nominally 10 200-mesh grid openings) and a single preparation with a maximum of 53 s/mm^2 for that same area.

4. Preparation areas for air samples must not only be separated from preparation areas for bulk samples, but they must be prepared in separate rooms.

5. Direct preparation techniques are required. The object is to produce an intact film containing the particulates of the filter surface which is sufficiently clear for TEM analysis.

a. TEM Grid Opening Area measurement must be done as follows:

i. The filter portion being used for sample preparation must have the surface collapsed using an acetone vapor technique.

ii. Measure 20 grid openings on each of 20 random 200-mesh copper grids by placing a grid on a glass and examining it under the PCM. Use a calibrated graticule to measure the average field diameters. From the data, calculate the field area for an average grid opening.

iii. Measurements can also be made on the TEM at a properly calibrated low magnification or on an optical microscope at a magnification of approximately 400X by using an eyepiece fitted with a scale that has been calibrated against a stage micrometer. Optical microscopy utilizing manual or automated procedures may be used providing instrument calibration can be verified.

b. TEM specimen preparation from polycarbonate (PC) filters. Procedures as described in Unit III.G. or other equivalent methods may be used.

c. TEM specimen preparation from mixed cellulose ester (MCE) filters.

i. Filter portion being used for sample preparation must have the surface collapsed using an acetone vapor technique or the Burdette procedure (Ref. 7 of Unit II.J.)

ii. Plasma etching of the collapsed filter is required. The microscope slide to which the collapsed filter pieces are attached is placed in a plasma asher. Because plasma ashers vary greatly in their performance, both from unit to unit and between different positions in the asher chamber, it is difficult to specify the conditions that should be used. Insufficient etching will result in a failure to expose embedded filters, and too much etching may result in loss of particulate from the surface. As an interim measure, it is recommended that the time for ashing of a

known weight of a collapsed filter be established and that the etching rate be calculated in terms of micrometers per second. The actual etching time used for the particulate asher and operating conditions will then be set such that a 1–2 μm (10 percent) layer of collapsed surface will be removed.

iii. Procedures as described in Unit III. or other equivalent methods may be used to prepare samples.

F. TEM Method

1. An 80–120 kV TEM capable of performing electron diffraction with a fluorescent screen inscribed with calibrated gradations is required. If the TEM is equipped with EDXA it must either have a STEM attachment or be capable of producing a spot less than 250 nm in diameter at crossover. The microscope shall be calibrated routinely for magnification and camera constant.

2. *Determination of Camera Constant and ED Pattern Analysis.* The camera length of the TEM in ED operating mode must be calibrated before ED patterns on unknown samples are observed. This can be achieved by using a carbon-coated grid on which a thin film of gold has been sputtered or evaporated. A thin film of gold is evaporated on the specimen TEM grid to obtain zone-axis ED patterns superimposed with a ring pattern from the polycrystalline gold film. In practice, it is desirable to optimize the thickness of the gold film so that only one or two sharp rings are obtained on the superimposed ED pattern. Thicker gold film would normally give multiple gold rings, but it will tend to mask weaker diffraction spots from the unknown fibrous particulate. Since the unknown d-spacings of most interest in asbestos analysis are those which lie closest to the transmitted beam, multiple gold rings are unnecessary on zone-axis ED patterns. An average camera constant using multiple gold rings can be determined. The camera constant is one-half the diameter of the rings times the interplanar spacing of the ring being measured.

3. *Magnification Calibration.* The magnification calibration must be done at the fluorescent screen. The TEM must be calibrated at the grid opening magnification (if used) and also at the magnification used for fiber counting. This is performed with a cross grating replica (e.g., one containing 2,160 lines/mm). Define a field of view on the fluorescent screen either by markings or physical boundaries. The field of view must be measurable or previously inscribed with a scale or concentric circles (all scales should be metric). A logbook must be maintained, and the dates of calibration and the values obtained must be recorded. The frequency of calibration depends on the past history of the particular microscope. After any maintenance of the microscope that involved adjustment of the power supplied to the lenses or the high-voltage system or the mechanical disassembly of the electron optical column apart from filament exchange, the magnification must be recalibrated. Before the TEM calibration is performed, the analyst must ensure that the cross grating replica is placed at the same distance from the objective lens as the specimens are. For instruments that incorporate a eucentric tilting specimen stage, all specimens and the cross grating replica must be placed at the eucentric position.

4. While not required on every microscope in the laboratory, the laboratory must have either one microscope equipped with energy dispersive X-ray analysis or access to an equivalent system on a TEM in another laboratory.

5. Microscope settings: 80–120 kV, grid assessment 250–1,000X, then 15,000–20,000X screen magnification for analysis.

6. Approximately one-half (0.5) of the predetermined sample area to be analyzed shall be performed on one sample grid preparation and the remaining half on a second sample grid preparation.

7. Individual grid openings with greater than 5 percent openings (holes) or covered with greater than 25 percent particulate matter or obviously having nonuniform loading must not be analyzed.

8. Reject the grid if:

a. Less than 50 percent of the grid openings covered by the replica are intact.

b. The replica is doubled or folded.

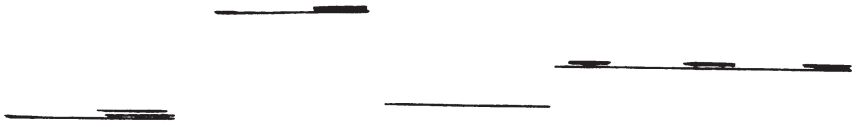
c. The replica is too dark because of incomplete dissolution of the filter.

9. *Recording Rules.*

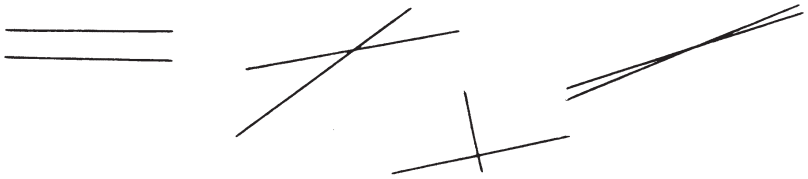
a. Any continuous grouping of particles in which an asbestos fiber with an aspect ratio greater than or equal to 5:1 and a length greater than or equal to 0.5 μm is detected shall be recorded on the count sheet. These will be designated asbestos structures and will be classified as fibers, bundles, clusters, or matrices. Record as individual fibers any contiguous grouping having 0, 1, or 2 definable intersections. Groupings having more than 2 intersections are to be described as cluster or matrix. An intersection is a nonparallel touching or crossing of fibers, with the projection having an aspect ratio of 5:1 or greater. See the following Figure 2:

FIGURE 2--COUNTING GUIDELINES USED IN DETERMINING ASBESTOS STRUCTURES

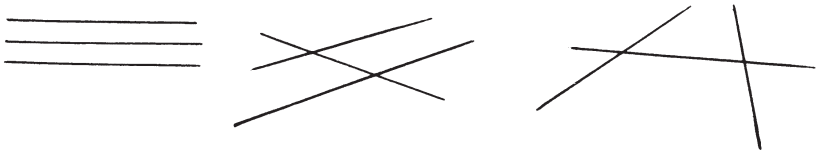
Count as 1 fiber; 1 Structure; no intersections.



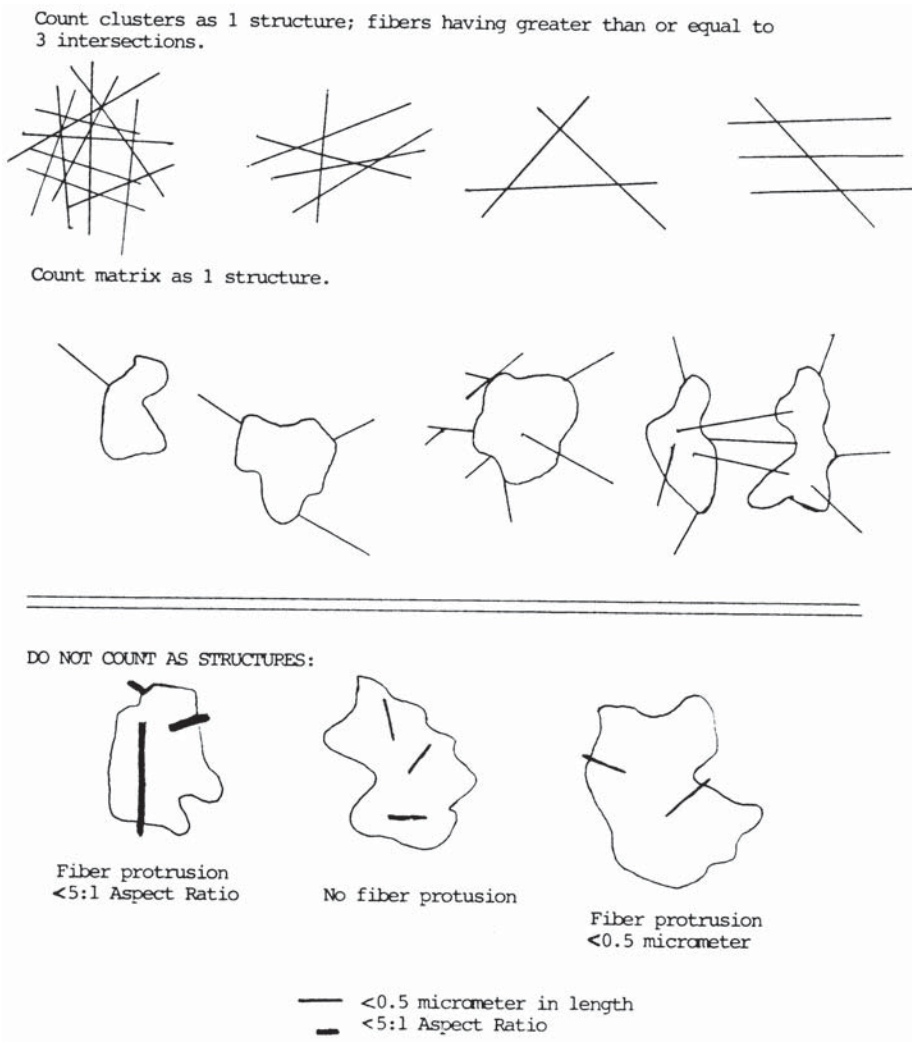
Count as 2 fibers if space between fibers is greater than width of 1 fiber diameter or number of intersections is equal to or less than 1.



Count as 3 structures if space between fibers is greater than width of 1 fiber diameter or if the number of intersections is equal to or less than 2.



Count bundles as 1 structure; 3 or more parallel fibrils less than 1 fiber diameter separation.



i. *Fiber.* A structure having a minimum length greater than or equal to 0.5 μm and an aspect ratio (length to width) of 5:1 or greater and substantially parallel sides. Note the appearance of the end of the fiber, i.e., whether it is flat, rounded or dovetailed.

ii. *Bundle.* A structure composed of three or more fibers in a parallel arrangement with each fiber closer than one fiber diameter.

iii. *Cluster.* A structure with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group. Groupings must have more than two intersections.

iv. *Matrix.* Fiber or fibers with one end free and the other end embedded in or hidden by a particulate. The exposed fiber must meet the fiber definition.

b. Separate categories will be maintained for fibers less than 5 μm and for fibers equal to or greater than 5 μm in length.

c. Record NSD when no structures are detected in the field.

d. Visual identification of electron diffraction (ED) patterns is required for each asbestos structure counted which would cause the

analysis to exceed the 70 s/mm^2 concentration. (Generally this means the first four fibers identified as asbestos must exhibit an identifiable diffraction pattern for chrysotile or amphibole.)

e. The micrograph number of the recorded diffraction patterns must be reported to the client and maintained in the laboratory's quality assurance records. In the event that examination of the pattern by a qualified individual indicates that the pattern has been misidentified visually, the client shall be contacted.

f. Energy Dispersive X-ray Analysis (EDXA) is required of all amphiboles which would cause the analysis results to exceed the 70 s/mm^2 concentration. (Generally speaking, the first 4 amphiboles would require EDXA.)

g. If the number of fibers in the non-asbestos class would cause the analysis to exceed the 70 s/mm^2 concentration, the fact that they are not asbestos must be confirmed by EDXA or measurement of a zone axis diffraction pattern.

h. Fibers classified as chrysotile must be identified by diffraction or X-ray analysis and recorded on a count sheet. X-ray analysis alone can be used only after 70 s/mm^2 have been exceeded for a particular sample.

i. Fibers classified as amphiboles must be identified by X-ray analysis and electron diffraction and recorded on the count sheet. (X-ray analysis alone can be used only after 70 s/mm^2 have been exceeded for a particular sample.)

j. If a diffraction pattern was recorded on film, record the micrograph number on the count sheet.

k. If an electron diffraction was attempted but no pattern was observed, record N on the count sheet.

l. If an EDXA spectrum was attempted but not observed, record N on the count sheet.

m. If an X-ray analysis spectrum is stored, record the file and disk number on the count sheet.

10. Classification Rules.

a. *Fiber*. A structure having a minimum length greater than or equal to 0.5 μm and an aspect ratio (length to width) of 5:1 or greater and substantially parallel sides. Note the appearance of the end of the fiber, i.e., whether it is flat, rounded or dovetailed.

b. *Bundle*. A structure composed of three or more fibers in a parallel arrangement with each fiber closer than one fiber diameter.

c. *Cluster*. A structure with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group. Groupings must have more than two intersections.

d. *Matrix*. Fiber or fibers with one end free and the other end embedded in or hidden by a particulate. The exposed fiber must meet the fiber definition.

11. After finishing with a grid, remove it from the microscope, and replace it in the appropriate grid holder. Sample grids must be stored for a minimum of 1 year from the date of the analysis; the sample cassette must be retained for a minimum of 30 days by the laboratory or returned at the client's request.

G. Sample Analytical Sequence

1. Under the present sampling requirements a minimum of 13 samples is to be collected for the clearance testing of an abatement site. These include five abatement area samples, five ambient samples, two field blanks, and one sealed blank.

2. Carry out visual inspection of work site prior to air monitoring.

3. Collect a minimum of 5 air samples inside the work site and 5 samples outside the work site. The indoor and outdoor samples shall be taken during the same time period.

4. Remaining steps in the analytical sequence are contained in Unit IV of this Appendix.

H. Reporting

1. The following information must be reported to the client for each sample analyzed:

a. Concentration in structures per square millimeter and structures per cubic centimeter.

b. Analytical sensitivity used for the analysis.

c. Number of asbestos structures.

d. Area analyzed.

e. Volume of air sampled (which must be initially supplied to lab by client).

f. Copy of the count sheet must be included with the report.

g. Signature of laboratory official to indicate that the laboratory met specifications of the method.

h. Report form must contain official laboratory identification (e.g., letterhead).

i. Type of asbestos.

I. Quality Control/Quality Assurance Procedures (Data Quality Indicators)

Monitoring the environment for airborne asbestos requires the use of sensitive sampling and analysis procedures. Because the test is sensitive, it may be influenced by a variety of factors. These include the supplies used in the sampling operation, the performance of the sampling, the preparation of the grid from the filter and the actual examination of this grid in the microscope. Each of these unit operations must produce a product of defined quality if the analytical result is to be a reliable and meaningful test result. Accordingly, a series of control checks and reference standards are to be performed along with the sample analysis as indicators that the materials used are adequate and the

operations are within acceptable limits. In this way, the quality of the data is defined and the results are of known value. These checks and tests also provide timely and specific warning of any problems which might develop within the sampling and analysis operations. A description of these quality control/quality assurance procedures is summarized in the following Table III:

TABLE III--SUMMARY OF LABORATORY DATA QUALITY OBJECTIVES

Unit Operation	QC Check	Frequency	Conformance Expectation
Sample receiving	Review of receiving report	Each sample	95% complete
Sample custody	Review of chain-of-custody record	Each sample	95% complete
Sample preparation	Supplies and reagents	On receipt	Meet specs. or reject
	Grid opening size	20 openings/20 grids/lot of 1000 or 1 opening/sample	100%
	Special clean area monitoring	After cleaning or service	Meet specs or reclean
	Laboratory blank	1 per prep series or 10%	Meet specs. or reanalyze series
	Plasma etch blank	1 per 20 samples	75%
	Multiple preps (3 per sample)	Each sample	One with cover of 15 complete grid sqs.
Sample analysis	System check	Each day	Each day
	Alignment check	Each day	Each day
	Magnification calibration with low and high standards	Each month or after service	95%
	ED calibration by gold standard	Weekly	95%
	EDS calibration by copper line	Daily	95%
Performance check	Laboratory blank (measure of cleanliness)	Prep 1 per series or 10% read 1 per 25 samples	Meet specs or reanalyze series
	Replicate counting (measure of precision)	1 per 100 samples	1.5 x Poisson Std. Dev.
	Duplicate analysis (measure of reproducibility)	1 per 100 samples	2 x Poisson Std. Dev.
	Known samples of typical materials (working standards)	Training and for comparison with unknowns	100%
	Analysis of NBS SRM 1876 and/or RM 8410 (measure of accuracy and comparability)	1 per analyst per year	1.5 x Poisson Std. Dev.
	Data entry review (data validation and measure of completeness)	Each sample	95%
	Record and verify ID electron diffraction pattern of structure	1 per 5 samples	80% accuracy
Calculations and data reduction	Hand calculation of automated data reduction procedure or independent recalculation of hand-calculated data	1 per 100 samples	85%

1. When the samples arrive at the laboratory, check the samples and documentation for completeness and requirements before initiating the analysis.

2. Check all laboratory reagents and supplies for acceptable asbestos background levels.

3. Conduct all sample preparation in a clean room environment monitored by laboratory blanks. Testing with blanks must also be done after cleaning or servicing the room.

4. Prepare multiple grids of each sample.

5. Provide laboratory blanks with each sample batch. Maintain a cumulative average of these results. If there are more than 53 fibers/mm^2 per 10 200-mesh grid openings, the system must be checked for possible sources of contamination.

6. Perform a system check on the transmission electron microscope daily.

7. Make periodic performance checks of magnification, electron diffraction and energy dispersive X-ray systems as set forth in Table III under Unit II.I.

8. Ensure qualified operator performance by evaluation of replicate analysis and standard sample comparisons as set forth in Table III under Unit II.I.

9. Validate all data entries.

10. Recalculate a percentage of all computations and automatic data reduction steps as specified in Table III under Unit II.I.

11. Record an electron diffraction pattern of one asbestos structure from every five samples that contain asbestos. Verify the identification of the pattern by measurement or comparison of the pattern with patterns collected from standards under the same conditions. The records must also demonstrate that the identification of the pattern has been verified by a qualified individual and that the operator who made the identification is maintaining at least an 80 percent correct visual identification based on his measured patterns.

12. Appropriate logs or records must be maintained by the analytical laboratory verifying that it is in compliance with the mandatory quality assurance procedures.

J. References

For additional background information on this method, the following references should be consulted.

1. ''Guidance for Controlling Asbestos-Containing Materials in Buildings,'' EPA 560/5–85–024, June 1985.

2. ''Measuring Airborne Asbestos Following an Abatement Action,'' USEPA, Office of Pollution Prevention and Toxics, EPA 600/4–85–049, 1985.

3. Small, John and E. Steel. Asbestos Standards: Materials and Analytical Methods. N.B.S. Special Publication 619, 1982.

4. Campbell, W.J., R.L. Blake, L.L. Brown, E.E. Cather, and J.J. Sjoberg. Selected Silicate Minerals and Their Asbestiform Varieties. Information Circular 8751, U.S. Bureau of Mines, 1977.

5. Quality Assurance Handbook for Air Pollution Measurement System. Ambient Air Methods, EPA 600/4–77–027a, USEPA, Office of Research and Development, 1977.

6. Method 2A: Direct Measurement of Gas Volume through Pipes and Small Ducts. 40 CFR Part 60 Appendix A.

7. Burdette, G.J., Health & Safety Exec. Research & Lab. Services Div., London, ''Proposed Analytical Method for Determination of Asbestos in Air.''

8. Chatfield, E.J., Chatfield Tech. Cons., Ltd., Clark, T., PEI Assoc., ''Standard Operating Procedure for Determination of Airborne Asbestos Fibers by Transmission Electron Microscopy Using Polycarbonate Membrane Filters,'' WERL SOP 87–1, March 5, 1987.

9. NIOSH Method 7402 for Asbestos Fibers, 12–11–86 Draft.

10. Yamate, G., Agarwall, S.C., Gibbons, R.D., IIT Research Institute, ''Methodology for the Measurement of Airborne Asbestos by Electron Microscopy,'' Draft report, USEPA Contract 68–02–3266, July 1984.

11. ''Guidance to the Preparation of Quality Assurance Project Plans,'' USEPA, Office of Pollution Prevention and Toxics, 1984.

III. Nonmandatory Transmission Electron Microscopy Method

A. Definitions of Terms

1. *Analytical sensitivity*—Airborne asbestos concentration represented by each fiber counted under the electron microscope. It is determined by the air volume collected and the proportion of the filter examined. This method requires that the analytical sensitivity be no greater than 0.005 s/cm^3.

2. *Asbestiform*—A specific type of mineral fibrosity in which the fibers and fibrils possess high tensile strength and flexibility.

3. *Aspect ratio*—A ratio of the length to the width of a particle. Minimum aspect ratio as defined by this method is equal to or greater than 5:1.

4. *Bundle*—A structure composed of three or more fibers in a parallel arrangement with each fiber closer than one fiber diameter.

5. *Clean area*—A controlled environment which is maintained and monitored to assure a low probability of asbestos contamination to materials in that space. Clean areas used in this method have HEPA filtered air under positive pressure and are capable of sustained operation with an open laboratory blank which on subsequent analysis has an average of less than 18 structures/mm^2 in an area of 0.057 mm^2 (nominally 10 200 mesh grid openings) and a maximum of 53 structures/mm^2 for no more than one single preparation for that same area.

6. *Cluster*—A structure with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group. Groupings must have more than two intersections.

7. *ED*—Electron diffraction.

8. *EDXA*—Energy dispersive X-ray analysis.

9. *Fiber*—A structure greater than or equal to 0.5 μm in length with an aspect ratio (length to width) of 5:1 or greater and having substantially parallel sides.

APPENDIX E – ANALYTICAL REQUIREMENTS SUMMARY SHEET

SUMMARY OF PREPARATION AND ANALYTICAL REQUIREMENTS FOR ASBESTOS

Document: O&M Sampling Guidance

DEQ Project Manager: Jason Rappe (406-444-6802, Jason.Rappe@mt.gov)

DEQ QA Manager: To be determined

Sampling Program Overview: The O&M sampling Guidance supports the collection and analysis of soil and building material samples to determine if cleanup actions are required, and soil and air samples to ensure LA-contaminated media is sufficiently removed to meet cleanup requirements.

PLM Preparation and Analytical Requirements:

Medium, Sample Type	Preparation Method	Analysis Method	Laboratory Modifications
Soil	PLM-9002	PLM-VE and PLM-Grav	LB-000073D, LB-000097A, LB-000103
Building Material	EPA/600/R-93/116	PLM-PC400	None
Building Material	PLM-9002	PLM-9002	LB-000073D, LB-000098

TEM Preparation and Analytical Requirements:

Medium, Sample Type	Preparation Details		Analysis Details		
	Indirect Prep?		Method	Recording Rules	Analytical Sensitivity
	With Ashing	Without Ashing			
Clearance Air	No[1]	No[1]	TEM-AHERA	All asbestos[2]; L: > 0.5µm AR: > 5:1	Examine a minimum of 2 GOs[3] in each of 2 grids. Continue examining GOs until one is achieved: 1) Target sensitivity of 0.005 cc^{-1} is achieved, or 2) 0.1 mm^2 of filter area has been examined, or 3) 25 LA structures are recorded (finish GO where 25th LA found)

[1] If any one clearance air sample in a set is overloaded, the laboratory will immediately notify DEQ QA Manager for instruction on how to proceed.
[2] Recording of chrysotile can stop after 25 chrysotile structures have been recorded (finish GO where 25th chrysotile found).
[3] Grid openings

APPENDIX F – EPA ANALYTICAL STANDARD OPERATING PROCEDURE

QUALITATIVE ESTIMATION OF ASBESTOS IN COARSE SOIL BY VISUAL EXAMINATION USING STEREOMICROSCOPY 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 — 9-21-12 — Date

Mark McDaniel — Print Name

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

TABLE OF CONTENTS

LIST OF ATTACHMENTS

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

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

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.

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.

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.)

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

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.

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,

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.

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

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 ≤ 1mm 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 > 1mm, separate them from the sample material.
- 14.2.5.3 Place and 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,

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

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

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.

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

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 ≤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

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 ≤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.

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

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.

TABLE OF CONTENTS

LIST OF ATTACHMENTS

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.

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

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

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.

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
- 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

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

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

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 ≤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

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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.

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

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.

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 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

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:

Pre-ash percent asbestos = (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

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 250μm. 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 > 250μm), 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.

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 Supplemental 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.

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

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

chrysotile should be done with the fibers mounted in the 1.550 liquid.

13.5.7.16 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 $Fe_7Si_8O_{22}(OH)_2$

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 $Na_2Fe_3^{2+}Fe_2^{3+}Si_8O_{22}(OH)_2$

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.

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 $(Mg,Fe)_7Si_8O_{22}(OH)_2$

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, $Ca_2(Mg,Fe)_5Si_8O_{22}(OH)_2$; winchite, $CaNaMg_4(Al,Fe^{3+})Si_8O_{22}(OH)_2$; richterite, $NaCaNa(Mg,Fe)_5Si_8O_{22}(OH)_2$; magnesio-arfvedsonite, $(Na,K)Na_2Mg_4Fe^{3+}Si_8O_{22}(OH)_2$; and magnesio-riebeckite, $Na_2Mg_3Fe^{3+}{}_2Si_8O_{22}(OH)_2$. 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.

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

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-

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

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.

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

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 weight[1] 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

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.

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

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

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 (α = 1.538 to 1.554, γ = 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 (α = 1.616 to 1.645, γ = 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

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 (α = 1.710 to 1.718, γ = 1.724 to 1.734).

13.8.5.4 Birefringence for kyanite ranges from 0.012 to 0.016.

13.8.6 Hornblende $(Ca,Na)_{2-3}(Mg,Fe,Al)_5Si_6(Si,Al)_2O_{22}(OH)_2$

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 (α = 1.60 to 1.70, γ = 1.62 to 1.73), but most hornblende has α = 1.645 to 1.665 and γ = 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, $(Ca,Na)(Mg,Fe,Al)(Si,Al)_2O_6$, and the end members Diopside, $CaMgSi_2O_6$, and Hedenbergite, $CaFeSi_2O_6$. 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 α = 1.66 to 1.75 and γ = 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

becomes ambiguous, but is generally length slow for smaller extinction angles.

13.8.8 Fiberglass (Amorphous Silica, SiO_2)

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.

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 be either mineral. However, polyethylene has a higher birefringence than chrysotile, and when placed on a hot plate, polyethylene will melt.

13.8.13 Rutile (TiO_2)

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 (α = 2.6 to 2.7, γ = 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

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.

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:

COC_Laboratory ID_Work Order Number_Analytical Method_Date Submitted to Client_EDD_Correction Number

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.

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

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

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 ≤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 ≥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 ≤1% LA. If both the original and inter-laboratory result is >1% LA, acceptance of the

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.

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17.12 Su, Shu-Chun (2004). Area Percentage Charts With Matrix and Area Percentage Charts With No Matrix.

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17.15 William D. Nesse (1991). Introduction to Optical Mineral Mineralogy. New York: Oxford University Press.

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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]
[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																															
					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 ____
					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 ____

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)				
	21°C	*23°C*	*25°C*	*27°C*	*29°C*	*21°C*	*23°C*	*25°C*	*27°C*	*29°C*
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)				
	21°C	*23°C*	*25°C*	*27°C*	*29°C*	*21°C*	*23°C*	*25°C*	*27°C*	*29°C*
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

λ_m (nm)	**1.635** (Cargille Series E) Cargille Glass 1.64 (Lot B)					**1.640** (Cargille Series E) Cargille Glass **1.64** (Lot B)				
	21°C	*23°C*	*25°C*	*27°C*	*29°C*	*21°C*	*23°C*	*25°C*	*27°C*	*29°C*
400	1.612	1.613	1.614	1.615	1.616	1.611	1.611	1.612	1.613	1.614
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

λ_m (nm)	**1.680** (Cargille Series B) Cargille Glass **1.68** (Lot A)					Cargille Glass **1.68** (Lot B or C)				
	21°C	*23°C*	*25°C*	*27°C*	*29°C*	*21°C*	*23°C*	*25°C*	*27°C*	*29°C*
400	1.633	1.634	1.635	1.636	1.637	1.634	1.635	1.636	1.637	1.638
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)				
21°C		*23°C*	*25°C*	*27°C*	*29°C*
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

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 1.604-1.612 1.599-1.612 1.6063	1.625-1.655 1.627-1.635 1.625-1.637 1.6343	0.017-0.028	Oblique (up to 21 °);	+ (length slow)
Actinolite[7]		1.600-1.628 1.612-1.668 1.613-1.628 1.6126	1.625-1.655 1.635-1.688 1.638-1.655 1.6393	0.017-0.028		+ (length slow)
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] **1.618-1.621**[2] 1.629[3] 1.636[4]	**1.634-1.642**[1] **1.634-1.637**[2] 1.650[3] 1.658[4]	**0.008-0.019**[1] **0.016**[2] 0.021[3] 0.022[4]	Oblique, **22°**[1] **15.8°**[2] Oblique, 7-29°[8]	+ (length slow)
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] 1.605-1.624[5] 1.615[6]	**1.638-1.639**[1] 1.627-1.641[5] 1.636[6]	**0.012-0.017**[1] 0.017-0.022[5]	Oblique, **21-22°**[1] Oblique, 5-45°[8]	+ (length slow)
Magnesio-riebeckite	Prismatic to fibrous aggregates. Blue, grey-blue, pale blue to yellow. Can be pleochroic.[8]	1.650-1.673[8]	1.662-1.676[8]	Up to 0.015[8]	Oblique, 8-40°[8]	- (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]	1.635-1.680[8]	0.012-0.026[8]	Oblique, 18-45°[8]	- (length fast)[8]

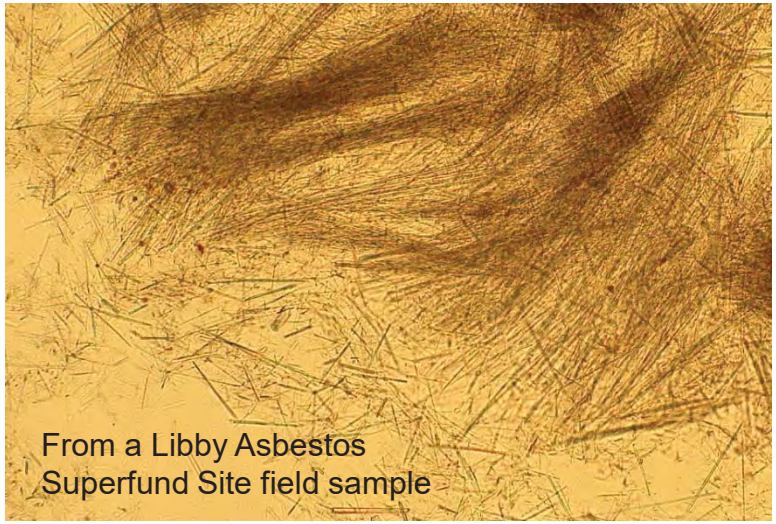
A. This table is adapted for use in SOP SRC-LIBBY-03 (Revision 3) from: Su, Shu-Chun, 2005. White paper: *Tables to Facilitate the Determination of Refractive Indices of Winchite and Richterite, (Libby, Montana) by Dispersion Staining*, August 8, 2005. Data on this table were compiled from data of amphiboles 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.

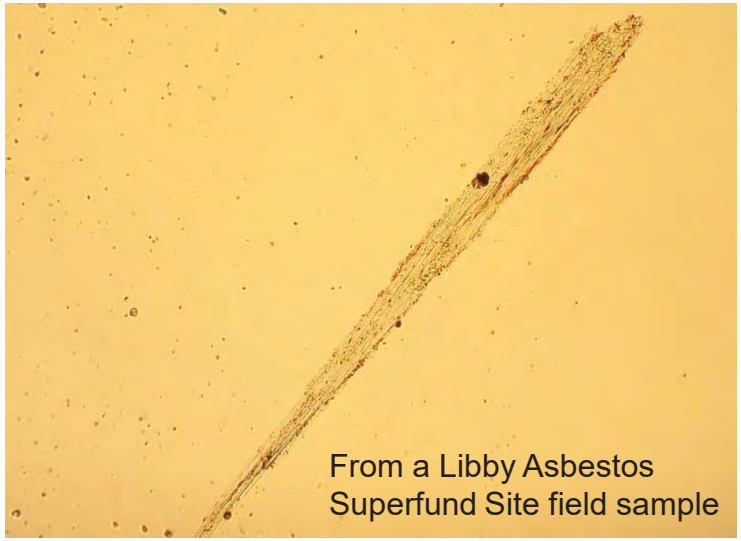
1. Bandli, B.R. et al. (2003) *Optical, compositional, morphological, and X-ray data on eleven particles of amphibole from Libby, Montana, U.S.A.* Canadian Mineralogist, 41, 1241-1253.
2. Wylie, A.G. and Verkouteren, J.R. (2000) *Amphibole asbestos from Libby, Montana: Aspects of nomenclature.* American 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.

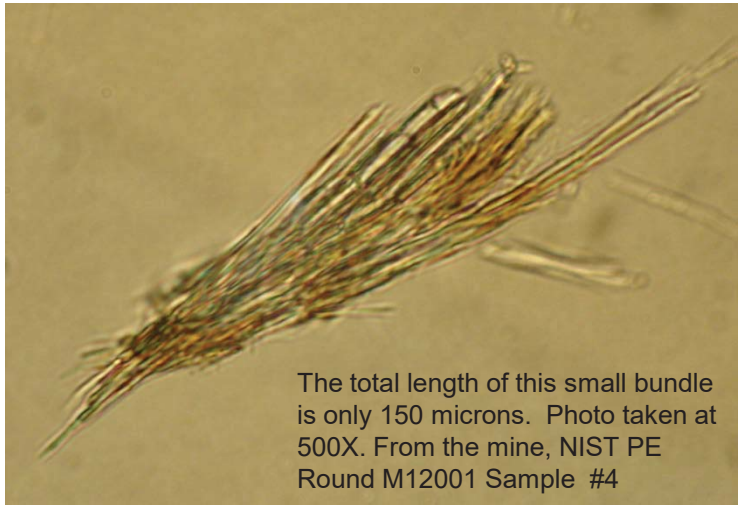
ATTACHMENT 5

PLM Photomicrographs Demonstrating a Wide Range of LA Habits

PLM Photomicrographs Demonstrating a Wide Range of Libby Amphibole Habits

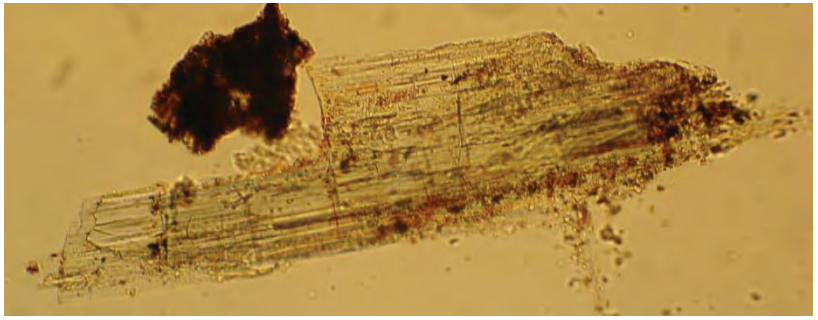






Prismatic Libby Amphibole

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.



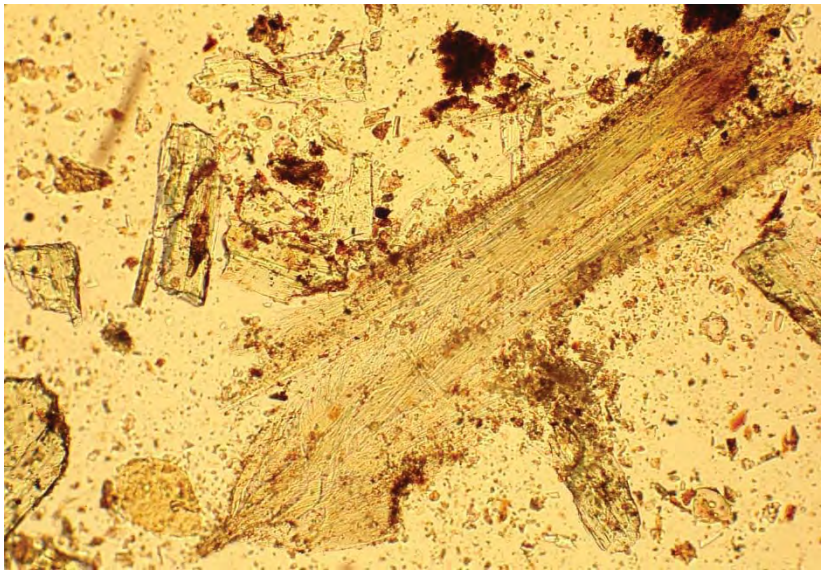
From a Libby Asbestos Superfund Site field sample

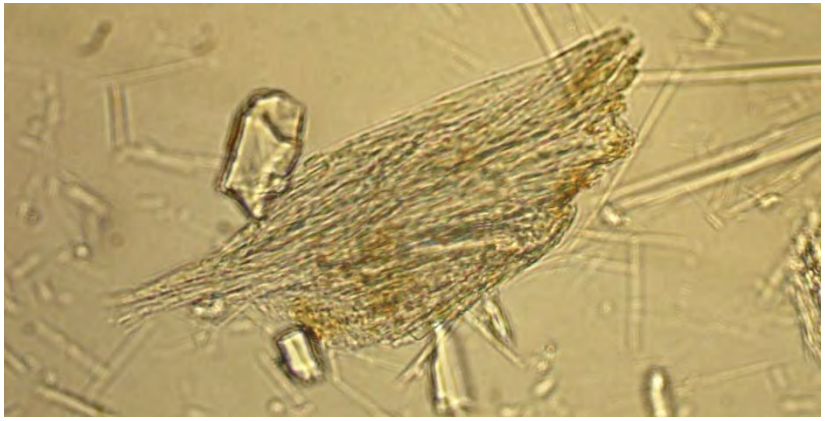
From the mine, NIST PE Round M12001 Sample #4

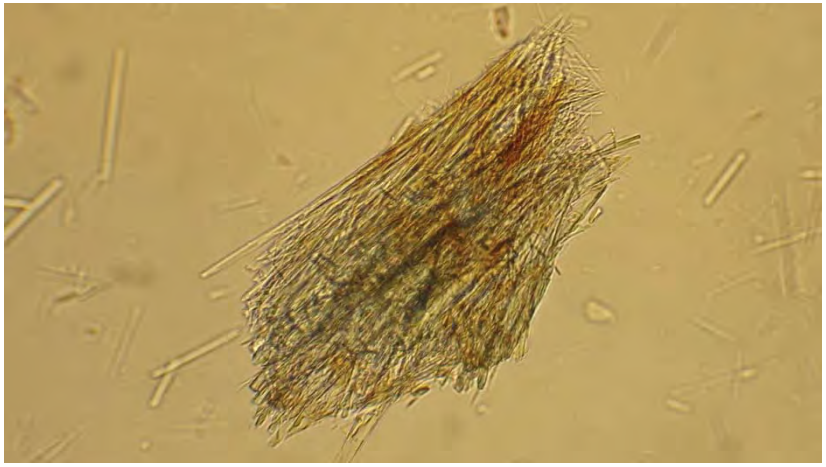
From the mine, NIST PE Round M12001 Sample #4

From a Libby Asbestos Superfund Site field sample

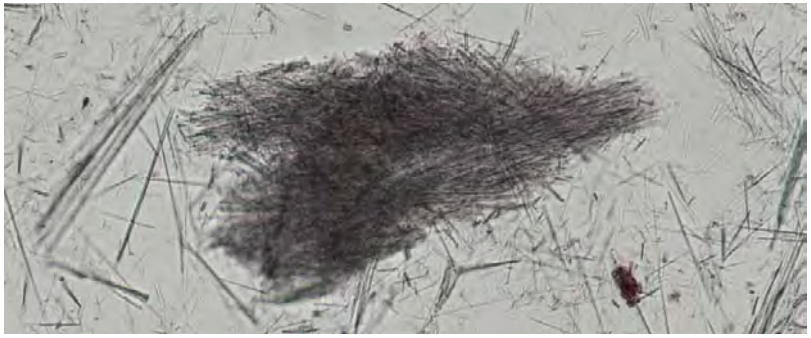
Some LA shows a “matted” or “felted” habit. The internal structure of these bundles is still fibrous. The green high-relief prismatic grains in the top right photo are hornblende. The bundles in the two top photos were found in Libby Asbestos Superfund Site field samples. The bundles in the lower two photos are from the NIST PE Round M12001 Sample #4, from the mine.



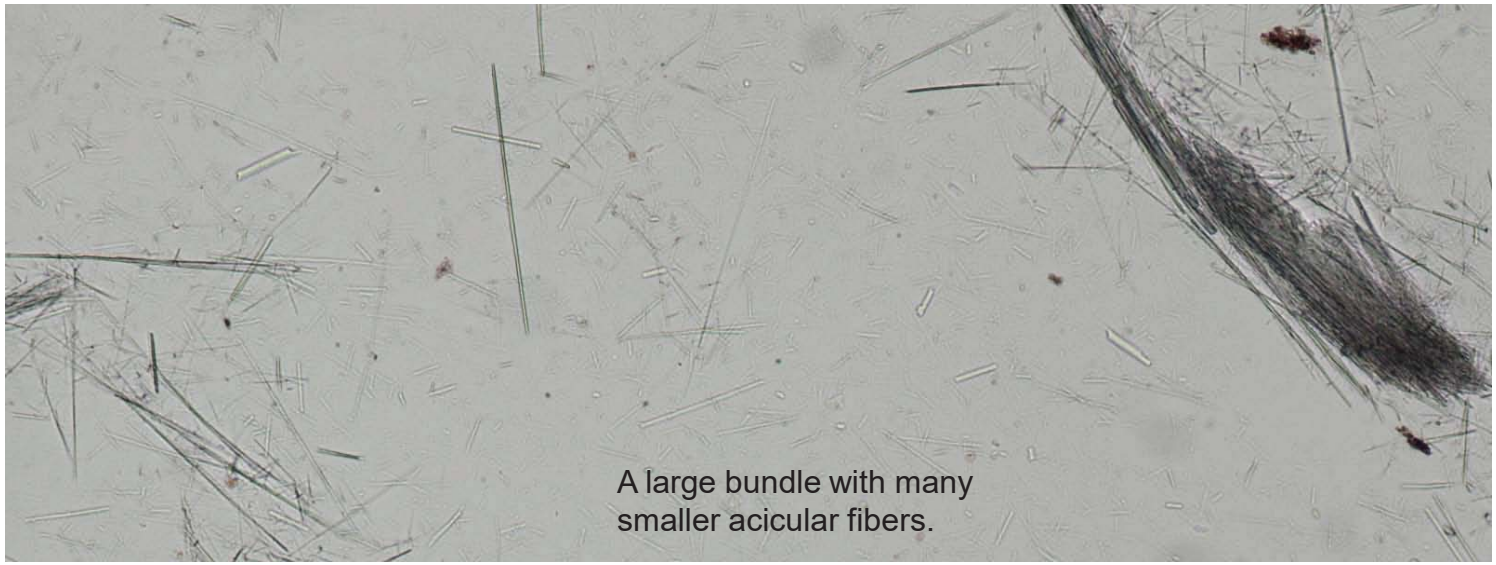




The fibers on the right side of this bundle are completely matted.



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.



A large bundle with many smaller acicular fibers.

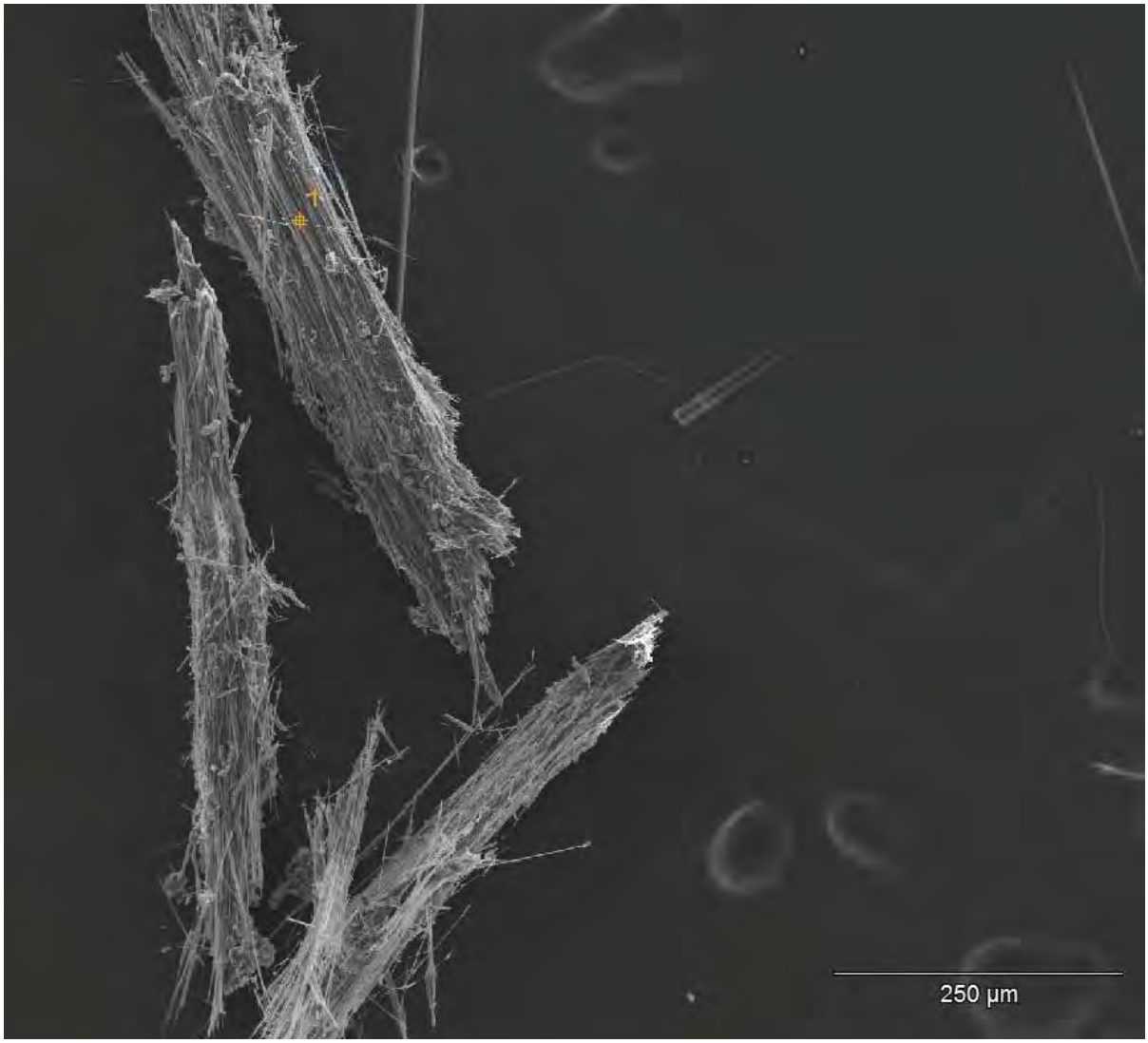
ATTACHMENT 6

SEM Photomicrographs of Representative Examples of LA Habits

SEM Photomicrographs of Representative Examples of Libby Amphibole Habits

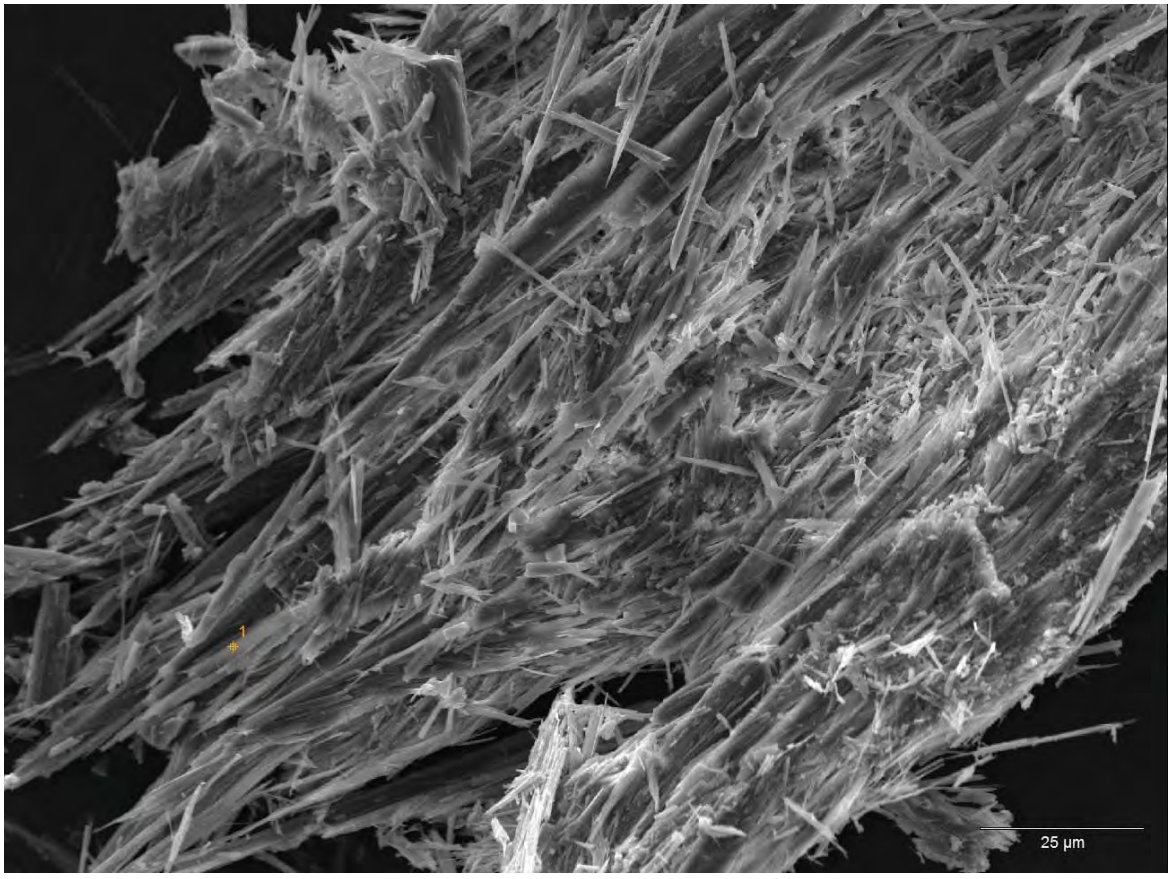
Individual bundles of Libby Amphibole (LA) were picked from soil samples at the ESAT Region 8 Laboratory and prepared for analysis by scanning electron microscopy (SEM). Slide mounts of these bundles were initially prepared in a refractive index (RI) liquid and the bundles were examined by PLM. Then 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.



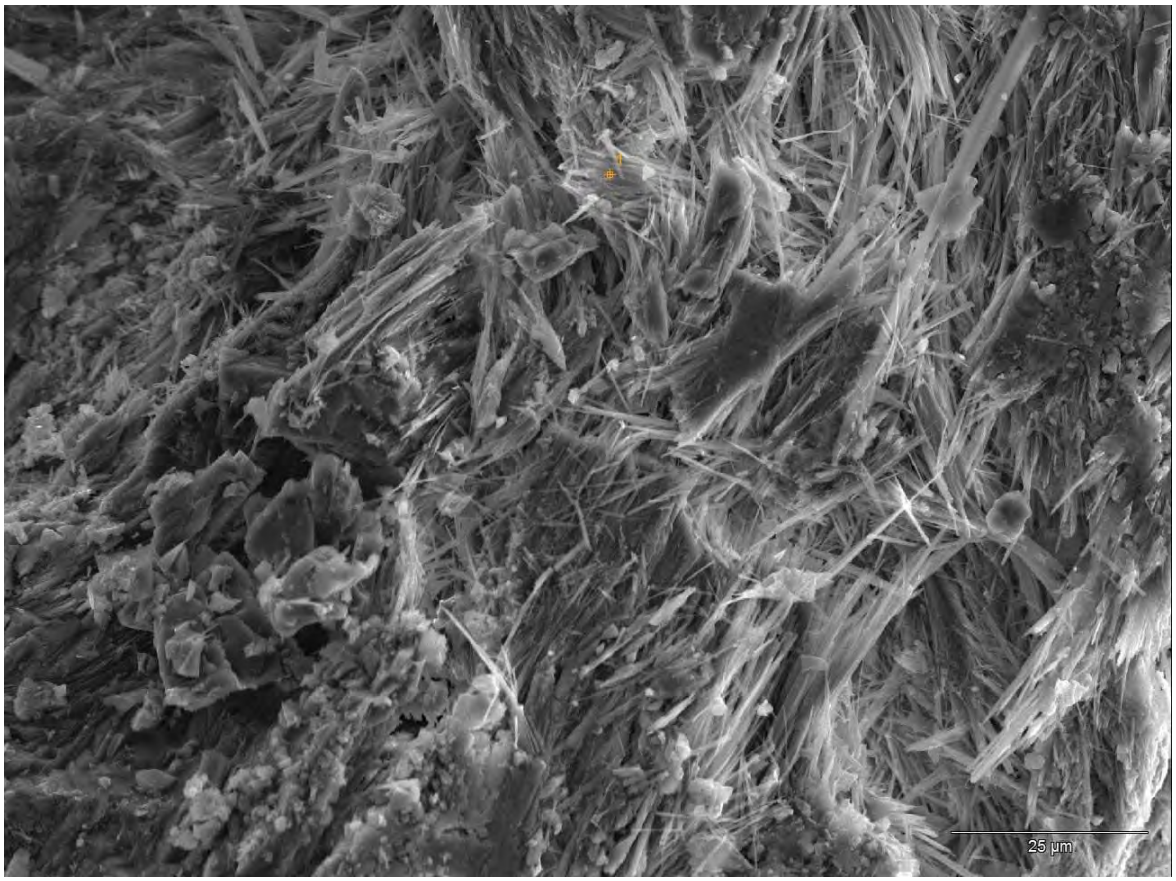
These are typical bundles of LA where the average aspect ratio of the fibers is high and most of the fibers are nearly parallel to one another. Note the scale in microns at the bottom of the photo. These three bundles are all of a size that can be seen with a stereomicroscope and picked out to be placed 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.



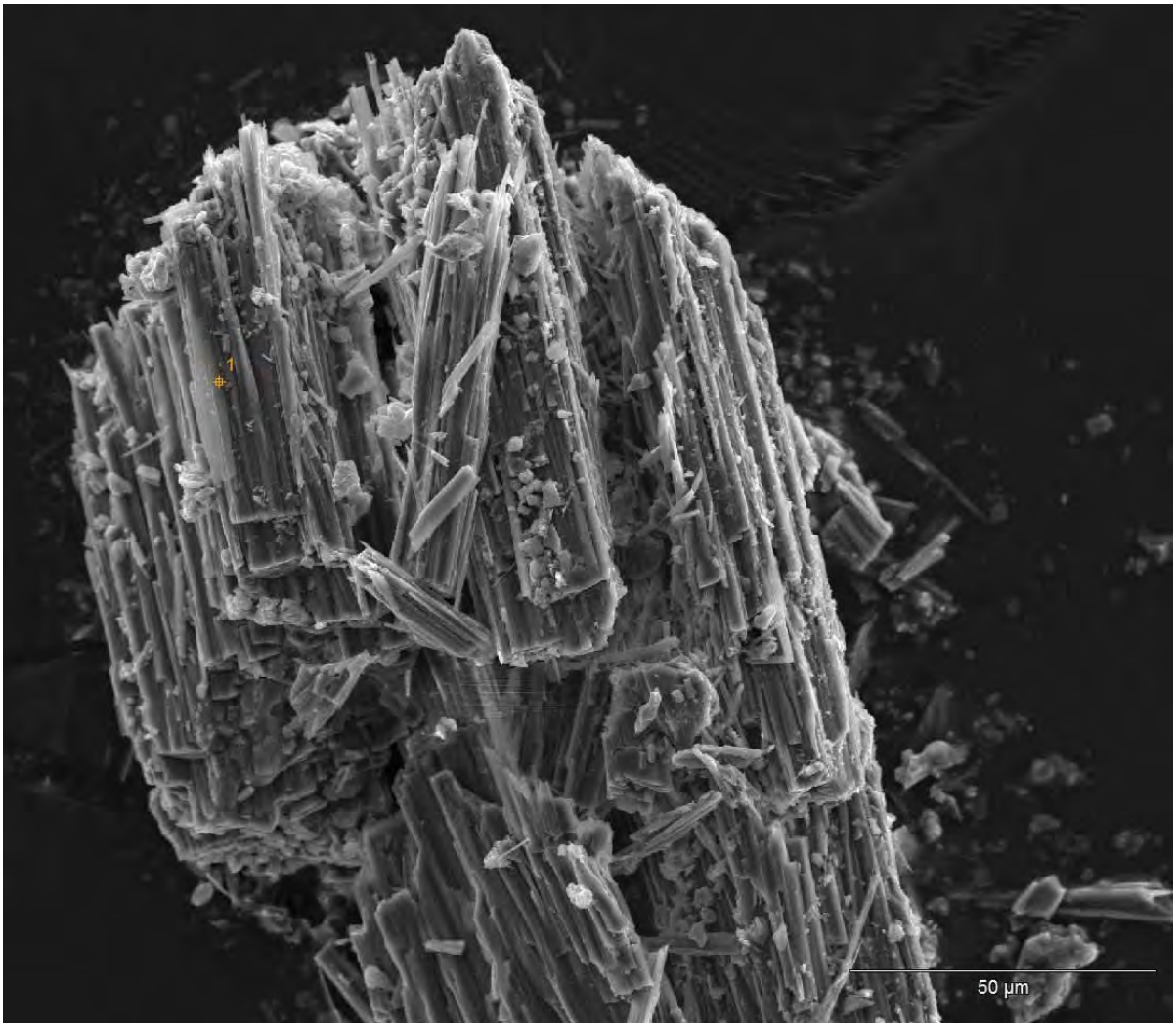
Varying degrees of parallelism can be seen in the fibers that compose bundles of LA. Note that the fibers in this bundle of LA are less parallel than the fibers in the bundles in the previous example.

Photograph provided by the USGS and used by permission.
Photo for use by the Libby Lab Team only- do not cite or distribute.



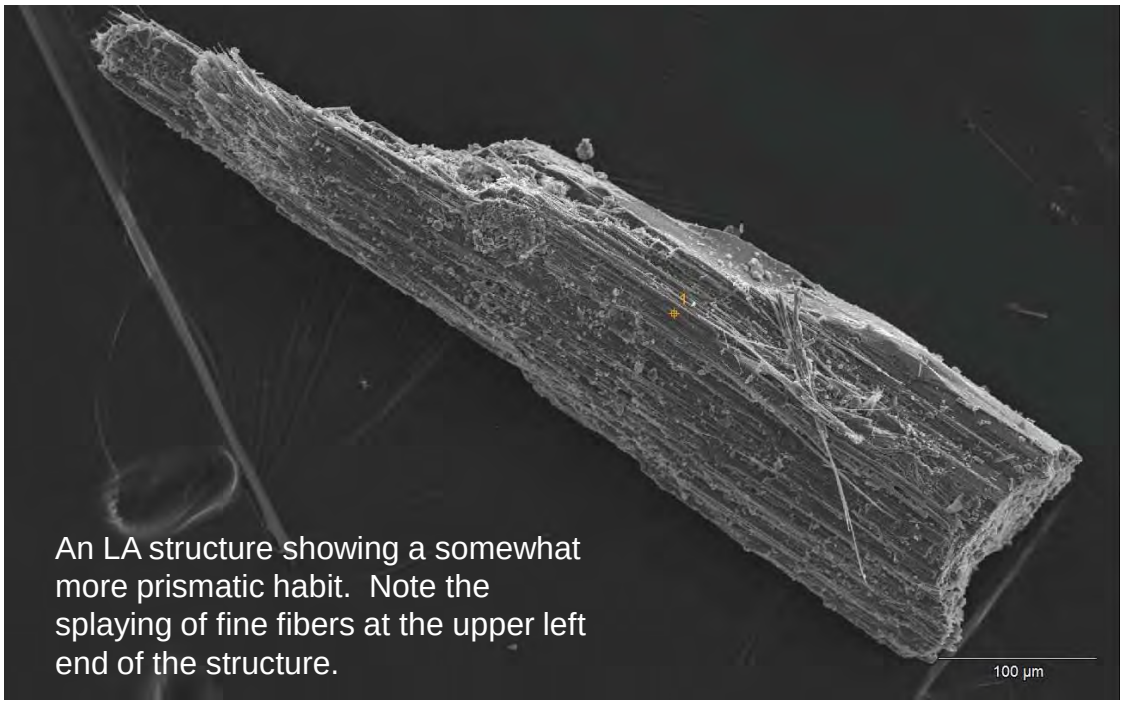
When this bundle of LA was viewed under PLM, its habit was described as “felted” or “matted” with the fibers crossing at high angles to one another. This is how the bundle appeared when it was subsequently viewed by SEM. The fibrous nature of the “felted” or “matted” habit is clear at this scale.

Photograph provided by the USGS and used by permission. Photo for use by the Libby Lab Team only- do not cite or distribute.

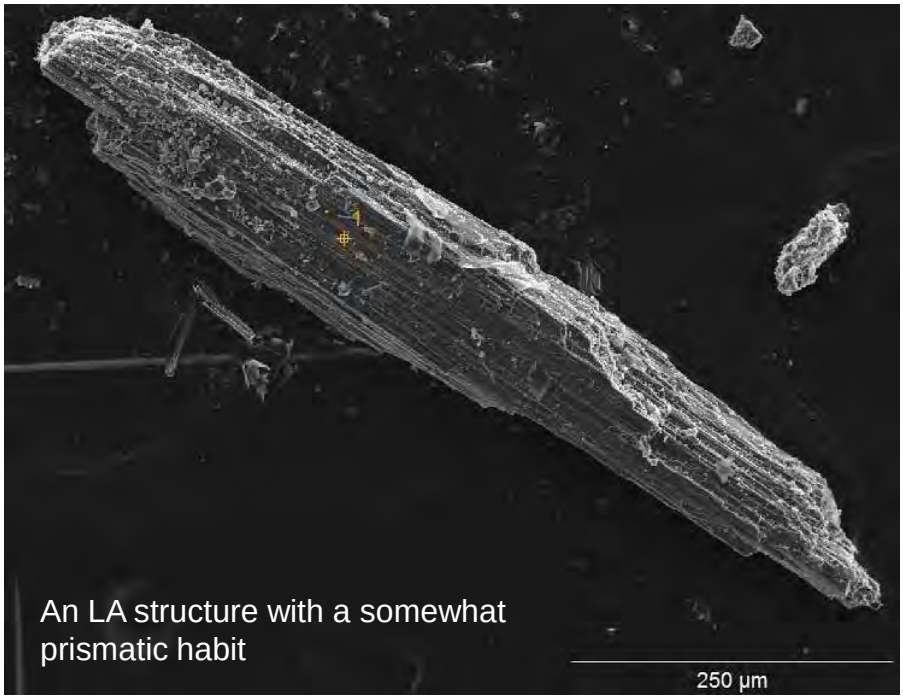


The average aspect ratio of the fibers in this bundle of LA is lower than those of the bundles in the previous examples. However, as seen by SEM, the bundle still splits readily into many small fibers.

Photograph provided by the USGS and used by permission. Photo for use by the Libby Lab Team only- do not cite or distribute.

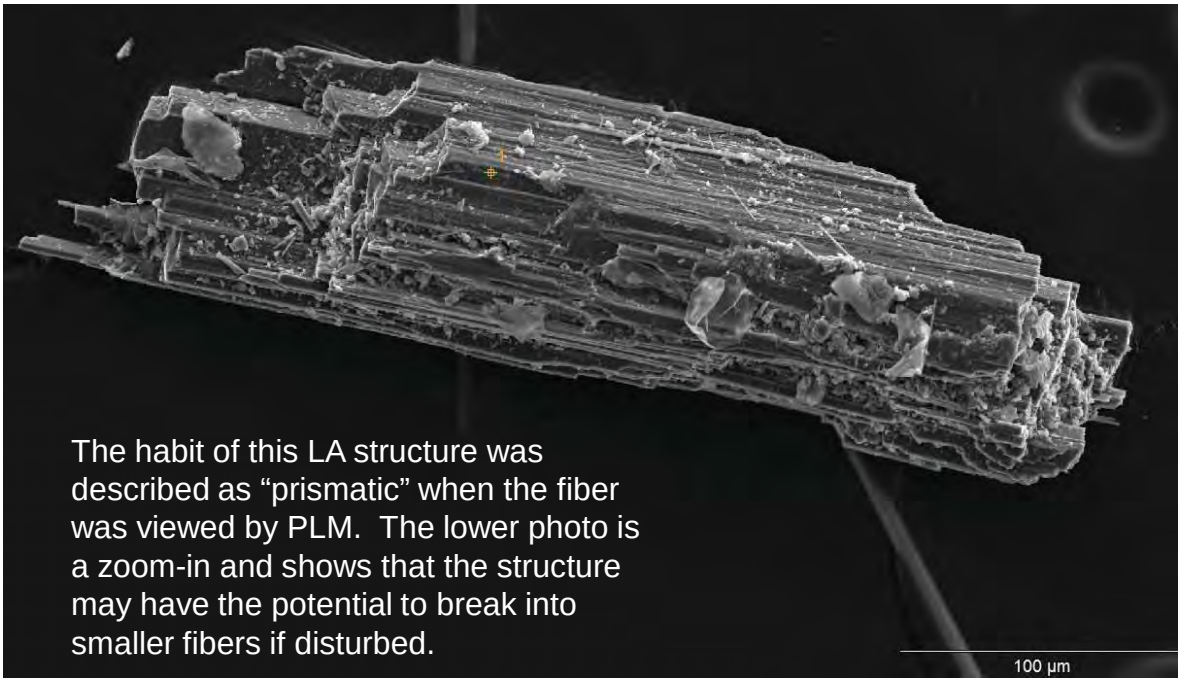


An LA structure showing a somewhat more prismatic habit. Note the splaying of fine fibers at the upper left end of the structure.

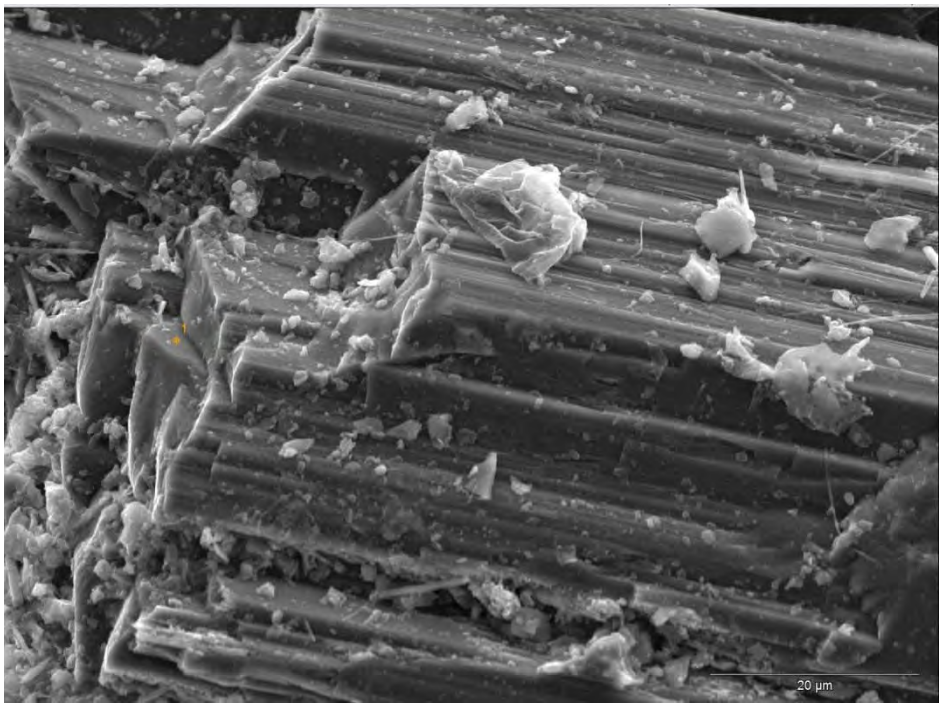


An LA structure with a somewhat prismatic habit

Photographs provided by the USGS and used by permission. Photos for use by the Libby Lab Team only- do not cite or distribute.



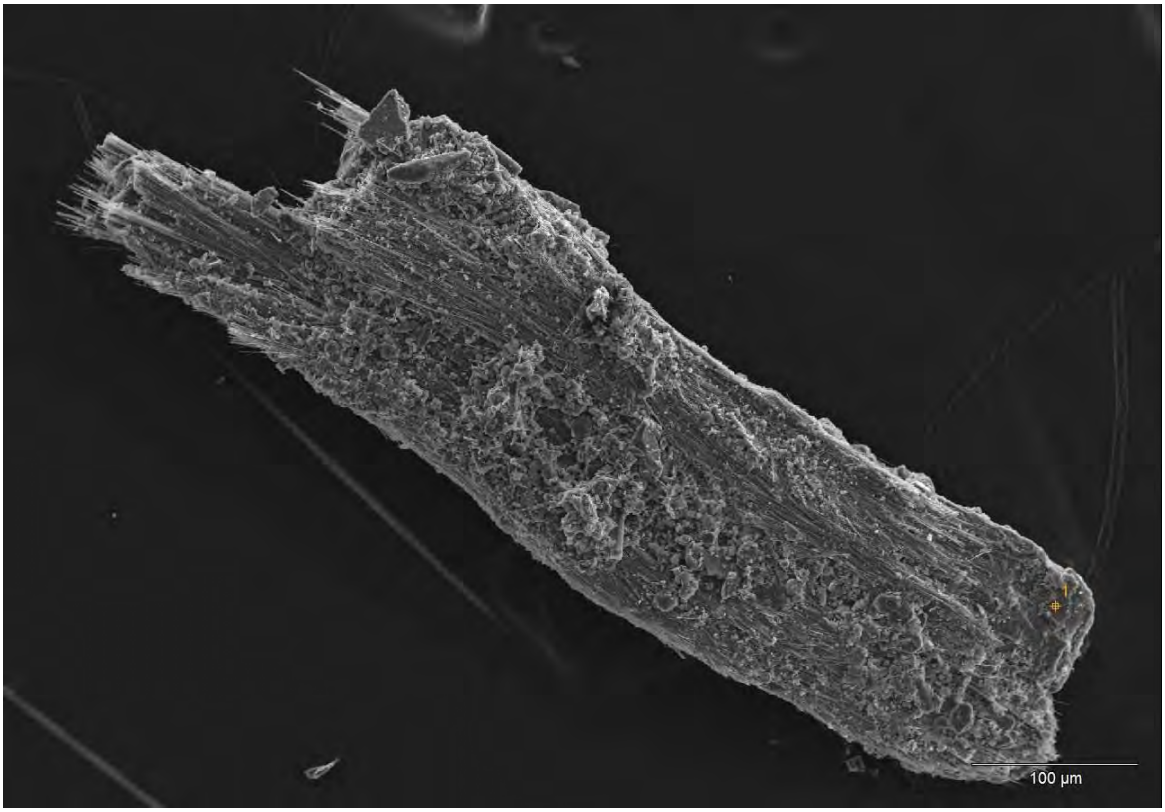
The habit of this LA structure was described as “prismatic” when the fiber was viewed by PLM. The lower photo is a zoom-in and shows that the structure may have the potential to break into smaller fibers if disturbed.



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.

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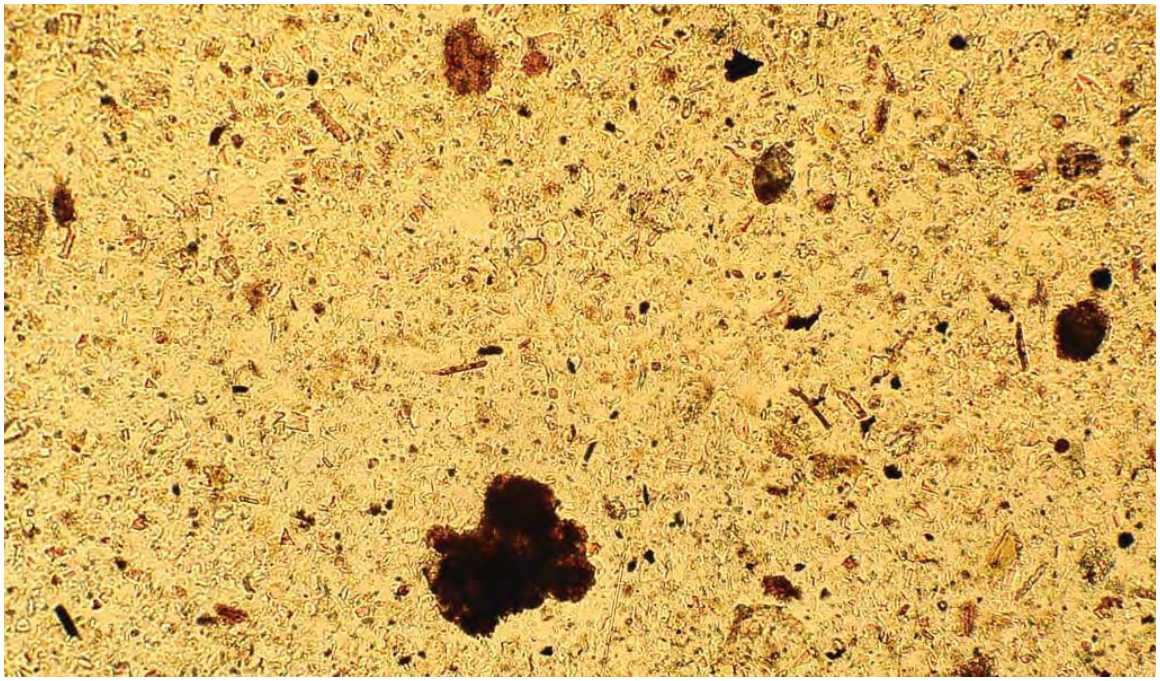
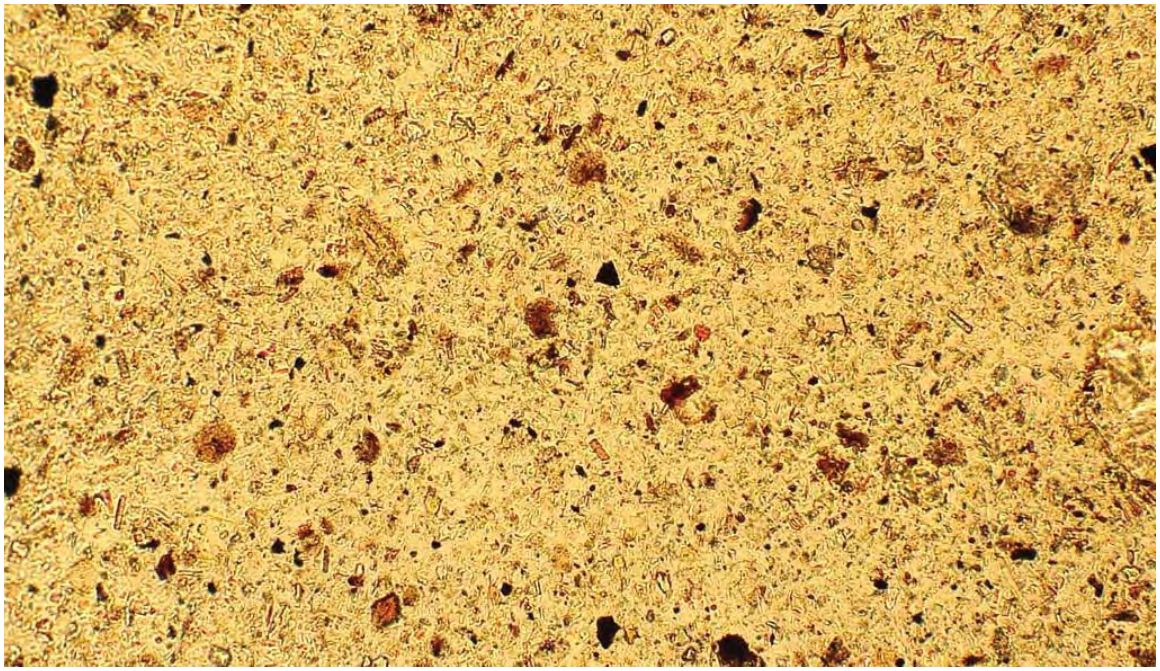
ATTACHMENT 7

Photomicrographs of Representative Fields of View of 0.2% and 1.0% LA Controlled PE Reference Materials

0.2% Libby Amphibole

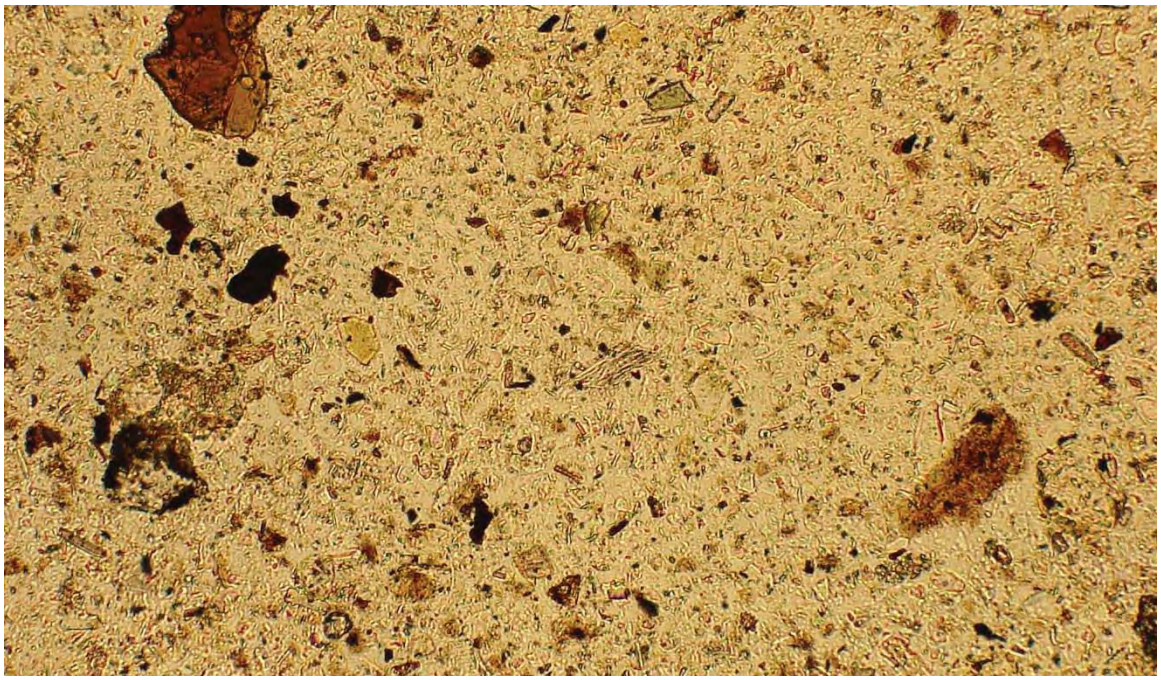
Photomicrographs of representative fields of view of the 0.2% Libby Amphibole by weight Controlled PE Reference Material. All photos taken at 100x, plane light in 1.55 refractive index oil. Width of each picture is approximately 1,500 microns.

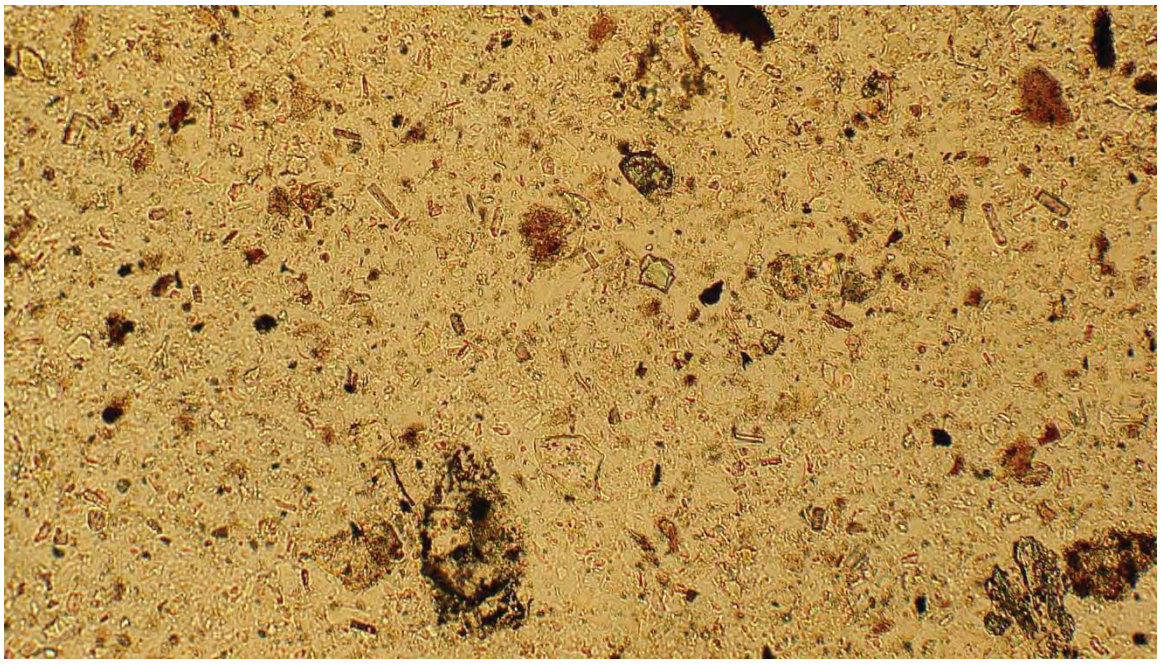




0.2% Libby Amphibole

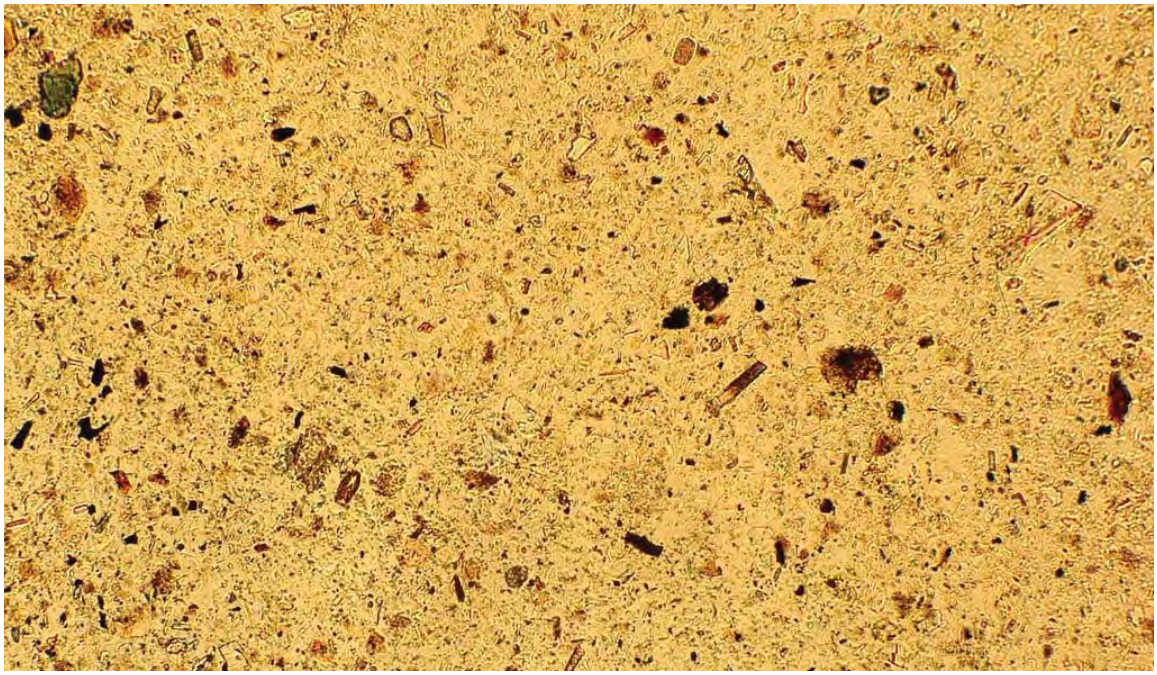
Photomicrographs of representative fields of view. Width of each picture is approximately 1,500 microns.

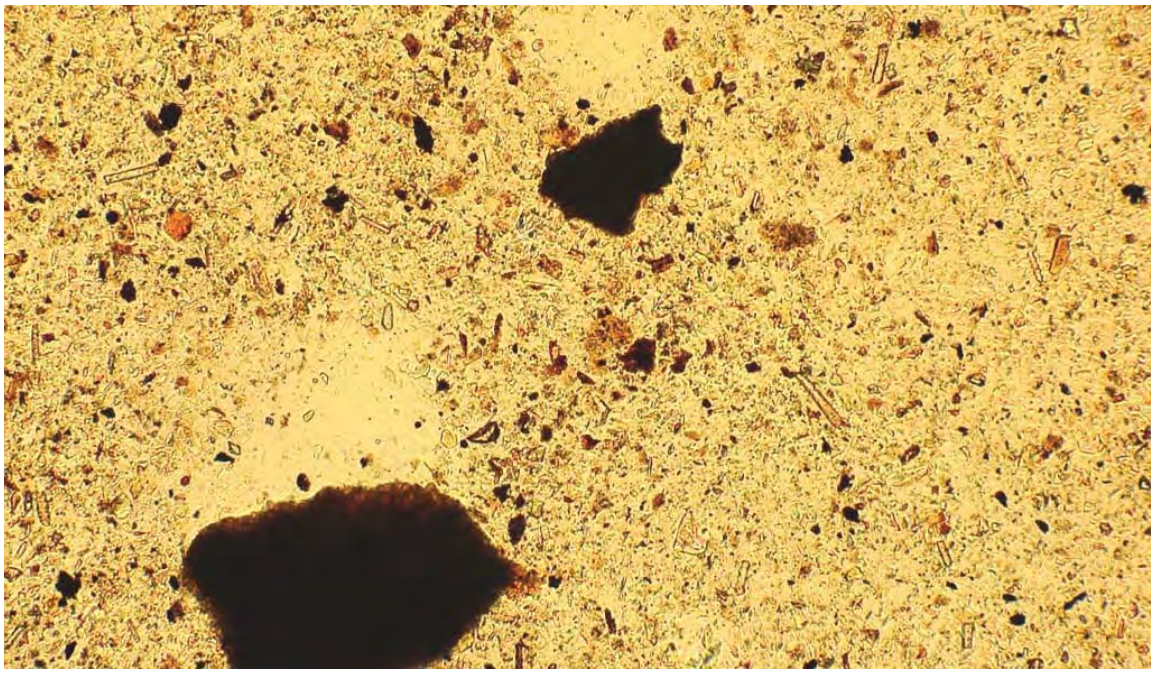




0.2% Libby Amphibole

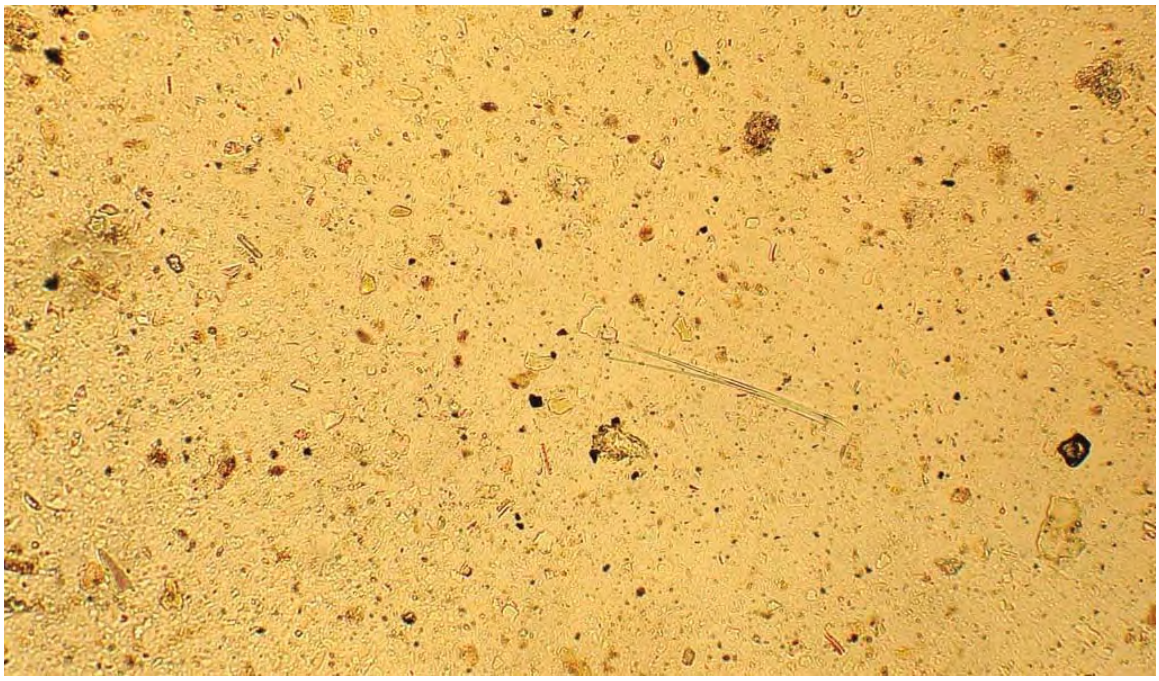
Photomicrographs of representative fields of view. Width of each picture is approximately 1,500 microns.

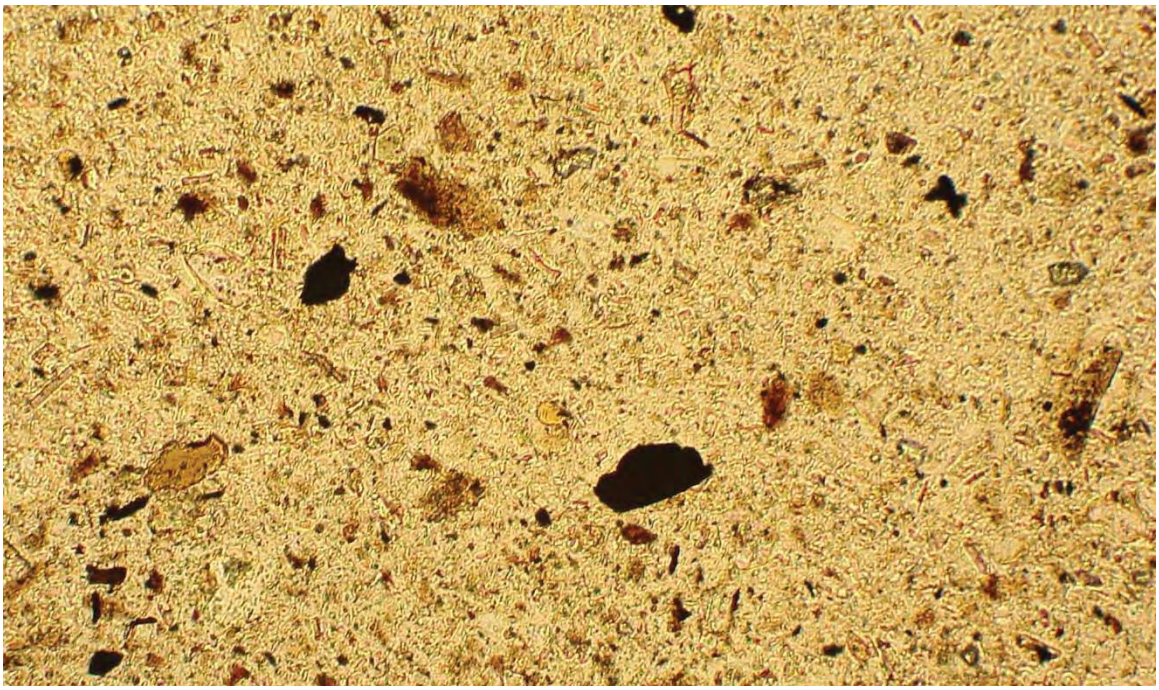




0.2% Libby Amphibole

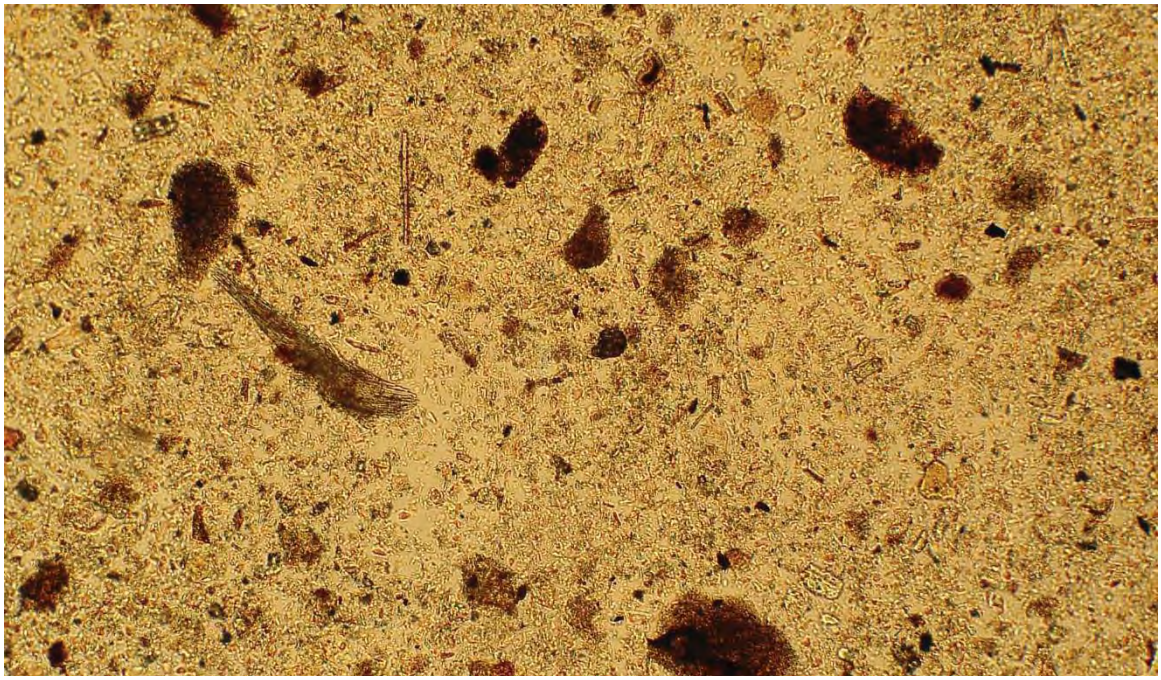
Photomicrographs of representative fields of view. Width of each picture is approximately 1,500 microns.

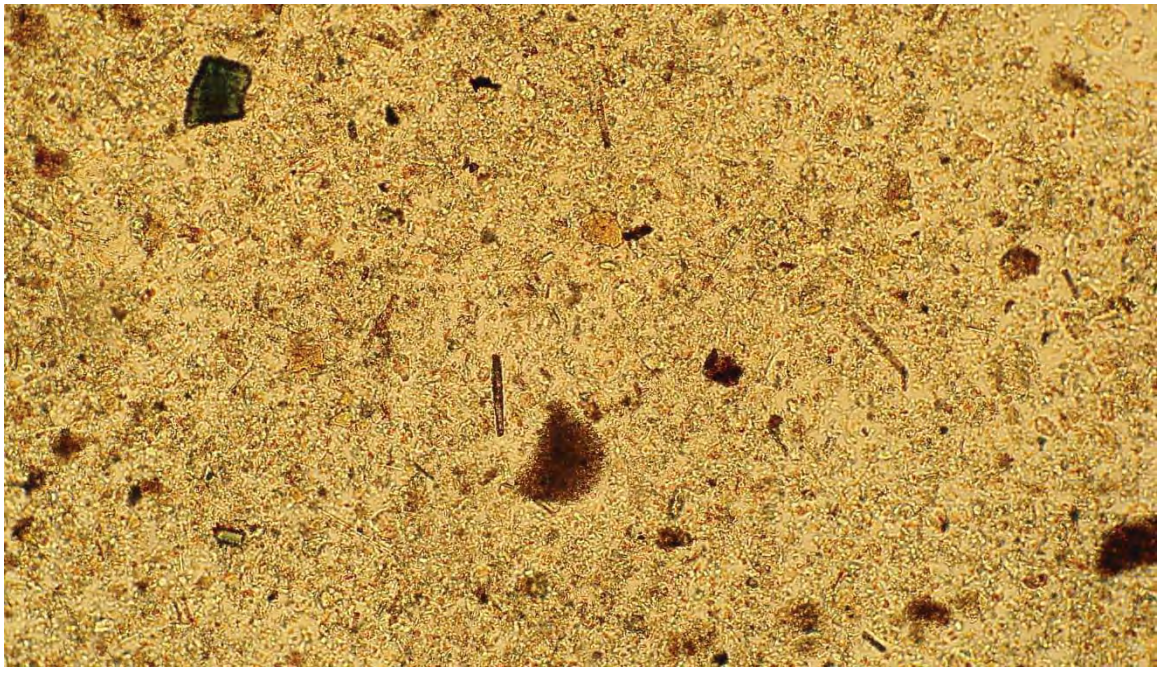




1.0% Libby Amphibole

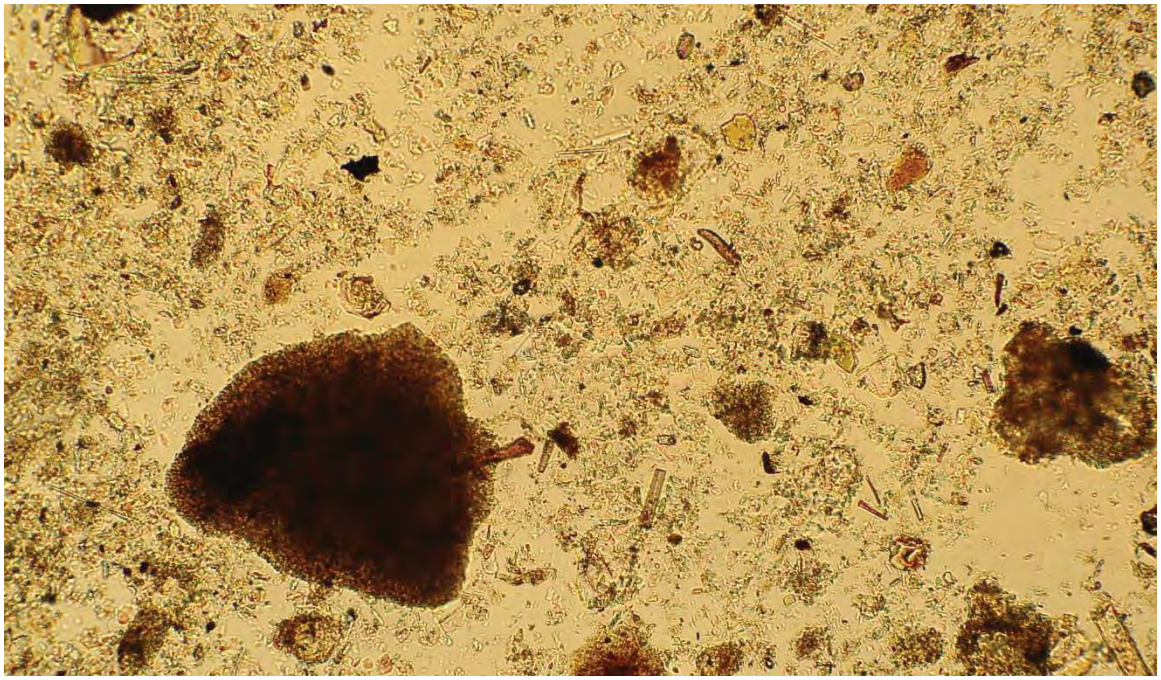
Photomicrographs of representative fields of view of the 1.0% Libby Amphibole by weight Controlled PE Reference Material. All photos taken at 100x, plane light in 1.55 refractive index oil. Width of each picture is approximately 1,500 microns.

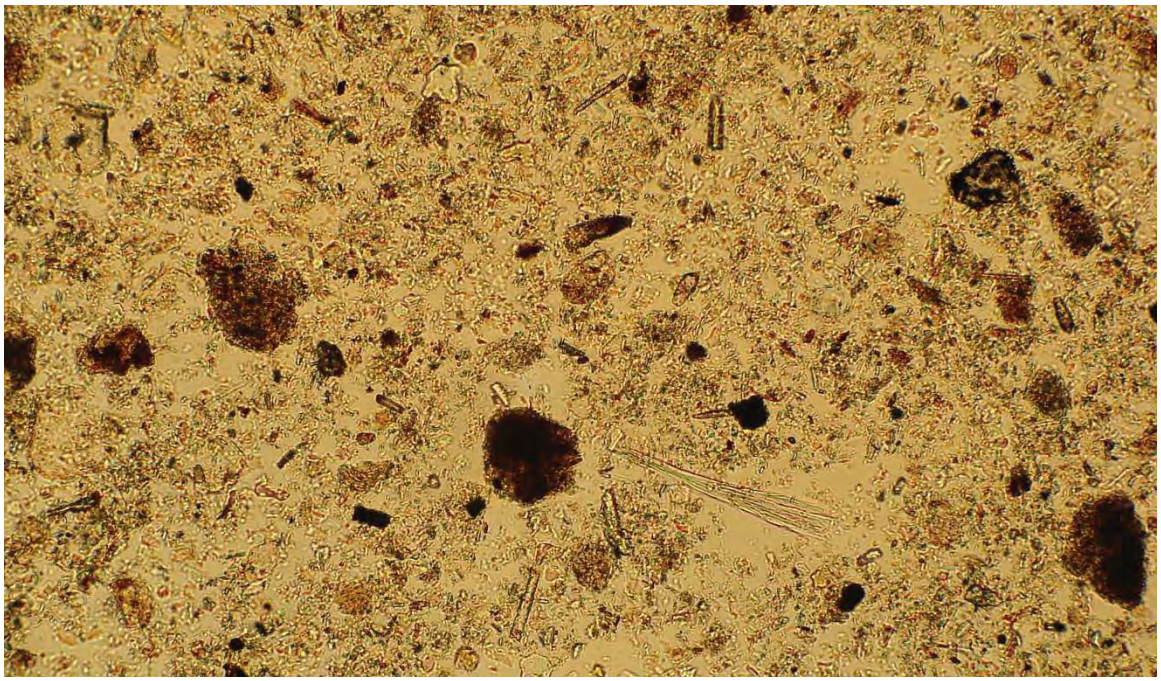




1.0% Libby Amphibole

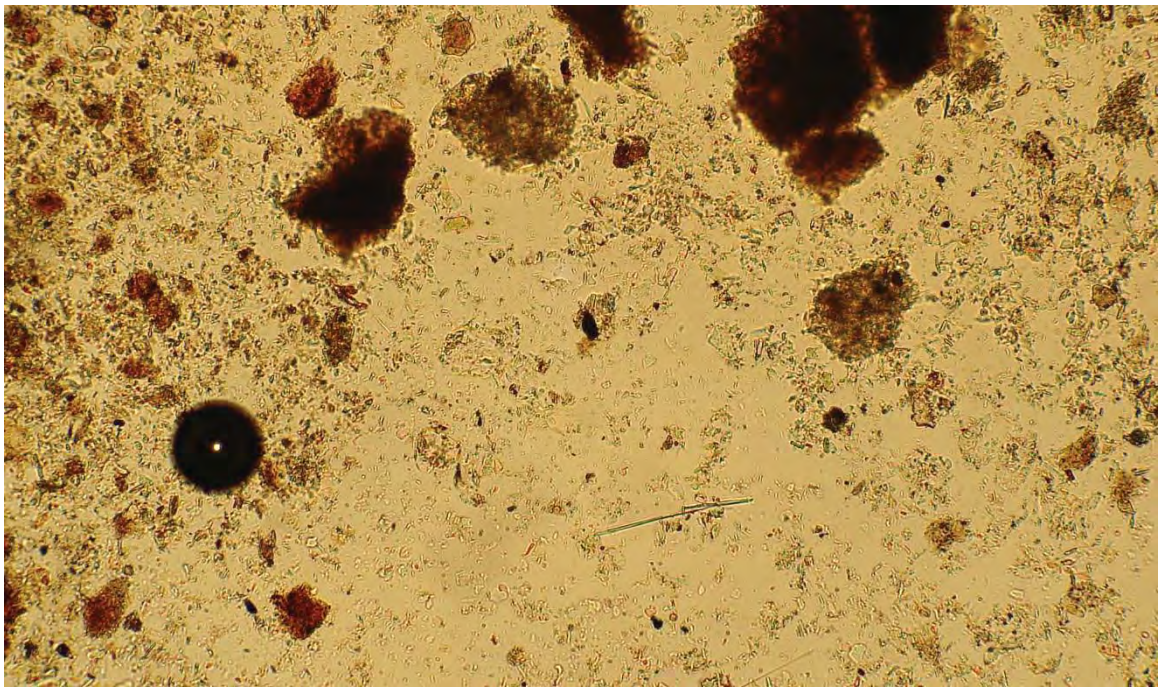
Photomicrographs of representative fields of view. Width of each picture is approximately 1,500 microns.

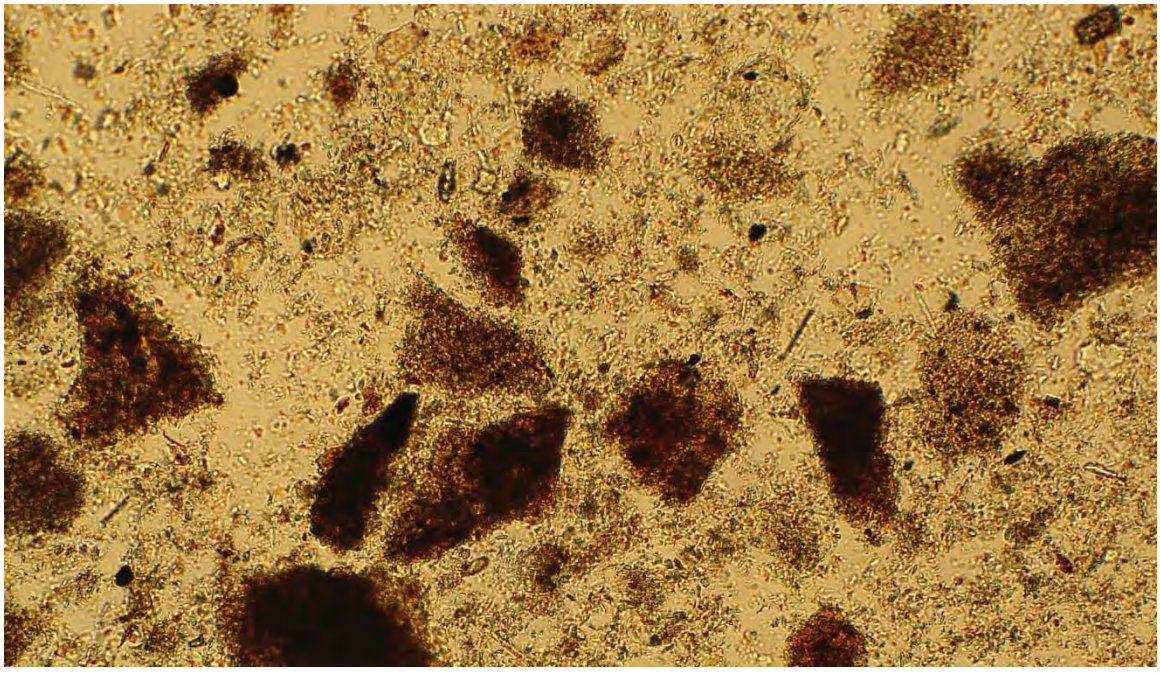




1.0% Libby Amphibole

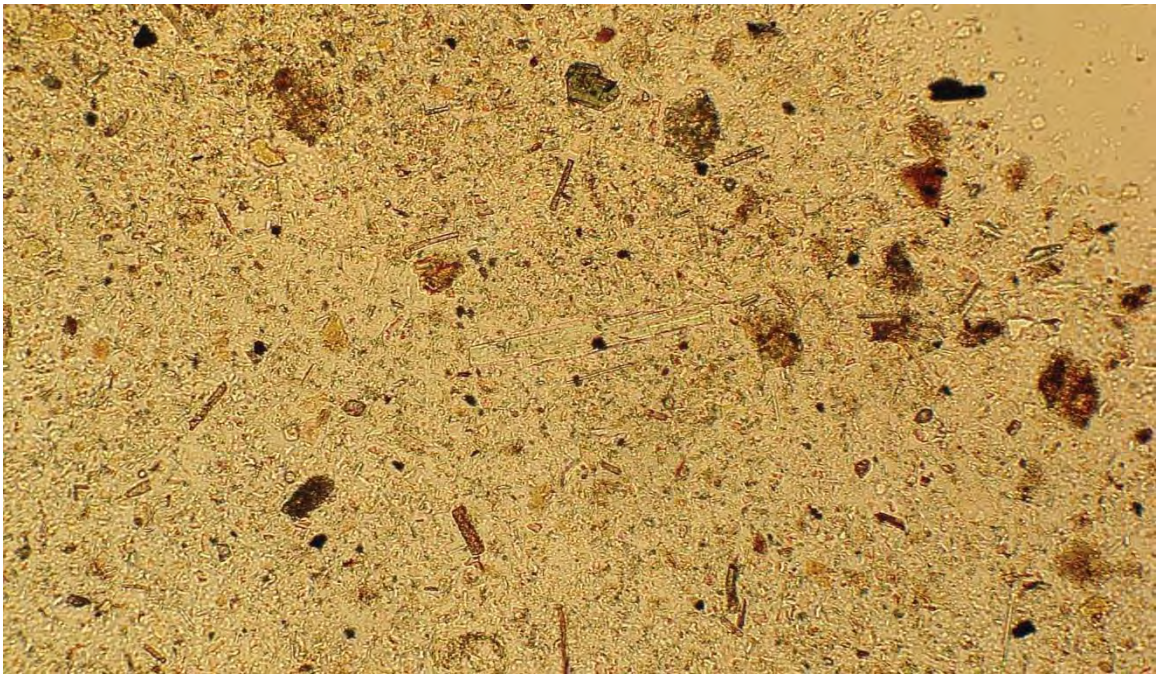
Photomicrographs of representative fields of view. Width of each picture is approximately 1,500 microns.

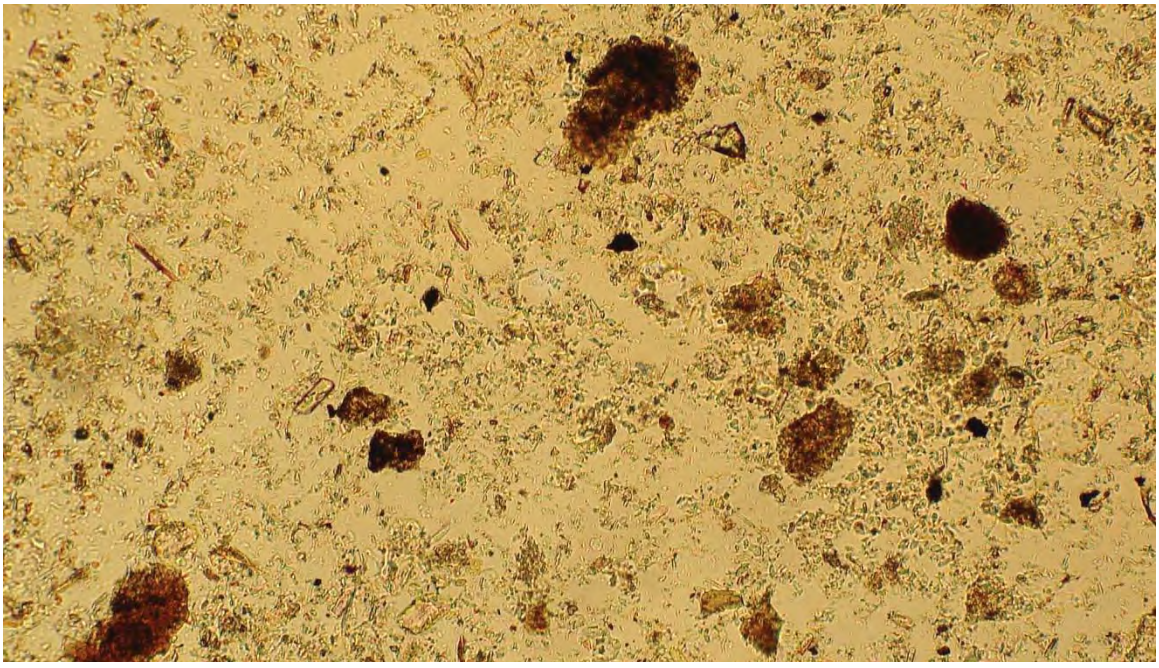




1.0% Libby Amphibole

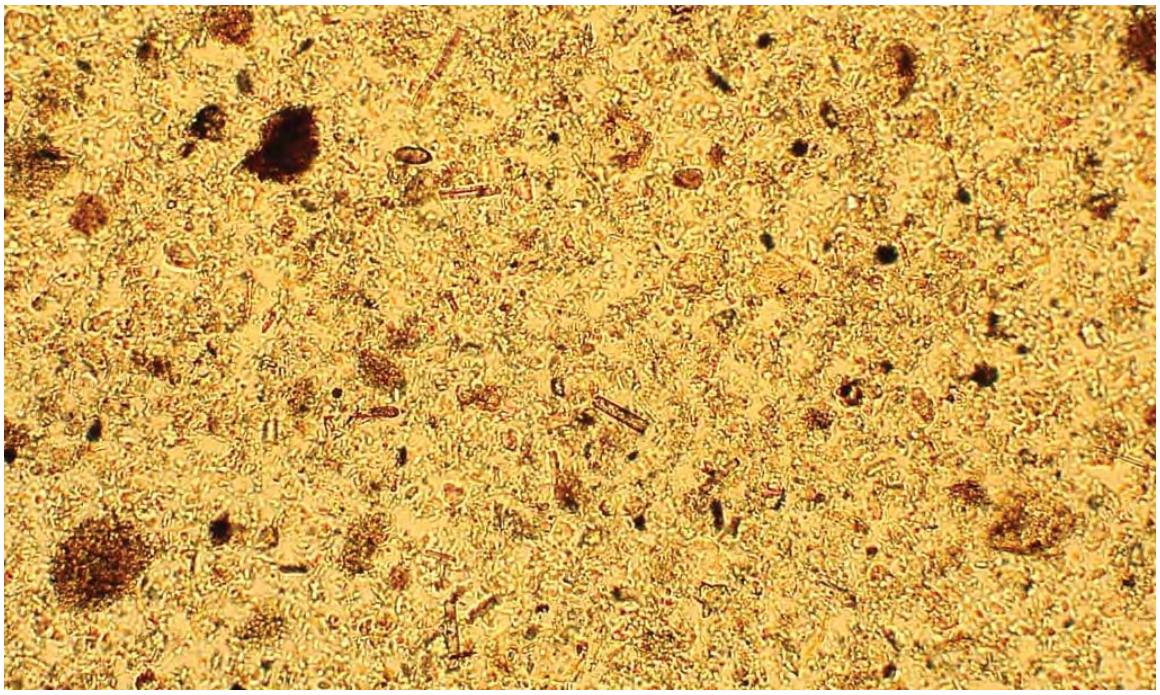
Photomicrographs of representative fields of view. Width of each picture is approximately 1,500 microns.

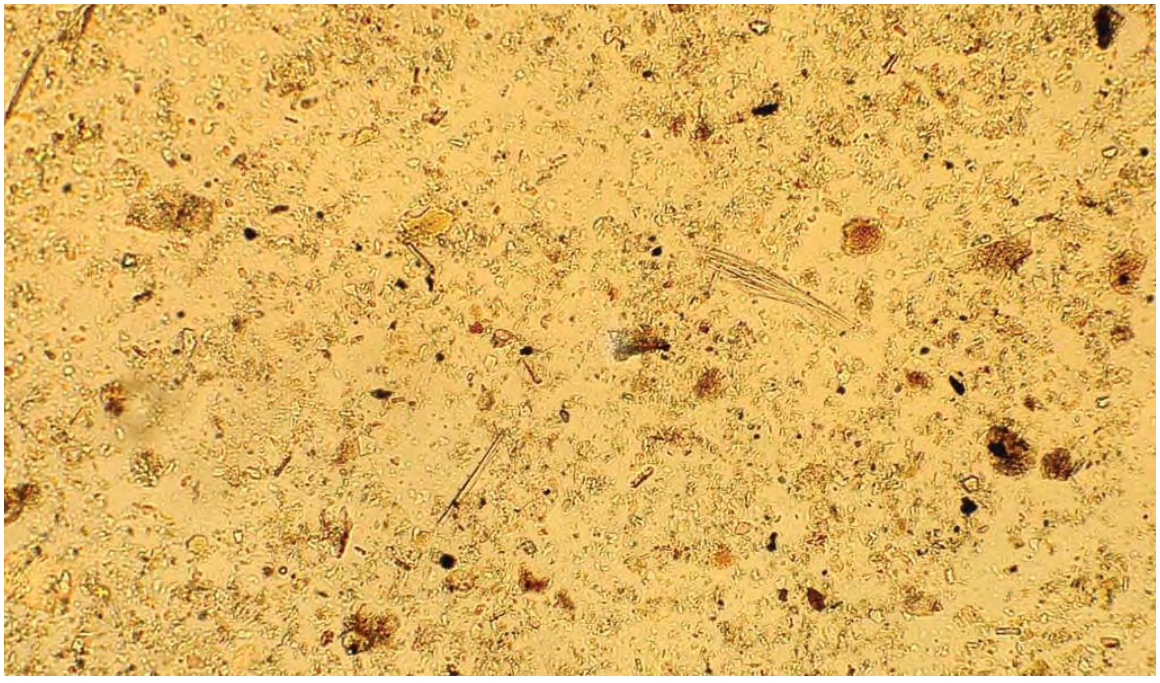




1.0% Libby Amphibole

Photomicrographs of representative fields of view. Width of each picture is approximately 1,500 microns.

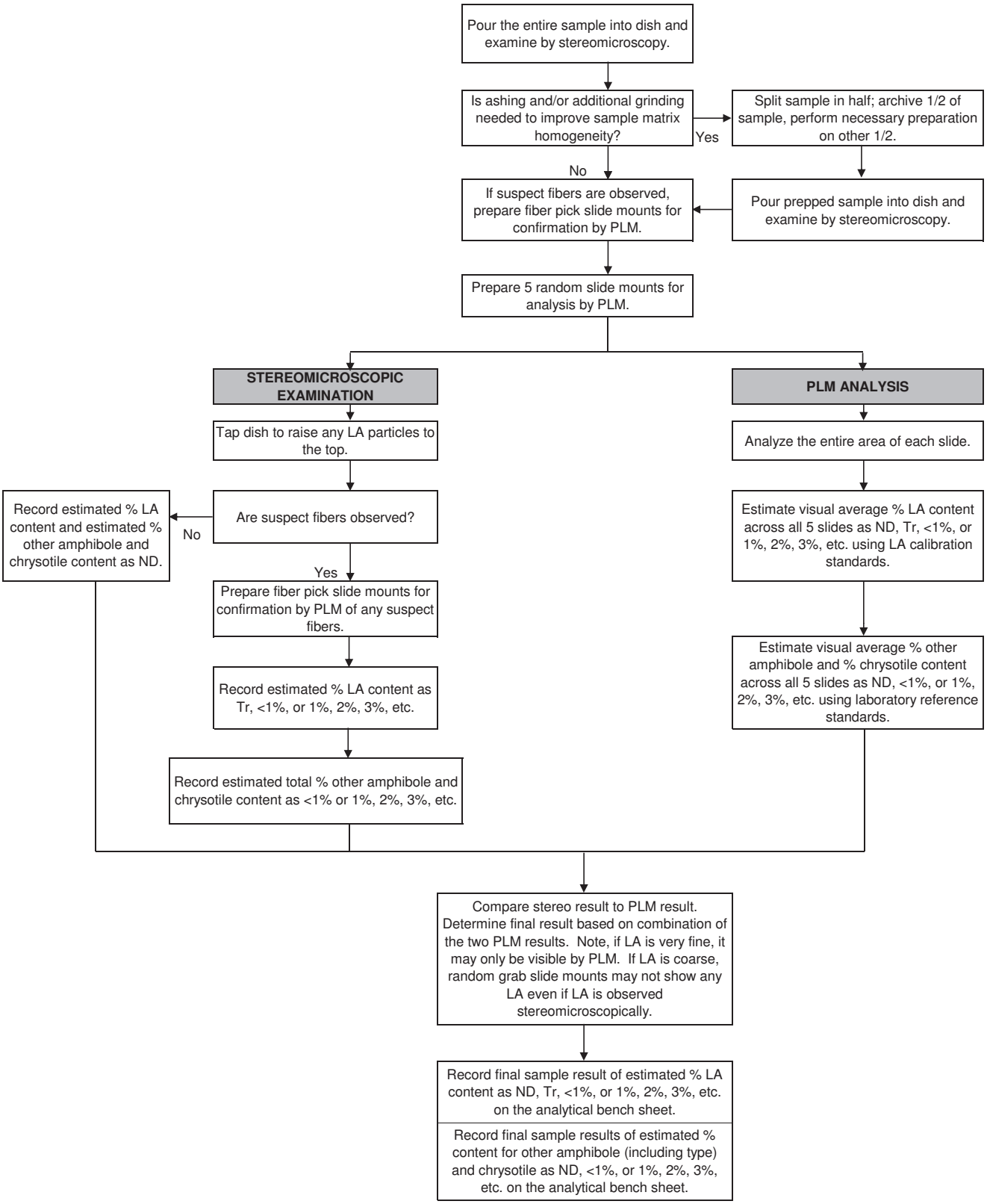




ATTACHMENT 8

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 Stereomicrosopic Examination and PLM Visual Estimation



ATTACHMENT 9

Becke Line Chart by F. D. Bloss

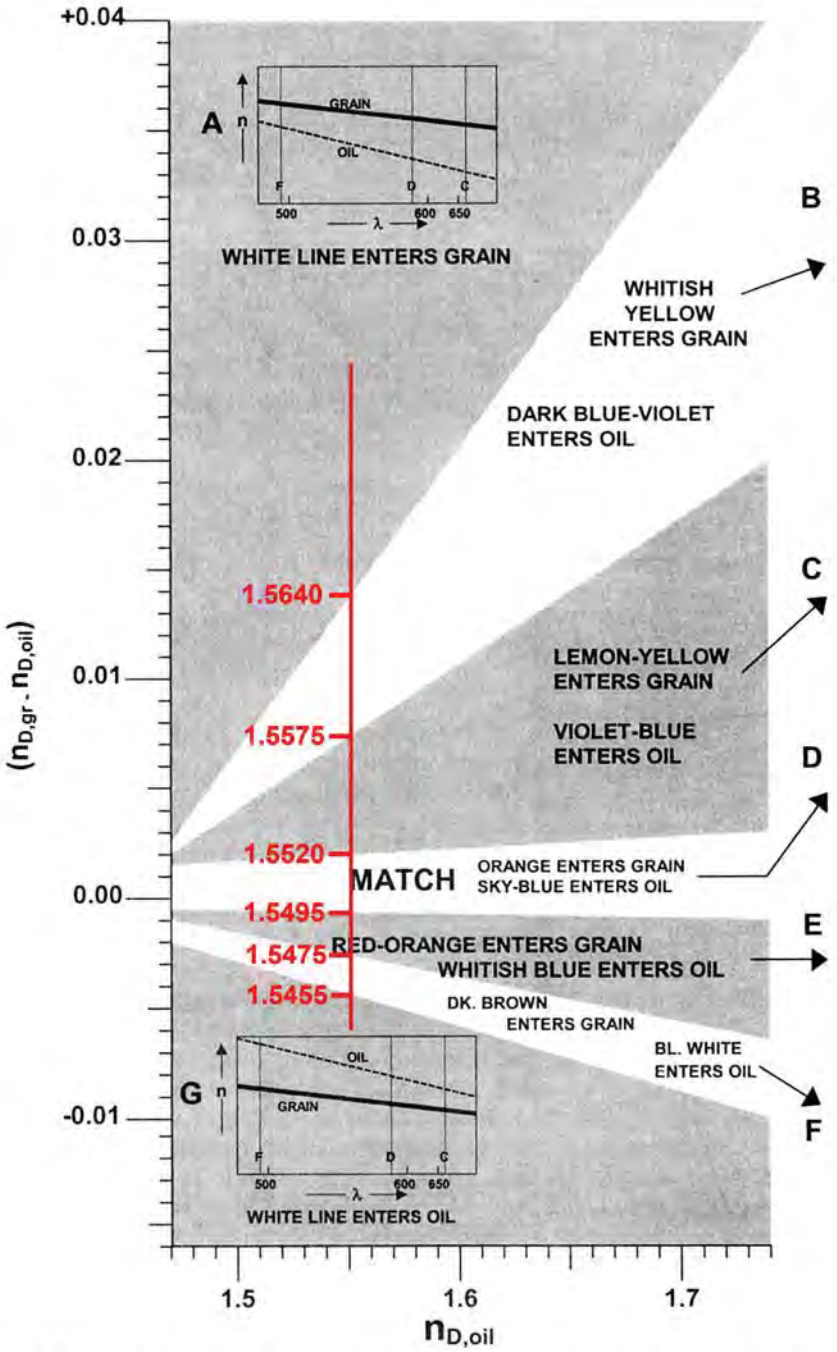


Figure 5-9. Use of the colors observed for the Becke lines or oblique illumination shadows to determine the amount by which n_D for the solid exceeds n_D for the liquid (if Cargille Series A or B liquids are being used). The Becke line movements are those observed when the objective is raised upward 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

Request for Modification
to
Laboratory Activities
LB-000073D

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.

All Labs Applicable Forms – copies to: EPA, QATS contractor, All Project Labs

Individual Labs Applicable Forms – copies to: EPA, QATS contractor, Initiating Lab

Method (circle one/those applicable): TEM-AHERA | TEM-ISO 10312 | PCM-NIOSH 7400 | EPA/600/R-93/116 | ASTM D5755 | TEM 100.2 Mod 20 | SRC-LIBBY-03 | SRC-LIBBY-01 | NIOSH 9002 | Other: _____

Revised by: Michael Lenkauskas Title: Scientist

Company: CB&I Federal Services, LLC (QATS) Date: June 22, 2015

Original Requester [LB-00073]: Lynn Woodbury, SRC Original Request Date: November 20, 2007

Original Requester [LB-00073A]: Lynn Woodbury, SRC Original Request Date: April 1, 2008

Original Requester [LB-00073B]: Lynn Woodbury, SRC Original Request Date: July 29, 2008

Original Requester [LB-00073C]: Lynn Woodbury, CDM Smith Original Request Date: December 6, 2012

Description of Modification:

The purpose of this modification is to provide permanent clarifications to inter-laboratory analyses for the Libby-specific PLM-VE (SRC-LIBBY-03) and PLM-Grav (SRC-LIBBY-01) methods. This modification standardizes the selection and analysis procedures for inter-laboratory samples of soil.

Reason for Modification:

This modification is needed to establish a standardized a real time inter-laboratory program for PLM-VE and PLM-Grav analyses across all Libby laboratories, and to ensure that all results are evaluated in real time and in accordance with a standard set of criteria.

Potential Implications of this Modification:

There are no potential negative implications resulting from this standardization of QC procedures.

Laboratory Applicability (circle one): ALL Individual(s) _____

This laboratory modification is (circle one): **NEW** **APPENDS to** _____ **SUPERSEDES** LB-000073, LB-000073A, LB-000073B, and LB-000073C

Duration of Modification (circle one):

Temporary Date(s): _____

Analytical Batch ID: _____

Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

Permanent (Complete Proposed Modification Section) Effective Date: June 22, 2015

Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

See attached.

See attached.

Data Quality Indicator (circle one) – Please reference definitions below for direction on selecting data quality indicators:

Not Applicable | Reject | Low Bias | Estimate | High Bias | **No Bias**

DATA QUALITY INDICATOR DEFINITIONS:

Reject - Samples associated with this modification form are not useable. The conditions outlined in the modification form adversely affect the associated sample to such a degree that the data are not reliable.

Low Bias - Samples associated with this modification form are useable, but results are likely to be biased low. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated low.

Estimate - Samples associated with this modification form are useable, but results should be considered approximations. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimates.

High Bias - Samples associated with this modification form are useable, but results are likely to be biased high. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated high.

No Bias - Samples associated with this modification form are useable as reported. The conditions outlined in the modification form suggest that associated sample data are reliable as reported.

Technical Review: _____ Date: 10/V15
(Laboratory Manager or designate)

Project Review and Approval: _____ Date: 11/2/15
(USEPA: Project Manager or designate)

Approved By: David L. Berry Date: 11/2/2015
(USEPA: Technical Assistance Unit Chief or designate)

Laboratory QC Type Definitions

An *inter-laboratory* analysis is a re-analysis of a soil sample (based on a second new fine ground or coarse aliquot) that is examined by an analyst from a different laboratory than who performed the initial analysis. The minimum frequency rate of inter-laboratory analyses is 1%, or about 1 per 100 analyses.

Selection Procedure for Inter-laboratory Samples

Samples for inter-laboratory comparisons will be selected in real-time by the staff at the Troy Sample Preparation Facility (SPF). Upon the selection of the 10th Sample for a given laboratory, a second Fine Ground (FG) aliquot will be shipped to a second laboratory in accordance with the Table 1 below. After the 10th sample, samples will be selected at the 100th, 200th etc. For Example, upon selection of the 10th sample for PLM-VE analysis for Lab #1, a second FG aliquot will be sent to Lab #2 and on the 100th a second FG will be sent to Lab #3.

Table 1 – PLM-VE

Originating Lab	Lab for IL Analysis #1	Lab for IL Analysis #2	Lab for IL Analysis #3	Lab for IL Analysis #4	Lab for IL Analysis #5	Lab for IL Analysis #6...
Lab #1	Lab #2	Lab #3	Lab #4	Lab #5	Lab #6	Repeat... (beginning with the Lab identified for IL Analysis #1)
Lab #2	Lab #3	Lab #4	Lab #5	Lab #6	Lab #1	
Lab #3	Lab #4	Lab #5	Lab #6	Lab #1	Lab #2	
Lab #4	Lab #5	Lab #6	Lab #1	Lab #2	Lab #3	
Lab #5	Lab #6	Lab #1	Lab #2	Lab #3	Lab #4	
Lab #6	Lab #1	Lab #2	Lab #3	Lab #4	Lab #5	

The same procedure described above for PLM-VE will also be applied for samples shipped for analysis by the PLM-Grav method, with the only difference being the PLM-Grav sample will be prepared by sieving of the associated Archive (A) Sample.

Table 2 – PLM-Grav

Originating Lab	Lab for IL Analysis #1	Lab for IL Analysis #2	Lab for IL Analysis #3	Lab for IL Analysis #4	Lab for IL Analysis #5	Lab for IL Analysis #6...
Lab #1	Lab #2	Lab #3	Lab #4	Lab #5	Lab #6	Repeat... (beginning with the Lab identified for IL Analysis #1)
Lab #2	Lab #3	Lab #4	Lab #5	Lab #6	Lab #1	
Lab #3	Lab #4	Lab #5	Lab #6	Lab #1	Lab #2	
Lab #4	Lab #5	Lab #6	Lab #1	Lab #2	Lab #3	
Lab #5	Lab #6	Lab #1	Lab #2	Lab #3	Lab #4	
Lab #6	Lab #1	Lab #2	Lab #3	Lab #4	Lab #5	

Optional post-Hoc Selection for PLM-VE

In addition to the above, to ensure a distribution of Bins B2 and C are represented, additional post-hoc samples could be selected by the EPA Quality Assurance Technical Support (QATS) contractor (or their designate) in accordance with the following procedure:

1. Inter-laboratory samples will be selected on a quarterly basis – 1st Quarter = January 1 - March 31, 2nd Quarter = April 1 - June 30, 3rd Quarter = July 1 - September 30, 4th Quarter = October 1 - December 31. Each quarter, the EPA QATS contractor (or their designate) will compile a list of all samples for which PLM-VE results were uploaded into the project database in the preceding quarter (e.g., on April 1st, the selection query would specify a date range of January 1st through March 31st). The project database query should be based on the upload date rather than the analysis date to ensure that analyses that are uploaded in a different quarter than they were analyzed are not excluded[1].

[1] Consider the case where the PLM analysis for sample X-12345 was performed on March 22 and the results were uploaded on April 3. The inter-laboratory selection query performed on April 1, if limited to all results analyzed from January 1 - March 31, would not capture the results for X-12345 because they had not yet been uploaded. The inter-

2. At a minimum, at least one sample will be selected for inter-laboratory analysis for each analytical laboratory each quarter from Bins B2 and C if samples are available:

Table 3- B2 and Selection				
Laboratory	Total # of PLM-VE Analyses		# of samples to select for Inter-lab*	
	B2	C	B2	C
Lab #1	0	0	0	0
Lab #2	0	0	0	0
Lab #3	0	1	0	1
Lab #4	0	0	0	0
Lab #5	2	0	1	0
Lab #6	7	1	1	1
Total	9	2	2	2
*calculated as Total # of analyses *0.01, rounded up to the nearest whole number.				

3. Exclude any samples that have already been selected for inter-laboratory evaluation previously.
4. Select the appropriate number of inter-laboratory samples from the available PLM-VE analyses by selecting equally from Bin B2 and C representing each analytical laboratory if samples are available with these results. In the above example (Table 3), if there are 9 Bin B2 results, and 2 Bin C results, one sample from each bin for each lab would be selected for a total of 4 inter-laboratory samples. The EPA QATS contractor (or their designate) will keep a running total of the number of samples selected by laboratory to ensure that the long-term frequency of inter-laboratory analysis for each laboratory is generally similar. A running summary of the number of samples in each bin will also be tracked to ensure that inter-laboratory samples are representative of the range of LA bins.
5. Submit the list of selected inter-laboratory analysis samples to the laboratory coordinator (LC). This list should also identify which laboratory will perform the inter-laboratory analysis in accordance with the following table:

Table 4 - Post-hoc B2 & C selection						
Originating Lab	Lab for IL Analysis #1	Lab for IL Analysis #2	Lab for IL Analysis #3	Lab for IL Analysis #4	Lab for IL Analysis #5	Lab for IL Analysis #6...
Lab #1	Lab #2	Lab #3	Lab #4	Lab #5	Lab #6	Repeat... (beginning with the Lab identified for IL Analysis #1)
Lab #2	Lab #3	Lab #4	Lab #5	Lab #6	Lab #1	
Lab #3	Lab #4	Lab #5	Lab #6	Lab #1	Lab #2	
Lab #4	Lab #5	Lab #6	Lab #1	Lab #2	Lab #3	
Lab #5	Lab #6	Lab #1	Lab #2	Lab #3	Lab #4	
Lab #6	Lab #1	Lab #2	Lab #3	Lab #4	Lab #5	

IL = inter-laboratory

6. Each quarter, the LC will provide the Troy Sample Preparation Facility (SPF) Manager with the list of samples selected for inter-laboratory analysis, and which laboratory will perform the inter-laboratory analysis for each selected sample.
7. The SPF Manager (or their designate) will obtain the appropriate fine ground aliquot (e.g., FG2) from the sample archive for each sample identified for inter-laboratory analysis (see Step 7 above) and submit this aliquot (under standard project chain of custody requirements) to the appropriate analytical laboratory for analysis by PLM-VE.

Note: Because the inter-laboratory samples are blind to the analytical laboratory, the inter-laboratory results will not be identified as "inter-laboratory" in the PLM-VE electronic data deliverable (EDD). Designation of the inter-laboratory status in the project database will need to be assigned *post hoc* by the database manager at the direction of EPA

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laboratory selection query performed on July 1, limited to all results analyzed from April 1 - June 30, would also not capture the results for sample X-12345 because the analysis date is outside of the specified range.

Evaluation of PLM-VE Inter-laboratory Analyses

Pair-wise Evaluation

The LA bin results of the IL analysis will be compared to the original analysis. Results will be ranked as concordant if both the original result and the IL result report the same semi-quantitative LA bin. Results will be ranked as weakly discordant if the original result and the inter-laboratory result differ by one semi-quantitative LA bin (e.g., Bin A vs. Bin B1). Results will be ranked as strongly discordant if the original result and the IL result differ by more than one semi-quantitative LA bin (e.g., Bin A vs. Bin B2).

In the table below, concordant pairs are shaded in dark gray and assigned a value of 0. Weakly discordant pairs are shaded in light gray and are assigned a value of -1 or +1, depending on the direction of the discordance (+1 if the original analysis reported a higher bin and -1 if the IL analysis reported a higher bin). Strongly discordant pairs are not shaded and are assigned a value of -3, -2, +2, or +3 depending upon the direction and magnitude of the difference in the reported bins.

		IL Analysis			
		Bin A	Bin B1	Bin B2	Bin C
Original Analysis	Bin A	0	-1	-2	-3
	Bin B1	+1	0	-1	-2
	Bin B2	+2	+1	0	-1
	Bin C	+3	+2	+1	0

If an individual pair is concordant or weakly discordant, no further action is warranted. If an individual pair is strongly discordant, the QATS contractor will determine the appropriate corrective action(s), in consultation with EPA, such as performing a re-examination of prepared slides (if available), analyzing a third aliquot of the archived fine ground soil, etc.

Program-wide Evaluation

The overall performance of the PLM-VE inter-laboratory program will be monitored by assembling summary statistics on inter-laboratory analyses, combining data within and across laboratories. The following tables illustrate how results may be summarized both within and across laboratories for the purposes of evaluating PLM-VE inter-laboratory results. In this table, the total number of samples is shown stratified by the reported LA bin:

		IL Laboratory (Lab #2)			
		Bin A	Bin B1	Bin B2	Bin C
Original Laboratory (Lab #1)	Bin A	66	1	0	0
	Bin B1	11	21	2	0
	Bin B2	1	7	2	1
	Bin C	0	2	0	2

In this example, 91 of 116 samples (78%) are ranked as concordant, 3 of 116 samples (3%) are ranked as strongly discordant, and 22 of 116 samples (19%) are ranked as weakly discordant. The average concordance value is calculated as follows:

$$[(91 * 0) + (4 * -1) + (18 * 1) + (3 * 2)] / 116 = 0.17$$

As shown, in this example, the original laboratory (Lab #1) has an overall tendency to be biased high relative to the IL laboratory (Lab #2). An analogous summary could be prepared in which the original laboratory (Lab #1) is compared to all other IL laboratories (i.e., Labs #2 through #6).

In this table, the average concordance value is shown by laboratory, which can be used to determine potential between laboratory differences in PLM-VE reporting:

		IL Laboratory						
		Lab #1	Lab #2	Lab #3	Lab #4	Lab #5	Lab #6	All
Original Laboratory	Lab #1		0.17					
	Lab #2							
	Lab #3							
	Lab #4							
	Lab #5							
	Lab #6							

The resulting concordance metrics will be tracked temporally for each laboratory (i.e., control charting) by the EPA QATS contractor to identify potential trends in strong and/or weak discordances or laboratory-specific biases. The program-wide goals for PLM-VE inter-laboratory samples will be interpreted as follows:

Metric	Good	Acceptable	Poor
% of inter-laboratory pairs ranked as strongly discordant	<5%	5-10%	>10%
% of inter-laboratory pairs ranked as weakly discordant	<20%	20-40%	>40%

If inter-laboratory results are ranked as good, no action is necessary. If inter-laboratory results are ranked as acceptable, the QATS contractor will investigate potential reasons for discordant results and may request corrective action(s), in consultation with EPA, such as re-training of laboratory analysts, increasing the frequency of inter-laboratory and/or performance evaluation analyses for the laboratory in question, etc. If inter-laboratory results are ranked as poor, the QATS contractor will investigate and request appropriate corrective action(s), in consultation with EPA. Corrective actions may include conducting a laboratory audit, re-training of laboratory analysts, performing a focused inter-laboratory assessment specific to that laboratory until acceptable proficiency can be demonstrated, and performing a re-analysis of field samples analyzed by the laboratory. As the project database continues to grow and we learn more, these program-wide goals may be revisited and revised. Changes to the program-wide goals will be accompanied by appropriate justification to support the change.

Evaluation of PLM-Grav Inter-laboratory Analyses

PLM-Grav samples should be evaluated as described in Section 17.2.5 of SOP SRC-LIBBY-01 (Revision 3), which states that for samples containing asbestos, LDS and LDC analyses are considered acceptable if results for both the original and QC analyses are ≤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.

Request for Modification
to
Laboratory Activities
LB-000097A

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.

All Labs Applicable Forms – copies to: EPA LC, QATS Contractor, All Project Labs

Individual Labs Applicable Forms – copies to: EPA LC, QATS Contractor, Initiating Lab

Method (circle one/those applicable): TEM-AHERA | TEM-ISO 10312 | PCM-NIOSH 7400 | EPA/600/R-93/116 | ASTM D5755 | TEM 100.2 Mod 20 | SRC-LIBBY-03 | SRC-LIBBY-01 | NIOSH 9002 | Other: _____

Requester: Talena Oliver Title: Senior PLM Analyst

Company: ECC/ESAT Region 8 Date: October 22, 2014

Original Requester [LB-000097]: Talena Oliver, ECC/ESAT Region 8 Original Request Date: June 5, 2014

Description of Modification:

The following items are procedures that must be followed by all laboratories using the PLM-VE SOP SRC-LIBBY-03, Revision 3, for Libby soil analysis.

1. The SOP does not specify what type of dish to pour the sample into, other than it must be an asbestos-free substrate. Moving forward, the sample dish must have a minimum diameter of 100mm and must be a low-form porcelain dish. The minimum diameter and low-form will ensure that the soil sample within is shallow enough to allow coarse particles to move to the surface during agitation, is not too deep which allows for ample soil surface area to be examined, and should be easily handled on any stereomicroscope. The porcelain substrate will allow for aggressive agitation of the sample within. Section 13.3 of the SOP mentions the use of a mortar and pestle to break up any large grains in the subsamples. Moving forward it is recommended, but not required, that all subsamples be gently ground in a mortar and pestle to ensure a more uniform particle size for even distribution and a thinner layer of sample material beneath each cover slip.

2. If non-disposable sample dishes and/or a mortar and pestle are being used during sample preparation, a daily contamination check must be performed by each analyst in each hood by preparing a non-asbestos material (such as fiberglass insulation, gypsum, etc.) using all equipment previously used that day.

3. Currently, the SOP does not state when the PLM-VE LDS slides should be made (during the original sample analysis by making 10 cover slips instead of 5, or as a completely new analysis). In order to test the soils for reproducibility, the LDS sample must be a complete re-preparation of the soil from start to finish. After completing the stereomicroscopic portion of the analysis and the prep hood is cleaned and ready for a new sample, the original sample must again be poured into the sample dish, and a complete stereomicroscopic examination must be performed and a new set of slides made, including any fiber picks. The original sample slides must be treated and analyzed separately from the LDS slides; fiber picks may not be shared between the two sets of slides. Results must be reported independently; what is seen during either stereomicroscopic examination of the original sample or analysis of the original slides must not be considered when reporting results for the LDS sample, and vice versa.

4. The current procedure for a PLM-VE LDC QC sample is for a second analyst to examine the original slides read by the original analyst. While this provides a QC measure on the original analysts' ability to observe and identify LA, it does not test the reproducibility of the entire procedure. The current LDC procedures will remain in place; however, they will be performed at a frequency of 4%, decreased from the previous 8%. A new type of LDC, called Lab Duplicate Cross-check RePrep (LDCR), will be performed at a frequency of

4%, keeping the overall LDC QC frequency at 8%. The LDCR will be a complete re-preparation and re-analysis of the original sample by a second analyst. As with the LDS QC samples, the original analysis and the LDC analysis must be completed independently of each other and their results must be reported separately.

5. In the case that a PLM-VE QC analysis is strongly discordant with the original analysis (i.e. results different by more than one bin category), a third analysis (Reconciliation, or RC) must be conducted by both the original and QC analysts working together to determine the "final" LA concentration. If necessary, additional analysts may be included in the RC analysis. When reporting the results, include the following analyses in the EDD: original ("NOT QC"); QC ("LDC" or "LDCR"); reconciliation ("RC"). It does not matter which analyst officially reports the RC analysis, so long as they are involved with the re-analysis.

6. It is important that the valid values for the optical property *Habit* are being used by all laboratories in the same manner, but these habits ("Acicular", "Fiber Bundles", "Prismatic", "Straight", and "Tapered") are not specifically defined in either the PLM-VE SOP or the EPA/600/R-93/116 method. However, the term "asbestiform" is defined in detail in the EPA method. To avoid creating definitions for all of the current *Habit* valid values (including length to width ratios for each), LA *Habit* will be recorded as either "Asbestiform" or "Non-Asbestiform" following the EPA method definition. For CH and OA, laboratories may use any habit at their discretion, because optical properties for CH and OA are only recorded on the analytical bench sheet and not included in the EDD uploaded to Scribe.

7. Section 3 of the SOP states that a first order red (550 nanometer) compensator plate is required equipment. However, in Section 5.5.3 of the NVLAP 150-3 checklist, the first order red compensator plate may range from 530 – 550nm retardation. Laboratories may use any plate that falls within the range dictated by NVLAP.

8. Section 16.4.4 of the SOP indicates that laboratories with only one primary analyst must send all LDC samples to another Libby laboratory for analysis. Moving forward, an effort will be made by the Lab Coordinator (LC) to not use laboratories that only have one analyst except in extenuating circumstances, which must be approved by the LC prior to any samples being shipped to that laboratory.

Reason for Modification:

Procedures need to be clarified in the SOP, as some sections leave room for interpretation by the analyst, and all laboratories must use the same procedures for all steps of the analysis in order for the results to be more comparable. The LDC QC samples were not testing the reproducibility of the method, only the repeatability of results produced from the same slides by two different analysts. In the case of strongly discordant results, laboratories must have a means of recording original, QC and reconciliation results.

Potential Implications of this Modification:

Improved QC procedures and consistency in preparation and analysis practices between laboratories.

Laboratory Applicability (circle one): ALL Individual(s) _____

This laboratory modification is (circle one): NEW APPENDS to _____ SUPERSEDES LB-00097

Duration of Modification (circle one):

Temporary Date(s): _____

Analytical Batch ID: _____

Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

Permanent (Complete Proposed Modification Section) Effective Date: December 17, 2014

Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of Method when applicable):

See Description of Modification section above.

REFERENCES

Data Quality Indicator (circle one) – Please reference definitions below for direction on selecting data quality indicators:

Not Applicable **Reject** **Low Bias** **Estimate** **High Bias** No Bias

DATA QUALITY INDICATOR DEFINITIONS:

Reject - Samples associated with this modification form are not useable. The conditions outlined in the modification form adversely affect the associated sample to such a degree that the data are not reliable.

Low Bias - Samples associated with this modification form are useable, but results are likely to be biased low. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated low.

Estimate - Samples associated with this modification form are useable, but results should be considered approximations. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimates.

High Bias - Samples associated with this modification form are useable, but results are likely to be biased high. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated high.

No Bias - Samples associated with this modification form are useable as reported. The conditions outlined in the modification form suggest that associated sample data are reliable as reported.

Technical Review: _____ Date: 12/16/2014
(Laboratory Manager or designate)

Project Review and Approval: _____ Date: 12/16/14
(USEPA: Project Manager or designate)

Approved By: _____ Date: 12/16/2014
(USEPA: Technical Assistance Unit Chief or designate)

Request for Modification
to
Laboratory Activities
LB-000098

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.

All Labs Applicable Forms – copies to: EPA LC, QATS Contractor, All Project Labs

Individual Labs Applicable Forms – copies to: EPA LC, QATS Contractor, Initiating Lab

Method (circle one/those applicable):	TEM-AHERA	TEM-ISO 10312	PCM-NIOSH 7400
	EPA/600/R-93/116	ASTM D5755	TEM 100.2 Mod 20	SRC-LIBBY-03
	SRC-LIBBY-01	NIOSH 9002	Other:_____	

Requester: Talena Oliver Title: Lead PLM Analyst

Company: ECC/ESAT Region 8 Date: January 7, 2014

Description of Modification:

The following items are procedures for all laboratories analyzing soils by the PLM-Grav SOP SRC-LIBBY-01, Revision 3.

1. Currently, Section 17 of the SOP states that an LDS and LDC consist of a complete re-weighing and re-examination of the original sample by the original analyst for LDS, or a second analyst for LDC. However, it is often the case that when asbestos (LA or otherwise) is observed in the coarse fraction, there is only a trace amount in the form of a single fiber bundle which is fiber picked for positive identification by PLM. During the original analysis, if a fiber is picked and verified as asbestos, it will be saved and used for the LDS or LDC sample (as there is likely no more asbestos in the sample to pick, and if there is more, it should remain in the same to be parted out and weighed). If there is no fiber picked in the original analysis, but there is a positively identified fiber pick in either the LDS or LDC, this will not be used to modify the original results and will only be used for the QC result.

2. Section 17.2.4 of the SOP indicates that laboratories with only one primary analyst must send all LDC samples to another Libby laboratory for analysis. Moving forward, an effort will be made by the Lab Coordinator (LC) to not use laboratories that only have one analyst except in extenuating circumstances, which must be approved by the LC prior to any samples being shipped to that laboratory.

Reason for Modification:

Procedures need to be clarified in the SOP, as some sections leave room for interpretation by the analyst, and all laboratories must use the same procedures for all steps of the analysis in order for the results to be more comparable.

Potential Implications of this Modification:

Improved QC procedures and consistency in analytical practices between laboratories.

Laboratory Applicability (circle one): ALL Individual(s) ______________________

This laboratory modification is (circle one): NEW APPENDS to ________ SUPERCEDES ________

Duration of Modification (circle one):

Temporary Date(s): ______________________

Analytical Batch ID ______________________

Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

Permanent (Complete Proposed Modification Section) Effective Date: 03/05/2014 TO, ESAT R8

Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts

Proposed Modification to Method (attach additional sheets if necessary, state section and page numbers of Method when applicable):

See Description of Modification section above.

REFERENCES

Data Quality Indicator (circle one) – Please reference definitions below for direction on selecting data quality indicators.

Not Applicable Reject Low Bias Estimate High Bias **No Bias**

DATA QUALITY INDICATOR DEFINITIONS:

Reject - Samples associated with this modification form are not useable. The conditions outlined in the modification form adversely affect the associated sample to such a degree that the data are not reliable.

Low Bias - Samples associated with this modification form are useable, but results are likely to be biased low. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated low.

Estimate - Samples associated with this modification form are useable, but results should be considered approximations. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimates.

High Bias - Samples associated with this modification form are useable, but results are likely to be biased high. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated high.

No Bias - Samples associated with this modification form are useable as reported. The conditions outlined in the modification form suggest that associated sample data are reliable as reported.

Technical Review: ______________________ Date: [illegible]/2014

(Laboratory Manager or designee)

Project Review and Approval: ______________________ Date: 3/5/2014

(CSETS Project Manager or designee)

Approved By: ______________________ Date: 03/05/2014

(USEPA, Technical Assistance Unit Chief or designee)

Request for Modification
to
Laboratory Activities
LB-000103

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.

All Labs Applicable Forms – copies to: EPA LC, QATS contractor, All Project Labs

Individual Labs Applicable Forms – copies to: EPA LC, QATS contractor, Initiating Lab

Method (circle all applicable):

	TEM-AHERA	TEM-ISO 10312	PCM-NIOSH 7400
EPA/600/R-93/116	ASTM 5755	TEM 100.2	SRC-LIBBY-03
SRC-LIBBY-01	NIOSH 9002	Other:	

Requester: Robyn Ray Title: Special Proj. Manager, EMSL

Company: EMSL Analytical, Inc. Date: 28 May 2015

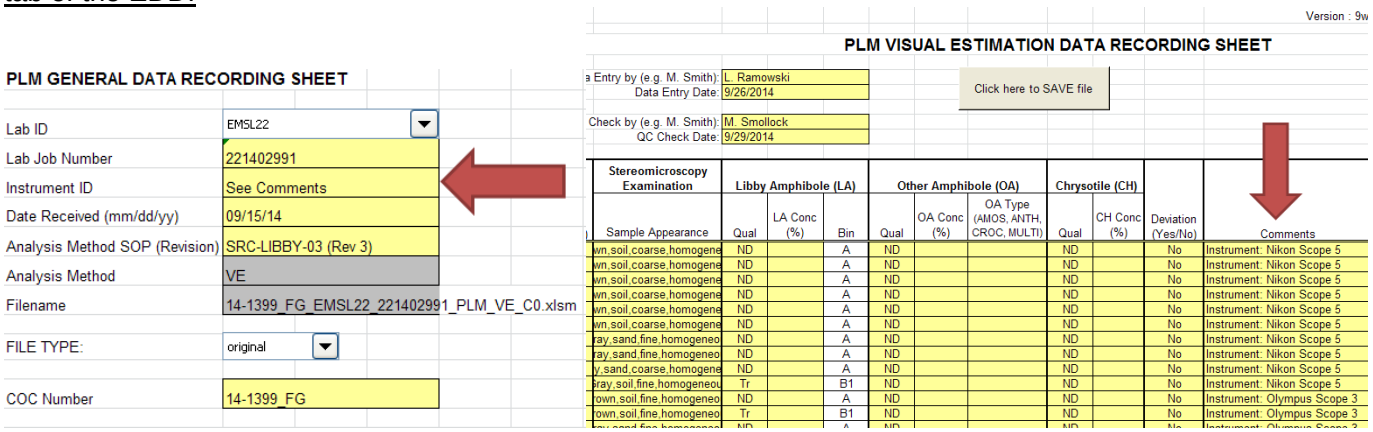
Original Requester: NA Original Request Date: NA

[only applicable if modification is a revision of an earlier modification]

Description of Modification:

There are times when a lab could use two PLM microscopes for one PLM job. Currently the microscope used is tracked at the order ID level. However, if multiple scopes are being used tracking would need to be done at the Sample level to ensure the traceability of the microscope calibrations. In order to document the use of multiple microscopes the labs must:

- For Field Samples- Put “See Comments” in the “Instrument ID” field, on the “General data entry” tab of the EDD. Then record the microscope number on the sample line comments section on the ”Visual Data Entry” tab of the EDD.



PLM GENERAL DATA RECORDING SHEET

Lab ID	EMSL22
Lab Job Number	221402991
Instrument ID	See Comments
Date Received (mm/dd/yy)	09/15/14
Analysis Method SOP (Revision)	SRC-LIBBY-03 (Rev 3)
Analysis Method	VE
Filename	14-1399_FG_EMSL22_221402991_PLM_VE_C0.xlsm
FILE TYPE:	original
COC Number	14-1399_FG

Version : 9w

PLM VISUAL ESTIMATION DATA RECORDING SHEET

Data Entry by (e.g. M. Smith): L. Ramowski
Data Entry Date: 9/26/2014
Click here to SAVE file
Check by (e.g. M. Smith): M. Smollock
QC Check Date: 9/29/2014

Stereomicroscopy Examination	Libby Amphibole (LA)			Other Amphibole (OA)			Chrysotile (CH)			
Sample Appearance	Qual	LA Conc (%)	Bin	Qual	OA Conc (%)	OA Type (AMOS, ANTH, CROC, MULTI)	Qual	CH Conc (%)	Deviation (Yes/No)	Comments
wn,soil,coarse,homogene	ND		A	ND			ND		No	Instrument: Nikon Scope 5
wn,soil,coarse,homogene	ND		A	ND			ND		No	Instrument: Nikon Scope 5
wn,soil,coarse,homogene	ND		A	ND			ND		No	Instrument: Nikon Scope 5
wn,soil,coarse,homogene	ND		A	ND			ND		No	Instrument: Nikon Scope 5
wn,soil,coarse,homogene	ND		A	ND			ND		No	Instrument: Nikon Scope 5
wn,soil,coarse,homogene	ND		A	ND			ND		No	Instrument: Nikon Scope 5
ray,sand,fine,homogeneo	ND		A	ND			ND		No	Instrument: Nikon Scope 5
ray,sand,fine,homogeneo	ND		A	ND			ND		No	Instrument: Nikon Scope 5
y,sand,coarse,homogene	ND		A	ND			ND		No	Instrument: Nikon Scope 5
Gray,soil,fine,homogeneou	Tr		B1	ND			ND		No	Instrument: Nikon Scope 5
rown,soil,fine,homogeneo	ND		A	ND			ND		No	Instrument: Olympus Scope 3
rown,soil,fine,homogeneo	Tr		B1	ND			ND		No	Instrument: Olympus Scope 3

- For QC samples- when only one microscope was used for the field analysis, it is understood that it would be a different scope and the lab need only to record the instrument ID in the sample line comments section on the ”Visual Data Entry” tab of the EDD.

Reason for Modification:

To ensure that data validators have all necessary microscope information when validating calibrations.

Potential Implications of this Modification:
There are no potential implications of this modification.

Laboratory Applicability (circle one): **All** **Individual(s)** _____

This laboratory modification is (circle one): **NEW** **APPENDS to** _____ **SUPERCEDES** _____

Duration of Modification (circle one):

Temporary Date(s): _____
Analytical Batch ID: _____

Temporary Modification Forms – Attach legible copies of approved form with all associated raw data packages

Permanent (Complete Proposed Modification Section) Effective Date: 28 May 2015

Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Proposed Modification to Method (attach additional sheets if necessary; state section and page numbers of method when applicable):
See above.

REFERENCES

None

Data Quality Indicator (circle one) – Please reference definitions below for direction on selecting data quality indicators:

Not Applicable **Reject** **Low Bias** **Estimate** **High Bias** **No Bias**

DATA QUALITY INDICATOR DEFINITIONS:

Reject - Samples associated with this modification form are not useable. The conditions outlined in the modification form adversely affect the associated sample to such a degree that the data are not reliable.

Low Bias - Samples associated with this modification form are useable, but results are likely to be biased low. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated low.

Estimate - Samples associated with this modification form are useable, but results should be considered approximations. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimates.

High Bias - Samples associated with this modification form are useable, but results are likely to be biased high. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated high.

No Bias - Samples associated with this modification form are useable as reported. The conditions outlined in the modification form suggest that associated sample data are reliable as reported.

Technical Review: _____ Date: 6/16/205
(Laboratory Manager or designate)

Project Review and Approval: _____ Date: 9/10/2015
(USEPA: Project Manager or designate)

Approved By: _____ Date: 9/10/2015
(USEPA: Technical Assistance Unit Chief or designate)

United States
Environmental Protection
Agency

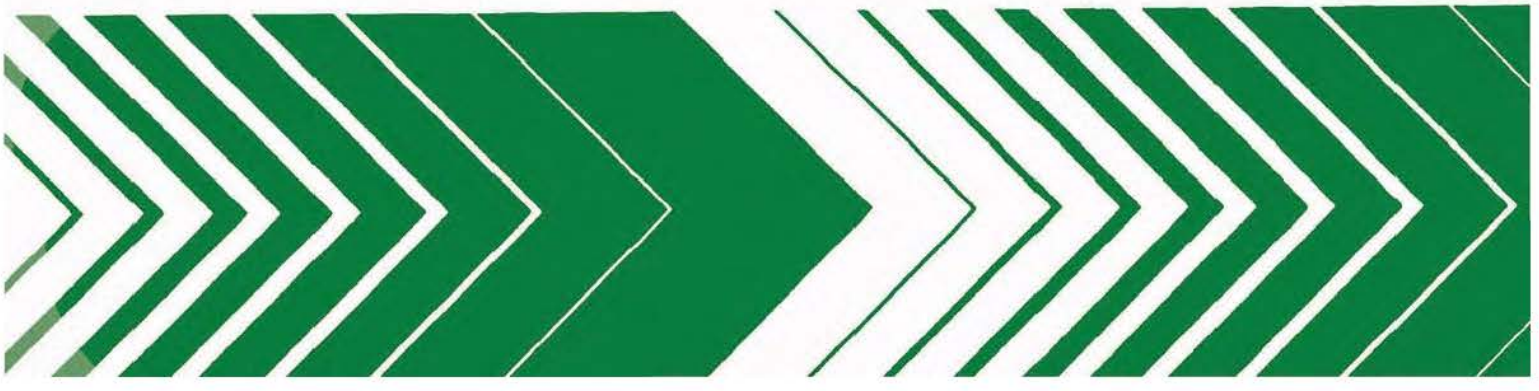
Office of Research and
Development
Washington, DC 20460

EPA/600/R-93/116
July 1993

EPA

Test Method

Method for the Determination of Asbestos in Bulk Building Materials



EPA/600/R-93/116
July 1993

TEST METHOD

METHOD FOR THE DETERMINATION OF ASBESTOS IN BULK BUILDING MATERIALS

by

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DISCLAIMER

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TABLE OF CONTENTS

TABLE OF CONTENTS (cont'd)

SECTION PAGE

TABLE OF CONTENTS (cont'd)

TABLES

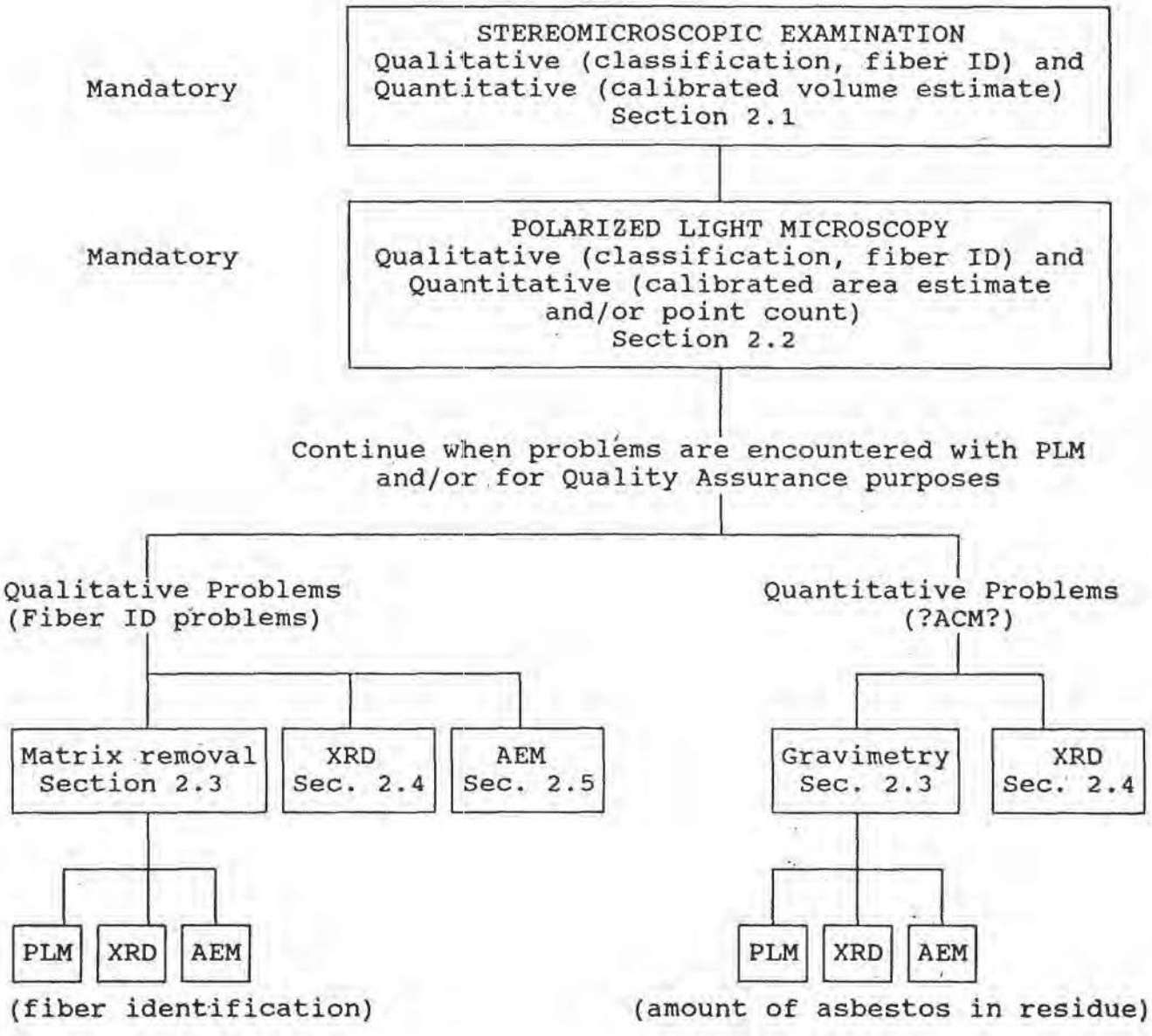
1.0 INTRODUCTION

Laboratories are now called upon to identify asbestos in a variety of bulk building materials, including loose-fill insulations, acoustic and thermal sprays, pipe and boiler wraps, plasters, paints, flooring products, roofing materials and cementitious products.

The diversity of bulk materials necessitates the use of several different methods of sample preparation and analysis. An analysis with a simple stereomicroscope is always followed by a polarized light microscopic (PLM) analysis. The results of these analyses are generally sufficient for identification and quantitation of major concentrations of asbestos. However, during these stereomicroscopic and PLM analyses, it may be found that additional techniques are needed to: 1) attain a positive identification of asbestos; 2) attain a reasonable accuracy for the quantity of asbestos in the sample; or 3) perform quality assurance activities to characterize a laboratory's performance. The additional techniques include x-ray diffraction (XRD), analytical electron microscopy (AEM), and gravimetry, for which there are sections included in the method. Other techniques will be considered by the Environmental Protection Agency (EPA) and may be added at some future time. Table 1-1 presents a simplified flowchart for analysis of bulk materials.

This Method for the Determination of Asbestos in Bulk Building Materials outlines the applicability of the various preparation and analysis methods to the broad spectrum of bulk building materials now being analyzed. This method has been evaluated by the EPA Atmospheric Research and Exposure Assessment Laboratory (EPA/AREAL) to determine if it offers improvements to current analytical techniques for building materials. This method demonstrated a capability for improving the precision and accuracy of analytical results. It contains significant revisions to procedures outlined in the Interim Method,[1] along with the addition of several new procedures. Each technique may reduce or introduce bias, or have some effect on the precision of the measurement, therefore results need to be interpreted judiciously. Data on each technique, especially those new to asbestos analysis, will be collected over time and carefully evaluated, with resulting recommendations for changes to the Method to be passed on to the appropriate program office within EPA.

TABLE 1-1. SIMPLIFIED FLOWCHART FOR ANALYSIS OF BULK MATERIALS



This is an analytical method. It is not intended to cover bulk material sampling, an area addressed previously[2,3,4,5] by the EPA. However, subsampling or sample splitting as it pertains to laboratory analysis procedures in this method, is discussed throughout.

1.1 References

1. **Interim Method for the Determination of Asbestos in Bulk Insulation Samples,** U.S. E.P.A. 600/M4-82-020, 1982.
2. **Asbestos-Containing Materials in School Buildings: A Guidance Document,** Part 1 and 2, U.S. E.P.A./O.T.S NO. C00090, 1979.
3. **Asbestos in Buildings: Simplified Sampling Scheme for Friable Surfacing Materials,** U.S. E.P.A. 560/5-85-030a, 1985.
4. **Guidance for Controlling Asbestos-Containing Materials in Buildings,** U.S. E.P.A. 560/5-85-024, 1985.
5. **Asbestos-Containing Materials in Schools: Final Rule and Notice,** 40 CFR Part 763, October, 1987.

2.0 METHODS

2.1 Stereomicroscopic Examination

A preliminary visual examination using a simple stereomicroscope is mandatory for all samples. A sample should be of sufficient size to provide for an adequate examination. For many samples, observations on homogeneity, preliminary fiber identification and semi-quantitation of constituents can be made at this point. Another method of identification and semi-quantitation of asbestos must be used in conjunction with the stereomicroscopic examination. A description of the suggested apparatus needed for stereomicroscopic examination is given in Appendix B.

The laboratory should note any samples of insufficient volume. A sufficient sample volume is sample-type dependent. For samples such as floor tiles, roofing felts, paper insulation, etc., three to four square inches of the layered material would be a preferred sample size. For materials such as ceiling tiles, loose-fill insulation, pipe insulation, etc., a sample size of approximately one cubic inch ($\sim$ 15cc) would be preferred. For samples of thin-coating materials such as paints, mastics, spray plasters, tapes, etc., a smaller sample

size may be suitable for analysis. Generally, samples of insufficient volume **should be rejected**, and further analysis curtailed until the client is contacted. The quantity of sample affects the sensitivity of the analysis and reliability of the quantitation steps. If there is a question whether the sample is representative due to inhomogeneity, the sample should be rejected, at least until contacting the client to see if: 1) the client can provide more material or 2) the client wishes the laboratory to go ahead with the analysis, but with the laboratory including a statement on the limited sensitivity and reliability of quantitation. If the latter is the case, the report of analysis should state that the client was contacted, that the client decided that the lab should use less material than recommended by the method, and that the client acknowledges that this may have limited the sensitivity and quantitation of the method. At the time the client is contacted about the material, he or she should be informed that a statement reflecting these facts will be placed in the report.

2.1.1 Applicability

Stereomicroscopic analysis is applicable to all samples, although its use with vinyl floor tile, asphaltic products, etc., may be limited because of small asbestos fiber size and/or the presence of interfering components. It does not provide positive identification of asbestos.

2.1.2 Range

Asbestos may be detected at concentrations less than one percent by volume, but this detection is highly material dependent.

2.1.3 Interferences

Detection of possible asbestos fibers may be made more difficult by the presence of other nonasbestos fibrous components such as cellulose, fiber glass, etc., by binder/matrix materials which may mask or obscure fibrous components, and/or by exposure to conditions (acid environment, high temperature, etc.) capable of altering or transforming asbestos.

2.1.4 Precision and Accuracy

The precision and accuracy of these estimations are material dependent and must be determined by the individual laboratory for the percent range involved. These values may be

determined for an individual analyst by the in-house preparation and analysis of standards and the use of error bars, control charts, etc.

The labs should also compare to National Voluntary Laboratory Accreditation Program (NVLAP) proficiency testing samples, if the lab participates in the Bulk Asbestos NVLAP, or to external quality assurance system consensus results such as from proficiency testing programs using characterized materials. However, at this time, consensus values for the quantity of asbestos have been shown to be unreliable. Only proficiency testing materials characterized by multiple techniques should be used to determine accuracy and precision.

2.1.5 Procedures

NOTE: Exposure to airborne asbestos fibers is a health hazard. Bulk samples submitted for analysis are oftentimes friable and may release fibers during handling or matrix reduction steps. All sample handling and examination must be carried out in a HEPA-filtered hood, a class 1 biohazard hood or a glove box with continuous airflow (negative pressure). Handling of samples without these precautions may result in exposure of the analyst to and contamination of samples by airborne fibers.

2.1.5.1 Sample Preparation

No sample preparation should be undertaken before initial stereomicroscopic examination. Distinct changes in texture or color on a stereomicroscopic scale that might denote an uneven distribution of components should be noted. When a sample consists of two or more distinct layers or building materials, each should be treated as a separate sample, when possible. Thin coatings of paint, rust, mastic, etc., that cannot be separated from the sample without compromising the layer are an exception to this case and may be included with the layer to which they are attached. Drying (by heat lamp, warm plate, etc.) of wet or damp samples is recommended before further stereomicroscopic examination and is mandatory before PLM examination. **Drying must be done in a safety hood.**

For nonlayered materials that are heterogeneous, homogenization by some means (mill, blender, mortar and pestle) may provide a more even distribution of sample components. It

may also facilitate disaggregation of clumps and removal of binder from fibers (rarely however, it may mask fibers that were originally discernable).

For materials such as cementitious products and floor tiles, breaking, pulverizing, or grinding may improve the likelihood of exposing fibrous components.

It may be appropriate to treat some materials by dissolution with hydrochloric acid to remove binder/matrix materials. Components such as calcite, gypsum, magnesite, etc., may be removed by this method. For materials found to possess a high organic content (cellulose, organic binders), ashing by means of a muffle furnace or plasma asher (for small, cellulosic samples), or dissolution by solvents may be used to remove interfering material. In either case, it is recommended that matrix removal be tracked gravimetrically.

Additional information concerning homogenization, ashing and acid dissolution may be found in Sections 2.2.5.1 and 2.3.

2.1.5.2 Analysis

Samples should be examined with a simple stereomicroscope by viewing multiple fields of view over the entire sample. The whole sample should be observed after placement in a suitable container (watchglass, weigh boat, etc.) substrate. Samples that are very large should be subsampled. The sample should be probed, by turning pieces over and breaking open large clumps. The purpose of the stereomicroscopic analysis is to determine homogeneity, texture, friability, color, and the extent of fibrous components of the sample. This information should then be used as a guide to the selection of further, more definitive qualitative and quantitative asbestos analysis methods. Homogeneity refers to whether each subsample made for other analytical techniques (e.g. the "pinch" mount used for the PLM analysis), is likely to be similar or dissimilar. Color can be used to help determine homogeneity, whether the sample has become wet (rust color), and to help identify or clarify sample labelling confusion between the building material sampler and the laboratory. Texture refers to size, shape and arrangement of sample components. Friability may be indicated by the ease with which the sample is disaggregated (see definitions in Appendix A) as received by the analyst. This does not necessarily represent the friability of the material as determined by the assessor at the collection site. The relative proportion of fibrous

components to binder/matrix material may be determined by comparison to similar materials of known fibrous content. For materials composed of distinct layers or two or more distinct building materials, each layer or distinct building material should be treated as a discrete sample. The relative proportion of each in the sample should be recorded. The layers or materials should then be separated and analyzed individually. Analysis results for each layer or distinct building material should be reported. If monitoring requirements call for one reported value, the results for the individual layers or materials should always be reported along with the combined value. Each layer or material should be checked for homogeneity during the stereomicroscopic analysis to determine the extent of sample preparation and homogenization necessary for successful PLM or other analysis. Fibers and other components should be removed for further qualitative PLM examination.

Using the information from the stereomicroscopic examination, selection of additional preparation and analytical procedures should be made. Stereomicroscopic examination should typically be performed again after any change or major preparation (ashing, acid dissolution, milling, etc.) to the sample. Stereomicroscopic examination for estimation of asbestos content may also be performed again after the qualitative techniques have clarified the identities of the various fibrous components to assist in resolving differences between the initial quantitative estimates made during the stereomicroscopic analysis and those of subsequent techniques. Calibration of analysts by use of materials of known asbestos content is essential.

The stereomicroscopic examination is often an iterative process. Initial examination and estimates of asbestos concentration should be made. The sample should then be analyzed by PLM and possibly other techniques. These results should be compared to the initial stereomicroscopic results. Where necessary, disagreements between results of the techniques should be resolved by reanalyzing the sample stereomicroscopically.

2.1.6 Calibration Materials

Calibration materials fall into several categories, including internal laboratory standards and other materials that have known asbestos weight percent content. These calibration materials could include:

- Actual bulk samples: asbestos-containing materials that have been characterized by other analytical methods such as XRD, AEM and/or gravimetry. (e.g. NVLAP test samples).

- Generated samples: in-house standards that can be prepared by mixing known quantities of asbestos and known quantities of asbestos-free matrix materials (by weight), and mixing (using blender, mill, etc.) thoroughly to achieve homogeneity; matrix materials such as vermiculite, perlite, sand, fiberglass, calcium carbonate, etc. may be used. A range of asbestos concentrations should be prepared (e.g. 1, 3, 5, 10, 20%, etc.). The relationship between specific gravities of the components used in standards should be considered so that weight/volume relationships may be determined.

- Photographs, drawings: photomicrographs of standards, computer-generated drawings, etc.

Suggested techniques for the preparation and use of in-house calibration standards are presented in Appendix C, and at greater length by Harvey et al.[1] The use of synthesized standards for analyst calibration and internal laboratory quality control is not new however, having been outlined by Webber et al.[2] in 1982.

2.1.7 References

1. Harvey, B. W., R. L. Perkins, J. G. Nickerson, A. J. Newland and M. E. Beard, "Formulating Bulk Asbestos Standards", **Asbestos Issues**, April 1991, pp. 22-29.

2. Webber, J. S., A. Pupons and J. M. Fleser, "Quality-Control Testing for Asbestos Analysis with Synthetic Bulk Materials". **American Industrial Hygiene Associations Journal**, 43, 1982, pp. 427-431.

2.2 Polarized Light Microscopy

2.2.1 Principle and Applicability

Samples of bulk building materials taken for asbestos identification should first be examined with the simple stereomicroscope to determine homogeneity and preliminary fiber identification. Subsamples should then be examined using PLM to determine optical properties of constituents and to provide positive identification of suspect fibers.

The principles of optical mineralogy are well-established.[1,2,3,4] A light microscope equipped with two polarizing filters is used to observe specific optical characteristics of a sample. The use of plane polarized light allows for the determination of refractive indices relative to specific crystallographic orientations. Morphology and color are also observed while viewing under plane polarized light. Observation of particles or fibers while oriented between polarizing filters whose privileged vibration directions are perpendicular (crossed polars) allows for determination of isotropism/anisotropism, extinction characteristics of anisotropic particles, and calculation of birefringence. A retardation plate may be placed in the polarized light path for verification of the sign of elongation. If subsamples are prepared in such a way as to represent all sample components and not just suspect fibers, semi-quantitative analysis may also be performed. Semi-quantitative analysis involves the use of calibrated visual area estimation and/or point counting. Visual area estimation is a semi-quantitative method that must relate back to calibration materials. Point counting, also semi-quantitative, is a standard technique used in petrography for determining the relative areas occupied by separate minerals in thin sections of rock. Background information on the use of point counting[3] and the interpretation of point count data[5] is available.

Although PLM analysis is the primary technique used for asbestos determination, it can show significant bias leading to false negatives and false positives for certain types of materials. PLM is limited by the visibility of the asbestos fibers. In some samples the fibers may be reduced to a diameter so small or masked by coatings to such an extent that they cannot be reliably observed or identified using PLM.

2.2.2 Range

The detection limit for visual estimation is a function of the quantity of sample analyzed, the nature of matrix interference, sample preparation, and fiber size and distribution. Asbestos may be detected in concentrations of less than one percent by area if sufficient material is analyzed. Since floor tiles may contain fibers too small to be resolved by PLM (< 0.25 μm in diameter), detection of those fibers by this method may not be possible. When point counting is used, the detection limit is directly proportional to the amount of sample analyzed, but is also limited by fiber visibility. Quantitation by area estimation, both visual and by point counting, should yield similar results if based on calibration standards.

2.2.3 Interferences

Fibrous and nonfibrous, organic and inorganic constituents of bulk samples may interfere with the identification and quantitation of the asbestos mineral content. Binder/matrix materials may coat fibers, affect color, or obscure optical characteristics to the extent of masking fiber identity. Many organic mastics are soluble in refractive index liquids and, unless removed prior to PLM examination, may affect the refractive index measurement of constituent materials. Fine particles of other materials may also adhere to fibers to an extent sufficient to cause confusion in identification. Gravimetric procedures for the removal of interfering materials are presented in Section 2.3.

2.2.4 Precision and Accuracy

Data obtained for samples containing a single asbestos type in a sample matrix have been reported previously by Brantley et al.[6] Data for establishing the accuracy and precision of the method for samples with various matrices have recently become available. Perkins,[7] Webber et al.[8] and Harvey et al.[9] have each documented the tendency for visual estimates to be high when compared to point-count data. Precision and accuracy must be determined by the individual laboratory for the percent range involved. If point counting and/or visual estimates are used, a table of reasonably expanded errors, such as those shown in Table 2-1, should be generated for different concentrations of asbestos.

If the laboratory cannot demonstrate adequate precision and accuracy (documented by control charts, etc), quantitation by additional methods, such as gravimetry, may be required. Refer to the Handbook for SRM Users[10] for additional information concerning the concepts of precision and accuracy.

TABLE 2-1. SUGGESTED ACCEPTABLE ERRORS FOR PLM ANALYSIS
(Based on 400 point counts of a reasonably homogeneous sample or 100 fields of view for visual estimate)

% Area Asbestos	Acceptable Mean Result	% Area Asbestos	Acceptable Mean Result
1	>0-3%	50	40-60%
5	>1-9%	60	50-70%
10	5-15%	70	60-80%
20	10-30%	80	70-90%
30	20-40%	90	80-100%
40	30-50%	100	90-100%

2.2.5 Procedures

NOTE: Exposure to airborne asbestos fibers is a health hazard. Bulk samples submitted for analysis are oftentimes friable and may release fibers during handling or matrix reduction steps. All sample and slide preparations must be carried out in a HEPA-filtered, a class 1 biohazard hood, or a glove box with continuous airflow (negative pressure). Handling of samples without these precautions may result in exposure of the analyst to and contamination of samples by airborne fibers.

2.2.5.1 Sample Preparation

Slide mounts are prepared for the identification and quantitation of asbestos in the sample.

2.2.5.1.1 Qualitative Analysis Preparation

The qualitative preparation must allow the PLM analysis to classify the fibrous components of the sample as asbestos or nonasbestos. The major goal of the qualitative

preparation is to mount easily visible fibers in appropriate refractive index liquids for complete optical characterization. Often this can be accomplished by making immersion grain mounts of random subsamples of the homogeneous material. Immersion liquids with refractive indices close to the suspected (see stereomicroscopic analysis) asbestos mineral should be used for the qualitative analysis so that n_D can be determined. Problem samples include those with inhomogeneities, coatings, small fibers, and interfering compounds. Additional qualitative preparations are often necessary for these types of samples. All samples, but especially those lacking homogeneity, may require picking of fibers from specific sample areas during the stereomicroscopic examination. Coatings on the fibers often need to be removed by mechanical or chemical means. Teasing the particles apart or use of a mortar and pestle or similar mechanical method often is sufficient to free fibers from coatings. Chemical means of removing some coatings and interfering compounds are discussed in Section 2.3, Gravimetry.

2.2.5.1.2 Quantitative Analysis Preparation

The major purpose of the quantitative preparation is to provide the analyst with a representative grain mount of the sample in which the asbestos can be observed and distinguished from the nonasbestos matrix. This is typically performed by using randomly selected subsamples from a homogeneous sample (see stereomicroscopic analysis). Particles should be mounted in a refractive index (RI) liquid that allows the asbestos to be visible and distinguished from nonasbestos components. Care should be taken to ensure proper loading and even distribution of particles. Both the qualitative and quantitative sample preparations are often iterative processes. Initial samples are prepared and analyzed. The PLM analysis may disclose problems or raise questions that can only be resolved by further preparations (e.g. through the use of different RI immersion liquids, elimination of interfering compounds, sample homogenization, etc.)

For layered materials, subsamples should be taken from each individual or discrete layer. Each of these subsamples should be treated as a discrete sample, but as stated in Section 2.1.5.2, the results for the individual layers or materials may be combined if called for by monitoring requirements.

Homogenization involves the use of any of a variety of devices, such as a mortar and pestle, mill, or blender to pulverize, disaggregate and mix heterogeneous, friable bulk materials. Selection of the appropriate device is dependent upon personal preference and the nature of the materials encountered. A blender or mortar and pestle may be adequate for homogenizing materials that lack appreciable amounts of tacky matrix/binder, and for separating interfering components from the fibers. For materials which are unusually sticky or tacky, or contain unusually long asbestos fibers, milling (especially freezer milling) may be more efficient. However, milling should be discontinued as soon as the material being milled appears homogeneous, in order to reduce the potential for mechanically reducing fiber size below the resolving power of the polarizing microscope. Hammer mills or cutting mills may also be used on these materials; however, the same precaution regarding reduction of fiber size should be taken. Blending /milling devices should be disassembled (to the extent possible) and thoroughly cleaned after each use to minimize contamination.

2.2.5.2 Analysis

Analysis of bulk building materials consists of the identification and semi-quantitation of the asbestos type(s) present, along with the identification, where possible, of fibrous nonasbestos materials, mineral components and matrix materials. If the sample is heterogeneous due to the presence of discrete layers or two or more distinct building materials, each layer or distinct material should be analyzed, and results reported. Total asbestos content may also be stated in terms of a relative percentage of the total sample.

2.2.5.2.1 Identification

Positive identification of asbestos requires the determination of the following optical properties:

- Morphology
- Color and, if present, pleochroism
- Refractive indices ($\pm$ .005)
- Birefringence
- Extinction characteristics
- Sign of elongation

Descriptions of the optical properties listed above for asbestos fibers may be found in Appendix A, Glossary of Terms. Table 2-2 lists the above properties for the six types of asbestos and Table 2-3 presents the central stop dispersion staining colors for the asbestos minerals with selected high-dispersion index liquids. Tables 2-4 and 2-5 list selected optical properties of several mineral and man-made fibers. All fibrous materials in amounts greater than trace should be identified as asbestos or nonasbestos, with all optical properties measured for asbestos and at least one optical property measured for each nonasbestos fibrous component that will distinguish each from asbestos. Small fiber size and/or binder may necessitate viewing the sample at higher magnification (400-500x) than routinely used (100x).

Although it is not the purpose of this section to explain the principles of optical mineralogy, some discussion of the determination of refractive indices is warranted due to its importance to the proper identification of the asbestos minerals. Following is a brief discussion of refractive index determination for the asbestos minerals.

All asbestos minerals are anisotropic, meaning that they exhibit different optical properties (including indices of refraction) in different directions. All asbestos minerals are biaxial, meaning that they have one principal refractive index parallel (or nearly parallel) to the length of the fiber and two principal refractive indices (plus all intermediate indices between these two) in the plane perpendicular (or nearly so) to the length of the fiber. Although chrysotile (serpentine) is classified as a biaxial mineral, it behaves as a uniaxial mineral (two principal refractive indices) due to its scrolled structure. Amosite and crocidolite, although also biaxial, exhibit uniaxial properties due to twinning of the crystal structure and/or random orientation of fibrils in a bundle around the long axis of the bundle. For all of the asbestos minerals except crocidolite, the highest refractive index (γ) is aligned with the fiber length (positive sign of elongation). For crocidolite, the lowest refractive index (α) is aligned with the fiber length (negative sign of elongation). A more complete explanation of the relationship of refractive indices to the crystallographic directions of the asbestos minerals may be found in References 1, 2, 4, 11 and 12. It should be noted that for the measurement of refractive indices in an anisotropic particle (e.g. asbestos fibers), the orientation of the particle is quite critical. Orientation with respect to rotation about the axis

of the microscope (and thus with respect to the vibration directions of the polarizer and analyzer) and also to the horizontal plane (plane of the microscope stage) will affect the determination of the correct values for refractive indices. The refractive index that is measured will always correspond to a direction perpendicular to the axis of the microscope (i.e., lying in the plane of the stage) and is the direction in that horizontal plane parallel to the vibration direction of the polarizer, by convention E-W.

To determine $\gamma(n\parallel)$ for chrysotile, anthophyllite and amosite, the index is measured when the length of the fiber is aligned parallel to the vibration direction of the polarizer (E-W). Under crossed polars, the fiber should be at extinction in this orientation. To determine the lowest refractive index, α $(n\perp)$, for chrysotile and amosite, the fiber should be oriented N-S (extinction position under crossed polars). The determination of $n\parallel$ and $n\perp$ with crocidolite is accomplished in the same manner as with amosite and chrysotile with the exception that the α and γ directions are reversed. For crocidolite, α is measured at the E-W position (parallel to the polarizer) and γ is measured at the N-S orientation (perpendicular to the polarizer). For anthophyllite, the fiber should be oriented N-S and the lowest and highest indices for this orientation should be measured. These correspond to α and β respectively.

The extinction behavior of tremolite-actinolite is anomalous compared to that of most monoclinic minerals due to the orientation of the optic axes relative to the crystallographic axes. This relationship is such that the refractive indices of the principal axes α and γ <u>are not measured</u> when the fiber is exhibiting the maximum extinction angle. The values measured at these positions are α' and γ' The fiber exhibits an extinction angle within a few degrees of the maximum throughout most of its rotation. A wide range of refractive indices from α' to α, and from γ' to γ, are observed. For tremolite-actinolite, β is measured on those fibers displaying parallel extinction when oriented in the N-S position. The refractive index for α is also measured when the fiber is oriented generally in the N-S position and exhibits <u>the true extinction</u> angle; true α will be the <u>minimum</u> index. To determine the refractive index for γ, the fibers should be oriented E-W and exhibit the true extinction angle; true γ will be the <u>maximum</u> value for this orientation.

When viewing single fibers, the analyst may often be able to manipulate the microscope slide cover slip and "roll" the fibers to positions that facilitate measuring the true values of refractive indices. When viewing a large population of fibers with the microscope in the dispersion staining mode, the analyst can easily detect fibers that exhibit the highest and lowest indices (β and α) in the N-S position and the highest indices (γ) in the E-W position. Since individual asbestos fibrils cannot generally be resolved using polarized light microscopy, refractive indices are most commonly measured on fiber bundles. Such measurements would not result in true values for the indices and therefore by convention should be reported as α' and γ'.

Asbestos types chrysotile, amosite and crocidolite are currently available as SRM 1866 and actinolite, tremolite and anthophyllite as SRM 1867 from the Office of Standard Reference Materials, National Institute of Standards and Technology.

2.2.5.2.2 Quantitation of Asbestos Content

As described in Sections 2.1.5 and 2.1.6, a calibrated visual volume estimation of the relative concentrations of asbestos and nonasbestos components should be made during the stereomicroscopic examination. In addition, quantitation of asbestos content should be performed on subsample slide mounts using calibrated visual area estimates and/or a point counting procedure. Section 2.1.6 and Appendix C discuss the procedures for preparation and use of calibration standards. After thorough PLM analysis in which the asbestos and other components of the bulk material are identified, several slides should be carefully prepared from randomly selected subsamples. If the sample is not homogeneous, some homogenization procedure should be performed to ensure that slide preparations made from small pinch samples are representative of the total sample. Homogenization may range from gentle mixing using a mortar and pestle to a brief period of mixing using a blender equipped with a mini-sample container. The homogenization should be of short duration ($\sim$ 15 seconds) if using the blender technique so as to preclude a significant reduction in fiber size. The use of large cover slips (22x30mm) allows for large subsamples to be analyzed. Each slide should be checked to ensure that the subsample is representative, uniformly dispersed, and loaded in a way so as not to be dominated by superimposed (overlapping) particles.

During the qualitative analysis of the sample, the analyst should decide on the appropriate optical system (including magnification) to maximize the visibility of the asbestos in the sample while still allowing the asbestos to be uniquely distinguished from the matrix materials. The analyst may choose to alter the mounting medium or the optical system to enhance contrast. During the quantitative analysis, slides should be scanned using an optical setup that yields the best visibility of the asbestos. Upon finding asbestos, the parameters that were selected in the qualitative analysis for uniquely distinguishing it from the matrix should be used for identification. These properties will vary with the sample but include any or all of the parameters required for the qualitative analysis. For instance, low magnification allows for concurrent use of dispersion staining (focal screening), but compromises resolution of extremely small diameter fibers; use of a compensator plate and crossed polarizers frequently enhances the contrast between asbestos fibers and matrix material.

Visual area estimates should be made by comparison of the sample to calibration materials that have similar textures and fiber abundance (see Section 2.1.6 and Appendix C). A minimum of three slide mounts should be examined to determine the asbestos content by visual area estimation. Each slide should be scanned in its entirety and the relative proportions of asbestos and nonasbestos noted. It is suggested that the ratio of asbestos to nonasbestos material be recorded for several fields for each slide and the results be compared to data derived from the analysis of calibration materials having similar textures and asbestos content.

For point counting, an ocular reticle (cross-line or point array) should be used to visually superimpose a point or points on the microscope field of view. The cross-line reticle is preferred. Its use requires the scanning of most, if not all, of the slide area, thereby minimizing bias that might result from lack of homogeneity in the slide preparation. In conjunction with this reticle, a click-stop counting stage can be used to preclude introducing bias during slide advancement. Magnification used will be dictated by fiber visibility. The slide should be examined along multiple parallel traverses that adequately cover the sample area. The analyst should score (count) only points directly over occupied (nonempty) areas. Empty points should not be scored on the basis of the closest particle. If an asbestos fiber and a nonasbestos particle overlap so that a point is superimposed on their visual intersection,

a point should be scored for both categories. If the point(s) is/are superimposed on an area which has several overlapping particles, the slide should be moved to another field. While not including them in the total asbestos points counted, the analyst should record the presence of any asbestos detected but not lying under the reticle cross-line or array points. A minimum of 400 counts (maximum of eight slides with 50 counts each to minimum of two slides with 200 counts each) per sample is suggested, but it should be noted that accuracy and precision improve with number of counts. Point counting provides a determination of the projected area percent asbestos. Conversion of area percent to dry weight percent is not feasible unless the specific gravities and relative volumes of the different materials are known. It should be noted that the total amount of material to be analyzed is dependent on the asbestos concentration, i.e. the lower the concentration of asbestos, the larger the amount of sample that should be analyzed, in both the visual estimation and point counting methods. Quantitation by either method is made more difficult by low asbestos concentration, small fiber size, and presence of interfering materials.

It is suggested that asbestos concentration be reported as volume percent, weight percent or area percent depending on the method of quantitation used. A weight concentration cannot be determined without knowing the relative specific gravities and volumes of the sample components.

Mineral	Morphology and Color[1]	Refractive Indices[2] α	Refractive Indices[2] γ[5]	Birefringence[6]	Extinction	Sign of Elongation
Chrysotile (asbestiform serpentine)	Wavy fibers. Fiber bundles have splayed ends and "kinks". Aspect ratio typically >10:1. Colorless[3]	1.493-1.546 1.532-1.549 1.529-1.559 1.544-1.553	1.517-1.557 1.545-1.556 1.537-1.567 1.552-1.561	0.004-0.017	Parallel	+ (length slow)
Amosite (asbestiform grunerite)	Straight to curved, rigid fibers. Aspect ratio typically >10:1. Colorless to brown, nonpleochroic or weakly so.[4] Opaque inclusions may be present	1.657-1.663 1.663-1.686 1.663-1.686 1.676-1.683	1.699-1.717 1.696-1.729 1.696-1.729 1.697-1.704	0.021-0.054	Usually parallel	+ (length slow)
Crocidolite (asbestiform riebeckite)	Straight to curved, rigid fibers. Aspect ratio typically > 10:1. Thick fibers and bundles common, blue to dark-blue in color. Pleochroic.	1.693 1.654-1.701 1.680-1.698	1.697 1.668-1.717 1.685-1.706	0.003-0.022	Usually parallel	- (length fast)
Anthophyllite-asbestos	Straight to curved fibers and bundles. Aspect ratio typically > 10:1. Anthophyllite cleavage fragments may be present with aspect ratios <10:1. Colorless to light brown.	1.598-1.652 1.596-1.694 1.598-1.674 1.6148[7]	1.623-1.676 1.615-1.722 1.615-1.697 1.6362[7]	0.013-0.028	Parallel	+ (length slow)
Tremolite-Actinolite-asbestos	Straight to curved fibers and bundles. Aspect ratio typically > 10:1. Cleavage fragments may be present with aspect ratios <10:1. Colorless to pale green	Tremolite 1.600-1.628 1.604-1.612 1.599-1.612 1.6063[7]	 1.625-1.655 1.627-1.635 1.625-1.637 1.6343[7]	 0.017-0.028	Parallel and oblique (up to 21°); Composite fibers show parallel extinction.	+ (length slow)
		Actinolite 1.600-1.628 1.612-1.668 1.613-1.628 1.6126[7]	 1.625-1.655 1.635-1.688 1.638-1.655 1.6393[7]	 0.017-0.028		

[1]Colors cited are seen by observation with plane polarized light.

[2]From references 2, 11, 12, and 18, respectively. Refractive indices for n_d at 589.3nm.

[3]Fibers subjected to heating may be brownish. (references 13, 14, and 15)

[4]Fibers subjected to heating may be dark brown and pleochroic. (references 13, 14, and 15)

[5]∥ to fiber length, except ⊥ to fiber length for crocidolite only.

[6]Maximum and minimum values from references 2, 11, 12, and 18 given.

[7]± 0.0007

TABLE 2-3. TYPICAL CENTRAL STOP DISPERSION STAINING COLORS[1]

Mineral	Cargille® RI Liquid	n∥	n⊥
Chrysotile	1.550HD	Magenta to light blue-green λ_0's ca. 520-620nm	Blue-green to pale blue λ_0's ca. 600-700nm
Amosite	1.680	Yellow to magenta λ_0's ca. 420-520nm	Blue magenta to light blue λ_0's ca. 560-660nm
Crocidolite	1.680	Yellow to magenta λ_0's ca. 420-520nm	Pale yellow to golden yellow λ_0's ca. 360-460nm
Anthophyllite-asbestos	1.605HD	Pale yellow to yellow λ_0's ca. 330-430nm	Golden yellow to light blue green λ_0's ca. 460-700nm
Tremolite-asbestos	1.605HD	Pale yellow to yellow λ_0's ca. 330-430nm	Golden yellow to light blue green λ_0's ca. 460-700nm
Actinolite-asbestos	1.605HD	Pale yellow λ_0's ca. 260-360nm	Pale yellow to golden yellow λ_0's ca. 360-460nm
	1.630HD	Yellow to magenta λ_0's ca. 420-520nm	Golden yellow to blue λ_0's ca. 450-600nm

[1]Modified from reference 16

TABLE 2-4. OPTICAL PROPERTIES OF MAN-MADE TEXTILE FIBERS[1,2]

Fiber Type	n∥	n⊥	n∥ − n⊥	Sign of Elongation
Polyester (Dacron®)	1.710	1.535	0.175	+
Polyamide (Nylon®)	1.582	1.514	0.063	+
Aramid (Kevlar®)	≈2.37	≈1.641	0.729	+
Olefin (Polyethylene)	1.556	1.512	0.044	+
Olefin (Polypropylene)	1.520	1.495	0.025	+
Viscose Rayon	1.535-1.555	1.515-1.535	0.020	+
Acetate	1.478-1.480	1.473-1.476	0.004-0.005	+
Acrylic (Orlon®)	1.505-1.515	1.507-1.517	0.004-0.002	
Modacrylic (Dynel®)	1.535	1.532	0.002	+

[1]Modified from reference 17

[2]Refractive indices for specific fibers; other fibers may vary

TABLE 2-5. OPTICAL PROPERTIES OF SELECTED FIBERS[1]

FIBER TYPE	MORPHOLOGY	REFRACTIVE INDICES	BIREFRINGENCE ($n\parallel - n\perp$)	EXTINCTION ANGLE	SIGN OF ELONGATION	DISPERSION STAINING COLORS
Paper (Cellulose)	Tapered, flat ribbons	$n\parallel$ – 1.580 $n\perp$ – 1.530	High (0.05)	Parallel and incomplete	+	in 1.550HD $n\parallel$: yellow (λ_o's < 400nm) $n\perp$: pale blue (λ_o's > 700nm)
Olefin (polyethylene)	Filaments or shredded like chrysotile	$n\parallel$ – 1.556 $n\perp$ – 1.512	Moderate (0.044)	Parallel	+	in 1.550HD $n\parallel$: yellow to magenta (λ_o's = 440-540nm) $n\perp$: pale blue (λ_o's > 700nm)
Brucite (nemalite)	Straight fibers	$n\parallel$ – 1.560-1.590 $n\perp$ – 1.580-1.600	Moderate (0.012-0.020)	Usually parallel	- occasionally +	in 1.550HD $n\parallel$: golden yellow (λ_o's 440-460nm) $n\perp$: yellow (λ_o's 400-440nm)
Heated amosite	Similar to unheated, (brittle and shorter) pleochroic: $n\parallel$-dark brown $n\perp$ yellow	$n\parallel$ and $n\perp$ >1.700[2]	High (> 0.05)	Usually parallel	+	in 1.680HD $n\parallel$ & $n\perp$: both pale yellow to white (λ_o's < 400nm)
Glass fibers, Mineral wool	Exotic shapes, tear drops, single filaments	1.515-1.700	Isotropic	------	-	in 1.550HD usually pale blue to blue (λ_o's 580 to > 700nm)
Wollastonite	Straight needles and blades	$n\parallel$ – 1.630 $n\perp$ – 1.632 $n\perp$ also 1.610	Moderate to low (0.018 to 0.002)	Parallel and oblique	+ and -	in 1.605HD $n\parallel$ & $n\perp$: yellow to pale yellow (λ_o's < 460nm)
Fibrous talc	Thin cleavage ribbons and wavy fibers	$n\parallel$ – 1.60 $n\perp$ – 1.54	High (0.06)	Parallel and oblique	+	in 1.550HD $n\parallel$: pale yellow (λ_o's <400nm) $n\perp$: pale blue (λ_o's >660nm)

[1]From reference 19

[2]From references 13, 14, and 15

2.2.5.2.3 Microscope Alignment

In order to accurately measure the required optical properties, a properly aligned polarized light microscope must be utilized. The microscope is aligned when:

1) the privileged directions of the substage polarizer and the analyzer are at 90° to one another and are represented by the ocular cross-lines;

2) the compensator plate's privileged vibration directions are 45° to the privileged directions of the polarizer and analyzer;

3) the objectives are centered with respect to stage rotation; and,

4) the substage condenser and iris diaphragm are centered in the optic axis.

Additionally, the accurate measurement of the refractive index of a substance requires the use of calibrated refractive index liquids. These liquids should be calibrated regularly to an accuracy of 0.004, with a temperature accuracy of 2°C using a refractometer or R.I. glass beads.

2.2.6 References

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2.3 Gravimetry

2.3.1 Principle and Applicability

Many components of bulk building materials, specifically binder components, can be selectively removed using appropriate solvents or, in the case of some organics, by ashing. The removal of these components serves the following purposes:

1) to isolate asbestos from the sample, allowing its weight to be determined;

2) to concentrate asbestos and therefore lower the detection limit in the total sample;

3) to aid in the detection and identification of fibrous components; and,

4) to remove organic (ashable) fibers which are optically similar to asbestos.

Common binder materials which are removed easily using the techniques described include: 1) calcite, gypsum, magnesite, brucite, bassanite, portlandite, and dolomite, using hydrochloric acid, and 2) vinyl, cellulose, and other organic components, by ashing. The removal of the binder components results in a residue containing asbestos, if initially present, and any other non-soluble or non-ashable components which were present in the original sample. Unless the procedures employed result in the loss of some asbestos, the weight percent of the residue is the upper limit for the weight percent of asbestos in the sample.

This section describes the procedure for removing acid-soluble and ashable components, and for determining the weight percent of the residue. However, the acid dissolution and ashing techniques can be used without the accompanying weight measurements to either liberate or clean fibers to aid in qualitative PLM or AEM analyses.

This technique is not an identification technique. Other methods, such as PLM, XRD, or AEM must be used to determine the identity of the components. A description of the suggested apparatus, reagents, etc. needed for the techniques described is included in Appendix B.

2.3.2 Interferences

Any components which cannot by removed from the sample by selective dissolution or ashing interfere with asbestos quantitation. These components include, but are not limited to, many silicates (micas, glass fibers, etc.) and oxides (TiO_2, magnetite, etc.). When interfering phases are present (the residue contains other phases in addition to asbestos), other techniques such as PLM, AEM, or XRD must be used to determine the percent of asbestos in the residue.

Care must be taken to prevent loss of or chemical/structural changes in the critical components (asbestos). Prolonged exposure to acids or excessive heating (above 500°C) can cause changes in the asbestos components in the sample and affect the optical properties.[1,2,3]

2.3.3 Quantitation

The weight of the residue remaining after solvent dissolution/ashing should be compared with the original weight of the material. Presuming no insoluble material is lost, the weight percent of the residue is the upper limit for the amount of asbestos in the sample. If the residue is comprised only of asbestos, then the weight percent of residue equals the weight percent of asbestos in the sample. If the residue contains other phases, then techniques such as PLM, XRD, or AEM must be employed to determine the relative abundance of asbestos in the residue.

The precision and accuracy of the technique are dependent upon the homogeneity of the material, the accuracy of the weight measurements, and the effectiveness of the sample reduction and filtering procedures. In practice, the precision can be equal to $\pm 1\%$, and the accuracy at 1 wt% asbestos can be less than or equal to $\pm 10\%$ relative.

The incomplete solution of components and the presence of other nonasbestos components in the residue contribute to producing a positive bias for the technique (falsely high percentages of asbestos).

2.3.4 Preliminary Examination and Evaluation

Stereomicroscopic and PLM examinations of the sample should already have been conducted prior to initiating this procedure. These examinations should have provided information about: 1) whether the sample contains components which can be removed by acid-washing, solvent dissolution, or ashing, and 2) whether the sample contains asbestos, or fibers that might be asbestos, or whether no asbestos was detected.

If the sample is friable and contains organic (ashable) components, the ashing procedure should be followed. If the sample is friable and contains HCl-soluble components, the acid dissolution procedure should be followed. If the sample is friable and contains both types of

components, the two procedures can be applied, preferably with acid dissolution following ashing.

If the sample is nonfriable (e.g. floor tiles), it is also recommended that the ashing procedure be used first, followed by the acid dissolution procedure. The ashing procedure reduces floor tiles to a material which is easily powdered, simplifying the sample preparation for acid dissolution.

2.3.5 Sample Preparation

2.3.5.1 Drying

Any moisture in the sample will affect the weight measurements, producing falsely low percentages of residue. If the sample is obviously wet, it should be dried at low temperature (using a heat lamp, or simply by exposure at ambient conditions, prior to starting the weighing procedure). If an oven is used, the drying temperature should not exceed 60°C. Drying by means of heat lamp or ambient air must be performed within a safety-filtered hood. Even if the sample appears dry, it can contain enough moisture to affect the precision and accuracy of the technique. The test for sample moisture involves placing the amount of sample to be used on the weighing pan; if the weight remains stable with time, then the sample is dry enough. If the weight decreases as the sample sits on the weighing pan, then the sample should be dried. Where conditions of moderate to high humidity are known to exist, all materials to be weighed should be allowed time to stabilize to these ambient conditions.

2.3.5.2 Homogenization/Grain Size Reduction

To increase the accuracy and precision of the acid dissolution technique, the sample should be homogenized prior to analysis. This reduces the grain size of the binder material and releases it from fiber bundles so that it may be dissolved in a shorter time period. Leaving the sample in the acid for a longer period of time to complete the dissolution process can adversely affect the asbestos components, and is not recommended. Homogenization of the sample also ensures that any material removed for analysis will more likely be representative of the entire sample.

Homogenization of friable samples prior to ashing may also accelerate the ashing process; however, the ashing time can simply be increased without affecting the asbestos in the sample. Nonfriable samples, such as vinyl floor tiles, can be broken or shaved into pieces to increase surface area and accelerate the ashing process.

Homogenization and grain size reduction can be accomplished in a variety of ways: 1) hand grinding in a mortar and pestle; 2) crushing with pliers or similar instrument; 3) mixing in a blender; 4) milling (i.e. Wylie mill, cryomill, etc.); or 5) any other technique which seems suitable. If the fibers are extremely long, a pair of scissors or similar implement can be used to reduce the fiber length.

2.3.6 Procedure for Ashing

1) **Weigh appropriate amount of material.**

   There is no restriction on the maximum weight of material used; however, a large amount of material may take longer to ash. Enough material should be used to avoid a significant contribution of weighing errors to the total accuracy and precision.

2) **Place material in crucible, weigh, and cover with lid.**

   Placing a lid on the crucible both minimizes the amount of oxygen available, slowing the rate of combustion of the sample, and prevents any foreign material from falling into the crucible during ashing.

3) **Place crucible into furnace, and ash for at least 6 hours.**

   The furnace temperature at the sample position should be at least 300°C but should not exceed 500°C. If the sample combusts (burns), the temperature of the sample may exceed 500°C. Chrysotile will decompose above approximately 500°C.

   The furnace area should be well-ventilated and the fumes produced by ashing should be exhausted outside the building.

   The ashing time is dependent on the furnace temperature, the amount of sample, and the surface area (grain size). Six hours at 450°C is usually sufficient.

4) **Remove crucible from furnace, allow contents to adjust to room temperature and humidity, and weigh.**

5) **Divide residue weight by starting weight and multiply by 100 to determine weight% residue.**

6) **Analyze residue and/or proceed to acid dissolution procedure.**

If the objective was to remove organic fibers that may be confused optically with asbestos, examine residue with PLM to determine whether any fibers remain.

If the sample is a floor tile, the acid dissolution procedure must now be performed. The residue does not have to be analyzed at this stage.

2.3.7 Use of Solvents for Removal of Organics

Solvent dissolution may be used as a substitute for low temperature ashing for the purpose of removing organic interferences from bulk building materials. However, solvent dissolution, because of the involvement of potentially hazardous reagents such as tetrahydrofuran, amyl acetate, 1-1-1, trichlorethane, etc., requires **that all work be performed with extreme caution inside a biohazard hood.** Material Safety Data Sheets should be reviewed before using any solvent. Solvent dissolution involves more apparatus than does ashing, and requires more time, mainly due to set-up and slow filtration resulting from viscous solvent/residue mixtures.

The following is a brief description of the solvent dissolution process.

1) **Weigh starting material.**

Place approximately 15-25ml of solvent in a 100ml beaker. Add 2.5-3.0 grams (carefully weighed for continued gravimetric tracking) of powdered sample.

2) **Untrasonicate sample.**

Place the beaker in an ultrasonic bath (or ultrasonic stirrer) for approximately 0.5 hours. The sample containers should be covered to preclude escape of an aerosol spray.

3) **Centrifuge sample.**

Weigh centrifuge vial before adding beaker ingredients. Wash beaker with an additional 10-15ml of solvent to remove any remaining concentrate. Then centrifuge

at approximately 2000-2500 rpm for 0.5 hour. Use solvent-resistant centrifuge tubes.

4) **Decant sample, reweigh.**

After separation by centrifuging, decant solvent by pipetting. Leave a small amount of solvent in the centrifuge vial to minimize the risk of decanting solid concentrate. Allow solid concentrate to dry in vial, then reweigh.

2.3.8 Procedure for Acid Dissolution

1) **Weigh starting material, transfer to acid resistant container.**

Small, dry sample weights between 0.1g and 0.5g are recommended (determined for 47mm filters adjust amount if different diameter filters are used). If too much material is left after acid dissolution the filter can get clogged and prevent complete filtration. Very small samples are also to be avoided, as the weighing errors will have a large effect on the total accuracy and precision of the technique.

2) **Weigh filter.**

3) **Add HCl to sample in container, stir, allow to sit for 2-10 minutes.**

Either concentrated or dilute HCl can be used. If concentrated HCl is used, add enough acid to completely soak the material, allow the reaction to proceed to completion, and then dilute with distilled water. Alternatively, a dilute solution, made by adding concentrated HCl to distilled water, can be used in the place of concentrated HCl. A solution of 1 part concentrated HCl to 3 parts distilled water (approximately 3N solution) has been found to be quite effective in removing components within 5 minutes. For a sample size less than 0.5g, 20-30 ml of a 3N HCl solution is appropriate. In either case (using concentrated or dilute HCl), the reaction will be more effective if the sample has been homogenized first. All obvious signs of reaction (bubbling) should cease before the sample is filtered. Add fresh acid, a ml or two at a time, to ensure complete reaction. It should be noted that if dolomite is present, a 15-20 minute exposure to concentrated HCl may be required to completely dissolve the carbonate materials.

NOTE: Other solvents may be useful for selective dissolution of nonasbestos components. For example, acetic acid will dissolve calcite, and will not dissolve asbestos minerals. If any solvent other than hydrochloric acid is used for the dissolution of inorganic components, the laboratory must be able to demonstrate that the solvent does not remove asbestos from the sample.

4) **Filter solution.**

Use the pre-weighed filter. Pour the solution into the vacuum filter assembly, then rinse all material from container into filter assembly. Rinse down the inside walls of the glass filter basin and check for particles clinging to the basin after removal.

5) **Weigh dried filter + residue, subtract weight of filter from total.**

6) **Divide residue weight by starting weight and multiply by 100 to determine weight% residue.**

7) **Analyze residue.**

Perform stereomicroscopic examination of residue (can be performed without removing the residue from the filter). Note in particular whether any binder material is still present.

Perform PLM, AEM, or XRD analysis of residue to identify fibers and determine concentration as described in the appropriate sections of this method.

8) **Modify procedure if necessary.**

If removal of the acid soluble components was not complete, start with a new subsample of material and try any of the following:

a) Decrease grain size of material (by grinding, milling, etc.)
b) Put solutions on hot plate warm slightly
c) Increase soak time (exercise caution)

9) **Calculate relative weight% asbestos in sample.**

wt% asbestos in sample = % asbestos in residue x wt% residue ÷ 100

For floor tiles, if the ashing procedure was used first, multiply the weight % of asbestos in the sample, as determined above, by the weight percent of the residue from the ashing procedure, then divide by 100.

Example:
A = wt% residue from ashing = 70%
B = wt% residue from HCl = 20%
C = wt% of asbestos in HCl residue = 50%

wt% asbestos after HCl dissolution = B x C ÷ 100 = 20 x 50 ÷ 100 = 10%

wt% asbestos in floor tile = (B x C ÷ 100) x A ÷ 100 = 10 x 70 ÷ 100 = 7%

If weights are expressed in decimal form, multiply the weight % of asbestos in the sample by the weight % of the residue from the ashing procedure, then multiply by 100.

wt% asbestos after HCl dissolution = B x C = 0.2 x 0.5 = 0.1 (x 100 = 10%)

wt% asbestos in floor tile = (B x C) x A = 0.1 x 0.7 = 0.07 (x 100 = 7%)

2.3.9 Determination of Optimal Precision and Accuracy

The precision of the technique can be determined by extracting multiple subsamples from the original sample and applying the same procedure to each. The optimal accuracy of the technique can be determined by applying gravimetric standards. Mixtures of calcite and asbestos (chrysotile, amosite, etc.) in the following proportions are recommended for testing the accuracy of the acid dissolution technique: 0.1 wt% asbestos/99.9 wt% calcite, 1.0 wt% asbestos/99.0 wt% calcite, and 10 wt% asbestos/90 wt% calcite. Mixtures of cellulose and asbestos are useful for testing the accuracy of the ashing technique.

Mixtures of only two components, as described above, are simplifications of "real-world" samples. The accuracy determined by analyzing these mixtures is considered optimal and may not apply directly to the measurement of each unknown sample. However, analyzing replicates and standards using the full laboratory procedure, including homogenization, ashing, acid dissolution, filtration, and weighing, may uncover steps that introduce significant bias or variation that the laboratory may then correct.

2.3.10 References

1. Kressler, J. R., "Changes in Optical Properties of Chrysotile During Acid Leaching", **The Microscope**, 31, 1983, pp. 165-172.

2. Prentice, J. and M. Keech, "Alteration of Asbestos with Heat", **Microscopy and Analysis,** March 1989.

3. Laughlin, G. and W. C. McCrone, "The Effect of Heat on the Microscopical Properties of Asbestos", **The Microscope,** 37, 1989, pp. 8-15.

2.4 X-Ray Powder Diffraction

2.4.1 Principle and Applicability

The principle of x-ray powder diffraction (XRD) analysis is well established.[1,2] Any solid crystalline material will diffract an incident beam of parallel, monochromatic x-rays whenever Bragg's Law,

$$\lambda = 2d \sin \theta,$$

is satisfied for a particular set of planes in the crystal lattice, where

λ = the x-ray wavelength, Å;
d = the interplanar spacings of the set of reflecting lattice planes, Å and
θ = the angle of incidence between the x-ray beam and the reflecting lattice planes.

By appropriate orientation of a sample relative to the incident x-ray beam, a diffraction pattern can be generated that will be uniquely characteristic of the structure of the crystalline phases present.

Unlike optical methods of analysis, however, XRD cannot determine crystal morphology. Therefore, in asbestos analysis, XRD does not distinguish between fibrous and nonfibrous forms of the serpentine and amphibole minerals (Table 2-6). However, when used in conjunction with methods such as PLM or AEM, XRD techniques can provide a reliable analytical method for the identification and characterization of asbestiform minerals in bulk materials.

For **qualitative** analysis by XRD methods, samples should initially be scanned over limited diagnostic peak regions for the serpentine ($\sim$7.4 Å) and amphibole (8.2-8.5 Å) minerals (Table 2-7). Standard slow-scanning methods for bulk sample analysis may be used for materials shown by PLM to contain significant amounts of asbestos (>5 percent). Detection of minor or trace amounts of asbestos may require special sample preparation and step-scanning analysis. All samples that exhibit diffraction peaks in the diagnostic regions for asbestiform minerals should be submitted to a full (5°-60° 2θ; 1° 2θ/min) qualitative XRD scan, and their diffraction patterns should be compared with standard reference powder

diffraction patterns[3] to verify initial peak assignments and to identify possible matrix interferences when subsequent quantitative analysis will be performed.

Accurate **quantitative** analysis of asbestos in bulk samples by XRD is critically dependent on particle size distribution, crystallite size, preferred orientation and matrix absorption effects, and comparability of standard reference and sample materials. The most intense diffraction peak that has been shown to be free from interference by prior qualitative XRD analysis should be selected for quantitation of each asbestiform mineral. A "thin-layer" method of analysis[5,6] can be used in which, subsequent to comminution of the bulk material to $\sim 10\ \mu m$ by suitable cryogenic milling techniques, an accurately known amount of the sample is deposited on a silver membrane filter. The mass of asbestiform material is determined by measuring the integrated area of the selected diffraction peak using a step-scanning mode, correcting for matrix absorption effects, and comparing with suitable calibration standards. Alternative "thick-layer" or bulk methods[7,8] are commonly used for **semi-quantitative analysis.**

TABLE 2-6. THE ASBESTOS MINERALS AND THEIR NONASBESTIFORM ANALOGS

Asbestiform	Nonasbestiform	Chemical Abstract Service No.
Serpentine		
Chrysotile	Antigorite, lizardite	12001-29-5
Amphibole		
Anthophyllite asbestos	Anthophyllite	77536-67-5
Cummingtonite-grunerite asbestos (Amosite)	Cummingtonite-grunerite	12172-73-5
Crocidolite	Riebeckite	12001-28-4
Tremolite asbestos	Tremolite	77536-68-6
Actinolite asbestos	Actinolite	77536-66-4

TABLE 2-7. PRINCIPAL LATTICE SPACINGS OF ASBESTIFORM MINERALS[1]

Minerals	Principal d-spacings (Å) and relative intensities			JCPDS Powder diffraction file[2] number
Chrysotile (Serpentine)	7.31_{100} 7.36_{100} 7.10_{100}	3.65_{70} 3.66_{80} 2.33_{80}	4.57_{50} 2.45_{65} 3.55_{70}	21-543[3] 25-645 22-1162 (theoretical)
Amosite (Grunerite)	8.33_{100} 8.22_{100}	3.06_{70} 3.060_{85}	2.756_{70} 3.25_{70}	17-745 (nonfibrous) 27-1170 (UICC)
Anthophyllite	3.05_{100} 3.06_{100}	3.24_{60} 8.33_{70}	8.26_{55} 3.23_{50}	9-455 16-401 (synthetic)
Crocidolite (Riebeckite)	8.35_{100} 8.40_{100}	3.10_{55} 3.12_{55}	2.720_{35} 2.726_{40}	27-1415 (UICC) 19-1061
Actinolite	2.72_{100}	2.54_{100}	3.40_{80}	25-157
Tremolite	8.38_{100} 2.706_{100} 3.13_{100}	3.12_{100} 3.14_{95} 2.706_{60}	2.705_{90} 8.43_{40} 8.44_{40}	13-437[3] 20-1310[3] (synthetic) 23-666 (synthetic mixture w/richterite)

1. This information is intended as a guide only. Complete powder diffraction data, including mineral type and source, should be referred to ensure comparability of sample and reference materials where possible. Additional precision XRD data on amosite, crocidolite, tremolite and chrysotile are available from the U.S. Bureau of Mines, Reference 4.

2. From Reference 3

3. Fibrosity questionable

This XRD method is applicable as a confirmatory method for identification and quantitation of asbestos in bulk material samples that have undergone prior analysis by PLM or other optical methods.

2.4.2 Range and Sensitivity

The range and sensitivity of the method have not been determined. They will be variable and dependent upon many factors, including matrix effects (absorption and interferences), diagnostic reflections selected and their relative intensities, preferred orientation, and instrumental limitations. A detection limit of one percent is feasible given certain sample characteristics.

2.4.3 Limitations

2.4.3.1 Interferences

Since the asbestiform and nonasbestiform analogs of the serpentine and amphibole minerals (Table 2-7) are indistinguishable by XRD techniques unless special sample preparation techniques and instrumentation are used,[9] the presence of nonasbestiform serpentines and amphiboles in a sample will pose severe interference problems in the identification and quantitative analysis of their asbestiform analogs.

The use of XRD for identification and quantitation of asbestiform minerals in bulk samples may also be limited by the presence of other interfering materials in the sample. For naturally-occurring materials, the commonly associated asbestos-related mineral interferences can usually be anticipated. However, for fabricated materials, the nature of the interferences may vary greatly (Table 2-8) and present more serious problems in identification and quantitation.[10] Potential interferences are summarized in Table 2-9 and include the following:

- **Chlorite** has major peaks at 7.19 Å and 3.58 Å that interfere with both the primary (7.31 Å) and secondary (3.65 Å) peaks for serpentine (chrysotile). Resolution of the primary peak to give good quantitative results may be possible when a step-scanning mode of operation is employed.

- **Vermiculite** has secondary peaks at 7.14 Å and 3.56 Å that could interfere with the primary peak (7.31 Å) and a secondary peak (3.65 Å) of serpentine (chrysotile).

TABLE 2-8. COMMON CONSTITUENTS IN BUILDING MATERIAL
(From Ref. 10)

A. Insulation Materials

Chrysotile
Amosite
Crocidolite
*Rock wool
*Slag wool
*Fiber glass
Gypsum ($CaSO_4 \cdot 2H_2O$)
Vermiculite (micas)
*Perlite
Clays (kaolin)
*Wood pulp
*Paper fibers (talc, clay carbonate filters)
Calcium silicates (synthetic)
Opaques (chromite, magnetite inclusions in serpentine)
Hematite (inclusions in "amosite")
Magnesite
*Diatomaceous earth

B. Flooring Materials

Calcite
Dolomite
Titanium Oxide
Quartz
Antigorite
Chrysotile
Anthophyllite
Tremolite
*Organic binders
Talc
Wollastonite

C. Spray Finishes or Paints

Bassanite
Carbonate minerals (calcite, dolomite, vaterite)
Talc
Tremolite
Anthophyllite
Serpentine (including chrysotile)
Amosite
Crocidolite
*Mineral wool
*Rock wool
*Slag wool
*Fiber glass
Clays (kaolin)
Micas
Chlorite
Gypsum
Quartz
*Organic binders and thickeners
Hydromagnesite
Wollastonite
Opaques (chromite, magnetite inclusion in serpentine)
Hematite (inclusions in "amosite")

D. Cementitious Materials

Chrysotile
Amosite
Crocidolite
Micas
Fiber glass
Cellulose
Animal hair
Quartz
Gypsum
Calcite
Dolomite
Calcium silicates

E. Roofing Materials

Chrysotile
Cellulose
Fiber glass
Mineral Wool
Asphalt
Quartz
Talc
Micas

* Amorphous materials--contribute only to overall scattered radiation and increased background radiation.

TABLE 2-9 INTERFERENCES IN XRD ANALYSIS OF ASBESTIFORM MINERALS

Asbestiform Mineral	Primary diagnostic peaks (approximate d spacings in Å)	Interference
Serpentine Chrysotile	7.3	Nonasbestiform serpentines, (antigorite, lizardite), chlorite, vermiculite, sepiolite, kaolinite, gypsum
	3.7	Nonasbestiform serpentines (antigorite, lizardite), chlorite, vermiculite, halloysite, cellulose
Amphibole Amosite (Grunerite) Anthophyllite Crocidolite (Riebeckite)	3.1	Nonasbestiform amphiboles (grunerite-cummingtonite, anthophyllite, riebeckite, tremolite), mutual interferences, talc, carbonates
Tremolite Actinolite	8.3	Nonasbestiform amphiboles (grunerite-cummingtonite, anthophyllite, riebeckite, tremolite), mutual interferences

- **Sepiolite** produces a peak at 7.47 Å which could interfere with the primary peak (7.31 Å) of serpentine (chrysotile).
- **Halloysite** has a peak at 3.63 Å that interferes with the secondary (3.65 Å) peak for serpentine (chrysotile).
- **Kaolinite** has a major peak at 7 15 Å that may interfere with the primary peak of serpentine (chrysotile) at 7.31 Å when present at concentrations of >10 percent. However, the secondary serpentine (chrysotile) peak at 3.65 Å may be used for quantitation.
- **Gypsum** has a major peak at 7.5 Å that overlaps the 7.31 Å peak of serpentine (chrysotile) when present as a major sample constituent. This may be removed by careful washing with distilled water, or by heating to 300°C to convert gypsum to plaster of paris (bassanite).
- **Cellulose** has a broad peak that partially overlaps the secondary (3.65 Å) serpentine (chrysotile) peak.[8]

- Overlap of major diagnostic peaks of the amphibole minerals, grunerite (amosite), anthophyllite, riebeckite (crocidolite), and tremolite, at approximately 8.3 Å and 3.1 Å causes mutual interference when these minerals occur in the presence of one another. In some instances adequate resolution may be attained by using step-scanning methods and/or by decreasing the collimator slit width at the x-ray port.

- **Carbonates** may also interfere with quantitative analysis of the amphibole minerals grunerite (amosite), anthophyllite, riebeckite (crocidolite), and tremolite-actinolite. Calcium carbonate ($CaCO_3$) has a peak at 3.035 Å that overlaps major amphibole peaks at approximately 3.1 Å when present in concentrations of >5 percent. Removal of carbonates with a dilute acid wash is possible; however, the time in acid should be no more than 20 minutes to preclude any loss of chrysotile.[11]

- A major **talc** peak at 3.12 Å interferes with the primary tremolite peak at this same position and with secondary peaks of actinolite (3.14 Å), riebeckite (crocidolite) (3.10 Å), grunerite (amosite) (3.06 Å), and anthophyllite (3.05 Å). In the presence of talc, the major diagnostic peak at approximately 8.3 Å should be used for quantitation of these asbestiform minerals.

The problem of intraspecies and matrix interference is further aggravated by the variability of the silicate mineral powder diffraction patterns themselves, which often makes definitive identification of the asbestos minerals by comparison with standard reference diffraction patterns difficult. This variability results from alterations in the crystal lattice associated with differences in isomorphous substitution and degree of crystallinity. This is especially true for the amphiboles. These minerals exhibit a wide variety of very similar chemical compositions, resulting in diffraction patterns characterized by having major (110) reflections of the monoclinic amphiboles and (210) reflections of orthorhombic anthophyllite separated by less than 0.2 Å.[12]

2.4.3.2 Matrix Effects

If a copper x-ray source is used, the presence of iron at high concentrations in a sample will result in significant x-ray fluorescence, leading to loss of peak intensity, increased background intensity, and an overall decrease in sensitivity. This situation may be corrected by use of an x-ray source other than copper; however, this is often accompanied both by loss of intensity and by decreased resolution of closely spaced reflections. Alternatively, use of a

diffracted beam monochromator will reduce background fluorescent radiation, enabling weaker diffraction peaks to be detected.

X-ray absorption by the sample matrix will result in overall attenuation of the diffracted beam and may seriously interfere with quantitative analysis. Absorption effects may be minimized by using sufficiently "thin" samples for analysis.[5,13,14] However, unless absorption effects are known to be the same for both samples and standards, appropriate corrections should be made by referencing diagnostic peak areas to an internal standard[7,8] or filter substrate (Ag) peak.[5,6]

2.4.3.3 Particle Size Dependence

Because the intensity of diffracted x-radiation is particle-size dependent, it is essential for accurate quantitative analysis that both sample and standard reference materials have similar particle size distributions. The optimum particle size (i.e., fiber length) range for quantitative analysis of asbestos by XRD has been reported to be 1 to 10 μm.[15] Comparability of sample and standard reference material particle size distributions should be verified by optical microscopy (or another suitable method) prior to analysis.

2.4.3.4 Preferred Orientation Effects

Preferred orientation of asbestiform minerals during sample preparation often poses a serious problem in quantitative analysis by XRD. A number of techniques have been developed for reducing preferred orientation effects in "thick layer" samples.[7,8,15] For "thin" samples on membrane filters, the preferred orientation effects seem to be both reproducible and favorable to enhancement of the principal diagnostic reflections of asbestos minerals, actually increasing the overall sensitivity of the method.[12,14] However, further investigation into preferred orientation effects in both thin layer and bulk samples is required.

2.4.3.5 Lack of Suitably Characterized Standard Materials

The problem of obtaining and characterizing suitable reference materials for asbestos analysis is clearly recognized. The National Institute of Standards and Technology can

provide standard reference materials for chrysotile, amosite and crocidolite (SRM 1866) and anthophyllite, tremolite and actinolite (SRM 1867).

In addition, the problem of ensuring the comparability of standard reference and sample materials, particularly regarding crystallite size, particle size distribution, and degree of crystallinity, has yet to be adequately addressed. For example, Langer et al.[18] have observed that in insulating matrices, chrysotile tends to break open into bundles more frequently than amphiboles. This results in a line-broadening effect with a resultant decrease in sensitivity. Unless this effect is the same for both standard and sample materials, the amount of chrysotile in the sample will be under-estimated by XRD analysis. To minimize this problem, it is recommended that standardized matrix reduction procedures be used for both sample and standard materials.

2.4.4 Precision and Accuracy

Neither the precision nor accuracy of this method has been determined. The individual laboratory should obtain or prepare a set of calibration materials containing a range of asbestos weight percent concentrations in combination with a variety of matrix/binder materials. Calibration curves may be constructed for use in semi-quantitative analysis of bulk materials.

2.4.5 Procedure

2.4.5.1 Sampling

Samples taken for analysis of asbestos content should be collected as specified by EPA[19]

2.4.5.2 Analysis

All samples must be analyzed initially for asbestos content by PLM. XRD may be used as an additional technique, both for identification and quantitation of sample components. **Note: Asbestos is a toxic substance. All handling of dry materials should be performed in a safety-hood.**

2.4.5.2.1 Sample Preparation

The method of sample preparation required for XRD analysis will depend on: (1) the condition of the sample received (sample size, homogeneity, particle size distribution, and overall composition as determined by PLM); and (2) the type of XRD analysis to be performed (qualitative or quantitative; thin-layer or bulk).

Bulk materials are usually received as heterogeneous mixtures of complex composition with very wide particle size distributions. Preparation of a homogeneous, representative sample from asbestos-containing materials is particularly difficult because the fibrous nature of the asbestos minerals inhibits mechanical mixing and stirring, and because milling procedures may cause adverse lattice alterations.

A discussion of specific matrix reduction procedures is given below. Complete methods of sample preparation are detailed in Sections 2.4.5.3 and 2.4.5.4. **Note: All samples should be examined microscopically before and after each matrix reduction step to monitor changes in sample particle size distribution, composition, and crystallinity, and to ensure sample representativeness and homogeneity for analysis.**

2.4.5.2.2 Milling

Mechanical milling of asbestos materials has been shown to decrease fiber crystallinity, with a resultant decrease in diffraction intensity of the specimen; the degree of lattice alteration is related to the duration and type of milling process.[20-23] Therefore, all milling times should be kept to a minimum.

For **qualitative analysis**, particle size is not usually of critical importance and initial characterization of the material with a minimum of matrix reduction is often desirable to document the composition of the sample as received. Bulk samples of very large particle size (>2-3 mm) should be comminuted to ~ 100 μm. A mortar and pestle can sometimes be used in size reduction of soft or loosely bound materials though this may cause matting of some samples. Such samples may be reduced by cutting with a razor blade in a mortar, or by grinding in a suitable mill (e.g., a microhammer mill or equivalent). When using a mortar for grinding or cutting, the sample should be moistened with ethanol, or some other

suitable wetting agent, to minimize exposure, and the procedure should be performed in a HEPA-filtered hood.

For accurate, reproducible **quantitative analysis**, the particle size of both sample and standard materials should be reduced to $\sim 10\ \mu m$. Dry ball milling at liquid nitrogen temperatures (e.g., Spex Freezer Mill®, or equivalent) for a maximum time of 10 minutes (some samples may require much shorter milling time) is recommended to obtain satisfactory particle size distributions while protecting the integrity of the crystal lattice.[5] Bulk samples of very large particle size may require grinding in two stages for full matrix reduction to $< 10\ \mu m$.[8,16]

Final particle size distributions should always be verified by optical microscopy or another suitable method.

2.4.5.2.3 Ashing

For materials shown by PLM to contain large amounts of cellulose or other organic materials, it may be desirable to ash prior to analysis to reduce background radiation or matrix interference. Since chrysotile undergoes dehydroxylation at temperatures between 550°C and 650°C, with subsequent transformation to forsterite,[24,25] ashing temperatures should be kept below 500°C. Use of a muffle furnace is recommended. In all cases, calibration of the furnace is essential to ensure that a maximum ashing temperature of 500°C is not exceeded (see Section 2.3).

2.4.5.2.4 Acid Washing

Because of the interference caused by gypsum and some carbonates in the detection of asbestiform minerals by XRD (see Section 2.4.3.1), it may be necessary to remove these interferences by a simple acid washing procedure prior to analysis (see Section 2.3).

2.4.5.3 Qualitative Analysis

2.4.5.3.1 Initial Screening of Bulk Material

Qualitative analysis should be performed on a representative, homogeneous portion of the sample, with a minimum of sample treatment, using the following procedure:

1. Grind and mix the sample with a mortar and pestle (or equivalent method, see Section 2.4.5.2.2) to a final particle size sufficiently small (~ 100 μm) to allow adequate packing into a sample holder.

2. Pack sample into a standard bulk sample holder. Care should be taken to ensure that a representative portion of the milled sample is selected for analysis. Particular care should be taken to avoid possible size segregation of the sample. **(Note: Use of back-packing method[26] for bulk sample preparation may reduce preferred orientation effects.)**

3. Mount the sample on the diffractometer and scan over the diagnostic peak regions for the serpentine (~ 7.4 Å) and amphibole (8.2-8.5 Å) minerals (see Table 2-7). The x-ray diffraction equipment should be optimized for intensity. A slow scanning speed of 1° 2θ/min is recommended for adequate resolution. Use of a sample spinner is recommended.

4. Submit all samples that exhibit diffraction peaks in the diagnostic regions for asbestiform minerals to a full qualitative XRD scan (5°-60° 2θ; 1° 2θ/min) to verify initial peak assignments and to identify potential matrix interferences when subsequent quantitative analysis is to be performed.

5. Compare the sample XRD pattern with standard reference powder diffraction patterns (i.e., JCPDS powder diffraction data[3] or those of other well-characterized reference materials). Principal lattice spacings of asbestiform minerals are given in Table 2-7; common constituents of bulk insulation and wall materials are listed in Table 2-8.

2.4.5.3.2 Detection of Minor or Trace Constituents

Routine screening of bulk materials by XRD may fail to detect small concentrations ($< 1\%$) of asbestos. The limits of detection will, in general, be improved if matrix absorption effects are minimized, and if the sample particle size is reduced to the optimal 1 to 10 μm range, provided that the crystal lattice is not degraded in the milling process. Therefore, in those instances when confirmation of the presence of an asbestiform mineral at very low levels is required, or where a negative result from initial screening of the bulk material by XRD (see Section 2.4.5.3.1) is in conflict with previous PLM results, it may be desirable to prepare the sample as described for quantitative analysis (see Section 2.4.5.4) and step-scan over appropriate 2θ ranges of selected diagnostic peaks (Table 2-7). Accurate

transfer of the sample to the silver membrane filter is not necessary unless subsequent quantitative analysis is to be performed.

2.4.5.4 Quantitative Analysis

The proposed method for quantitation of asbestos in bulk samples is a modification of the NIOSH-recommended thin-layer method for chrysotile in air.[6] A thick-layer bulk method involving pelletizing the sample may be used for semi-quantitative analysis;[7,8] however, this method requires the addition of an internal standard, use of a specially fabricated sample press, and relatively large amounts of standard reference materials. Additional research is required to evaluate the comparability of thin- and thick-layer methods for quantitative asbestos analysis.

For quantitative analysis by thin-layer methods, the following procedure is recommended:

1. Mill and size all or a substantial representative portion of the sample as outlined in Section 2.4.5.2.2.

2. Dry at 60°C for 2 hours; cool in a desiccator.

3. Weigh accurately to the nearest 0.01 mg.

4. Samples shown by PLM to contain large amounts of cellulosic or other organic materials, gypsum, or carbonates, should be submitted to appropriate matrix reduction procedures described in Sections 2.4.5.2.3 and 2.4.5.2.4. After ashing and/or acid treatment, repeat the drying and weighing procedures described above, and determine the percent weight loss, L.

5. Quantitatively transfer an accurately weighed amount (50-100 mg) of the sample to a 1-L volumetric flask containing approximately 200 mL isopropanol to which 3 to 4 drops of surfactant have been added.

6. Ultrasonicate for 10 minutes at a power density of approximately 0.1 W/mL, to disperse the sample material.

7. Dilute to volume with isopropanol.

8. Place flask on a magnetic-stirring plate. Stir.

9. Place silver membrane filter on the filtration apparatus, apply a vacuum, and attach the reservoir. Release the vacuum and add several milliliters of isopropanol to the reservoir. Vigorously hand shake the asbestos suspension and immediately withdraw

an aliquot from the center of the suspension so that total sample weight, W_T, on the filter will be approximately 1 mg. Do not adjust the volume in the pipet by expelling part of the suspension; if more than the desired aliquot is withdrawn, discard the aliquot and repeat the procedure with a clean pipet. Transfer the aliquot to the reservoir. Filter rapidly under vacuum. Do not wash the reservoir walls. Leave the filter apparatus under vacuum until dry. Remove the reservoir, release the vacuum, and remove the filter with forceps. (Note: Water-soluble matrix interferences such as gypsum may be removed at this time by careful washing of the filtrate with distilled water. Extreme care should be taken not to disturb the sample.)

10. Attach the filter to a flat holder with a suitable adhesive and place on the diffractometer. Use of a sample spinner is recommended.

11. For each asbestos mineral to be quantitated, select a reflection (or reflections) that has (have) been shown to be free from interferences by prior PLM or qualitative XRD analysis and that can be used unambiguously as an index of the amount of material present in the sample (see Table 2-7).

12. Analyze the selected diagnostic reflection(s) by step-scanning in increments of 0.02° 2θ for an appropriate fixed time and integrating the counts. (A fixed count scan may be used alternatively; however, the method chosen should be used consistently for all samples and standards.) An appropriate scanning interval should be selected for each peak, and background corrections made. For a fixed time scan, measure the background on each side of the peak for one-half the peak-scanning time. The net intensity, I_a, is the difference between the peak integrated count and the total background count.

13. Determine the net count, I_{Ag}, of the filter 2.36 Å silver peak following the procedure in step 12. Remove the filter from the holder, reverse it, and reattach it to the holder. Determine the net count for the unattenuated silver peak, I°_{Ag}. Scan times may be less for measurement of silver peaks than for sample peaks; however, they should be constant throughout the analysis.

14. Normalize all raw, net intensities (to correct for instrument instabilities) by referencing them to an external standard (e.g., the 3.34 Å peak of an α-quartz reference crystal). After each unknown is scanned, determine the net count, I°_r, of the reference specimen following the procedure in step 12. Determine the normalized intensities by dividing the peak intensities by I°_r:

$$\hat{I}_a = \frac{I_a}{I_I^\circ}, \quad \hat{I}_{Ag} = \frac{I_{Ag}}{I_I^\circ}, \text{ and } \quad \hat{I}_{Ag}^\circ = \frac{I_{Ag}^\circ}{I_I^\circ}$$

2.4.6 Calibration

2.4.6.1 Preparation of Calibration Standards

1. Mill and size standard asbestos materials according to the procedure outlined in Section 2.4.5.2.2. **Equivalent standardized matrix reduction and sizing techniques should be used for both standard and sample materials.**

2. Dry at 100°C for 2 hours; cool in a desiccator.

3. Prepare two suspensions of each standard in isopropanol by weighing approximately 10 and 50 mg of the dry material to the nearest 0.01 mg. Transfer each to a 1-L volumetric flask containing approximately 200 mL isopropanol to which a few drops of surfactant have been added.

4. Ultrasonicate for 10 minutes at a power density of approximately 0.1 W/mL, to disperse the asbestos material.

5. Dilute to volume with isopropanol.

6. Place the flask on a magnetic stirring plate. Stir.

7. Prepare, in triplicate, a series of at least five standard filters to cover the desired analytical range, using appropriate aliquots of the 10 and 50 mg/L suspensions. For each standard, mount a silver membrane filter on the filtration apparatus. Place a few mL of isopropanol in the reservoir. Vigorously hand shake the asbestos suspension and immediately withdraw an aliquot from the center of the suspension. Do not adjust the volume in the pipet by expelling part of the suspension; if more than the desired aliquot is withdrawn, discard the aliquot and resume the procedure with a clean pipet. Transfer the aliquot to the reservoir. Keep the tip of the pipet near the surface of the isopropanol. Filter rapidly under vacuum. Do not wash the sides of the reservoir. Leave the vacuum on for a time sufficient to dry the filter. Release the vacuum and remove the filter with forceps.

2.4.6.2 Analysis of Calibration Standards

1. Mount each filter on a flat holder. Perform step scans on selected diagnostic reflections of the standards and reference specimen using the procedure outlined in Section 2.4.5.4, step 12, and the same conditions as those used for the samples.

2. Determine the normalized intensity for each peak measured, $\hat{I}^{\circ}_{std}$, as outlined in Section 2.4.5.4, step 14.

2.4.7 Calculations

For each asbestos reference material, calculate the exact weight deposited on each standard filter from the concentrations of the standard suspensions and aliquot volumes. Record the weight, w, of each standard. Prepare a calibration curve by regressing $\hat{I}^{\circ}_{std}$, on w. Poor reproducibility ($\pm$15 percent RSD) at any given level indicates problems in the sample preparation technique, and a need for new standards. The data should fit a straight-line equation.

Determine the slope, m, of the calibration curve in counts/microgram. The intercept, b, of the line with the $\hat{I}^{\circ}_{std}$ axis should be approximately zero. A large negative intercept indicates an error in determining the background. This may arise from incorrectly measuring the baseline or from interference by another phase at the angle of background measurement. A large positive intercept indicates an error in determining the baseline or that an impurity is included in the measured peak.

Using the normalized intensity, $\hat{I}_{Ag}$ for the attenuated silver peak of a sample, and the corresponding normalized intensity from the unattenuated silver peak $\hat{I}^{\circ}_{Ag}$, of the sample filter, calculate the transmittance, T, for each sample as follows:[27,28]

$$T = \frac{\hat{I}_{Ag}}{\hat{I}^{\circ}_{Ag}}$$

Determine the correction factor, f(T), for each sample according to the formula:

$$f(T) = \frac{-R(\ln T)}{1 - T^R}$$

where

$$R = \frac{\sin \theta_{Ag}}{\sin \theta_a}$$

θ_{Ag} = angular position of the measured silver peak (from Bragg's Law), and

θ_a = angular position of the diagnostic asbestos peak.

Calculate the weight, W_a, in micrograms, of the asbestos material analyzed for in each sample, using the absorption corrections:

$$W_a = \frac{\hat{I}_a f(t) - b}{m}$$

Calculate the percent composition, P_a, of each asbestos mineral analyzed for in the parent material, from the total sample weight, W_T, on the filter:

$$P_a = \frac{W_a(1 - .01L)}{W_T} \times 100$$

where

P_a = percent asbestos mineral in parent material;

W_a = mass of asbestos mineral on filter, in μg;

W_T = total sample weight on filter, in μg;

L = percent weight loss of parent material on ashing and/or acid treatment (see Section 2.4.5.4).

2.4.8 References

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2.5 Analytical Electron Microscopy

2.5.1 Applicability

Analytical electron microscopy (AEM) can often be a reliable method for the detection and positive identification of asbestos in some bulk building materials, both friable and nonfriable. The method is particularly applicable to bulk materials that contain a large amount of interfering materials that can be removed by ashing and/or dissolution and contain asbestos fibers that are not resolved by PLM techniques. Many floor tiles and plasters would be included in this type of sample. In combination with suitable specimen preparation techniques, the AEM method can also be used to quantify asbestos concentrations.

2.5.2 Range

The range is dependent on the type of bulk material being analyzed. The upper detection limit is 100%, and the lower detection limit can be as low as 0.0001% depending on the extent to which interfering materials can be separated during the preparation of AEM

specimens, the sophistication of the AEM preparation, and the amount of labor expended on AEM examination.

2.5.3 Interferences

The presence of a large amount of binder/matrix materials associated with fibers can make it difficult to positively identify fibers as asbestos. The portion of the fiber examined by either electron diffraction or energy dispersive x-ray analysis (EDXA) must be free of binder/matrix materials.

2.5.4 Precision and Accuracy

The precision and accuracy of the method have not been determined.

2.5.5 Procedures

The procedures for AEM specimen preparation depend on the data required. In analysis of floor tiles, the weighed residue after removal of the matrix components (see Section 2.3, Gravimetry) is often mostly asbestos, and the task is primarily to identify the fibers. In this situation the proportion of asbestos in the residue can be estimated by AEM and this estimate can be used to refine the gravimetric result. For many floor tiles, the final result is not very sensitive to errors in this estimation because the proportion of asbestos in the residue is very high. For samples in which this is not the case, precise measurements can be made using a quantitative AEM preparation, in which each grid opening of the specimen grid corresponds to a known weight of the original sample or of a concentrate derived from the original sample. Asbestos fibers on these grids are then identified and measured, using a fiber counting protocol which is directed towards a precise determination of mass concentration. This latter procedure is suitable for samples of low asbestos concentration, or for those in which it is not possible to remove a large proportion of the matrix material.

2.5.5.1 AEM Specimen Preparation for Semi-Quantitative Evaluation

The residual material from any ashing or dissolution procedures (see Section 2.3) used (usually trapped on a membrane filter) should be placed in a small volume of ethanol or another solvent such as acetone or isopropyl alcohol, in a disposable beaker, and dispersed

by treatment in an ultrasonic bath. A small volume of this suspension (approximately 3μl) should be pipetted onto the top of a carbon-coated TEM grid. The suspension should be allowed to dry under a heat lamp. The grid is then ready for examination.

Samples that are not conducive to ashing or dissolution may also be prepared in this way for AEM analysis. A few milligrams of the sample may be ground in a mortar and pestle or milled, dispersed in ethanol or another solvent using an ultrasonic bath, and pipetted onto a grid as described previously.

2.5.5.2 AEM Specimen Preparation for Quantitative Evaluation

The objective of this preparation is to obtain a TEM grid on which a known weight of the bulk sample is represented by a known area of the TEM grid. A known weight of the bulk sample, or of the residue after extraction, should be dispersed in a known volume of distilled water. Aliquots of this dispersion should then be filtered through 0.22 μm pore-size MCE or 0.2 μm pore-size PC filters, using filtration techniques as described for analysis of water samples.[1] In order to obtain filters of appropriate particulate loading for AEM analysis, it may be necessary to perform serial dilutions of the initial dispersion. TEM grids should then be prepared from appropriately-loaded filters, using the standard methods.[2]

Determination of the mass concentration of asbestos on the TEM grids requires a different fiber counting protocol than that usually used for determination of numerical fiber concentrations. Initially, the grids should be scanned to determine the dimensions of the largest asbestos fiber or fiber bundle on the specimens. The volume of this fiber or bundle should be calculated. The magnification of the AEM should be set at a value for which the length of this fiber or bundle just fills the fluorescent screen. Asbestos fiber counting should then be continued at this magnification. The count should be terminated when the volume of the initial large fiber or bundle represents less than about 5% of the integrated volume of all asbestos fibers detected. This counting strategy ensures that the fiber counting effort is directed toward those fibers which contribute most to the mass, and permits a precise mass concentration value to be obtained.

2.5.5.2.1 Identification

To document the positive identification of asbestos in a sample, the analyst should record the following physical properties: morphology data, electron diffraction data, EDXA data, and any other distinguishing characteristics observed. For fibrous structures identified as nonasbestos, the unique physical property or properties that differentiate the material from asbestos should be recorded.

The purpose of the identification data collected is to prevent or limit false negatives and false positives. This can be accomplished by having a system for measuring and recording the d-spacings and symmetry of the diffraction patterns, determining the relative abundance of the elements detected by EDXA, and comparing these results to reference data. The laboratory should have a set of reference asbestos materials from which a set of reference diffraction patterns and x-ray spectra have been developed. Also, the laboratory should have available reference data on the crystallography and chemical composition of minerals that might analytically interfere with asbestos.

2.5.6 References

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2.6 Other Methodologies

Additional analytical methods (e.g. Scanning Electron Microscopy) may be applicable for some bulk materials. However, the analyst should take care to recognize the limitations of any analytical method chosen. Conventional SEM, for example, cannot detect small diameter fibers ($\sim < 0.2\mu m$), and cannot determine crystal structure. It is, however, very useful for observing surface features in complex particle matrices, and for determining elemental compositions.

3.0 QUALITY CONTROL/QUALITY ASSURANCE OPERATIONS- PLM

A program to routinely assess the quality of the results produced by the PLM laboratory must be developed and implemented. **Quality Control** (QC) is a system of activities whose purpose is to control the quality of the product or service so that it meets the need of the users. This also includes **Quality Assessment**, whose purpose is to provide assurance that the overall quality control is being done effectively. While the essential elements of a quality control system are described in detail elsewhere,[1,2,3,4,5,6] only several of the elements will be discussed here. **Quality Assurance (QA)** is comprised of Quality Control and Quality Assessment and is a system of activities designed to provide assurance that a product or service meets defined standards of quality.

The purpose of the Quality Assurance program is to minimize failures in the analysis of materials prior to submitting the results to the client. Failures in the analysis of asbestos materials include false positives, false negatives, and misidentification of asbestos types. False positives result from identification or quantitation errors. False negatives result from identification, detection, or quantitation errors.

For the stereomicroscopic and PLM techniques, the quality control procedures should characterize the accuracy and precision of both individual analysts and the techniques. Analysts should demonstrate their abilities on calibration materials, and also be checked routinely on the analysis of unknowns by comparison with results of a second analyst. The limitations of the stereomicroscopic and PLM techniques can be determined by using a second analytical technique, such as gravimetry, XRD, or AEM. For example, stereomicroscopic and PLM techniques can fail in the analysis of floor tiles because the asbestos fibers in the sample may be too small to be resolved by light microscopy. An XRD or AEM analysis is not subject to the same limitations, and may indicate the presence of asbestos in the sample.

The accuracy, precision, and detection limits of all analytical techniques described in this method are dependent on the type of sample (matrix components, texture, etc.), on the preparation of the sample (homogeneity, grain size, etc.), and the specifics of the method (number of point counts for PLM, mass of sample for gravimetry, counting time for XRD,

etc.). These should be kept in mind when designing quality control procedures and characterizing performance, and are variables that must be tracked in the quality assurance system.

3.1 General Considerations

3.1.1 Training

Of paramount importance in the successful use of this or any other analytical method is the well-trained analyst. It is highly recommended that the analyst have completed course work in optical mineralogy on the collegiate level. That is not to say that others cannot successfully use this method, but the classification error rate[7] may, in some cases, be directly attributable to level of training. In addition to completed course work in optical mineralogy, specialized course work in PLM and asbestos identification by PLM is desirable. Experience is as important as education. A good laboratory training program can be used in place of course work. Analysts that are in training and not yet fully qualified should have all analyses checked by a qualified analyst before results are released. A QC Plan for asbestos identification would be considered incomplete without a detailed description of the analyst training program, together with detailed records of training for each analyst.

3.1.2 Instrument Calibration and Maintenance

Microscope alignment checks (alignment of the polarizer at 90° with respect to the analyzer, and coincident with the cross-lines, proper orientation of the slow vibration direction of the Red I compensator plate, image of the field diaphragm focussed in the plane of the specimen, centering of the central dispersion staining stop, etc.) should be performed with sufficient frequency to ensure proper operations. Liquids used for refractive index determination and those optionally used for dispersion staining should have periodic refractive index checks using a refractometer or known refractive index solids. These calibrations must be documented.

Microscopes and ancillary equipment should be maintained daily. It is recommended that at least once per year each microscope be thoroughly cleaned and re-aligned by a professional microscope service technician. Adequate inventories of replaceable parts

(illumination lamps, etc.) should be established and maintained. All maintenance must be documented.

3.2 Quality Control of Asbestos Analysis

3.2.1 Qualitative Analysis

All analysts must be able to correctly identify the six regulated asbestos types (chrysotile, amosite, crocidolite, anthophyllite, actinolite, and tremolite) using combined stereomicroscopic and PLM techniques. Standards for the six asbestos types listed are available from NIST, and should be used to train analysts in the measurement of optical properties and identification of asbestos. These materials can also be used as identification standards for XRD and AEM.

Identification errors between asbestos types (e.g. reporting amosite when tremolite is present) implies that the analyst cannot properly determine optical properties and is relying on morphology as the identification criteria. This is not acceptable. Each analyst in the lab should prove his or her proficiency in identifying the asbestos types; this can be checked through use of calibration materials (NVLAP proficiency testing materials, materials characterized by an independent technique, and synthesized materials) and by comparing results with another analyst. The identification of all parameters (e.g. refractive indices, birefringence, sign of elongation, etc.) leading to the identification should fall within control limits determined by the laboratory. In addition, a subset of materials should be analyzed using another technique to confirm the analysis.

As discussed earlier, the qualitative analysis is dependent upon matrix and asbestos type and texture. Therefore, the quality assurance system should monitor for samples that are difficult to analyze and develop additional or special steps to ensure accurate characterization of these materials. When an analyst is found to be out of the control limits defined by the laboratory, he or she should undergo additional training and have confirmatory analyses performed on all samples until the problem has been corrected.

3.2.2 Quantitative Analysis

The determination of the amount of asbestos in a sample can be accomplished using the various techniques outlined in this method. The mandatory stereomicroscopic and PLM examinations provide concentrations in terms of volume, area, or weight, depending upon the calibration procedure. Gravimetric and quantitative XRD techniques result in concentrations in units of weight percent. Specific guidelines for determining accuracy and precision using these techniques are provided in the appropriate sections of this method. In general, however, the accuracy of any technique is determined through analysis of calibration materials which are characterized by multiple independent techniques in order to provide an unbiased value for the analyte (asbestos) in question. The precision of any technique is determined by multiple analyses of the sample. The analyst is the detector for stereomicroscopic and PLM techniques, as opposed to gravimetric and XRD techniques, and therefore must be calibrated as an integral part of the procedure.

As in the qualitative analysis, the laboratory should determine its accuracy and precision for quantitative asbestos analysis according to the type of material analyzed and the technique used for analysis. For example, the laboratory may determine that its analysts have a problem with calibrated area estimates of samples containing cellulose and chrysotile and therefore needs to make or find special calibration materials for this class of sample.

Calibration materials for quantitative analysis of asbestos are available through the Bulk Asbestos NVLAP as proficiency testing materials for those laboratories enrolled in NVLAP. In a report provided following a test round, the concentration of asbestos in each sample is given in weight percent with 95%/95% tolerance limits, along with a description of the major matrix components. Materials from other round robin and quality assurance programs for asbestos analysis may not have been analyzed by independent techniques; the concentrations may represent consensus PLM results that could be significantly biased. Therefore, values from these programs should <u>not</u> be used as calibration materials for quantitative analysis.

Calibration materials for quantitative analysis can also be synthesized by mixing asbestos and appropriate matrix materials, as described in Appendix C of this method. These

materials are usually simplifications of "real world" samples; therefore the accuracy and precision determined from analysis of these materials are probably ideal.

Limits on permissible analytical variability must be established by the laboratory prior to QC implementation. It is recommended that a laboratory initially be at 100% quality control (all samples reanalyzed.) The proportion of quality control samples can later be lowered gradually, as control indicates, to a minimum of 10%. Quantitative results for standards including the mean and error estimate (typically 95% confidence or tolerance intervals) should be recorded. Over time these data can be used to help determine control limits for quality control charts.

The establishment and use of control charts is extensively discussed elsewhere in the literature. [1,2,3,4,5] Several cautions are in order:

- Control charts are based on the assumption that the data are distributed normally. Using rational subgrouping, the means of the subgroups are approximately normally distributed, irrespective of the distribution of the individual values in the subgroups. Control charts for asbestos analysis are probably going to be based on individual measurements, not rational subgroups. Check the data for normality before proceeding with the use of control charts. Ryan[8] suggests a minimum of 50 analyses before an attempt is made to establish control limits. However, for this analysis, consider setting "temporary" limits after accumulating 20-30 analyses of the sample.

- Include both prepared slides as well as bulk samples in your reference inventory.

- Make certain that sample quantities are sufficient to last, and that the act of sampling will not alter the composition of the reference sample.

Data on analytical variability can be obtained by having analysts repeat their analyses of samples and also by having different analysts analyze the same samples.

3.3 Interlaboratory Quality Control

The establishment and maintenance of an interlaboratory QC program is fundamental to continued assurance that the data produced within the laboratory are of consistent high quality. Intralaboratory programs may not be as sensitive to accuracy and precision error, especially if the control charts (see Section 3.2.2) for all analysts in the laboratory indicate small percent differences. A routine interlaboratory testing program will assist in the detection of internal bias and analyses may be performed more frequently than proficiency

testing. Arrangements should be made with at least two (preferably more) other laboratories that conduct asbestos identification by PLM. Samples (the number of which is left to the participating laboratories, but at least 4-10) representing the types of samples and matrices routinely submitted to the lab for analysis should be exchanged with sufficient frequency to determine intralaboratory bias. Both reference slides and bulk samples should be used. Results of the interlaboratory testing program should be evaluated by each of the participating laboratories and corrective actions, if needed, identified and implemented. Since quantitation problems are more pronounced at low concentrations ($\leq$ 5%), it would be prudent to include approximately 30-50% from this concentration range in the sample selection process.

3.4 Performance Audits

Performance audits are independent quantitative assessments of laboratory performance. These audits are similar to the interlaboratory QC programs established between several laboratories, but with a much larger cohort (the EPA Asbestos Bulk Sample Analysis Quality Assurance Program had as many as 1100 participating laboratories). Participation in this type of program permitted assessment of performance through the use of "consensus" test materials, and served to assist in assessing the bias relative to individual interlaboratory, as well as intralaboratory programs. Caution should be exercised in the use of "consensus" quantitation results, as they are likely to be significantly responsible for the propagation of high bias in visual estimates. The current NIST/NVLAP[9] for bulk asbestos laboratories (PLM) does not use concensus quantitation results. Results are reported in weight percent with a 95% tolerance interval. The American Industrial Hygiene Association (AIHA)[10] also conducts a proficiency testing program for bulk asbestos laboratories. Quantitation results for this program are derived from analyses by two reference laboratories and PLM, XRD and gravimetric analysis performed by Research Triangle Institute.

3.5 Systems Audits

Where performance audits are quantitative in nature, systems audits are qualitative. Systems audits are assessments of the laboratory quality system as specified in the Laboratory

Quality Assurance Manual. Such an audit might consist of an evaluation of some facet of the QA Manual, or the audit may be larger in scope. For example, the auditor might request specific laboratory data sheets which will be evaluated against written procedures for data recording in the laboratory. Or, the auditor might request air monitoring or contamination control data to review for frequency of sampling, analysis methodology, and/or corrective actions taken when problems were discovered. The audit report should reflect the nature of the audit as well as the audit results. Any recommendations for improvement should also be reflected in such a report.

3.6 References

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6. Bulk Asbestos Handbook, National Institute of Standards and Technology, National Voluntary Laboratory Accreditation Program, NISTIR 88-3879, October 1988.

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8. Ryan, T.P., **Statistical Techniques for Quality Improvement,** John Wiley & Sons, Inc., New York, 1989.

9. National Institute of Standards & Technology (NIST) National Voluntary Laboratory Accreditation Program (NVLAP), Building 411, Room A124, Gaithersburg, MD 20899, telephone (301) 975-4016.

10. American Industrial Hygiene Association (AIHA), 2700 Prosperity Avenue, Suite 250, Fairfax, VA 22031, (703) 849-8888.

APPENDIX A

Glossary Of Terms

APPENDIX A. GLOSSARY OF TERMS

Accuracy The degree of agreement of a measured value with the true or expected value.

Anisotropic Refers to substances that have more than one refractive index (e.g. are birefringent), such as nonisometric crystals, oriented polymers, or strained isotropic substances.

Asbestiform (morphology) Said of a mineral that is like asbestos, i.e., crystallized with the habit of asbestos. Some asbestiform minerals may lack the properties which make asbestos commercially valuable, such as long fiber length and high tensile strength. With the light microscope, the asbestiform habit is generally recognized by the following characteristics:

- Mean aspect ratios ranging from 20:1 to 100:1 or higher for fibers longer than 5μm. Aspect ratios should be determined for fibers, not bundles.
- Very thin fibrils, usually less than 0.5 micrometers in width, and
- Two or more of the following:

  Parallel fibers occurring in bundles,

  Fiber bundles displaying splayed ends,

  Matted masses of individual fibers, and/or

  Fibers showing curvature

These characteristics refer to the population of fibers as observed in a bulk sample. It is not unusual to observe occasional particles having aspect ratios of 10:1 or less, but it is unlikely that the asbestos component(s) would be dominated by particles (individual fibers) having aspect ratios of $<$20:1 for fibers longer than 5μm. If a sample contains a fibrous component of which most of the fibers have aspect ratios of $<$20:1 and that do not display the additional asbestiform characteristics, by definition the component should not be considered asbestos.

Asbestos - A commercial term applied to the asbestiform varieties of six different minerals. The asbestos types are chrysotile (asbestiform serpentine), amosite (asbestiform grunerite), crocidolite (asbestiform riebeckite), and asbestiform anthophyllite, asbestiform tremolite, and asbestiform actinolite. The properties of asbestos that caused it to be widely used commercially are: 1) its ability to be separated into long, thin, flexible fibers; 2) high tensile strength; 3) low thermal and electrical conductivity; 4) high mechanical and chemical durability, and 5) high heat resistance.

Becke Line - A band of light seen at the periphery of a specimen when the refractive indices of the specimen and the mounting medium are different; it is used to determine refractive index.

Bias - A systematic error characterized by a consistent (non-random) measurement error.

Binder - With reference to a bulk sample, a component added for cohesiveness (e.g. plaster, cement, glue, etc.).

Birefringence - The numerical difference between the maximum and minimum refractive indices of an anisotropic substance. Birefringence may be estimated, using a Michel-Levy chart, from the interference colors observed under crossed polarizers. Interference colors are also dependent on the orientation and thickness of the grain, and therefore are used qualitatively to determine placement in one of the four categories listed below.

Qualitative	Quantitative(N-n)
none	0.00 or isotropic
low	≤ 0.010
moderate	0.011-0.050
high	> 0.050

Bulk Sample - A sample of building material taken for identification and quantitation of asbestos. Bulk building materials may include a wide variety of friable and nonfriable materials.

Bundle - Asbestos structure consisting of several fibers having a common axis of elongation.

Calibration Materials - Materials, such as known weight % standards, that assist in the calibration of microscopists in terms of ability to quantitate the asbestos content of bulk materials.

Color - The color of a particle or fiber when observed in plane polarized light.

Compensator - A device with known, fixed or variable retardation and vibration direction used for determining the degree of retardation (hence the thickness or value of birefringence) in an anisotropic specimen. It is also used to determine the sign of elongation of elongated materials. The most common compensator is the first-order red plate (530-550nm retardation).

Control Chart - A graphical plot of test results with respect to time or sequence of measurement, together with limits within which they are expected to lie when the system is in a state of statistical control.

Detection Limit The smallest concentration/amount of some component of interest that can be measured by a single measurement with a stated level of confidence.

Dispersion Staining (focal masking) An optical means of imparting apparent or virtual color to transparent substances by the use of stops in the objective back focal plane; ir it is used to determine refractive indices.

Error Difference between the true or expected value and the measured value of a quantity or parameter.

Extinction The condition in which an anisotropic substance appears dark when observed between crossed polars. This occurs when the vibration directions in the specimen are parallel to the vibration directions in the polarizer and analyzer. Extinction may be complete or incomplete; common types include parallel, oblique, symmetrical and undulose.

Extinction Angle For fibers, the angle between the extinction position and the position at which the fiber is parallel to the polarizer or analyzer privileged directions.

Fiber With reference to asbestiform morphology, a structure consisting of one or more fibrils.

Fibril The individual unit structure of fibers.

Friable Refers to the cohesiveness of a bulk material, indicating that it may be crumbled or disaggregated by hand pressure.

Gravimetry Any technique in which the concentration of a component is determined by weighing. As used in this document, it refers to measurement of asbestos-containing residues after sample treatment by ashing, dissolution, etc.

Homogeneous Uniform in composition and distribution of all components of a material, such that multiple subsamples taken for analysis will contain the same components in approximately the same relative concentrations.

Heterogeneous Lacking uniformity in composition and/or distribution of material; components not uniform. Does not satisfy the conditions stated for homogenous; e.g., layered or in clumps, very coarse grained, etc.

Isotropic Refers to substances that have a single refractive index such as unstrained glass, un-oriented polymers and unstrained substances in the isometric crystal system.

Lamda Zero (λ_0) The wavelength (λ_0) of the dispersion staining color shown by a specimen in a medium; both the specimen and medium have the same refractive index at that wavelength.

Matrix Nonasbestos, nonbinder components of a bulk material. Includes such components as cellulose, fiberglass, mineral wool, mica, etc.

Michel-Levy Scale of Retardation colors A chart plotting the relationship between birefringence, retardation and thickness of anisotropic substances. Any one of the three variables can be determined if the other two are known.

Morphology The structure and shape of a particle. Characterization may be descriptive (platy, rod-like, acicular, etc) or in terms of dimensions such as length and diameter (see asbestiform).

Pleochroism The change in color or hue of colored anisotropic substance when rotated relative to the vibration direction of plane polarized light.

Point Counting A technique used to determine the relative projected areas occupied by separate components in a microscope slide preparation of a sample. For asbestos analysis, this technique is used to determine the relative concentrations of asbestos minerals to nonasbestos sample components.

Polarization Colors Interference colors displayed by anisotropic substances between two polarizers. Birefringence, thickness and orientation of the material affect the colors and their intensity.

Precision The degree of mutual agreement characteristic of independent measurements as the result of repeated application of the process under specified conditions. It is concerned with the variability of results.

Reference Materials Bulk materials, both asbestos-containing and nonasbestos-containing, for which the components are well-documented as to identification and quantitation.

Refractive Index (index of refraction) The ratio of the velocity of light in a vacuum relative to the velocity of light in a medium. It is expressed as n and varies with wavelength and temperature.

Sign of Elongation Referring to the location of the high and low refractive indices in an elongated anisotropic substance, a specimen is described as positive when the higher refractive index is lengthwise (length slow), and as negative when the lower refractive index is lengthwise (length fast).

Standard Reference Material (SRM) A reference material certified and distributed by the National Institute of Standards and Technology.

Visual Estimate An estimation of concentration of asbestos in a sample as compared to the other sample components. This may be a volume estimate made during stereomicroscopic examination and/or a projected area estimation made during microscopic (PLM) examination.

APPENDIX B

Apparatus For Sample Preparation And Analysis

B1.0 INTRODUCTION

The following lists the apparatus and materials required and suggested for the methods of sample preparation and analysis described in the test method.[1,2,3]

B2.0 STEREOMICROSCOPIC EXAMINATION

The following are suggested for routine stereomicroscopic examination.

- HEPA-filtered hood or class 1 biohazard hood, negative pressure
- Microscope: binocular microscope, preferably stereoscopic, 5-60X magnification (approximate)
- Light source: incandescent or fluorescent
- Tweezers, dissecting needles, scalpels, probes, etc. (for sample manipulation)
- Glassine paper, glass plates, weigh boats, petri dishes, watchglasses, etc. (sample containers)

The following are suggested for sample preparation.

- Mortar and pestle, silica or porcelain-glazed
- Analytical balance (readability less than or equal to one milligram) (optional)
- Mill or blender (optional)

B3.0 POLARIZED LIGHT MICROSCOPY

The laboratory should be equipped with a polarized light microscope (preferably capable of Köhler or Köhler-type illumination if possible) and accessories as described below.

- Ocular(s) binocular or monocular with cross hair reticle, or functional equivalent, and a magnification of at least 8X
- 10X, 20X, and 40X objectives, (or similar magnification)

- Light source (with optional blue "day-light" filter)
- 360-degree rotatable stage
- Substage condenser with iris diaphragm
- Polarizer and analyzer which can be placed at 90 degrees to one another, and can be calibrated relative to the cross-line reticle in the ocular.
- Accessory slot for wave plates and compensators (or demonstrated equivalent).
- Wave retardation plate (Red I compensator) with approximately 550 nanometer retardation, and with known slow and fast vibration directions.
- Dispersion staining objective or a demonstrated equivalent. (optional)
- Monochromatic filter (n_D), or functional equivalent. (optional)

In addition, the following equipment, materials and reagents are required or recommended.[1]

- NIST traceable standards for the major asbestos types (NIST SRM 1866 and 1867)
- Class I biohazard hood or better (see "Note", Section 2.2.5)
- Sampling utensils (razor knives, forceps, probe needles, etc.)
- Microscope slides and cover slips
- Mechanical Stage
- Point Counting Stage (optional)
- Refractive index liquids: 1.490-1.570, 1.590-1.720 in increments of less than or equal to 0.005; high dispersion, (HD) liquids are optional; however, if using dispersion staining, HD liquids are recommended.
- Mortar and pestle
- Distilled water
- HCl, ACS reagent grade concentrated

- Muffle furnace (optional)
- Mill or blender (optional)
- Beakers and assorted glassware (optional)
- Other reagents (tetrahydrofuran, amyl acetate, acetone, sodium hexametaphosphate, etc.) (optional)

B4.0 GRAVIMETRY

The following equipment, materials, and reagents are suggested.

- Scalpels
- Crucibles, silica or porcelain-glazed, with lids
- Muffle furnace temperature range at least to 500°C, temperature stable to $\pm$ 10°C, temperature at sample position calibrated to $\pm$ 10°C
- Filters, 0.4 μm pore size polycarbonate
- Petri dishes
- Glass filtration assembly, including vacuum flask, water aspirator, and/or air pump
- Analytical balance, readable to 0.001 gram
- Mortar and pestle, silica or porcelain-glazed
- Heat lamp or slide warmer
- Beakers and assorted glassware
- Centrifuge, bench-top
- Class I biohazard hood or better
- Bulb pipettes
- Distilled water
- HCl, reagent-grade concentrated

- Organic solvents (tetrahydrofuran, amyl acetate,etc)
- Ultrasonic bath

B5.0 X-RAY DIFFRACTION

Sample Preparation

Sample preparation apparatus requirements will depend upon the sample type under consideration and the kind of XRD analysis to be performed.

- Mortar and pestle: agate or porcelain
- Razor blades
- Sample mill: SPEX, Inc., freezer mill or equivalent
- Bulk sample holders
- Silver membrane filters: 25-mm diameter, 0.45-μm pore size. Selas Corp. of America, Flotronics Div., 1957 Pioneer Road, Huntington Valley, PA 19006
- Microscope slides
- Vacuum filtration apparatus: Gelman No. 1107 or equivalent, the side-arm vacuum flask
- Microbalance
- Ultrasonic bath or probe: Model W140, Ultrasonics, Inc., operated at a power density of approximately 0.1 W/mL, or equivalent
- Volumetric flasks: 1-L volume
- Assorted pipets
- Pipet bulb
- Nonserrated forceps
- Polyethylene wash bottle
- Pyrex beakers: 50-mL volume

- Desiccator
- Filter storage cassettes
- Magnetic stirring plate and bars
- Porcelain crucibles
- Muffle furnace or low temperature asher
- Class 1 biohazard hood or better

Sample Analysis

Sample analysis requirements include an x-ray diffraction unit, equipped with:

- Constant potential generator; voltage and mA stabilizers
- Automated diffractometer with step-scanning mode
- Copper target x-ray tube: high intensity; fine focus, preferably
- X-ray pulse height selector
- X-ray detector (with high voltage power supply): scintillation or proportional counter
- Focusing graphite crystal monochromator; or nickel filter (if copper source is used, and iron fluorescence is not a serious problem)
- Data output accessories:
  Strip chart recorder
  Decade scaler/timer
  Digital printer

  or

  PC, appropriate software and Laser Jet Printer
- Sample spinner (optional)
- Instrument calibration reference specimen: α-quartz reference crystal (Arkansas quartz standard, #180-147-00, Philips Electronics Instruments, Inc., 85 McKee Drive, Mahwah, NJ 07430) or equivalent.

Reagents, etc.

Reference Materials The list of reference materials below is intended to serve as a guide. Every attempt should be made to acquire pure reference materials that are comparable to sample materials being analyzed.

- Chrysotile: UICC Canadian, NIST SRM 1866 (UICC reference material available from: UICC, MRC Pneumoconiosis Unit, Llandough Hospital, Penarth, Glamorgan, CF61XW, UK); (NIST Standard Reference Materials available from the National Institute of Standards and Technology, Office of Reference Standards, Gaithersburg, MD 20899)
- Crocidolite: UICC, NIST SRM 1866.
- "Amosite": UICC, NIST SRM 1866.
- Anthophyllite-Asbestos: UICC, NIST SRM 1867
- Tremolite Asbestos: Wards Natural Science Establishment, Rochester, NY; Cyprus Research Standard, Cyprus Research, 2435 Military Ave., Los Angeles, CA 900064 (washed with dilute HCl to remove small amount of calcite impurity); Indian tremolite, Rajasthan State, India; NIST SRM 1867.
- Actinolite Asbestos: NIST SRM 1867

Adhesive Tape, petroleum jelly, etc. (for attaching silver membrane filters to sample holders).

Surfactant 1 Percent aerosol OT aqueous solution or equivalent.

Isopropanol ACS Reagent Grade.

B6.0 ANALYTICAL ELECTRON MICROSCOPY

AEM equipment requirements will not be discussed in this document; it is suggested that equipment requirements stated in the AHERA regulations be followed. Additional information may be found in the NVLAP Program Handbook for Airborne Asbestos Analysis.[3]

The following additional materials and equipment are suggested:

- Analytical balance, readable to 0.001 gram
- Ultrasonic bath
- Glass filtration assembly (25mm), including vacuum flask and water aspirator
- Mixed cellulose ester (MCE) filters (0.22μm pore size) or 0.2μm pore size polycarbonate filters
- MCE backing filters (5μm pore size)
- Silica mortar and pestle
- Beakers glass and disposable
- Pipettes, disposable, 1,5, and 10 ml

B7.0 REFERENCES

1. National Institute of Standards and Technology (NIST) National Voluntary Laboratory Accreditation Program (NVLAP) Bulk Asbestos Handbook, NISTIR 88-3879, 1988.
2. **Interim Method for the Determination of Asbestos in Bulk Insulation Samples,** U.S. E.P.A. 600/M4-82-020, 1982.
3. National Institute of Standards and Technology (NIST) National Voluntary Laboratory Accreditation Program (NVLAP) Program Handbook for Airborne Asbestos Analysis, NISTIR 89-4137, 1989.

APPENDIX C

Preparation and Use of Bulk Asbestos Calibration Standards

C1.0 INTRODUCTION

Evaluation of the results from national proficiency testing programs for laboratories analyzing for asbestos in bulk materials indicates that laboratories have had, and continue to have, problems with quantitation of asbestos content, especially with samples having a low asbestos concentration.[1] For such samples, the mean value of asbestos content reported by laboratories may be four to ten times the true weight percent value. It is assumed that the majority of the laboratories quantify asbestos content by visual estimation, either stereomicroscopically or microscopically; therefore, the problem of quantitation must be attributed to lack of or inadequate calibration of microscopists.

As calibration standards for asbestos-containing bulk materials are not currently commercially available, laboratories should consider generating their own calibration materials. This may be done rather easily and inexpensively.

C2.0 MATERIALS AND APPARATUS

Relatively pure samples of asbestos minerals should be obtained. Chrysotile, amosite and crocidolite (SRM 1866) and anthophyllite, tremolite and actinolite (SRM 1867) are available from NIST. A variety of matrix materials are commercially available; included are calcium carbonate, perlite, vermiculite, mineral wool/fiberglass, and cellulose. Equipment, and materials needed to prepare calibration bulk materials are listed below.

- Analytical balance, readable to 0.001 gram
- Blender/mixer; multi-speed, ~ one quart capacity
- Filtration assembly, including vacuum flask, water aspirator and/or air pump (optional)
- HEPA-filtered hood with negative pressure
- Filters, 0.4μm pore size polycarbonate (optional)
- Beakers and assorted glassware, weigh boats, petri dishes, etc.
- Hot/warm plate

- Asbestos minerals
- Matrix materials
- Distilled water.

C3.0 MATERIAL FORMULATION PROCEDURES

The formulation procedure involves first weighing appropriate quantities of asbestos and matrix material to give the desired asbestos weight percent. The following formula may be used to determine the weights of asbestos and matrix materials needed to give a desired weight percent asbestos.

$$\frac{WTa}{Wa} = \frac{WTm}{Wm}$$

Where:

WTa = weight of asbestos in grams (to 0.001 gram)
WTm = weight of matrix materials in grams (to 0.001 gram)
Wa = weight percent asbestos
Wm = weight percent matrix

Example: The desired total weight for the calibration sample is ~ 10 grams containing 5% asbestos by weight. If 0.532 grams of asbestos are first weighed out, what corresponding weight of matrix material is required?

WTa = 0.532 grams
Wa = 5%
Wm = 95%

$$\frac{0.532}{5} = \frac{WTm}{95}$$

Then: WTm = 10.108 grams

The matrix is then placed into the pitcher of a standard over-the-counter blender, the pitcher being previously filled to approximately one-fourth capacity (8-10 ounces) with distilled water. Blending is performed at the lowest speed setting for approximately ten seconds which serves to disaggregate the matrix material. The asbestos is then added, with additional blending of approximately 30 seconds, again at the lowest speed setting. Caution should be taken not to overblend the asbestos-matrix mixture. This could result in a significant reduction in the size of the asbestos fibers causing a problem with detection at normal magnification during stereomicroscopic and microscopic analyses. Ingredients of the

pitcher are then poured into a filtering apparatus, with thorough rinsing of the pitcher to ensure complete material removal. After filtering, the material is transferred to a foil dish which is placed on a hot plate. The material is covered and allowed to sit over low heat until drying is complete; intermittent stirring will speed the drying process. For fine-grained matrix materials such as gypsum, calcium carbonate, clays, etc., the sample is not filtered after the blending process. Instead, the ingredients in the pitcher are transferred into a series of shallow, glass (petri) dishes. The ingredients should be stirred well between each pouring to minimize the possible settling (and over-representation) of some components. The dishes are covered and placed on a hot plate until the contents are thoroughly dried. For small quantities of any matrix materials (15 grams or less), air-drying without prior filtering is generally very suitable for removing water from the prepared sample. For each material, the final step involves placing all formulated, dried subsamples into a plastic bag (or into one petri dish, for small quantities), where brief hand-mixing will provide additional blending and help to break up any clumps produced during drying. **All operations should be performed in a safety-hood with negative pressure.**

C4.0 ANALYSIS OF MATERIALS

All formulations should be examined with the stereomicroscope to determine homogeneity. Gravimetric analysis (ashing and/or acid dissolution) should be performed on those materials containing organic and/or acid-soluble components. Matrix materials to which no asbestos has been added should be analyzed by gravimetric analysis to determine the amount of nonashable or insoluble materials that are present. Several subsamples of each material should be analyzed by the gravimetric technique to provide information concerning the uniformity of the prepared materials. Experience has shown that the previously described formulation procedure results in relatively homogeneous materials.[2]

C4.1 Stereomicroscopic Analysis

Visual estimation of sample components using the stereomicroscope is in reality a comparison of the relative volumes of the components.[3] Therefore, differences in specific gravity between asbestos and matrix material must be considered and the relationship

between weight percent and volume percent must be determined.[4] Materials such as expanded vermiculite, perlite, and cellulose have specific gravities significantly lower than asbestos minerals. Table C1 lists the specific gravities for the three most commonly encountered asbestos varieties and several common matrix materials.

TABLE C1. SPECIFIC GRAVITIES OF ASBESTOS VARIETIES AND MATRIX MATERIALS

Asbestos Type	Specific Gravity	Matrix Type	Specific Gravity
Chrysotile	2.6	Calcium Carbonate	2.7
		Gypsum	2.3
Amosite	3.2	Perlite	~0.4
		Vermiculite (expanded)	~0.3
		Mineral Wool	~2.5
Crocidolite	3.3	Fiberglass	~2.5
		Cellulose	~0.9

The conversion of weight percent asbestos to equivalent volume percent asbestos is given by the following formula:

$$\frac{\frac{Wa}{Ga}}{\frac{Wa}{Ga} + \frac{Wm}{Gm}} \times 100 = Va$$

where:

Wa = weight percent asbestos
Ga = specific gravity of asbestos
Wm = weight percent matrix
Gm = specific gravity of matrix
Va = volume percent asbestos

Example: Chrysotile and perlite have been combined to form a 5% asbestos calibration standard, by weight. What is the equivalent volume percent asbestos?

$$Wa = 5\% \quad Ga = 2.6 \quad Wm = 95\% \quad Gm = 0.4$$

$$Va = \frac{\frac{5}{2.6}}{\frac{5}{2.6} + \frac{95}{0.4}} \times 100 = 0.8\%$$

Conversely, to convert volume percent asbestos to equivalent weight percent, the following formula may be used.

$$\frac{(Va)(Ga)}{(Va)(Ga)+(Vm)(Gm)} \times 100 = Wa$$

Vm = volume percent matrix

Example: A calibration standard consisting of amosite and cellulose is estimated to contain 2% asbestos, by volume. What is the equivalent weight percent asbestos?

$$Va = 2\% \quad Ga = 3.2 \quad Vm = 98\% \quad Gm = 0.9$$

$$Wa = \frac{(2)(3.2)}{(2)(3.2)+(98)(0.9)} \times 100 = 6.77\%$$

Volume percentages should be calculated for all calibration materials prepared so that visual estimates determined by examination with the stereomicroscope may be compared to true volume concentrations.

Figure C1 illustrates the relationship between volume percent and weight percent of chrysotile mixed with vermiculite and cellulose respectively. It should be noted that when asbestos in a low weight percentage is mixed with matrix materials having low specific gravities (vermiculite, perlite), the resulting volume concentration of asbestos is very low For example, a mixture containing three percent chrysotile by weight in a cellulose matrix would result in a volume percent asbestos of approximately 1.1%; in a vermiculite matrix, the resulting volume percent asbestos would be approximately 0.4%. In the latter case especially, an analyst might possibly fail to detect the asbestos or consider it to be present in only trace amounts.

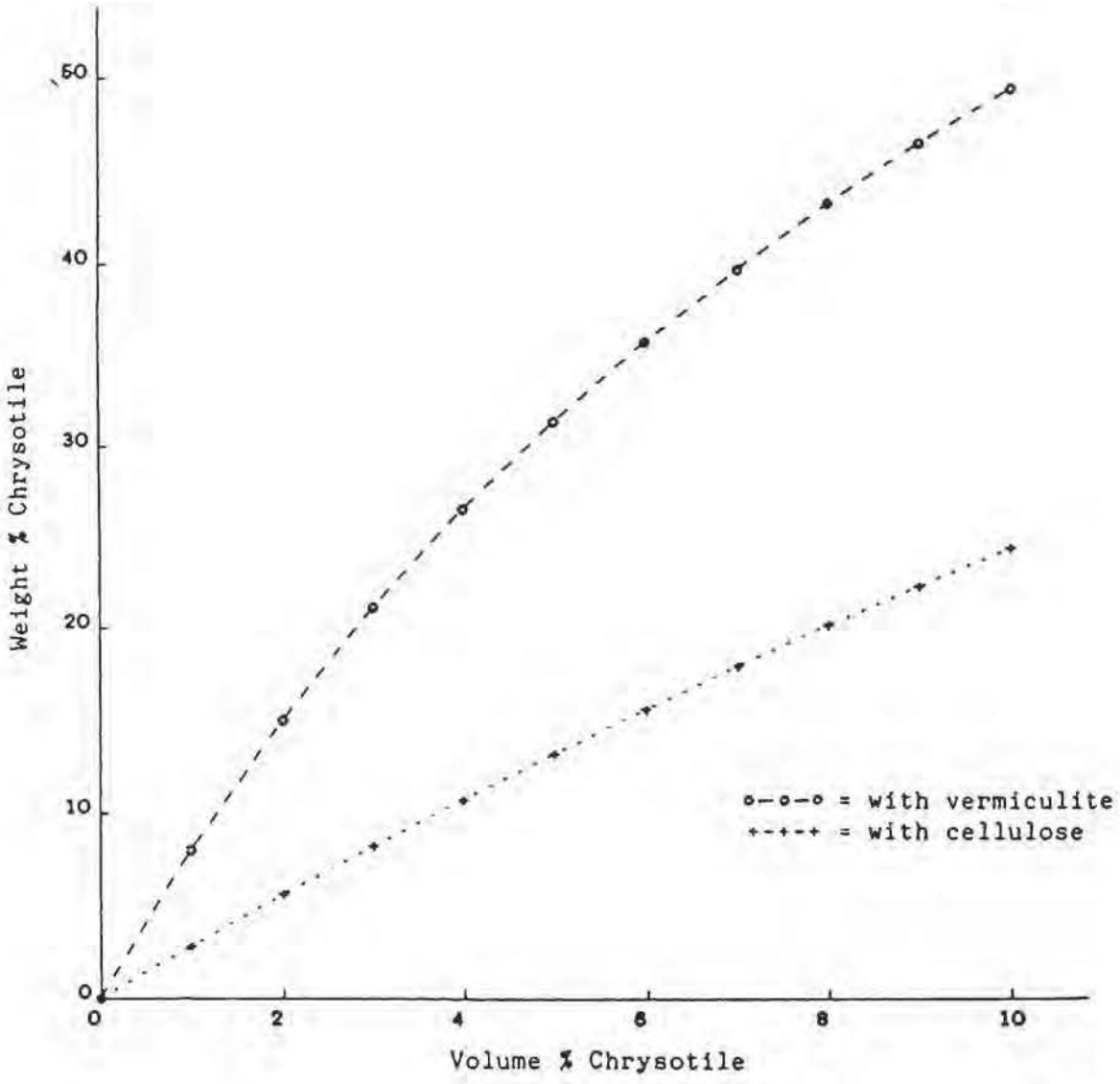


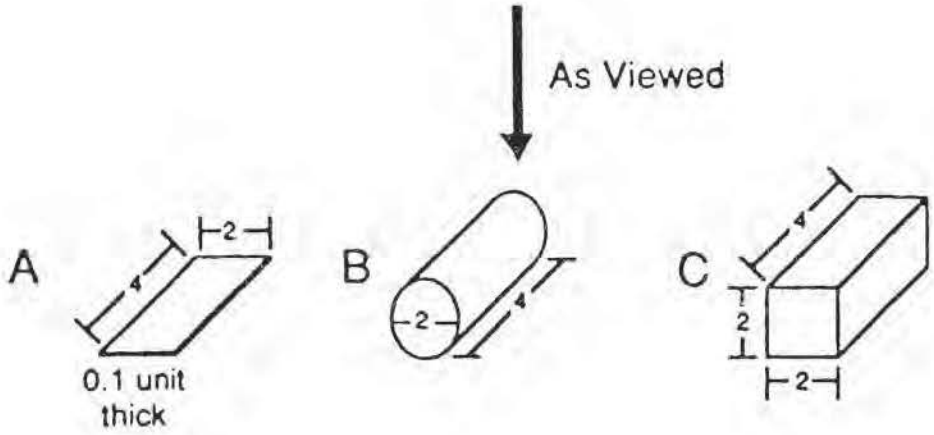
Figure C1. Relationship between volume % and weight % of chrysotile mixed with a)vermiculite and b) cellulose.

C4.2 Microscopical Analysis (PLM)

The polarized light microscope may be used to quantify asbestos and other components of a sample. Slide mounts are prepared from "pinch" samples of the calibration material and asbestos content is determined by visual area estimate and/or point counting. Both of these quantitation techniques are in fact estimates or measurements of the relative projected areas of particles as viewed in two dimensions on a microscope slide. For quantitation results to be meaningful, the following conditions should be met:

- The sample should be homogeneous for slide preparations, which are made from small pinches of the sample, to be representative of the total sample.
- Slide preparation should have an even distribution of particles and approach a one particle thickness (seldom achieved) to avoid particle overlap.
- All materials used should be identified and specific gravities determined in order to relate area percent to volume and/or weight percent.
- The size (thickness) relationship between matrix particles and asbestos fibers should be determined if the results based on projected area are to be related to volume and/or weight percent.

Particle characteristics can greatly affect the quantitation results obtained by visual area estimation or point counting. Figure C2 illustrates three hypothetical particle shapes of identical length and width (as viewed from above). Although the three-dimensional shape is different, the projected area is equal for all particles. The table accompanying Figure C2 presents data for each particle in terms of thickness, volume and projected area. It should be noted that although the projected areas may be equal, the volumes represented by the particles may vary by a factor of 20(0.8 vs 16 cubic units). It is obvious that quantitation of a sample consisting of a mixture of particles with widely ranging particle thicknesses could result in different results. For example, if a sample contained relatively thick bundles of asbestos and a fine-grained matrix such as clay or calcium carbonate, the true asbestos content (by volume) would likely be underestimated. Conversely, if a sample contained thick "books" of mica and thin bundles of asbestos, the asbestos content (by volume) would likely be overestimated.



Particle	Thickness	Volume	Projected Area
A	0.1 units	0.8 cubic units	8 sq. units
B	2 units	12.6 cubic units	8 sq. units
C	2 units	16 cubic units	8 sq. units

Note that although all particles have the same projected area, particle C volume is 20x that of particle A.

Figure C2. Relationship of projected area to volume and thickness for three different particles as viewed on a slide mount.

Table C2 illustrates several examples of expected results from area estimates or point counting of samples in which the asbestos fibers and matrix particles differ in thickness.

TABLE C2. RELATIONSHIP OF WEIGHT PERCENT, VOLUME PERCENT AND PARTICLE THICKNESS TO QUANTITATION RESULTS

Composition of Sample In Wt. %	Theoretical Vol. % Asbestos	Thickness Factor* (Matrix/Asbestos)	Expected Area %
1% Amosite 99% Calcium Carbonate	0.9	0.5	0.4
1% Amosite 99% Calcium Carbonate	0.9	1	0.9
1% Amosite 99% Calcium Carbonate	0.9	2	1.8
1% Amosite 99% Vermiculite	0.1	1	0.1
1% Amosite 99% Vermiculite	0.1	10	1.0
1% Amosite 99% Vermiculite	0.1	20	2.0
1% Amosite 99% Vermiculite	0.1	30	2.9

* Value represents the relationship between the mean thickness of the matrix particles compared to the mean thickness of the asbestos particles.

It should be noted that it is not uncommon for matrix particle thickness to differ greatly from asbestos fiber thickness, especially with matrix materials such as vermiculite and perlite; vermiculite and perlite particles may be 20 - 30 times as thick as the asbestos fibers.

The general size relationships between matrix particles and asbestos fibers may be determined by scanning slide mounts of a sample. A micrometer ocular enables the microscopist to actually measure particle sizes.

If a thickness factor can be determined for a calibration sample of known volume proportions of asbestos and matrix materials, an expected equivalent projected area asbestos can be calculated using the following formula:

$$\frac{Va}{\frac{Vm}{T} + Va} \times 100 = Aa$$

where:

Va = true volume percent asbestos
Vm = true volume percent matrix
T = thickness factor (mean size matrix particle/mean size asbestos fiber)
Aa = expected projected area percent asbestos

Example: A calibration standard of known weight percent asbestos is determined, by factoring in component specific gravities, to be 5.0% asbestos by volume. The matrix particles are estimated to be ten times thicker than the asbestos fibers. What would be the expected projected area percentage of asbestos?

Va = 5%
Vm = 95%
T = 10

$$Aa = \frac{5}{\frac{95}{10} + 5} \times 100 = 34.5\%$$

Conversely, to convert projected area percent asbestos to equivalent volume percent, the following formula may be used:

$$\frac{Aa}{T(Am) + Aa} \times 100 = Va$$

Where: Am = projected area matrix

Example: A slide containing a subsample of an amosite/mineral wool calibration standard is determined by point counting to have a projected area asbestos of 18.6%. If the mineral wool fibers are estimated to be six times the asbestos fibers, in diameter, what is the equivalent volume percent asbestos?

Am = 81.4%
Aa = 18.6%
T = 6

$$Va = \frac{(18.6)}{6(81.4) + 18.6} \times 100 = 3.67\%$$

Based on specific gravity values listed in Table 1C and on the above volume asbestos determination, what is the equivalent weight percent asbestos in the sample?

Va = 3.67%
Ga = 3.2
Vm = 96.33%
Gm = 2.5

$$Wa = \frac{(3.67)(3.2)}{(3.67)(3.2) + (96.33)(2.5)} \times 100 = 4.7\%$$

C5.0 USE OF CALIBRATION STANDARDS FOR QA/QC

Once the materials have been formulated and thoroughly characterized by all techniques to determine their suitability as calibration standards, a system for incorporating them into the QA/QC program should be established. Someone should be designated (QA officer, lab supervisor, etc.) to control the distribution of standards and to monitor the analysis results of the microscopists. Both precision and accuracy may be monitored with the use of suitable standard sets.

Records such as range charts, control charts, etc. may be maintained for volume (stereomicroscopic estimates), area (PLM) estimates and point counts. For point counts and area estimates, relatively permanent slides may be made using epoxy or Melt Mount *. Such slides may be very accurately quantified over time as to point count values, and due to their very long shelf life, may be used for QA/QC purposes almost indefinitely.

C6.0 REFERENCES

1. "Analysis Summaries for Samples used in NIST Proficiency Testing", National Institute of Standards and Technology (NIST) National Voluntary Laboratory Accreditation Program (NVLAP) for Bulk Asbestos, January 1989 to present.

2. Harvey, B. W., R. L. Perkins, J. G. Nickerson, A. J. Newland and M. E. Beard, "Formulating Bulk Asbestos Standards", **Asbestos Issues**, April 1991.

3. Perkins, R. L. and M. E. Beard, "Estimating Asbestos Content of Bulk Materials", **National Asbestos Council Journal**, Vol. 9, No. 1, 1991, pp. 27-31.

4. **Asbestos Content in Bulk Insulation Samples: Visual Estimates and Weight Composition**, U.S. Environmental Protection Agency 560/5-88-011, 1988.

APPENDIX D

Special-Case Building Materials

Asbestos laboratories are now called upon to analyze many types of bulk building materials that are very difficult to characterize by routine PLM analysis. These materials are dominantly nonfriable and can be grouped into the following categories:

- Cementitious Products (pipe, sheeting, etc.)
- Viscous Matrix Products (adhesives, cements, coatings, etc.)
- Vinyl Materials (vinyl floor tile, sheeting)
- Asphaltic Roofing Materials (shingles, roll roofing)
- Miscellaneous Products (paints, coatings, friction plates, gaskets, etc.)

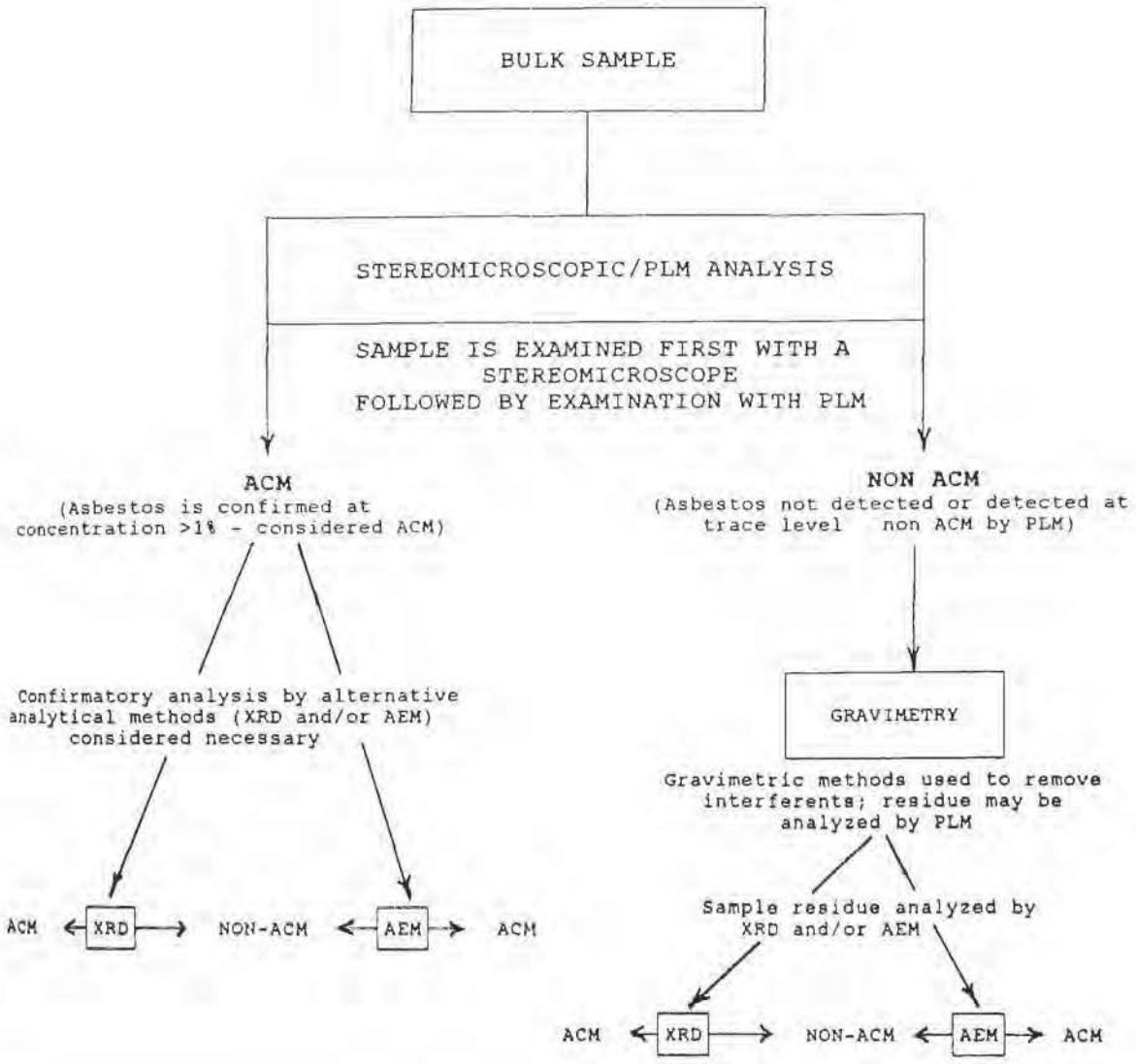
Materials characterized by interfering binder/matrix, low asbestos content, and/or small fiber size may require that additional sample treatment(s) and analysis be performed beyond routine PLM analysis. The sample treatment(s) required is(are) determined by the dominant nonasbestos sample components (see Section 2.3, Gravimetry). Materials containing an appreciable amount of calcareous material may be treated by dissolution with hydrochloric acid. Samples containing organic binders such as vinyl, plasticizers, esters, asphalts, etc. can be treated with organic solvents or ashed in a muffle furnace (preferred method) or low temperature plasma asher to remove unwanted components. Materials containing cellulose, synthetic organic fibers, textiles, etc. may also be ashed in a muffle furnace or low temperature plasma asher.

The method chosen for analysis of a sample after treatment is dependent on asbestos concentration and/or fiber size. An examination of the sample residue by PLM may disclose asbestos if the fibers are large enough to be resolved by the microscope, but additional analytical methods are required if the sample appears negative. Analysis by XRD is not fiber-size dependent, but may be limited by low concentration of asbestos and the presence of interfering mineral phases. In addition, the XRD method does not differentiate between fibrous and nonfibrous varieties of a mineral. Analysis by AEM is capable of providing positive identification of asbestos type(s) and semi-quantitation of asbestos content.

The following flowchart illustrates a possible scheme for the analysis of special-case building materials.

NOTE: Preliminary studies indicate that the XRD method is capable of detecting serpentine (chrysotile) in floor tile samples without extensive sample preparation prior to XRD analysis. XRD analysis of small, intact sections of floor tile yielded diffraction patterns that confirmed the presence of serpentine, even at concentrations of ~one percent by weight. TEM analysis of these same tiles confirmed the presence of chrysotile asbestos. With further investigation, this method may prove applicable to other types of nonfriable materials.

FLOWCHART FOR QUALITATIVE ANALYSIS OF SPECIAL CASE BUILDING MATERIALS SUCH AS FLOOR TILES, ASPHALTIC MATERIALS, VISCOUS MATRIX MATERIALS, ETC.*



*Although this flowchart is applicable to all bulk materials, it is primarily intended to be used with known problem materials that are difficult to analyze by PLM due to low asbestos concentration, and/or small fiber size, and/or interfering binder/matrix. In addition to being qualitative, the results may also be semi-quantitative. It should not be assumed that all samples need to be analyzed by AEM and XRD. The flowchart simply illustrates options for methods of analysis. Alternate methods such as SEM may be applicable to some bulk materials.

*U.S. GOVERNMENT PRINTING OFFICE 1993 -750-002/80237

APPENDIX G – DATA QUALITY OBJECTIVES

Data Quality Objectives for Investigation Sampling

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.

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

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.

During O&M, there may be properties encountered where it is unknown whether LA contamination exists.

This O&M sampling guidance was developed to reflect current investigation and sampling methods and is designed to determine if LA and/or LA source materials are present, and if so, the extent of the contamination.

Step 2 - Identify the Decision

The principal study questions are as follows:

1. Is vermiculite-containing materials (e.g., chinking, plaster, mortar, and other materials on boilers, pipes, or other appurtenances) present in buildings?
2. Is LA detected at levels greater than or equal to 1% in any surface soil samples collected from individual properties?
    a. What is the extent of LA contamination in the soils?
3. Is vermiculite insulation present in buildings?

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 1.

Table 1 –Investigation Inputs to Resolve Study Questions and Use of Input Information

Principal Study Question	Input to Resolve Question	Use of Input to Resolve Questions
Is vermiculite-containing material observed in building materials?	Visual Inspection/ Building Material Samples	Attics, living spaces, walls, and understructures will be inspected for vermiculite-containing building materials to the extent possible. Samples will be collected if friable materials are observed do determine if LA source material is present. If so, the results of the visual inspection will be used to determine the extent of LA source materials for removal planning.
Is LA detected at levels greater than or equal to 1% in any surface soil samples collected from individual properties?	Soil Samples	Surface soil samples will be collected from use areas (e.g., SUAs, CUAs, LUAs, etc.). The results of the surface soil samples will be used to determine if LA contamination is present at individual properties and the extent of LA source materials for removal planning.
Is vermiculite insulation present in property buildings?	Visual Inspection	The results of the visual inspection will be used to determine the extent of LA source materials for removal planning.

Step 4 – Define the Boundaries of the Study

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

Spatial Bounds

The information gathered to answer the investigation objectives will be collected from properties within the Superfund site boundaries of OUs 1, 2, 4, 5, 7, and 8 during O&M as directed by DEQ. The boundaries are the property boundaries that the investigation is occurring (the entire building or structure) and the depth of soil samples collected, approximately 6 inches below ground surface.

Temporal Bounds

For each property, the temporal boundaries of this investigation include the time from when an investigation begins to the time it is determined whether LA or LA source materials exist on the property.

Step 5 – Develop Decision Rules

The purpose of this step is to describe the method that DEQ will use to determine if 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 2.

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 investigation sampling, two types of decision errors are possible:

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

Type I errors may leave humans exposed to unacceptable levels of LA. Type II errors may result in unnecessary expenditures for abatement during O&M. Table 3 summarizes the consequences of making a decision error.

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 this O&M Sampling Guidance.

Table 2 – Decision Rules

Principal Study Question	Input to Resolve Question	Input Requirements	Action Level	Decision Rule
Is vermiculite-containing material observed in building materials?	Visual Inspection	Presence or absence of vermiculite-containing building materials	Presence of vermiculite	If vermiculite is observed in any friable building material, collect samples. If vermiculite is not observed, take no action.
	Building Material Samples	Analysis: PLM NIOSH 9002, PLMPC400 Reported Result: %LA	≥ 0.25 % LA	If levels of LA are ≥ 0.25 % LA by polarized light microscopy using point counting (400 points examined) (PLM-PC400), the location will be documented for subsequent removal action. If levels of LA < 0.25 %, take no action.
Is LA detected above the action level in any surface soil samples collected from individual residential or commercial or park/school properties?	Soil Samples	Analysis PLM-VE and PLM-Grav (w/ Libby-specific modifications) Reported Result: % LA Analytical Sensitivity: 0.2%	For frequently used areas: ≥ 0.2% LA, regardless of spatial extent *or* ≤0.2% (trace) if the spatial extent is more than 25% of the total soil exposure area at a property For infrequently used areas: ≥ 0.2% LA	If levels of LA in surface soil samples are above action level, the location will be documented for subsequent removal action. If levels of LA in surface soil samples are below action level, take no action.
Is LA detected above the action level in any surface soil samples collected from individual transportation corridors or industrial properties?	Soil Samples	Analysis PLM-VE and PLM-Grav (w/ Libby-specific modifications) Reported Result: % LA Analytical Sensitivity: 0.2%	≥ 0.2% LA	If levels of LA in surface soil samples are above action level, the location will be documented for subsequent removal action. If levels of LA in surface soil samples are below action level, take no action.
Is vermiculite insulation present in property buildings?	Visual Inspection	Presence or absence of vermiculite insulation	Presence of vermiculite	If vermiculite is observed, the location will be documented for subsequent removal action. If vermiculite is not observed, take no action.

Table 3 – Limits on Decision Errors

Principal Study Question	Null Hypothesis	Type I Error Will Result in:	Type II Error Will Result In:
Is vermiculite-containing material observed in building materials?	Friable building materials contain vermiculite	Determining that friable building materials are not contaminated with LA when they actually are. This may result in no subsequent removal action, and in turn, resulting in increased human health risk.	Determining that friable building materials contain unacceptable levels of LA when they actually do not. This would result in unnecessarily performing removal action planning and/or removal actions.
Is LA detected above the action level in any surface soil samples collected from individual properties?	Surface soils are contaminated with LA	Determining that surface soils are not contaminated with LA when they actually are. This may result in no subsequent removal action, and in turn, resulting in increased human health risk.	Determining that soils contain unacceptable levels of LA when they actually do not. This would result in unnecessarily performing removal action planning and/or removal actions.
Is vermiculite insulation present in property 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, resulting in increased human health risk.	Determining that property buildings contain vermiculite insulation when they actually do not. This would result in unnecessarily performing removal action planning and/or removal actions.

Confirmation Sampling Data Quality Objectives

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.

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 Boundaries of the Study
5. Develop Decision Rules
6. Specify Tolerable Limits on Decision Errors
7. Optimize the Design for Obtaining Data

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.

After removal activities (e.g., excavation of contaminated soil, removal of vermiculite insulation, etc.), confirmation and/or clearance samples will be collected to determine if the response actions meet project-specific goals. Therefore, the overall response action sampling program must address whether abatement goals following removal activities were achieved.

This O&M sampling guidance was developed to reflect current abatement sampling methods used to collect data of sufficient quality and representativeness to evaluate each of these items.

Step 2 - Identify the Decision

The principal study questions related to the achievement of the abatement goals following removal activities are as follows:

1. Are LA structures detected in the air within an NPE where vermiculite insulation was removed?
2. Is LA detected in the soil surface or sidewalls of the excavated area?

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 1.

Table 1 –Investigation Inputs to Resolve Study Questions and Use of Input Information

Principal Study Question	Input to Resolve Question	Use of Input to Resolve Questions
Are LA structures detected in the air within the abatement area where vermiculite insulation or building materials were removed?	Air Clearance Samples	For each property undergoing vermiculite insulation removal, clearance air samples will be collected from within the abatement area where the vermiculite insulation or building materials were removed. The results of the clearance air samples will be used to determine if LA contamination was removed to applicable clearance criteria.
Is LA detected in the soil surface or sidewalls of the excavated area?	Soil Confirmation Samples	For each property undergoing contaminated soil removal, confirmation soil samples will be collected from the surface of the excavated area. The results of the confirmation soil samples will be used to determine if LA contamination was removed to applicable clearance criteria.

Step 4 – Define the Boundaries of the Study

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

Spatial Bounds

The information gathered to answer the abatement objectives will be collected from properties within the Superfund site boundaries of OUs 1, 2, 4, 5, 7, and 8 during O&M as directed by DEQ. The boundaries are the property boundaries that the abatement action is occurring (the entire building or structure) and to the deepest excavation depth, typically 12-18 inches below ground surface.

Temporal Bounds

For each property, the temporal boundaries of this investigation include the time from when response actions begin at each property to the time clearance or confirmation samples are collected and meet applicable clearance criteria.

Step 5 – Develop Decision Rules

The purpose of this step is to describe the method that DEQ will use to determine if 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 2.

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 investigation sampling, two types of decision errors are possible:

- A Type I (false negative) decision error would occur if a risk manager decides that an sample does not contain 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 levels of LA above a level of concern, when in fact they are not.

Type I errors may leave humans exposed to unacceptable levels of LA. Type II errors may result in unnecessary expenditures for abatement during O&M. Table 3 summarizes the consequences of making a decision error.

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 this O&M Sampling Guidance.

Table 2 – Decision Rules

Principal Study Question	Input to Resolve Question	Input Requirements	Action Level (Clearance Criteria)	Decision Rule
Are LA structures detected in the air within the abatement area where vermiculite insulation or building materials were removed above the action level?	Air Clearance Samples	Analysis: TEM AHERA with Libby-specific modifications AS: 0.005 s/cc Minimum Volume:1200 L/sample Collect: 5 samples of disturbed air within abatement area	Indoor Living Space: All 5 clearance air samples have total LA conc. that are non-detect Indoor Non-Living Space: All 5 clearance air samples have an average total LA air conc. less than 0.005 s/cc	Following vermiculite insulation removal activities, if sample results exceed the action level or samples are overloaded, then the area will be re-cleaned by the removal contractor. Collect additional air samples. If sample results are below the action level, then the area is acceptably cleaned.
Is LA detected in the soil surface or sidewalls of the excavated area above the action level?	Soil Confirmation Samples	Analysis: PLM-VE and PLM-Grav (w/ Libby-specific modifications) Reported Result: % LA Analytical Sensitivity: 0.2%	For frequently used areas: <0.2% LA *and* No more than 25% of the total soil exposure area can be <0.2% (trace) For infrequently used areas: < 0.2% LA	If levels of LA detected in soils at less than the excavation depth defined in the SOW are above the action level, then excavation will advance to the SOW-defined depth to the extent possible, unless limited by tree roots, building foundations, etc. Those areas will be noted on property documentation. If levels of LA detected in surface soil samples at depths greater than or equal to the excavation depth defined in the SOW, DEQ will determine next steps. If levels of LA are below action levels, take no action and the area is deemed acceptably cleaned.

Table 3 – Limits on Decision Errors

Principal Study Question	Null Hypothesis	Type I Error Will Result in:	Type II Error Will Result In:
Are LA structures detected in the air within the abatement area where vermiculite insulation or building materials were removed above the action level?	The building that was previously contaminated with LA, may still be contaminated with LA after removal.	Determining that the building that previously contained LA is not contaminated with LA after removal when it potentially is. May result in an increased risk to human health.	Determining that the building that previously contained LA is contaminated with LA after removal when it is not. May result in unnecessary re-cleaning of the building, adding unnecessary abatement costs.
Is LA detected in the soil surface or sidewalls of the excavated area above the actin level?	The soils below an excavation are still contaminated with LA	Determining that surface soils below an excavation are not contaminated with LA when they actually are. May result in increased human health risk.	Determining that surface soils below an excavation contain unacceptable levels of LA when they actually do not. May result in excavation of additional soils when it is unnecessary, adding unnecessary abatement costs.

APPENDIX H – REIMBURSEMENT ELIGIBILITY INFORMATION

ELEMENT SPECIFIC ELIGIBILITY GUIDANCE

1.0 Investigation Sampling

During a property evaluation, ARP will review previous investigation data and property information (including that of neighboring properties) and make a recommendation to DEQ whether additional investigation sampling is needed in order to evaluate the current or proposed use against the RALs. If additional characterization is recommended, ARP will develop an investigation sampling SOW and provide it to DEQ for review and approval.

For properties being developed by a commercial developer, investigation sampling is not eligible for reimbursement. This practice is consistent with EPA's '*Enforcement Discretion Guidance Regarding Statutory Criteria for Those Who May Qualify as CERCLA Bona Fide Prospective Purchasers, Contiguous Property Owners, or Innocent Landowners ("Common Elements")*' dated July 29, 2019. While the investigation sampling is not eligible for reimbursement, a SOW should still be developed and approved by DEQ to ensure data can be used for subsequent abatement actions, if determined necessary.

When writing the investigation sampling SOW, ARP should consider the following:

- For changes in use, additional sampling will only occur in the areas that use has or is anticipated to change (i.e., the entire property will not be re-investigated).
- Previously collected 5-point composite samples do not need to be re-sampled using 30-point criteria.
- Un-disturbed land may not require characterization if it is reasonable to believe there is no potential for encountering mine-related LA contamination (i.e. only naturally occurring LA may be present.)

If a third-party contractor collects samples, labor time should be recorded. Sample collection costs (i.e. reasonable labor hours, materials, etc.) will be reimbursed by DEQ in accordance with the approved SOW. Laboratory analytical costs are not eligible for reimbursement as they will be paid directly by DEQ to the specified laboratory.

At private residential properties, where previous sampling results identify the presence of LA, but not at levels high enough to require cleanup, and a change in use area (i.e. home addition, adding a building, etc.) is proposed, EPA recommends that federal O&M funds be available for sampling only after the changes are complete. State funds, however, can be utilized to conduct investigation sampling prior to the proposed changes occurring.

2.0 Self-Perform vs. Contractor Perform

If work is self-performed, material costs may be eligible for reimbursement, subject to the limitations described. Labor costs are only eligible if performed by a third-party contractor. Documentation of labor costs will need to be submitted.

3.0 Site Preparation

Site preparation work is ineligible for O&M reimbursement funds. The property owner will be responsible for removing all items within the proposed work area not in direct contact with LA and or LA source materials prior to work commencement, including, but not limited to furniture, appliances,

fixtures, trim, cabinetry, etc. In addition, the owners and/or tenants will be responsible for the security of personal belongings.

4.0 Temporary Relocations and Per Diem

EPA does not recommend O&M funding be used for temporary relocation or per diem for properties undergoing remodeling, damage, repair due to fire, etc. However, if a "miss" is identified due to error or oversight during the original inspections and at an EPA-led cleanup, these costs are eligible for federal reimbursement provided EPA concurs with the "miss" determination.

5.0 Equipment

Depending on the magnitude of proposed remedial activities, rental of equipment may be necessary. Rental costs are eligible only in circumstances where the equipment is required solely for the purpose of LA abatement activities. In those instances, information on equipment type(s), hours of use, rental cost, etc. must be provided.

6.0 Backfill Material

EPA-provided backfill materials should be utilized and provided at no cost to the property owner. However, if the property owner chooses to import material from another approved source, they may be eligible for state-only reimbursement at a reasonable cost based on evaluation of identified unit costs and quantities.

7.0 ARP Supplied Materials

ARP-supplied materials, such as poly, asbestos disposal bags, HEPA vacuums, etc. are not eligible for property reimbursement.

8.0 Disposal of Contaminated Materials

Disposal of eligible LA-contaminated materials at the Lincoln County landfill will be paid directly by DEQ. Non-LA containing materials should be segregated whenever possible. If materials must be disposed together, an estimate of the approximate % of LA materials must be identified prior to transport to the landfill. Additionally, trace soils must be segregated when transferring to the landfill so that they can be utilized as cover for the asbestos cell. Payment to the landfill will be made based on the landfill's bill of lading and the County's current tipping fee costs. Because disposal of LA materials will be paid directly by DEQ, reimbursement for contaminated material disposal is ineligible for reimbursement. Disposal of other materials is also ineligible for reimbursement.

9.0 Non-Maintained Controls

Homeowners have a responsibility to maintain previously installed engineered controls and/or boundary conditions. If ARP determines, through a site visit and discussion with the property owner, that maintenance activities have been grossly ignored, federal funds may not be used for reimbursement. State-funds may be utilized on a case-by-case basis, only if approved by DEQ.

Considerations for lack of maintenance includes knowledge by property owner of engineered controls and maintenance requirements, time lack of maintenance has occurred, and circumstances surrounding damage to existing controls.

10.0 Restoration Activities

Restoration activities for planned disturbances and/or changes in use are not eligible for reimbursement. For unplanned disturbances such as accidents, floods, fire, vandalism, etc., restoration costs may be eligible if necessary to restore previous function and use.

11.0 Investment Properties

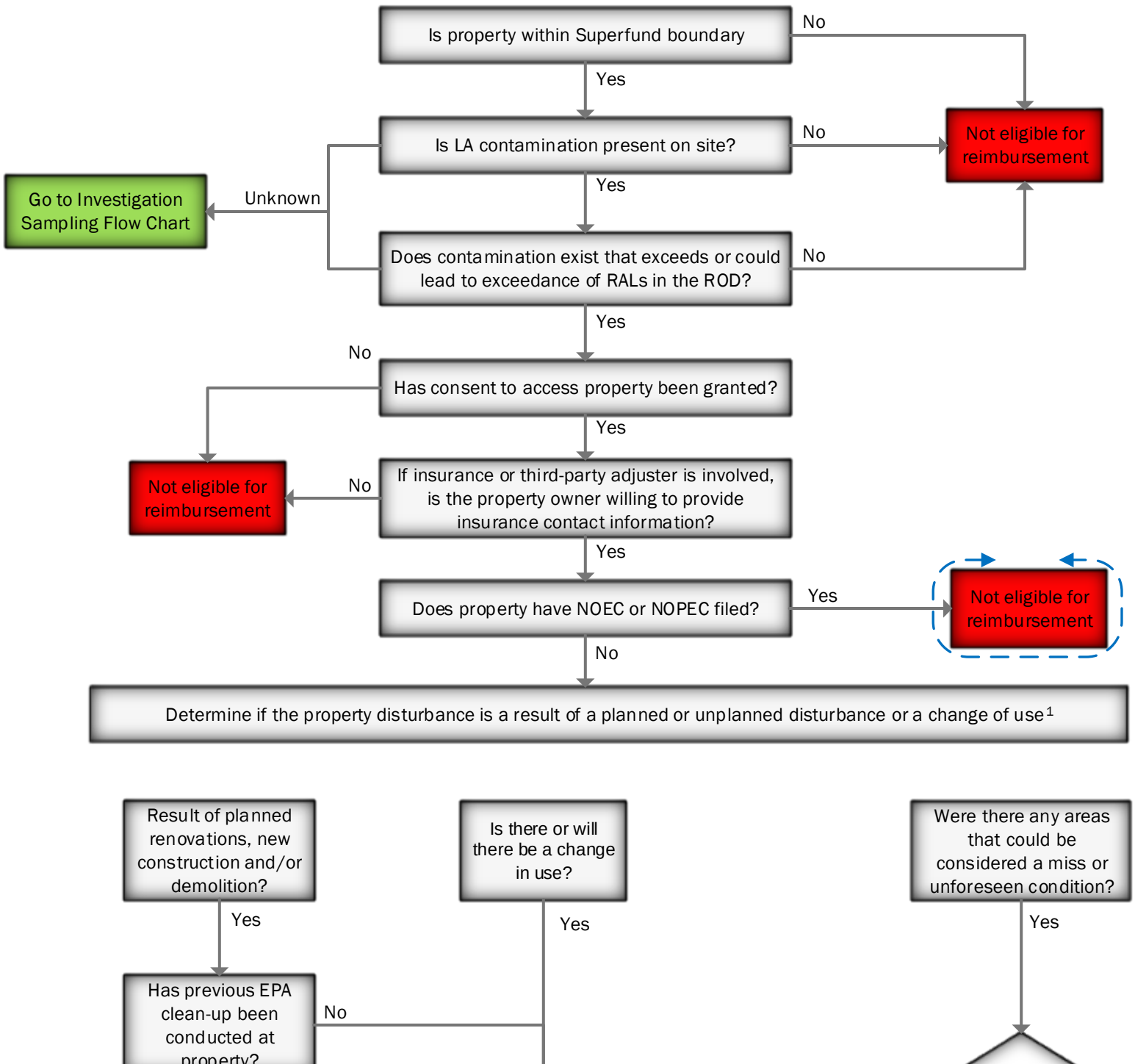
Since NOEC and NOPECs are intended to alert the buyer that there may be certain environmental conditions that could adversely affect the property, the price of the property is likely depressed. As a result, DEQ may not reimburse remediation costs for a commercial developer that may benefit by obtaining a property at a reduced price (i.e., windfall situation).

12.0 Materials Recommended to be Left in Place

Contaminated materials may be left in place if they are located in an inaccessible area or in areas not likely to be disturbed. If the property owner elects to remove contaminated materials from these areas, costs associated with this work will not be eligible for reimbursement.

Property Reimbursement Eligibility Flow Chart for Non-Federal Properties

03/20/20



[1]If scenario is catastrophic or unusual, case-by case review may be required.

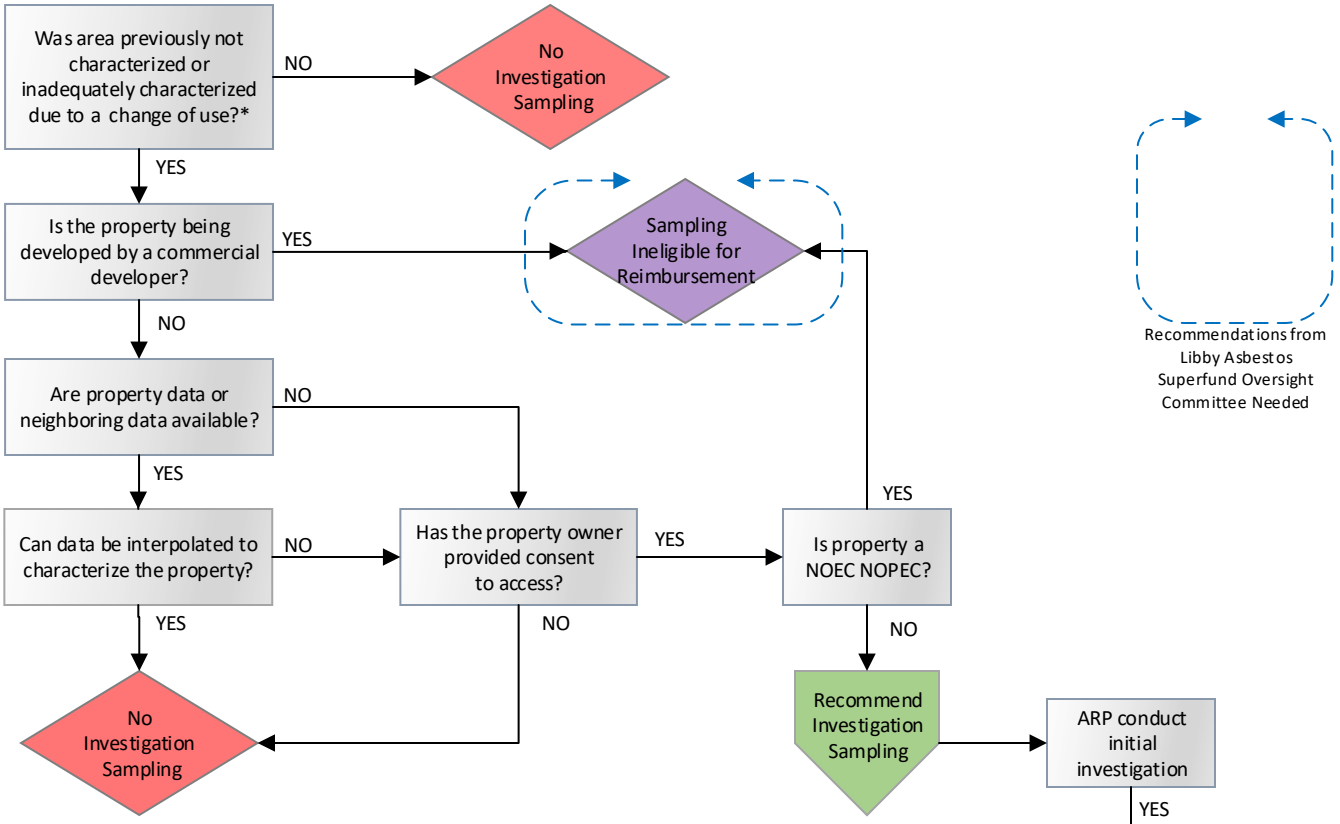
[2]Defined per ASTM E1527-13 § 3.2.12

[3]Miss does not include limits of inspection

Recommendations from Libby Asbestos Superfund Oversight Committee Needed

03/20/2020

Investigation Sampling Flow Chart
For Non-Federal Properties



Appropriate Sample Collection Costs Eligible for Reimbursement:

- Third-party purchased sampling materials
- Shipping costs
- Reasonable sample collection labor time

Sample Collection Costs Ineligible for Reimbursement:

- Laboratory costs (to be paid directly by MDEQ)
- ARP-supplied materials
- Per diem

*Inadequate characterization does not include previous 5-pt composite samples.
**Third-party can be homeowner, contractor, or designee.

APPENDIX I – “NO CALL” LIST

Below is the list of AD#'s from the Do Not Call list:

DO NOT CALL - LIBBY
AD-000080
AD-000136
AD-001003
AD-001728
AD-001897
AD-001914
AD-002516
AD-002538
AD-002541
AD-002589
AD-002604
AD-002889
AD-003051
AD-003085
AD-003086
AD-003116
AD-005248
AD-005295
AD-005309
AD-005669
AD-006345
AD-006348
AD-006553
AD-006556
AD-006657
AD-006684
AD-006704
AD-007906
AD-008234
AD-008457
AD-008458
AD-008483

DO NOT CALL - TROY
AD-200106
AD-200112
AD-200117
AD-200118
AD-200119
AD-200120
AD-200122
AD-200182
AD-200342
AD-200343
AD-200344
AD-200347
AD-200349
AD-200354
AD-200363

APPENDIX J – ANNUAL INSPECTION INFORMATION

OU 4/7 Property File Review Checklist

Property Address: **Geounit:**
AD Number: **BD Number(s):**
Land Use Category: ☐ Residential ☐ Commercial
☐ Park/School ☐ Transportation Corridor
Reviewer: **Date of Review:**

1. Compare Response Manager and Libby Asbestos Site Viewer data.
    a. Do the address and geounit match? ☐ Yes ☐ No
    b. If no, has database been updated? ☐ Yes ☐ No
2. Is there a status assigned to the property? ☐ Yes ☐ No
    a. If yes, what is the status?
        ☐ Inspection Complete
        ☐ Inspection Required
        ☐ Removal Complete
        ☐ Removal Required
        ☐ Refusal
    b. If no, has database been updated? ☐ Yes ☐ No
3. What year(s) were activities conducted at the property?
4. What is most recent type of inspection?
    ☐ PDI/EIC ☐ SI ☐ DI ☐ CSS ☐ TAPE ☐ Phase I
5. Review documents/reports in Response Manager.
    a. Has entire exterior of property been sampled? ☐ Yes ☐ No
        i. If applicable, identify areas that were not sampled:
    b. Are sample results available? ☐ Yes ☐ No
        i. If yes, where are sample results stored?
        ☐ Response Manager ☐ Scribe
    c. Is there an interior report? ☐ Yes ☐ No
    d. Is removal well defined? ☐ Yes ☐ No
    e. Are there areas sealed in place? ☐ Yes ☐ No
    f. Are there inaccessible areas? ☐ Yes ☐ No
        i. If applicable, identify areas that are inaccessible:
        ☐ Attic ☐ Crawl Space ☐ Other:
    g. Does work meet remedy requirements in Record of Decision? ☐ Yes ☐ No
6. Summary of Findings:
7. Deficiencies Noted:
8. Recommendations (if applicable):

Acronym Key

ABS= Activity Based Sampling
Aliq= aliquot
Conc= concentration
COMM-REC= Record of Communication
Comfort Ltr= Comfort Letter
EIC=Exterior Inspection Checklist
ERS-IAC= Emergency Response Specialist Initial Assessment Checklist
GPI= General Property Investigation
LA= Libby Amphibole Asbestos
LA BIN= bin assigned by lab that corresponds to LA result
Lab QC Type= indicates type of sample analyzed (e.g. field sample, field quality control sample, lab quality control sample, etc.)
Location Area= area inspected and/or sampled (in SQ FT)
ND: Non- detect for LA
PCC= Property Closeout Checklist
PLM= Polarized Light Microscopy
PLM Grav= (Gravimetric Method)
PLM-VE= (Visual Estimation Method) Sample Analysis
PLM-PC400= approach previously used to perform point counting using PLM; not an EPA standard method.
PLM 9002= (NOSHA method) Clearance Sample at depth
QRSW= Quick Response Statement of Work
RR-Agreement= Removal and Restoration Agreement
RR-COMPLETE=Removal and Restoration Completion form and EPA Letter
RWAP= Response Action Work Plan
SIIC= Supplemental Interior Inspection Checklist
TR: Trace (<0.2%) LA Detected
VV= visible vermiculite
<1%: Greater than or equal to 0.2% LA and less than 1% LA detected
>1%: Greater than or equal to 1% LA detected

Report Naming Conventions

1-XXXXX: Phase 1
CS-XXXXX: CSS samples
TT-XXXXX: TAPE
XD-XXXXX: PDI
XG-XXXXX: GPI
XR-XXXXX: Removal Clearance
EX-XXXXX: Activity-base sampling
C-XXXXX:
HW-XXXXX:

Remedial Action Levels from ROD

Contaminated Soil

Residential/Commercial

Frequently Used Areas

The RALs for addressing LA contamination in surface soil for frequently used areas at residential/commercial properties are as follows:

- LA soil concentrations of Bin B2 or Bin C by PLM-VE (i.e., LA present at levels greater than or equal to 0.2 percent) (regardless of spatial extent) or
- LA soil concentrations of Bin B1 by PLM-VE (i.e., LA is present at levels less than 0.2 percent), if the spatial extent of the Bin B1 area is more than 25 percent of the total soil exposure area at a property.

Infrequently Used Areas

The RAL for addressing LA contamination in surface soils for infrequently used areas at residential/commercial properties is as follows:

- LA soil concentrations of Bin B2 or Bin C by PLM-VE (i.e., LA present at levels greater than or equal to 0.2 percent).

Industrial

The RAL for addressing LA contamination in surface soil for industrial properties is as follows:

- LA soil concentrations of Bin C by PLM-VE (i.e., LA is present at levels greater than or equal to 1 percent).

Transportation Corridors

The RAL for addressing LA contamination in surface soil for transportation corridors is as follows:

- LA soil concentrations of Bin C by PLM-VE (i.e., LA is present at levels greater than or equal to 1 percent).

Parks/Schools

The RAL for addressing LA contamination in surface soil for park/school properties is as follows:

- LA soil concentrations of Bin B2 or Bin C by PLM-VE (i.e., LA present at levels greater than or equal to 0.2 percent).

Contaminated Building Materials

The RALs for addressing contaminated building materials regardless of land use category is as follows:

- Presence of accessible LA-containing vermiculite insulation in any quantity in living spaces, non-living spaces, and/or secondary structures or
- Presence of accessible friable and/or deteriorated building materials containing greater than or equal to 0.25 percent LA by PLM-PC400 (e.g., chinking, plaster, mortar, and other materials on boilers, pipes, or other appurtenances).

OU4/OU7 Phone Interview Questions

Property Address: **Geounit:**
AD Number: **BD Number(s):**
Person Contacted: **Phone Number:**
Person Conducting Interview: **Date of Interview:**

Goals and objectives of the call: To verify that the property owner is familiar with the Libby Superfund Site, to ensure the remedy remains protective (e.g. property owner responsibilities), to inform the owner of potential ICs that are put in place, and to ensure the owner is familiar with education and outreach opportunities. Prior to the call, staff will review the property file and Response Manager.

Interview Questions

1) What remediation work was completed at your property?
2) Do you have documents from EPA showing the work that was completed?
   ☐ Yes ☐ No
3) Has there been any breaches (i.e. flower beds, gardens, trees, crawl space, attics) and/or home improvements completed since the remedial action? ☐ Yes ☐ No
4) Has there been any changes to the use of your property? ☐ Yes ☐ No
   a. If yes, please describe those changes:
5) Have you encountered any suspicious material that may not have been evaluated?
   ☐ Yes ☐ No
   a. If so, what steps did you take?
6) Do you have any plans to change your property? ☐ Yes ☐ No
7) Are you aware of the resources available to you? ☐ Yes ☐ No Do you have any ongoing concerns or questions? ☐ Yes ☐ No
   a. If so, please describe:

RECOMMENDED ANNUAL O&M /REMEDY EVALUATION CHECKLIST

Introduction and Purpose

Effective operation and maintenance (O&M) at Superfund sites generally is critical to ensure that remedies remain protective of human health and the environment.

The recommended Annual O&M Remedy Evaluation Checklist has been designed to help the Remedial Project Manager (RPM) capture data routinely collected during O&M in a way that can better evaluate the efficiency and effectiveness of the remedial action. This recommended checklist may also be used to evaluate an operating remedy prior to transferring the site to the State for O&M. In addition, remedy performance summarized using this recommended checklist can be used to communicate remedy progress to the local community, highlight potential issues before they become problems and help the RPM complete five-year reviews more efficiently.

The information that you collect using this recommended form should help you answer the following questions:

- Is the remedy achieving the remedial action objectives (RAOs), maintaining cleanup goals and/or achieving technology-specific performance goals?
- If the remedy is not achieving the established objectives and goals, what must I do to correct this and how can I document this?
- If the remedy is achieving the performance goals, objectives and performance standards, are there any opportunities to optimize the remedy to make it work more efficiently?

This recommended checklist is intended to be completed annually. It is recommended that any data that you use to complete this evaluation be attached to the checklist, as this will make completing the next year's evaluation easier.

This recommended checklist does not recommend the level of review carried out in the U.S. Environmental Protection Agency (EPA) five-year review process. However the recommended checklist contains review elements that are consistent with a five-year review process.

Instructions:

The recommended checklist is in Microsoft Word and was designed to be completed electronically. Most questions involve a short answer, yes/no response or simply checking the box. Questions that involve a short answer will have an expandable text box. For responses that ask to you to "select one," please double click on "select one" and choose the correct answer. If the information is not available for a particular question, please indicate this with a N/A. A site visit is strongly encouraged, but not required prior to completing the recommended checklist.

1. This evaluation is intended to be completed yearly once O&M activities have begun at a site and can be stored and maintained in an electronic format.
2. For large complex sites, consider completing a separate checklist for each Operable Unit (OU).
3. This evaluation should be based on information and documentation (e.g., O&M reports and monitoring data) that is readily available to the RPM.
4. Section VIII, "Technical Data and Remedy Performance," provides specific instructions regarding what data and information are important for this section. Data entered in Section VIII are used to evaluate the specific technology used in that remedial action (RA). Please note: *Section VIII, Appendix E, Other Remedy Types/Components* was designed to be used by the RPM for the annual review of O&M remedies and remedy components that are not addressed in Appendices A through D or by the separate *Recommended Annual O&M Remedy Evaluation Checklist for Contaminated Sediment Remedies*, OSWER #9355.0-118.
5. When you have completed the recommended checklist, please sign and date page 1 and place the completed document in the site file. Additionally, we recommend that you save the completed checklist electronically for use in completing the next year's evaluation.

Generally, including the Recommended Annual O&M/Remedy Evaluation Checklist in the site repository can provide the community with information about O&M status and remedy performance and can demonstrate that the Region is tracking performance to ensure that the remedy remains protective.

Acronym List

AS	Air Sparging	PCOR	Preliminary Close Out Report
CSM	Conceptual Site Model	PRGs	Preliminary Remediation Goals
GAC	Granular Activated Carbon	PRP	Potentially Responsible Party
ICs	Institutional Controls	RAO	Remedial Action Objective
LEL	Lower Explosive Limit	ROD	Record of Decision
LTRA	Long-Term Response Action	RPM	Remedial Project Manager
MNA	Monitored Natural Attenuation	RSE	Remediation System Evaluation
NPL	National Priorities List	SVE	Soil Vapor Extraction
O&F	Operational and Functional	TI Waivers	Technical Impracticability Waivers
O&M	Operation and Maintenance	USACE	U.S. Army Corps of Engineers
OSHA	Occupational Safety and Health Administration	VEB	Vertical Engineered Barrier
OU	Operable Unit	VOCs	Volatile Organic Compounds

RECOMMENDED ANNUAL O&M /REMEDY EVALUATION CHECKLIST

Please save electronically and send this completed checklist and any attachments to the site file and site repository.

I. SIGNATURES AND APPROVALS					
RPM			**RPM (If appropriate)**		
Name:			Name:		
Telephone:			Telephone:		
Signature:		Date:	Signature:		Date:
State Contact (if appropriate)					
Name:					
Telephone:					
Signature:					Date:

II. GENERAL SITE INFORMATION			
Site Name:			
State:			
Period Covered:	to		EPA Site ID:
Site Lead:	(Select one)	Other, specify:	
Organization responsible for O&M operations:		(Select one)	
Other, specify:			
Site Remedy Components (ref. Section VIII):			
Preliminary Close Out Report (PCOR) date:			
Operational & Functional (O&F) date:			
Last five-year review date:			
NPL deletion date:			
Did you make a site visit during this review?	☐ Yes ☐ No		Date:
If no, why:			
Date of next planned checklist evaluation:			
Location of Administrative Record/Site Files:			
During the site visit, was monitoring equipment operational?			☐ Yes ☐ No ☐ N/A
Please elaborate:			
Has an Optimization Study been conducted at the site?	☐ N/A ☐ Yes ☐ No		Date:
If not, is one planned?			

List all site events since the last evaluation that impact or may impact remedy performance.
Chronology of events since last report (e.g., site visits, receipt of reports, equipment failures, shutdowns, vandalism, storm events):
Elaborate on significant site events or visits to site:

III. DOCUMENTS AND RECORDS

Because these documents may be required for the five-year review, verify what documents are currently available on-site, or note off-site location:

Document	Required	Not required	On-site	Off-site (indicate where)
O&M Manual	☐	☐	☐	☐
O&M Maintenance Logs	☐	☐	☐	☐
O&M Annual Reports	☐	☐	☐	☐
RA as-built drawings modified during O&M	☐	☐	☐	☐
Site-Specific Health and Safety Plan	☐	☐	☐	☐
Contingency/Emergency Response Plan	☐	☐	☐	☐
O&M/Occupational Safety and Health Administration (OSHA) Training Records	☐	☐	☐	☐
Settlement Monument Records	☐	☐	☐	☐
Gas Generation Records	☐	☐	☐	☐
Ground Water Monitoring Records	☐	☐	☐	☐
Surface Water/Sediment/Fish Monitoring Records**	☐	☐	☐	☐
Cap/Cover System Inspection Records	☐	☐	☐	☐
Leachate Extraction Records	☐	☐	☐	☐
Discharge Compliance Records	☐	☐	☐	☐
Institutional Controls (ICs) Review	☐	☐	☐	☐
Other(s) (Please name each)	☐	☐	☐	☐
	☐	☐	☐	☐
	☐	☐	☐	☐
	☐	☐	☐	☐
	☐	☐	☐	☐

** Note: A separate O&M checklist has been developed for surface water/sediment remedies. For completeness, answer this question regarding documentation requirements and availability, and enter more detailed information in the surface water/sediment checklist.

IV. ADMINISTRATIVE ISSUES

Check all that apply:	Date Initiated:
☐ Explanation of Significant Differences in progress	
☐ Record of Decision (ROD) Amendment in progress	
☐ Site in O&F period	
☐ Long-Term Response Action (LTRA) in progress	
☐ LTRA Transition to O&M in progress	
☐ Notice of Intent to Delete site in progress	
☐ Partial Site Deletion in progress	
☐ Technical Impracticability (TI) Waivers in progress	
☐ Reuse Assessment or Reuse Plan in progress	
☐ Revised Risk Assessment in progress ☐ Ecological OR ☐ Human Health	
☐ Other administrative issues:	

VI. O&M COSTS

The purpose of this section is to document what is known about O&M costs for this site. It is realized that not all cost information will be readily available, but to the extent possible, please provide the following information, as this will help identify cost increases and flag potential budget issues before they arise.

What was the total annual O&M cost for the previous year?	
What is the expected total annual O&M cost for the upcoming year?	
Please provide an approximate breakout of the previous year's O&M costs below.	**Use either $ or %**
Analytical (e.g., lab costs):	
Materials (e.g., treatment chemicals, cap materials):	
Oversight (e.g., project management):	
Monitoring (e.g., ground water sampling):	
Utilities (e.g., electric, gas, phone, water):	
ICs (implementation and enforcement):	
Other (e.g., capital improvements, equipment repairs):	

Describe any unanticipated/unusually high or low O&M costs and potential future O&M funding issues.

VII. INSTITUTIONAL CONTROLS (ICs)**

The purpose of the IC evaluation at the O&M phase is to determine if the ICs are implemented, effective and durable. The following references may be useful for completing this evaluation:

- *Institutional Controls Bibliography: Institutional Control, Remedy Selection, and Post Construction Completion Guidance and Policy* (OSWER 9355.0110, December 2005);
- *Supplement to the Comprehensive Five-Year Review Guidance; Evaluation of Institutional Controls* (OSWER 9355.7-12, working draft 3/17/05);
- *National IC Strategy to Ensure Institutional Controls Implementation at Superfund Sites* (OSWER 9355.0-106, September 2004); and
- *Institutional Controls: A Site Manager's Guide to Identifying, Evaluating and Selecting Institutional Controls at Superfund and RCRA Corrective Action Cleanup* (OSWER 9355.0-7-4FS-P, September 2000).

** Note: A separate O&M checklist has been developed for surface water/sediment remedies. For completeness, answer this question regarding ICs, and enter more detailed information in the surface water/sediment checklist.

Identify each IC (media, objective, and instrument) implemented/to be implemented at the site. Attach an extra sheet if necessary.	
Are the ICs adequate to minimize the potential for human exposure and protect the integrity of the remedy? If no, please explain.	☐ Yes ☐ No
Please identify the party responsible for compliance and enforcement of the IC.	
Please describe what the ICs are intended to accomplish, who they are designed to inform, the source document for the IC, and where the IC information is located.	
Please identify the date when the ICs were implemented. If the ICs have yet to be implemented, please identify the party responsible for implementing the ICs and the scheduled implementation date.	
If the ICs have been implemented, are they still in place? If the ICs remain in place, please identify whether there is a planned termination date and, if so, what it is.	
Are there reasons to clarify or modify the appropriate decision document(s) to improve the effectiveness and/or durability of the ICs? If yes, please explain and describe any plans to clarify/modify the document(s).	☐ Yes ☐ No

VIII. TECHNICAL DATA AND REMEDY PERFORMANCE

The purpose of this section is to help prompt questions about remedy performance over the past year, the adequacy of monitoring activities to assess remedy performance, and changes in field conditions or understanding that could affect the remedy. Specific sections also prompt questions about remedy optimization. Addressing these questions on an annual basis can help to flag opportunities and potential issues to watch in the coming year and help inform future improvements in remedy O&M. The collection of annual checklists can also serve as documentation of when a potential issue was first identified, what was done to address it, and when it was addressed. Thus, an annual checklist can be a useful, succinct source of information to help RPMs recount O&M history.

Questions for specific remedy types (e.g., ground water pump-and-treat) are contained in Appendices A through D at the end of the form. Appendix E contains general questions that can be used to document technical data and remedy performance for remedies and remedy components that do not fit within the specific categories identified in the remainder of this checklist. Identify the remedy types in Section VIII.A, below, and complete a copy of each appendix that is applicable to the site. If the site includes multiple remedies or remedy components of the same type, please complete a copy of the applicable appendix for each remedy/component (e.g., if the remedy includes two separately managed containment areas, complete two copies of Appendix C, one for each area). A separate O&M checklist has been developed for surface water/sediment remedies and remedy components. If the site includes a surface water/sediment remedy, note this below and complete the surface water/sediment checklist.

A. Please identify the type(s) of remedy(ies) this Annual O&M Remedy Evaluation Checklist addresses:

- ☐ Ground Water Pump-and-Treat (please complete Appendix A)
- ☐ Ground Water Monitored Natural Attenuation (MNA) (please complete Appendix B)
- ☐ Ground Water or Soil Containment (please complete Appendix C)
- ☐ Soil Vapor Extraction/Air Sparging (please complete Appendix D)
- ☐ Other Remedy Types (please complete Appendix E)

IX. RECOMMENDATIONS

New Recommendations, from this annual review:

Recommendation	Party Responsible	Milestone Date

APPENDICES

TECHNICAL DATA AND REMEDY PERFORMANCE ANNUAL O&M /REMEDY EVALUATION CHECKLIST

RECOMMENDED APPENDIX A. GROUND WATER PUMP-AND-TREAT REMEDIES

The following checklist is an abbreviated set of questions that could be used by an EPA RPM for annually reviewing the O&M of a ground water pump-and-treat remedy, including pump-and-treat remedies designed for hydraulic containment. This checklist was developed using concepts presented in EPA guidance, *Elements for Effective Management of Operating Pump and Treat Systems* (EPA 542-R-02-009, December 2002). This guidance is part of a series of fact sheets that EPA OSRTI has prepared as guidance to the ground water remediation community on effectively and efficiently designing and operating long-term ground water remedies. For more information, including the guidance *O&M Report Template for Ground Water Remedies (with Emphasis on Pump and Treat Systems)* (EPA 542-R-05-010, April 2005) and report *Pilot Project to Optimize Superfund-Financed Pump and Treat Systems: Summary Report and Lessons Learned* (EPA 542-R-02-008a), visit EPA's CLU-IN Website (www.cluin.org/).

A. Remedy Goals and Conceptual Site Model (CSM)

1. Review of the current remedy goals and measurements: Remedy goals may be expressed in terms of a broad, long-term purpose or intent specified in a decision document (e.g., cleanup to a specified concentration), a performance-based metric or milestone intermediate in duration (e.g., a 20% decrease in monthly influent concentrations within 24 months of operation); or a specific and short-term objective (e.g., demonstration of plume containment).

List the short-term objectives and intermediate system goals:	
List the final system goals:	
What metrics (performance criteria) are being implemented to measure project progress towards meeting each goal?	
What schedule has been established for measuring and reporting each metric?	
Based on new information or events since the last O&M review, is there a reason to re-evaluate the system goals? Note: this might be due to factors such as regulatory framework has been revised; better technology/strategy alternatives available; existing goals appear unrealistic; costs greater than originally anticipated; extent of plume has changed; new sources of contamination removed and/or discovered; or land use or ground water production near site has changed. If yes, identify the remedy goals that should be re-evaluated, the rationale, and any plans for re-evaluating the goals.	☐ Yes ☐ No

2. Review of changes to the CSM: The CSM is a combination of text and figures that describe the hydrogeologic system, the cause of the ground water impacts, and the fate and transport of the ground water contaminants. If monitoring data during active remediation do not agree with expectations, this could point to a gap in the conceptual model that should be addressed with a focused investigation. This does not imply a return to the "remedial investigation" phase. The CSM should evolve over time, including during active remediation, as more information about the site becomes available. The following questions may be used to evaluate the need for updating the CSM:

Since the last time you completed the O&M checklist for this system, have new contaminant sources been identified or have previously suspected contaminant sources been eliminated from further consideration? If yes, use this space to comment.	☐ Yes ☐ No
Since the last time you completed an O&M checklist for this system, have new contaminants been identified in the ground water that could affect remedy effectiveness? If yes, use this space to comment.	☐ Yes ☐ No
Based on your answers to the above questions, would it be useful to update the CSM at this time?	☐ Yes ☐ No

If yes, please describe any plans to update the CSM.	

B. Remedy Performance Assessment

1. Evaluate remedy effectiveness: The following questions are intended to review whether the ground water pump-and-treat remedy is performing as intended and whether there are opportunities for optimizing the remedy.

Plume Capture

When addressing these questions, it may be useful to refer to *A Systematic Approach for Evaluation of Capture Zones at Pump and Treat Systems* (EPA 600/R-08/003, January 2008).

Has a three-dimensional target capture zone been clearly defined? If no, use this space to explain why not.	☐ Yes ☐ No
If not clearly defined, describe plans to better define the target capture zone.	
What lines of evidence have been used to evaluate actual capture achieved (e.g., flow budget and/or capture zone width calculations, potentiometric surface maps, water elevation pairs, concentration trends at wells beyond the target capture zone, particle tracking in conjunction with ground water modeling, tracer tests)	

System Equipment/Structures (e.g., extraction wells, collection systems)

Since the last time you completed an O&M checklist for this system, has the downtime associated with non-routine operations and maintenance exceeded expectations? If yes, what systems have been responsible for unplanned downtime (e.g., extraction pumps, wastewater facilities)? If yes, what corrections have been or are being made to minimize downtime?	☐ Yes ☐ No
Since the last time you completed the O&M checklist for this remedy/remedy component, have any major repairs to the pump-and-treat system(s) been required? If yes, describe the repairs, their impact on progress toward remediation milestones, and actions taken to minimize similar repairs in the future.	☐ Yes ☐ No
Since the last time you completed an O&M checklist for this system, have the extraction/injection well rates changed significantly? If yes, describe the known/suspected source of the change, if identified. If yes, is the change reflective of a long-term condition and, if so, how will this be addressed in the O&M of the system?	☐ Yes ☐ No
Since the last time an O&M checklist was completed for this system, have air emissions from the system met permit requirements, if any? If not, what is being done to meet the permit requirements?	☐ Yes ☐ No ☐ N/A
Since the last time an O&M checklist was completed for this system, has effluent discharge met permit requirements? If not, what was (is) the problem and what was (or will be) done to correct it?	☐ Yes ☐ No

Optimization

Has an optimization study been conducted for this system?	☐ Yes ☐ No
If an optimization study has been conducted, have any of the optimization recommendations been implemented since the last time an O&M checklist was completed for this system?	☐ Yes ☐ No ☐ N/A
If optimization recommendations have been implemented (during this or prior review periods), describe any new results observed or conclusions drawn since the last time an O&M checklist was completed for this system.	
If optimization recommendations have not been implemented, why not?	

2. Evaluate collection and analysis of performance monitoring data	
Do the approaches used to interpret ground water monitoring data (e.g., concentration trend analyses, plume contour and/or bubble maps, plume cross-sections, potentiometric surface maps) provide adequate information to assess the performance of the pump-and-treat remedy? If no, describe plans, if any, to implement new approaches.	☐ Yes ☐ No
Based on information collected since the last O&M review, is there a need to re-evaluate the parameters, sampling methods, sampling frequency, and monitoring locations used to evaluate remedy performance?	☐ Yes ☐ No
Are ground water data managed electronically? If no, use this space to explain why not.	☐ Yes ☐ No
Are performance-monitoring reports of sufficient quality and frequency to evaluate the efficacy of the remedy and recognize protectiveness problems in time for effective action? If no, what actions, if any, have been taken or are planned to address this situation?	☐ Yes ☐ No
C. Cost Effectiveness	
Are actual parameters consistent with design parameters (based on process monitoring)? If not, how do they differ? (check all that apply) ☐ Influent rate to treatment plant ☐ Influent concentrations ☐ Mass loading to the system ☐ Removal efficiency for each treatment component ☐ Air to water ratio (air strippers) ☐ Materials usage (e.g., granular activated carbon (GAC), chemicals) ☐ Other (please explain)	☐ Yes ☐ No
Based on the above comparisons, have any above ground systems or process monitoring procedures been evaluated/implemented to reduce costs? If yes, please identify which of the following have been done to reduce costs. (check all that apply) ☐ Ensuring proper maintenance and efficiency of equipment ☐ Replacing treatment components with alternate technologies (e.g., replace UV/Oxidation with air stripping) or more appropriately sized components ☐ Eliminating unnecessary or redundant treatment components that are no longer needed (e.g., metals removal or GAC polishing system) ☐ Changing discharge ☐ Automating system to reduce labor ☐ Optimizing ground water extraction rates and/or locations ☐ Other (please explain)	☐ Yes ☐ No
D. Remedial Decisions: Indicate which of the following remedial decisions is appropriate at the present time and provide the basis for the decision.	
☐ No Change to the System ☐ Modify/Optimize System ☐ Modify/Optimize Monitoring Program ☐ IC Modifications ☐ Implementation of Contingency/Alternative Remedy	
Basis for decision:	

RECOMMENDED APPENDIX B. GROUND WATER MONITORED NATURAL ATTENUATION (MNA) REMEDIES

The following checklist is an abbreviated set of questions that could be used by an EPA RPM for annually reviewing the O&M of a MNA remedy for ground water. This MNA guidance checklist was developed using concepts presented in EPA guidance, *Performance Monitoring of MNA Remedies for* [volatile organic compounds] *(VOCs) in Ground Water* (EPA/600/R-04/027; April 2004). For some approaches, a more detailed remedy optimization study or remediation system evaluation (RSE) may be beneficial. For guidance on remedy optimization studies or RSEs, visit EPA's CLU-IN Website (www.cluin.org/) or the U.S. Army Corps of Engineers (USACE) Hazardous, Toxic and Radioactive Waste Center of Expertise RSE Website (www.environmental.usace.army.mil/)

A. Remedy Goals and Conceptual Site Model (CSM)	
1. Review of the current remedy goals and measurements: The remedy goals may be expressed in the ROD as remedial action objectives (RAOs) and preliminary remediation goals (PRGs). RAOs provide a general description of what the cleanup will accomplish (e.g., restoration of ground water). PRGs are the more specific statements of the desired endpoint concentrations or risk levels, for each exposure route, that are believed to provide adequate protection of human health and the environment.	
List the intermediate system goals (RAOs and PRGs).	
List the final system goals (RAOs and PRGs).	
What metrics (performance criteria) are being implemented to measure project progress towards meeting each goal?	
What schedule has been established for measuring and reporting each metric?	
Based on new information or events since the last review, is there a need to re-evaluate the remedy goals? Note: this might be due to factors such as whether the regulatory framework has been revised, whether existing goals appear realistic, and if there have been changes to land use or ground water production near the site. If yes, identify the remedy goals that should be re-evaluated, the rationale, and any plans for re-evaluating the goals.	☐ Yes ☐ No
2. Review of changes to the CSM: The CSM for natural attenuation is the site-specific qualitative and quantitative description of the migration and fate of contaminants with respect to possible receptors and the geologic, hydrologic, biologic, geochemical and anthropogenic factors that control contaminant distribution. Because the CSM provides the basis for the remedy and monitoring plan, it can be reevaluated as new data are developed throughout the lifetime of the remedy. The following questions may be used to evaluate the need for updating the CSM:	
Have new contaminant sources been identified or have previously suspected contaminant sources been eliminated from further consideration since the last time you completed the O&M checklist for this remedy? If yes, use this space to comment.	☐ Yes ☐ No
Has there been an increase or decrease in size of the plume since the last time you completed an O&M checklist for this remedy? Comments (e.g., what is the nature and magnitude of the change).	☐ Increase ☐ Decrease ☐ No change
Has there been an increase or decrease in vertical extents of the plume since the last time you completed an O&M checklist for this remedy? Comments (e.g., what is the nature and magnitude of the change).	☐ Increase ☐ Decrease ☐ No change
Has there been an increase or decrease in the maximum contaminant concentrations in the plume since the last time you completed an O&M checklist for this remedy? Comments (e.g., have maximum concentrations changed for all or a subset of contaminants, which ones, and by how much).	☐ Increase ☐ Decrease ☐ No change
What types of reaction zone(s) are present in the plume (aerobic, anaerobic, or both)?	

Based on information collected since the last O&M review, is there a need to re-evaluate the number and/or location of monitoring points in the reaction zone(s)? If yes, use this space to comment.	☐ Yes ☐ No
Based on information collected since the last O&M review, is there a need to re-evaluate the number and/or location of monitoring points in the target zones? If yes, use this space to comment.	☐ Yes ☐ No
Has there been a change in ground water flow rate or direction that may suggest monitoring frequency or locations may need to be reevaluated? If yes, use this space to comment.	☐ Yes ☐ No
Is there evidence of periodic pulses of residual contamination from the vadose zone that suggest new monitoring points should be added in the vadose zone? If yes, use this space to comment.	☐ Yes ☐ No
If there is reason to re-evaluate the number and location of monitoring points and/or monitoring frequency (as indicated in above responses), identify any plans for re-evaluating the monitoring program.	
Based on your responses to the above questions, would it be useful to update the CSM at this time? If yes, please describe any plans to update the CSM.	☐ Yes ☐ No

B. Remedy Performance Assessment

1. Review performance monitoring objectives. The OSWER Directive 9200.4-17P (U.S. EPA, 1999a) provides eight specific objectives for the performance-monitoring program of an MNA remedy.

For each of the following eight performance monitoring objectives, identify which are currently being met, which are currently being met but could benefit from further review, and which are currently not being met.

Objective	Status		
	Being met	*Benefit from review*	*Not being met*
1) Demonstrate that natural attenuation is occurring according to expectations	☐	☐	☐
2) Detect changes in environmental conditions that may reduce the efficacy of any of the natural attenuation processes	☐	☐	☐
3) Identify any potentially toxic and/or mobile transformation products	☐	☐	☐
4) Verify that the plume(s) is not expanding downgradient, laterally or vertically	☐	☐	☐
5) Verify no unacceptable impact to downgradient receptors	☐	☐	☐
6) Detect new releases of contaminants to the environment that could impact the effectiveness of the natural attenuation remedy	☐	☐	☐
7) Demonstrate the efficacy of ICs that were put in place to protect potential receptors	☐	☐	☐
8) Verify attainment of remediation objectives	☐	☐	☐

If any of these objectives are not being met or would benefit from review, please describe (e.g., in what way is the objective not being met, why might the objective benefit from further review).

Describe any plans to review and/or change the location, frequency or types of samples and measurements to meet this (these) objective(s).

2. Evaluate remedy effectiveness: The following questions are intended to review whether the MNA remedy is performing as intended, or whether there may be a need to implement a contingency remedy. A contingency remedy is a cleanup technology or approach that functions as a backup remedy in the event that the selected remedy fails to perform as anticipated.	
Since the last O&M review, have contaminant concentrations in soil or ground water at specified locations exhibited an increasing trend not originally predicted during remedy selection?	☐ Yes ☐ No
Since the last O&M review, have near-source wells exhibited large concentration increases indicative of a new or renewed release?	☐ Yes ☐ No
Since the last O&M review, have contaminants been detected in monitoring wells located outside of the original plume boundary or other compliance-monitoring boundary?	☐ Yes ☐ No
Since the last O&M review, have analyses concluded that the rate of decrease of contaminant concentrations may be inadequate to meet the remediation objectives?	☐ Yes ☐ No
Since the last O&M review, have changes in land and/or ground water use been suggested and or implemented that have the potential to reduce the protectiveness of the MNA remedy?	☐ Yes ☐ No
Since the last review, have contaminants been identified in locations that pose or have the potential to pose unacceptable risk to receptors?	☐ Yes ☐ No
If you answered yes to any of the above questions, did the information suggest the need for immediate action or is the condition being monitored to evaluate the need for future action? Use this space to comment.	☐ Immediate action ☐ Monitored for future ☐ N/A
Based on your answers to the above questions, is there reason to evaluate the need for a contingent remedy at this time? If yes, use this space to comment.	☐ Yes ☐ No
3. Evaluate collection and analysis of performance monitoring data	
What evidence has been used to evaluate actual plume dissipation (e.g., temporal trends in individual wells, estimation of mass reduction, comparisons of observed contaminant distributions with predictions and required milestones, comparison of field-scale attenuation rates)?	
Since the last O&M review, has it been necessary to modify the site-specific plans (e.g., Sampling and Analysis Plan, Quality Assurance Project Plan, Data Management Plan) to account for new information and/or unforeseen circumstances? If yes, use this space to comment.	☐ Yes ☐ No
Does information collected since the last O&M review suggest the need to evaluate whether field parameters that are critical to an MNA evaluation (e.g., dissolved oxygen, redox potential) are being collected at appropriate monitoring points? If yes, use this space to comment.	☐ Yes ☐ No
Do the approaches used to interpret ground water monitoring data (e.g., concentration trend analyses, plume contour and/or bubble maps, plume cross-sections, potentiometric surface maps) provide adequate information to assess the performance of the natural attenuation remedy? If no, describe plans, if any, to implement new approaches.	☐ Yes ☐ No
Does information collected since the last O&M review suggest the need to re-evaluate the ground water and soil-monitoring program to more accurately delineate and monitor the plume boundary? If yes, use this space to comment.	☐ Yes ☐ No
Since the last O&M review, has it been necessary to modify the data quality assessment, including statistical tests (if appropriate), regression analysis, scatter plots, etc. to account for new information and/or unforeseen circumstances? If yes, use this space to comment.	☐ Yes ☐ No
Are ground water data managed electronically? If no, use this space to explain why not.	☐ Yes ☐ No

If statistical tests are used, do the data meet the assumptions of the statistical test?	☐ Yes ☐ No
If no, does this suggest the need to change the monitoring program or re-evaluate the statistical approach? Use this space to comment.	☐ Evaluate monitoring program ☐ Evaluate statistical approach ☐ Neither
Is high variability in the data interfering with or preventing a meaningful interpretation of the data?	☐ Yes ☐ No
If yes, could this situation be mitigated by increasing the density or frequency of sampling? Use this space to comment.	☐ Yes ☐ No
Are performance-monitoring reports of sufficient quality and frequency to evaluate the efficacy of MNA as a remedy and recognize protectiveness problems in time for effective action? If no, what actions, if any, have been taken or are planned to address this situation?	☐ Yes ☐ No
Are techniques or models being used to evaluate adequacy/redundancy of individual wells in the monitoring network, and adequacy/redundancy of sampling frequency? Note that techniques may range from statistical trend analysis to application of ~~a~~ decision support tool.	☐ Yes ☐ No
If no, are there plans to evaluate the adequacy/redundancy of individual monitoring wells and/or sampling frequency? Use this space to comment.	☐ Yes ☐ No
C. Cost Effectiveness: Key considerations in looking at cost-effectiveness of an MNA remedy are the list of parameters for monitoring, as well as the frequency and location of monitoring. Decreases in monitoring parameters, frequency or locations may be appropriate and allow for reductions in project monitoring costs. For example, decreases in monitoring frequency for certain parameters may be warranted if the remedy is proceeding according to expectations and trends are stable after evaluation of data from a sufficient number of monitoring periods (e.g., many years). To support such a decision, the available data generally cover a time period sufficient to allow for an evaluation of seasonal trends and other long-term cycles and trends.	
Does information collected since the last O&M review suggest opportunities to eliminate monitoring points (e.g., because of redundancy, unreliability, or changes in program objectives)? If yes, use this space to comment.	☐ Yes ☐ No
Does information collected since the last O&M review suggest opportunities to replace current analytical and sampling methods with less expensive methods and still meet the data quality objectives? If yes, use this space to comment.	☐ Yes ☐ No
Can the analyte list be shortened to focus on the known contaminants of concern?	☐ Yes ☐ No
D. Remedial Decisions: Following data evaluation, decisions are routinely made regarding the effectiveness of the MNA remedy, monitoring program, and ICs, and the need for contingency or alternative remedies. The following remedial decisions are discussed in Section 4 of the EPA guidance document *Performance Monitoring of MNA Remedies for VOCs in Ground Water* (EPA/600/R-04/027; April 2004). Indicate which of the following remedial decisions is appropriate at the present time and provide the basis for the decision.	
☐ No Change to the Monitoring Program ☐ Modify/Optimize Monitoring Program ☐ IC Modifications ☐ Implementation of Contingency/Alternative Remedy ☐ Terminate Performance Monitoring and Initiate Verification Monitoring	
Basis for decision:	

RECOMMENDED APPENDIX C. CONTAINMENT REMEDIES

The following checklist is an abbreviated set of questions that could be used by a EPA RPMs for an annual review of the O&M of a containment remedy and associated off-gas treatment system. This checklist focuses on engineered containment remedies, including landfill caps, covers, and vertical engineered barriers (VEB). Containment by other means such as hydraulic control and in-situ sediment containment remedies are not addressed by this appendix. See separate surface water/sediment remedy checklist for sediment remedies. Although the checklist includes items for off-gas systems, it focuses on off-gas collection. The checklist does not address off-gas management using combustion systems because such systems are uncommon at Superfund sites.

A. Remedy Description, Goals and Conceptual Site Model (CSM)

1. Review of the current remedy

Identify the containment systems in place:

- ☐ Cap/cover
- ☐ VEB
- ☐ Liner
- ☐ Landfill gas collection
- ☐ Landfill gas management
- ☐ Leachate detection
- ☐ Leachate collection
- ☐ Leachate management
- ☐ Other (Describe:)

Identify the O&M components:

- ☐ Inspection
- ☐ Monitoring
- ☐ Testing
- ☐ Ground water monitoring
- ☐ Surface water monitoring
- ☐ Landfill gas monitoring
- ☐ Vapor intrusion monitoring
- ☐ Leachate monitoring
- ☐ Other (Describe:)

2. Review of the current remedy goals

Identify the remedy goals (RAOs):

- ☐ Prevent direct contact with a contaminant source
- ☐ Prevent migration of a contaminant source to:
  - ☐ A drinking water aquifer
  - ☐ Surface water
  - ☐ Soil or other solid media☐
  - ☐ Air (via wind-borne material)
  - ☐ Air (via volatilization)
  - ☐ Other (Describe:)
- ☐ Prevent migration of contaminated ground water
- ☐ Prevent vapor intrusion or indoor air exposure
- ☐ Control off-gas
- ☐ Other remedy goals (Describe:)

What metrics (performance criteria) are being implemented to measure project progress towards meeting each goal?

What schedule has been established for measuring and reporting each metric?

Based on new information or events since the last O&M review, is there a need to re-evaluate the remedy goals? This might be due to factors such as whether the regulatory framework has been revised, whether existing goals appear to be realistic, and whether there have been changes in land use or ground water production near the site. If yes, identify the remedy goals that should be re-evaluated, the rationale, and any plans for re-evaluating the goals.	☐ Yes ☐ No

3. Review of changes to the CSM: The CSM for a containment remedy is the site-specific, qualitative and quantitative description of the migration and fate of contaminants with respect to possible receptors and the geologic, hydrologic, biological, geochemical and anthropogenic factors that control contaminant distribution. Because the CSM provides the basis for the remedy and the post-closure maintenance plan or O&M plan, the model should be re-evaluated as new data are collected throughout the lifetime of the remedy.	
Does new information gathered or conclusions reached since the last time the O&M checklist was completed indicate a change in understanding about the sources, types, migration, and fate of contaminants? *Note that indicators could include (1) the remedy not functioning as designed, (2) unexpected contaminants or contaminant concentrations above the required levels at the point of compliance, (3) unexpected trends in contaminant concentrations, (4) unexpected changes in the flow rate or direction of ground water, (5) unexpected changes in off-gas characteristics, or (6) unexpected evidence of vapor intrusion in nearby structures.*	☐ Yes ☐ No
Based on new information and/or conclusions, would it be useful to update the CSM at this time? If yes, please describe any plans to update the CSM.	☐ Yes ☐ No
B. Remedy Performance Assessment This section contains a series of questions that can be used to help assess a containment remedy's effectiveness and evaluate the collection and analysis of performance monitoring data. For each potential problem identified, an analysis should be performed to determine what, if anything should be done.	
1. Evaluate remedy effectiveness: The following questions are intended to review whether the containment remedy is performing as intended or whether there is a need to implement a contingency remedy. A contingency remedy is a cleanup technology or approach that functions as a backup remedy in the event that the selected remedy fails to perform as anticipated. A contingency remedy may be considered if there is a "yes" answer to one or more of the following three questions. *Note that additional measures and methods for evaluating the effectiveness of containment remedies can be found in "EPA/USACE Draft Technical Guidance for RCRA/CERCLA Final Covers" (EPA 540-R-04-007) and "EPA Comprehensive 5-Year Review Guidance, Appendix D, Five-Year Review Site Inspection Checklist" (OSWER Directive 9355.7-03B-P).*	
Since the last O&M review, has inspection or testing of the cap, cover, liner, or VEB indicated that the system is failing or could eventually fail?	☐ Yes ☐ No
Since the last O&M review, have changes in land, surface water, or ground water use been suggested and or implemented that have the potential to reduce the protectiveness of the containment remedy?	☐ Yes ☐ No
Since the last O&M review, have contaminants been identified in new locations or at higher concentrations where they pose or have the potential to pose unacceptable risks to receptors?	☐ Yes ☐ No
If you answered yes to any of the above questions, did the information suggest the need for immediate action or is the condition being monitored to evaluate the need for future action? Use this space to comment. What actions, if any, have been taken and/or are planned in response to the new information?	☐ Immediate action ☐ Monitored for future ☐ N/A
For VEB Only: Note that additional measures and methods for evaluating VEB effectiveness can be found in "EPA Evaluation of Subsurface Engineered Barriers at Waste Sites".	
Have bulk integrity tests been performed since the last O&M review?	☐ Yes ☐ No

If bulk integrity tests have been performed since the last review, do test results indicate that need to evaluate possible breaches or excessive leakage in the VEB over the short and long terms? If yes, what actions have been taken and/or are planned in response?	☐ Yes ☐ No ☐ N/A
Based on information collected since the last O&M review, do contaminant concentrations upgradient of the VEB indicate the need to evaluate actions to prevent possible contaminant migration? If yes, what actions have been taken and/or are planned in response?	☐ Yes ☐ No
Does information collected since the last O&M review suggest the need to evaluate hydraulic controls as an additional measure to control possible contaminant migration around the VEB (answer N/A if hydraulic controls are already part of the remedy)? If yes, what actions have been taken and/or are planned in response?	☐ Yes ☐ No ☐ N/A
For Off-Gas Collection Management Only: Note that additional measures and methods for evaluating off-gas collection and management effectiveness can be found in "USACE Landfill Off-Gas Treatment, Thermal Oxidation Checklist".	
Since the last O&M review for this system, have off-gas volume and composition been consistently within equipment design parameters? If no, what actions have been taken and/or are planned in response?	☐ Yes ☐ No
Since the last O&M review for this system, have off-gas system operational characteristics, such as required temperatures and pressures, been maintained within system design parameters? If no, what actions have been taken and/or are planned in response?	☐ Yes ☐ No
Since the last time an O&M checklist was completed for this system, have off-gas emissions met all federal, state, and local regulatory requirements? If no, what is being done to meet these requirements?	☐ Yes ☐ No
Based on information collected since the last O&M review, is there any evidence of unacceptable vapor intrusion in nearby structures? If yes, what actions have been taken and/or are planned in response?	☐ Yes ☐ No
Based on information collected since the last O&M review, have concentrations of off-gases inside buildings or at the site fence line suggested the need to assess safety and human health threats? If yes, what actions have been taken and/or are planned in response?	☐ Yes ☐ No
2. Evaluate collection and analysis of performance monitoring data Note that more detailed information about performance parameters can be found in the following documents: ▪ "EPA/USACE Draft Technical Guidance for RCRA/CERCLA Final Covers" (EPA 540-R-04-007) ▪ "EPA Comprehensive 5-Year Review Guidance, Appendix D, Five-Year Review Site Inspection Checklist" (OSWER Directive 9355.7-03B-P) ▪ "USACE Landfill Off-Gas Treatment, Thermal Oxidation Checklist" ▪ "EPA Evaluation of Subsurface Engineered Barriers at Waste Sites" (EPA 542-R-98-005; August 1998).	
Since the last O&M review, has it been necessary to modify planned inspections, sampling events, and sample analyses, as reflected in the site post-closure maintenance plan or O&M plans, to account for new information and/or unforeseen circumstances? If yes, use this space to comment.	☐ Yes ☐ No
Has information collected since the last O&M review suggested the need to re-evaluate whether performance parameters that are critical to evaluation of the containment remedy are being collected at appropriate monitoring points? If yes, what actions have been taken and/or are planned in response?	☐ Yes ☐ No

Are ground water and off-gas system monitoring data managed electronically? If no, use this space to explain why not.	☐ Yes ☐ No
Since the last O&M review, have monitoring data been analyzed to identify trends and their significance? If no, use this space to explain why not.	☐ Yes ☐ No
Is high variability in the data interfering with or preventing a meaningful interpretation of the data?	☐ Yes ☐ No
If yes, could this situation be mitigated by increasing the density or frequency of data collection? Use this space to comment.	☐ Yes ☐ No
Are inspection and performance monitoring reports of sufficient quality and frequency to evaluate the efficacy of containment as a remedy and recognize protectiveness problems in time for effective action? If no, what actions, if any, have been taken or are planned to address this situation?	☐ Yes ☐ No
C. Cost-Effectiveness	
If off-gas is currently being treated, can it be vented to the atmosphere without treatment in compliance with all applicable federal, state, and local regulations?	☐ Yes ☐ No ☐ N/A
If yes, has the possibility of discontinuing off-gas treatment been explored? Use this space to comment.	☐ Yes ☐ No ☐ N/A
If leachate is currently being collected and treated, is operation of the leachate system necessary for proper functioning of the containment system?	☐ Yes ☐ No ☐ N/A
If no, has the possibility of discontinuing leachate collection and treatment been explored? Use this space to comment.	☐ Yes ☐ No ☐ N/A
If hydraulic controls are being used in conjunction with a VEB, would the VEB provide passive containment without these controls?	☐ Yes ☐ No ☐ N/A
If yes, has the possibility of discontinuing the hydraulic controls been explored? Use this space to comment.	☐ Yes ☐ No ☐ N/A
D. Remedial Decisions: Indicate which of the following remedial decisions is appropriate at the present time and provide the basis for the decision.	
☐ No change to the remedy ☐ Modify or optimize remedy ☐ Modify or optimize O&M ☐ Modify ICs ☐ Implement contingency or alternative remedy ☐ Terminate inspections or monitoring	
Basis for decision:	

RECOMMENDED APPENDIX D. SOIL VAPOR EXTRACTION/AIR SPARGING REMEDIES

- The following checklist is an abbreviated set of questions that EPA RPMs could use when conducting an annual review of the O&M of a soil vapor extraction (SVE), air sparging (AS), or combined SVE/AS remedy. This checklist does not represent the level of review used in EPA's five-year review process to determine whether the remedy is or will be protective of human health and the environment. However, the checklist does contain review elements regarding the performance of SVE and/or AS remedies that are consistent with the comprehensive five-year review process.

A. Remedy Description, Goals and Conceptual Site Model (CSM)

1. Review of the current remedy

Identify the current remedy:

- ☐ SVE
- ☐ AS

How many extraction wells or trenches are used for SVE (if applicable)?

How many injection wells are used for AS (if applicable)?

2. Review of the current remedy goals

List the remedy goals (RAOs):

- ☐ Prevent migration of a contaminant source to:
  - ☐ A drinking water aquifer
  - ☐ Surface water
  - ☐ Soil or other solid media
- ☐ Prevent migration of contaminated ground water
- ☐ Restore ground water
- ☐ Other (Describe:)

List the short-term objectives and intermediate system goals.

List the long-term soil and ground water cleanup goals.

What metrics (performance criteria) are being implemented to measure project progress towards meeting each goal?

What schedule has been established for measuring and reporting each metric?

Based on new information or events since the last O&M review, is there a reason to re-evaluate the remedy goals? Note that this might be due to factors such as whether the regulatory framework has been revised, whether existing goals appear to be realistic, and whether there have been changes in land or ground water use near the site. If yes, identify the remedy goals that should be re-evaluated, the rationale, and any plans for re-evaluating the goals.	☐ Yes ☐ No

3. Review of changes to the CSM: The CSM for a SVE/AS remedy is the site-specific, qualitative and quantitative description of the migration and fate of contaminants with respect to possible receptors and the geologic, hydrologic, biological, geochemical and anthropogenic factors that control contaminant distribution. Because the CSM provides the basis for the remedy and the O&M plan, the model should be re-evaluated as new data are collected throughout the lifetime of the remedy.	
Does new information gathered or conclusions reached since the last time the O&M checklist was completed indicate a change in understanding about the sources, types, migration, and fate of contaminants? *Note that indicators could include: (1) the remedy not functioning as designed, (2) unexpected contaminants or contaminant concentrations above the required levels at the point of compliance, (3) unexpected trends in contaminant concentrations, (4) unexpected changes in the flow rate or direction of ground water, (5) unexpected changes in off-gas characteristics, (6) unexpected evidence of vapor intrusion in nearby structures; or (7) identification of new sources.*	☐ Yes ☐ No
Based on new information and/or conclusions, would it be useful to update the CSM at this time? If yes, please describe any plans to update the CSM.	☐ Yes ☐ No
B. Remedy Performance Assessment This section contains a series of questions that can be used to help assess a SVE/AS remedy's effectiveness and evaluate the collection and analysis of performance monitoring data.	
1. Evaluate remedy effectiveness: The following questions are intended to review whether the SVE/AS remedy is performing as intended, or whether there is a need to implement a contingency remedy. A contingency remedy is a cleanup technology or approach that functions as a backup remedy in the event that the selected remedy fails to perform as anticipated. A contingency remedy may be considered if there is a "yes" answer to either of the following five questions.	
Based on information collected since the last O&M review, do monitoring data indicate that the system is failing or could eventually fail to meet remedy goals?	☐ Yes ☐ No
Since the last O&M review, has the areal extent of contamination (or plume) increased in a manner not originally predicted during remedy selection?	☐ Yes ☐ No
Since the last O&M review, have monitoring data exhibited trends indicative of a new or renewed release?	☐ Yes ☐ No
Since the last O&M review, have changes in land and/or ground water use been suggested and or implemented that have the potential to reduce the protectiveness of the SVE/AS remedy?	☐ Yes ☐ No
Since the last O&M review, have contaminants been identified in new locations or at higher concentrations where they pose or have the potential to pose unacceptable risks to receptors?	☐ Yes ☐ No
If you answered yes to any of the above questions, did the information suggest the need for immediate action or is the condition being monitored to evaluate the need for future action? Use this space to comment. What actions, if any, have been taken and/or are planned in response to the new information?	☐ Immediate action ☐ Monitored for future ☐ N/A
Based on your answers to the above questions, is there reason to evaluate the need for a contingent remedy at this time? If yes, use this space to comment.	☐ Yes ☐ No

Blowers and Piping	
Since the last O&M review for this system, has evidence of excessive corrosion of system components been observed? If yes, what actions have been taken and/or are planned in response?	☐ Yes ☐ No
Since the last O&M review, if blowers are operated intermittently, do VOC concentrations increase after they are shut off? How has this information been interpreted and what actions, if any, have been taken and/or are planned in response?	☐ Yes ☐ No ☐ N/A
Since the last O&M review, have blower operational characteristics, such as flow rate, pressure, and discharge temperatures, been consistently within equipment design parameters? If no, what actions have been taken and/or are planned in response?	☐ Yes ☐ No
Since the last O&M review, if water is manually removed from the extraction blower water separator, has water accumulation been observed that could adversely impact blower operation? If yes, what actions have been taken and/or are planned in response?	☐ Yes ☐ No ☐ N/A
Since the last O&M review, have all blowers, water separators, valves, and piping components been consistently operational?	☐ Yes ☐ No
Has the downtime associated with non-routine operations and maintenance of the blowers since the last time you completed an O&M checklist for this system exceeded expectations? If yes, what have been identified as the causes? If yes, what corrections have been or are being made to minimize downtime?	☐ Yes ☐ No
Does the operational history suggest that the preventative maintenance plan for the blowers needs to be re-evaluated? If yes, what actions have been taken and/or are planned in response?	☐ Yes ☐ No

Soil Vapor Extraction System	
Identify the SVE system characteristics, if any, that have deviated consistently/frequently from operational expectations since the last time an O&M checklist was completed for this system: ☐ Vapor flow rates at one or more extraction wells ☐ Vapor compositions (VOCs, CO_2, O_2) at one or more extraction wells ☐ Pressures at one or more extraction wells ☐ Flow at blower (prior to entry of any dilution air if used) ☐ Accumulation of water in the water separator	
Does this (do these) deviation(s) indicate a new condition since the last O&M review or an ongoing trend?	☐ New condition ☐ Ongoing trend ☐ N/A
What has been identified as the cause for this (these) deviation(s)?	
What actions, if any, have been or are being taken in response to this (these) deviation(s)?	
Based on information collected since the last O&M review, is there any evidence of unacceptable vapor intrusion in nearby structures? If yes, what actions have been taken and/or are planned in response?	☐ Yes ☐ No

Since the last O&M review, have gas concentrations in the blower discharge been running close enough to the lower explosive limit (LEL) or shown an increasing trend that suggests the need for action? *Note that specific compound LEL data are available in many chemistry texts as well as National Fire Protection Agency guidelines.* What actions, if any, have been taken and/or are planned in response to the new information?	☐ Yes ☐ No
Air Sparging System	
Since the last O&M review of the AS system, have flow rates at each injection well been consistently maintained within system design parameters? If no, what actions, if any, have been or are being taken in response?	☐ Yes ☐ No
Based on information collected since the last O&M review, have dissolved oxygen concentrations been maintained at a level sufficient to promote biological activity? If no, what actions, if any, have been or are being taken in response?	☐ Yes ☐ No
Since the last O&M review, are measured dissolved oxygen concentrations consistently indicative of good air/water contact rates (i.e., are concentrations near saturation)? If no, what actions, if any, have been or are being taken in response?	☐ Yes ☐ No
VOC Control System	
If the SVE system contains a VOC control device, has the device consistently met performance and compliance monitoring requirements (e.g., total VOC emission limits, specific compound limits, monitoring, air permit) since the last O&M review for this system? If no, what actions have been taken and/or planned in response?	☐ Yes ☐ No ☐ N/A
Since the last O&M review, has the VOC control system consistently meet required destruction and removal efficiencies? If no, what actions have been taken and/or planned in response?	☐ Yes ☐ No
Since the last O&M review, have any violations of air permits been reported? If yes, what has been or is being done to meet permit requirements?	☐ Yes ☐ No
Since the last time you completed an O&M checklist for this system, has the VOC control system been responsible for downtime associated with non-routine operations and maintenance? If yes, ▪ What was (were) the cause(s) for unplanned shutdown(s)? ▪ What has been done or is being done to minimize future downtime?	☐ Yes ☐ No
Thermal Oxidizers	
Since the last O&M review for this system, have the operational characteristics (e.g., LEL history of feed gas, operating temperature, inlet flow, oxygen level in flue gas, fuel use) been consistently within equipment design parameters? If no, what actions, if any, have been or are being taken in response?	☐ Yes ☐ No ☐ N/A
Since the last O&M review, has there been any indication of improper operation of flashback protection equipment (e.g., detonation arrestor, sealed drum)? If yes, what actions have been taken and/or planned in response?	☐ Yes ☐ No
Since the last O&M review, has there been any indication of improper operation of safety interlocks (e.g., high LEL, high oxidizer temperature, loss of flame, low fuel pressures)? If yes, what actions have been taken and/or planned in response?	☐ Yes ☐ No

If acid gases are present, have scrubber operations (e.g., scrubber liquid flow and pH, caustic use, scrubber blowdown and its treatment) been consistent with operational expectations since the last O&M review? If no, what actions have been taken and/or planned in response?	☐ Yes ☐ No
Carbon Adsorbers	
Does the unit have humidity controls?	☐ Yes ☐ No
Since the last O&M review for this system, have the operational characteristics (e.g., relative humidity data at adsorber inlet, adsorber operating temperature, carbon breakthrough, carbon change out history, operating velocity through adsorbers, adsorber discharge VOC data) been consistently within equipment design parameters? If no, what actions, if any, have been or are being taken in response?	☐ Yes ☐ No ☐ N/A
Other Control Devices	
Since the last O&M review for this system, have the operational characteristics (e.g., biofiltration media surface loading rate, temperature controls, nutrient addition rate) been consistently within equipment design parameters? If no, what actions, if any, have been or are being taken in response?	☐ Yes ☐ No ☐ N/A
2. Evaluate collection and analysis of performance monitoring data	
Since the last O&M review, has it been necessary to modify sampling frequency relative to the original O&M plan to account for new information and/or unforeseen circumstances? If yes, use this space to comment.	☐ Yes ☐ No
Does soil and/or ground water data collected since the previous O&M review (e.g., VOCs concentrations, ground water elevations) suggest the need to re-evaluate other aspects of the monitoring program (e.g., monitoring locations, test parameters) to account for new information/unforeseen circumstances? If yes, use this space to comment.	☐ Yes ☐ No
C. Cost Effectiveness: Key considerations in looking at cost-effectiveness are the O&M costs incurred relative to design and reduction in VOC removal rates. Opportunities to reduce costs can be potentially found in the following areas:	
Does information collected since the last O&M review suggest that flows could be redistributed to speed overall remediation (i.e., reduce or eliminate flow to/from wells where removals have reached near asymptotic conditions or where cleanup goals have been achieved)? Use this space to comment.	☐ Yes ☐ No
Does information collected since the last O&M review show evidence of diffusion-limited VOC movement?	☐ Yes ☐ No
If yes, has the idea of modifying operation to pulsing (intermittent) been considered to speed overall remediation? Use this space to comment.	☐ Yes ☐ No
Does information collected since the last O&M review show reduced VOC removal rates that might warrant a reduction in monitoring frequencies? Use this space to comment.	☐ Yes ☐ No
Does information collected since the last O&M review suggest that VOC recovery rates have been reduced to the extent that the VOC control device can be eliminated? Use this space to comment.	☐ Yes ☐ No ☐ N/A

Does information collected since the last O&M review suggest that an alternative, lower cost VOC control device could be used? Use this space to comment.	☐ Yes ☐ No
Does information collected since the last O&M review suggest that operation of the VOC control device could be modified to reduce costs, e.g., operate thermal oxidizer at lower temperatures or lower dilution air flows (e.g., when LEL basis no longer requires design flow) or use larger carbon beds to reduce carbon supplier charges for change outs? Use this space to comment.	☐ Yes ☐ No
Has maintenance history since the last O&M review identified high-maintenance equipment that could be replaced? Use this space to comment.	☐ Yes ☐ No

E. Remedial Decisions: Indicate which of the following remedial decisions are appropriate at the present time and provide a basis for each decision:

- ☐ Continue current remedy
- ☐ Goals have been achieved -- system can be shutdown in favor of MNA
- ☐ Modify/optimize remedial system(s) – use intermittent operation; optimize flows to/from wells to promote increased removals; increase use of sparging to promote biodegradation; add new wells if contaminant movement is indicated to areas currently not being influenced; implement cost reduction measures; conduct more detailed evaluation of the contaminated zone using a tool such as Pneulog.
- ☐ Modify/optimize O&M – increase monitoring to provide additional data for more definitive assessment at the next review
- ☐ Modify ICs
- ☐ Implement contingent or alternative remedy

Basis for decision:

RECOMMENDED APPENDIX E. OTHER REMEDY TYPES/COMPONENTS

The following checklist is a set of questions that may be used by EPA RPMs for an annual review of the O&M of remedies and remedy components that are not addressed in Appendices A through D or the separate surface water/sediment remedy O&M checklist. This could include remedies/components that involve a technology that is not covered in these other materials or remedies/components where the O&M can be more efficiently reviewed using the more streamlined questions below. If the site includes multiple remedy components that are not covered elsewhere, multiple copies of this appendix, each applying to a different component or related set of components, could be completed.

A. Remedy Description and Goals

1. Review of current remedy goals, and measurements

The following questions can be used to document basic information about the remedy and remedy goals to provide context for the remainder of the information in this appendix.

Identify the remedy component(s) and associated systems and technologies being covered on this form:	
What are the intermediate and final system goals?	
What metrics (performance criteria) are being implemented to measure project progress towards meeting each goal?	
What schedule has been established for measuring and reporting each metric?	
Based on new information or events since the last O&M review of this system/technology, is there a need to re-evaluate the remedy goals? If yes, identify the remedy goals that should be re-evaluated, the rationale, and any plans for re-evaluating the goals.	☐ Yes ☐ No

2. Review of changes to the CSM

The following questions ask about changes in contamination and other field conditions that could affect the monitoring program, system operations, and other aspects of O&M. They provide context for questions in subsequent sections that ask whether action should be taken to modify the O&M program.

Do monitoring data indicate trends/patterns that are inconsistent with the CSM (or similar conceptual understanding of site conditions) that was used as the basis for design of the remedy/remedial component(s)? If yes, use this space to comment.	☐ Yes ☐ No
Have there been changes in field conditions (e.g., change in land/water use) that differ significantly from the conditions incorporated in the CSM (or similar conceptual understanding of site conditions) that was used as the basis for design of the remedy/remedial component(s)? If yes, use this space to comment.	☐ Yes ☐ No
Have new contaminant sources been identified? If yes, please describe the new sources and how they are they being addressed:	☐ Yes ☐ No

B. Remedy Performance Assessment

This section contains a series of questions that can be used to help assess whether the monitoring program and remediation systems O&M should be adjusted.

1. Monitoring Program

Describe changes to the monitoring program that have been made since the last time you completed the O&M checklist for this remedy component.	
Are the baseline data and post-remedy data adequate to perform statistical comparisons and evaluate remedy performance? If no, what actions have been or are being taken in response?	☐ Yes ☐ No

Is high variability in the data interfering with or preventing a meaningful interpretation of the data?	☐ Yes ☐ No
If yes, could this situation be mitigated by increasing the density or frequency of data collection? Use this space to comment.	☐ Yes ☐ No
Based on changes in contamination or field conditions (see A.2 of this appendix), is there reason to modify the monitoring program? If yes, describe changes to the monitoring program that are most necessary.	☐ Yes ☐ No
Has the adequacy/redundancy and cost-effectiveness of the monitoring program been evaluated, including evaluation of sampling locations, frequency, sampling and analytical methods, monitoring parameters, and test methods? Use this space to comment.	☐ Yes ☐ No
Is there reason to modify the monitoring program to address inadequacies, remove redundancies, and/or improve its cost-effectiveness? If yes, describe changes to the monitoring program that would likely have the greatest impact.	☐ Yes ☐ No
Do you have adequate documentation (e.g., good quality O&M reports) and tools (e.g., software) to effectively manage and interpret monitoring data? If no, please explain how documentation and/or tools could be improved.	☐ Yes ☐ No
2. System Operations	
Describe changes to system operations that have been made since the last time you completed the O&M checklist for this remedy component.	
Is (are) the remedial system(s) covered under this appendix performing as expected relative to the remediation milestones and goal(s)? If no, what actions have been or are being taken in response?	☐ Yes ☐ No
Do monitoring data indicate trends/patterns that are consistent with remedial design expectations? If no, what actions have been or are being taken in response?	☐ Yes ☐ No
Based on observations regarding contamination or field conditions (see A.2 of this appendix and previous questions in this section), is there reason to modify systems operations to improve remedy performance? If yes, describe changes to system operations that are most necessary.	☐ Yes ☐ No
Has an optimization study been conducted for the remedy/remedy component(s)? Use this space to comment.	☐ Yes ☐ No
Has the downtime associated with non-routine operations and maintenance exceeded expectations? If yes, what actions have been or are being taken to minimize downtime?	☐ Yes ☐ No
Based on optimization and downtime considerations, is there reason to modify systems operations to improve remedy performance? If yes, describe changes to system operations that are most necessary.	☐ Yes ☐ No
3. Maintenance	
Are routine maintenance activities adequate to ensure the reliable operation of the remedial system(s)? If no, what changes to the maintenance program are most necessary?	☐ Yes ☐ No

Have any major repairs to the remedial system(s) been required since the last time you completed the O&M checklist for this remedy/remedy component? If yes, describe the repairs, their impact on progress toward remediation milestones, and actions taken to minimize similar repairs in the future.	☐ Yes ☐ No
C. Cost Effectiveness	
Does information collected since the last O&M review suggest opportunities to reduce costs associated with equipment operations and maintenance? If yes, use this space to comment.	☐ Yes ☐ No
Does information collected since the last O&M review suggest opportunities to reduce costs associated with the monitoring program? If yes, use this space to comment.	☐ Yes ☐ No
D. Remedial Decisions: Indicate which of the following remedial decisions is appropriate at the present time and provide the basis for the decision.	
☐ No Change ☐ Modify/Optimize System ☐ Modify/Optimize Monitoring Program ☐ Modify ICs ☐ Implement Contingency/Alternative Remedy	
Basis for decision:	